UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

GISLENE CRISTINA VERTUAN

**Evaluation of physicochemical properties of a new epoxy resin-
based root canal sealer**

**Avaliação das propriedades físico-químicas de um novo cimento
obturador de canais radiculares à base de resina epóxica**

BAURU
2018

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Evaluation of physicochemical properties of a new epoxy resin-based root canal sealer

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Dissertação constituída por artigo apresentada a Faculdade de Odontologia de Bauru da Universidade de São Paulo para obtenção do título de Mestre em Ciências no Programa de Ciências Odontológicas Aplicadas, na área de concentração Endodontia.

Orientador: Prof. Dr. Rodrigo Ricci Vivan

BAURU

2018

Vertuan, Gislene Cristina.

V617e Evaluation of physicochemical properties of a new epoxy resin-based root canal sealer / Gislene Cristina Vertuan. – Bauru, 2018.

57p. : il. ; 31cm.

Dissertação (Mestrado) – Faculdade de Odontologia de Bauru. Universidade de São Paulo

Orientador: Prof. Dr. Rodrigo Ricci Vivan

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DADOS CURRICULARES

GISLENE CRISTINA VERTUAN

Nascimento	26 de junho de 1987, Macatuba, São Paulo
Filiação	Irineu Vertuan Fátima dos Santos Vertuan
2005 – 2008	Graduação em Comunicação Social – Publicidade e Propaganda Universidade Paulista – Bauru/SP
2010 – 2014	Graduação em Odontologia Universidade Sagrado Coração – Bauru/SP
2015 – 2015	Aperfeiçoamento em Endodontia Associação Paulista de Cirurgiões Dentistas – Bauru/SP
2015 – 2016	Pós-graduação Lato Sensu – Especialização em Endodontia Fundação Bauruense de Estudos Odontológicos (FUNBEO- FOB/USP) – Bauru/SP
2016 – 2018	Pós-graduação Stricto Sensu – Mestrado em Endodontia Faculdade de Odontologia de Bauru, Universidade de São Paulo (FOB/USP) – Bauru/SP

DEDICATÓRIA

Primeiramente à **Deus**, pelo dom da minha vida e por ter me concedido àqueles que sempre estão ao meu lado: minha família!

Dedico também este trabalho para vocês amados pais **Fátima** e **Irineu**, amada irmã **Fernanda**.

AGRADECIMENTOS

Neste momento de grande gratidão, esta é a oportunidade de demonstrar o que sinto por tantas pessoas e que muitas vezes não consigo dizer.

Agradeço em primeiro lugar à **Deus** pela minha vida e de minha família. Agradeço pelo sustento e força diária que Ele me concedeu para que eu pudesse trilhar mais esse caminho, lutar nas dificuldades e comemorar nas vitórias. Sem sua permissão e benção meu Deus, nada disso seria possível.

Agradeço aos meus pais **Fátima e Irineu** e a minha irmã **Fernanda** pela razão e sentido que dão à minha vida. Obrigada pelo apoio e paciência. Obrigada por tudo que são e que somos juntos. Um por todos e todos por um!

Agradeço a vocês por estarem ao meu lado em todos os momentos, me oferecendo o melhor que tem. Agradeço desde um simples olhar de apoio, uma palavra de incentivo, um gesto de compreensão, uma atitude de segurança. Se hoje cheguei até aqui é porque vocês acreditaram em mim e caminharam ao meu lado. Foram meu sustento e meu suporte nos momentos de dificuldade. Sempre me aconselharam nas decisões; compartilharam dos momentos de angústia e de alegria; aguentaram minha preocupação, mesmo quando acompanhada de estresse, nervoso e mau-humor, em várias situações. Obrigada por acreditarem em mim, me apoiarem e incentivarem. Vocês são essenciais em minha vida, são meu alicerce e porto seguro. Obrigada pai pela sua responsabilidade com nossa família e por abrir mão de seus sonhos para que a gente pudesse buscar os nossos. Obrigada mãe pela preocupação e oração diária. Obrigada gêmea por ser minha vida. Vocês são tudo para mim! E agora contamos com mais um integrante em nossa família, meu cunhado **Elder**.

Agradeço aos familiares – **Tia Nair, Adriana, Ana Carolina, Cristiano, Marina, Dona Liu, Tia Dinei, Sandra, Keila e Vó Amélia** – pelo incentivo na realização de mais uma etapa profissional em minha vida, no apoio constante através das ligações e mensagens de preocupação, motivação e por todos os momentos de oração. Obrigada pelo carinho e pelo amor de vocês.

À querida **Dona Fátima** eu agradeço por me ouvir e por me aconselhar, não somente nesse período, mas desde que nos conhecemos. Agradeço por poder contar com a senhora nos bons e maus momentos e, principalmente, por todas suas orações, pois sem elas e sem a proteção dos anjos eu não poderia chegar até aqui. Muito obrigada por cuidar de mim e de minha família como uma mãe. Obrigada pelo seu carinho e por seu amor.

Agradeço à minha amiga **Angélica Cristina Fonseca**, companheira da graduação para à vida, por nos entendermos e podermos dividir nossos momentos de tensão e angústia. Obrigada mais uma vez por ter sido minha grande parceira de clínica, por termos errado e acertado, por termos aprendido juntas, por termos trazido em nossa bagagem nossos medos e receios a serem superados e também nossos sonhos a serem realizados. Agradeço por fazer parte de minha vida pessoal, mesmo agora que passamos muito menos tempo uma ao lado da outra, mas sempre presente no coração.

E assim, faço desse trabalho, que é mais uma conquista em minha e em nossas vidas, um instrumento de gratidão e reconhecimento por tudo quanto recebi de vocês. Creio que o suporte de Deus esteve presente em minha vida através do cuidado, carinho e amor de cada um.

Ao meu orientador Professor **Dr. Rodrigo Ricci Vivan**, agradeço pela confiança em me orientar, pela ideia de realizar esta pesquisa, por acreditar em mim, pelos seus conselhos, paciência e correção na concretização desse trabalho. Agradeço também por ter me dedicado seu tempo em conversas e incentivos, para que eu pudesse acreditar mais em mim mesma, encarar e superar desafios. Obrigada pelos conhecimentos e momentos compartilhados. Suas atitudes, ensinamentos, exemplos e incentivos foram muito importantes para minha formação. Como já te falei algumas vezes, então não é segredo, que minha escolha pela endodontia tem relação com a admiração que sentia ao assistir suas aulas ainda na graduação. Seu entusiasmo e paixão pela profissão, a didática ao ministrar o conteúdo, sua confiança e eloquência ao expor o conhecimento sobre determinado assunto ou simplesmente se comunicar. Características que me deixaram admirada e posteriormente me fizeram buscar o caminho acadêmico. Para mim, ser professor é admirável. Poder

ensinar o que se sabe, dividir o conhecimento, fazer parte do crescimento pessoal e profissional de outra pessoa, é admirável. Obrigada pela oportunidade!

Professor **Dr. Clóvis Monteiro Bramante**. Se eu não acreditava que um dia poderia conhecê-lo pessoalmente, quem diria ser aluna e poder conviver esse tempo da pós-graduação, com um dos ícones da endodontia. Que honra e orgulho para mim, poder admirar de perto seu amor e dedicação à profissão, seu exemplo a ser seguido, clínica, científica e didaticamente. Obrigada por todos apontamentos durante os seminários. Obrigada por todas as vezes que o senhor me recebeu em sua sala e eu pude questionar sobre algum assunto ou discutir algum caso clínico. O senhor é uma pessoa incrível!

Professora **Dra. Flaviana Bombarda de Andrade**. Agradeço primeiramente pela sua amizade, disposição em me ouvir, aconselhar, incentivar. Agradeço também por ter conferido a mim algumas vezes essa função. Sua paixão pela microbiologia é admirável e só quem trabalha nessa área pode compreender os desafios que ela oferece. Sua trajetória de vida e seu exemplo de dedicação à profissão, de superação de obstáculos, em busca de seus objetivos, são inspirações de como insistir, persistir e não desistir. Obrigada pela atenção, paciência e por todos ensinamentos.

Professor **Dr. Marco Antonio Hungaro Duarte**, agradeço pela convivência e ensinamentos transmitidos. É sabido que o senhor com muito esforço e dedicação conquistou o seu espaço, tornando-se um dos maiores e mais reconhecidos pesquisadores, visando uma endodontia de sucesso, alicerçada em evidências científicas e que atenda padrões técnicos e biológicos adequados. Nesse sentido, a atividade de pesquisa é contínua no desenvolvimento ou aprimoramento de técnicas e materiais, bem como mais atrativos clinicamente. O senhor tem visão, ideias, competência e grande capacidade intelectual. Sinto-me honrada em ter sido sua aluna. Obrigada pelos seus ensinamentos.

Professor **Dr. Norberti Bernardinelli**, agradeço pelos elogios, críticas e sugestões de melhoria nos seminários, contribuindo com minha formação didática e científica na pós-graduação.

Professor **Dr. Roberto Brandão Garcia**. Um verdadeiro gentleman. Sempre muito respeitoso, educado, atencioso, gentil e solícito. Sempre disposto a ensinar, seguindo uma coerente linha de raciocínio, desde seu prefácio. Disposto também a desenhar e repetir, não uma, mas quantas vezes fossem necessárias até que o aluno pudesse entender. Uma didática maravilhosa e uma pessoa admirável!

Aos demais **Professores do Programa de Pós-Graduação** pelos conhecimentos transmitidos através das disciplinas cursadas durante o mestrado, muito obrigada.

Não posso deixar de agradecer também a todos aqueles que foram meus **Professores de Graduação** e que fizeram parte da minha caminhada. Cada um deles teve sua significativa contribuição com minha formação profissional, teórica, clínica e didaticamente. Agradeço àqueles que não pouparam esforços para dividir seu conhecimento, se dispondo a ensinar, trocar experiências, solucionar dúvidas, discutir casos clínicos. Muito obrigada.

Aos professores sugeridos para composição da **Banca Examinadora** eu expresso meus agradecimentos a cada um. Cada nome foi pensado com muito carinho. Aos titulares, eu agradeço por prontamente terem aceito o convite, atenção com que o receberam e disposição em conciliar as datas na agenda. Agradeço também as correções e considerações, pois são de muita contribuição em minha formação. Obrigada por participarem desse momento especial de minha vida.

A todos os professores que tive ao longo de minha vida, que muito me ensinaram, saibam que foram essenciais para minha formação profissional sólida e ética, humana e responsável com o próximo, e colaboraram para que eu chegasse até aqui. Muito obrigada.

Aos meus companheiros de turma de Mestrado 2016-2018, **Mariana Maciel Batista Borges, Maricel Rosario Cárdenas Cuellar, Melissa Esther Rivera Peña, Renan Diego Furlan e Yahir Muñoz Valencia**. Cada um de vocês, com seu jeito ímpar, pessoal e profissionalmente, tem espaço em minha memória e coração. Cada um de vocês contribuiu muito comigo nesse caminho. A cada novo passo que a gente

dava. A cada aula que cursávamos na mesma turma. A cada seminário que aprendíamos em conjunto, com nossos erros e acertos, buscando nos aperfeiçoar cada vez mais. Durante esse tempo em que estivemos juntos, de maior ou menor convívio, certamente cada um de vocês deixou um pouco de si comigo que quero levar pela vida toda, seja com palavras, exemplos, suporte emocional, ensinamentos e aprendizagem. Muito obrigada por tudo! Meu coração na pós-graduação tem 1/6 de cada um de nós!

Aos colegas de pós-graduação do departamento de Endodontia, agradeço pelos momentos compartilhados, de convivência e aprendizado, e desejo um caminho de saúde, felicidade e sucesso à todos vocês: **Arthur Costa Lemos, Bruno Martini Guimarães, Bruno Piazza, Clarissa Rodrigues Teles, Denise Ferracioli Oda, Ericson Janólio de Camargo, Ericke Mucke Silva, Francine Cesário, Gláucia Beatriz Gonçalves, Jussaro Alves Duque, Lyz Cristina Furquim Canali, Marcelo Pomini, Michel Espinosa Klymus, Milena Perraro Martins, Murilo Priori Alcalde, Pedro César Gomes Titato, Rafaela Fernandes Zancan, Raquel Midená Zanin, Talita Tartari, Thaís Cristina Pereira, Vanessa Abreu Sanches Marques e Victor de Moraes Cruz.**

Ao colega de doutorado **Michel Espinosa Klymus** agradeço pela paciência, apoio e incentivo. Por nossas conversas e suas dicas, conselhos e exemplos. Obrigada por confiar em mim na execução desse trabalho, junto ao Professor Rodrigo.

Ao colega de pós-doutorado Professor **Dr. Bruno Carvalho de Vasconcelos**, da Universidade Federal do Ceará, meus agradecimentos pelos ensinamentos e pela oportunidade, por ter confiado a mim parte da execução de seu trabalho experimental, paralelo a minha dissertação de mestrado.

O conhecimento é o único bem que possuímos e que ninguém pode nos roubar. O único bem que ao ser dividido com outras pessoas, ele é também multiplicado. Não estamos aqui para competir ou para ser concorrentes e sim companheiros de uma jornada. Em equipe, formando um time, para que todos ganhem juntos. O que vamos levar dessa vida, se não o bem que fizemos, que plantamos e que colhemos. O reconhecimento pelo que ajudamos ao outro e a gratidão pelo que nos ajudaram.

Quando escolhemos ser cirurgiões-dentistas, escolhemos atuar na área da saúde. Ser dentista não é somente uma profissão. Seja clínico, professor ou pesquisador. Buscamos oferecer qualidade de vida para as pessoas. Buscamos ofertar aos pacientes o melhor tratamento possível. E é nessa busca constante, de melhor desempenho clínico, que são contínuas as pesquisas no desenvolvimento ou aprimoramento de técnicas e materiais. A cada dia nos é dada uma oportunidade, uma nova chance de fazer o bem, de se doar para o próximo, para que ele possa ter uma vida melhor, mais saudável e feliz, com a grata satisfação de cada sorriso que iremos receber e a certeza de que fizemos o nosso melhor.

Aos funcionários **Suely Regina Bettio** e **Edimauro de Andrade**, do departamento de Endodontia, agradeço pela amizade e carinho com que me acolheram na pós-graduação e pela convivência harmoniosa durante esses anos.

A funcionária **Thelma Lopes Silva**, do Laboratório de Bioquímica, por toda atenção, paciência e ensinamentos com as análises químicas realizadas neste trabalho de dissertação. Obrigada por sempre estar disposta a ajudar.

A funcionária **Márcia Sirlene Zardin Graeff** do Centro Integrado de Pesquisa, por toda atenção e paciência nos agendamentos e realização das imagens de microscopia confocal de varredura a laser, que fizeram parte do meu projeto de pesquisa, embora não constem nessa dissertação. Obrigada por se mostrar disposta a ajudar e por comemorar comigo a contaminação intratubular dos grupos estudados.

De uma maneira geral, agradeço a todos **funcionários** desta instituição que, direta ou indiretamente, colaboraram com minha formação pessoal e profissional. Em especial, aos funcionários da **Secretaria de Pós-Graduação** e **Biblioteca** pelas informações e auxílios durante esse período.

A todos que de uma forma ou de outra contribuíram para o desenvolvimento desse trabalho, muito obrigada! Que Deus os abençoe!

AGRADECIMENTOS INSTITUCIONAIS

À **Universidade de São Paulo**, por meio do atual reitor Professor Dr. Vahan Agopyan.

À **Faculdade de Odontologia de Bauru**, por meio do atual diretor Professor Dr. Carlos Ferreira dos Santos.

À **Comissão do Programa de Pós-Graduação** por meio da atual presidente Professora Dra. Izabel Regina Fischer Rubira de Bullen.

Expresso meus agradecimentos por proporcionarem a estrutura necessária para formar seus alunos em profissionais de qualidade e também pelo estímulo e apoio à pesquisa.

À **Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES)** pelo apoio financeiro para a execução desse trabalho.

AGRADECIMENTO ESPECIAL

A pós-graduação me presenteou com alguém muito especial, que eu tenho verdadeira admiração, como pessoa e profissional, incrível que é. De uma simplicidade incomparável, para falar sobre assuntos corriqueiros do dia-a-dia, ou para ensinar temas complexos, transformando o difícil em fácil. Consegue transmitir seu conhecimento de maneira simples e objetiva. Um verdadeiro mestre! Um exemplo a ser seguido por nós pós-graduandos. Além de ser extremamente humilde, não exaltar suas inúmeras conquistas e contribuições para a endodontia. Agradeço por todas as oportunidades de uma boa e produtiva conversa. Uma pessoa maravilhosa e que merece tudo de melhor, por ser do jeitinho que é!

Querido Professor **Dr. Ivaldo Gomes de Moraes**.

Lembro-me do dia que li essa mensagem, e pensei “eu conheço uma pessoa assim: é o Professor Ivaldo”. Imediatamente eu a enviei para o senhor. Desconheço o autor desse texto, mas pelas palavras que nos emocionaram, eu o transcrevo aqui como forma de agradecimento especial para esse grande homem que o senhor é. O pai que a ciência me deu. E com o respeito de uma filha para com um pai, permita-me dizer: eu amo o senhor!

“Existem pessoas boas. Boas de verdade. Pessoas que não se contaminam e sabem segurar a nossa mão como seguram seu mundo. Pessoas assim, além de sua generosidade, não destilam falsidade, não destilam veneno. Elas nos notam, nos acomodam, sorriem de um jeito diferente. Jeito de quem nos entende e sabemos que podemos contar nas horas boas e ruins.

Existem pessoas que transcendem o espaço, a vida, transcendem tudo em prol do bem. Elas vivem em outra esfera, vivem de um modo mais profundo e menos ressentido. Elas sabem distribuir carinho, gentileza. Não usam aspereza e sabem muito bem lidar com a cartilha da vida. Essas pessoas não nos consomem, pelo contrário: somam, multiplicam suas raízes, que se fincam e ficam. Tornam-se especiais. Onde a gente se achega e descansa debaixo daquela quantidade imensa de paz e luz. Elas dão frutos, dão flores, nos trazem mais cor durante a palidez de

alguns dias. Elas nos ensinam, nos corrigem, não nos delimitam. Suavizam nosso caminho por vezes machucado ou sofrido. Mostram-nos a nossa capacidade de evoluir, de sermos pessoas melhores. Transformam tudo a nossa volta.

Existem pessoas que não têm idade na alma. Que o semblante mostra a que veio sem precisar de espelho. Tudo nelas reflete o que o coração precisa sentir. Talvez sejam anjos, talvez sejam espíritos muito mais evoluídos. Talvez sejam missionários que aqui estão para nos ensinar a ter mais paz e humildade. Estão aqui para servirem de ponte para que possamos encontrar um lugar melhor para descansar os nossos cansaços. Talvez sejam nossos melhores amigos de outras vidas e que vieram nos cuidar nessa.

Existem pessoas que não merecem ir embora. Que não merecem passar pela nossa porta para dizer sequer adeus. São pessoas que nos conservam, nos alimentam a alma, nos tocam. Fazem-nos brotar mais entendimento e conhecimento.

Se você conhece ou convive com pessoas assim é porque Deus lhe capacitou para ser protegido por um grande manto de luz. É sinal de que Ele lhe enviou uma legião de espiritualidade. Uma legião de amor-amigo incondicional. Encante-se com a beleza rara que vem de dentro. Ore por elas. Vibre por elas. Agradeça pela existência delas.”

ABSTRACT

Evaluation of physico-chemical properties of a new epoxy resin-based root canal sealer

Introduction: The aim of this study was to evaluate some of the physico-chemical properties of a new root canal sealer. **Methodology:** The sealers tested were Sealer Plus (MKLife, Porto Alegre, Rio Grande do Sul, Brazil), compared as AH Plus (Dentsply, DeTrey GmbH, Konstanz, Alemanha). For the radiopacity, flow, solubility and fabrication of test specimens relative to setting times the ANSI/ADA No. 57 (2000) and ISO 6876 (2012) specifications were followed. To measure the initial and final setting times the ASTM C266/2008 standard was used. pH was evaluated in the time intervals of 3, 24, 72 and 168 hours). Statistical tests were applied to the results obtained at a level of significance of 5%. **Results:** The results demonstrated that the Sealer Plus sealer showed a lower radiopacity value than AH Plus sealer ($p < 0.05$), however, this was higher than the minimum value recommended by the specifications; that is 3 mm Al. Relative to flow, the value for Sealer Plus was 19.19 mm and for AH Plus, 19.81 mm ($p > 0.05$). Sealer plus presented initial and final setting times of 138 min. and 210 min., respectively, while the values for AH Plus were 437 min. and 849 min., respectively, ($p < 0.05$). Relative to solubility, Sealer Plus presented 0.21% and AH Plus, 0.27% ($p > 0.05$). None of the sealers showed a significant increase in pH ($p > 0.05$). **Conclusion:** Sealer Plus sealer presented physico-chemical properties in accordance with ANSI/ADA (2000) No.57 and ISO 6876 (2012) specifications.

Key words: Endodontic sealers, materials testing, physical and chemical properties, resin epoxy, root canal obturation

RESUMO

Introdução: Este estudo objetivou avaliar algumas propriedades físico-químicas de um novo cimento obturador de canais radiculares. **Metodologia:** Para os testes de radiopacidade, escoamento, solubilidade e confecção dos corpos de prova para os tempos de presa foram seguidas as especificações nº57 da ANSI/ADA (2000) e ISO 6876 (2012). Para a medição dos tempos de presa, inicial e final, foi empregada a norma ASTM C266/2008. O pH foi avaliado nos períodos de 3, 24, 72 e 168 horas. Sobre os resultados foram aplicados testes estatísticos com nível de significância de 5%. **Resultados:** Os resultados demonstraram que o cimento Sealer Plus apresentou menor radiopacidade do que o cimento AH Plus ($p<0.05$), porém, superior ao mínimo recomendado pelas especificações, ou seja, de 3 mm de Al. Com relação ao escoamento, o do Sealer Plus foi 19,19 mm e do AH Plus 19,81 mm ($p>0.05$). O Sealer Plus apresentou tempos de presa inicial e final de 138 min. e 210 min., respectivamente, enquanto o AH Plus, 437 min. e 849 min., respectivamente, ($p<0.05$). Em relação à solubilidade, o Sealer Plus apresentou 0,21% e o AH Plus 0,27% ($p>0.05$). Nenhum dos cimentos apresentou aumento significativo do pH ($p>0.05$). **Conclusão:** O cimento Sealer Plus apresentou propriedades físico-químicas de acordo com as especificações nº 57 da ANSI/ADA (2000) e ISO 6876 (2012).

Palavras-chave: Cimentos endodônticos, propriedades físicas e químicas, resina epóxi, obturação do canal radicular

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1 INTRODUCTION

1 INTRODUCTION

Even with such innovation and progress in endodontics, with new instruments, improvement of techniques, better materials, the basic precepts of endodontic treatment remain the same, based on cleaning, shaping and filling of the root canals (SOUSA-NETO et al., 2018). In this way, endodontic treatment is comprised of a sequence of important steps interdependent with each other and requiring the same attention to be performed satisfactorily. This is aimed at success, not only from a clinical point of view, but also biological (VIVAN et al., 2013).

The goal of the root canal treatment is prevention and healing of apical periodontitis (HARGREAVES; COHEN, 2011), by means of controlling the infection of the root canal system by pathogenic micro-organisms and their communication with the periapical tissues (FLORES et al., 2011). Existence of bacteria is the main cause of periapical inflammation (SALEH et al., 2004). For the long-term success of endodontic treatment is very important to reduce these bacteria from root canal system and for this it is necessary an adequate cleaning, shaping and filling, comprising a set of operative procedures performed in an orderly sequence with a view to achieving a tridimensional sealing (SCHILDER, 1967; HARGREAVES; COHEN, 2011).

The survival of bacteria from the infected root canal system isn't desirable. Eliminating of these microorganisms is achieved through mechanical cleaning, irrigation with antibacterial solutions, as well as temporary sealing between appointments and adequate filling of the root canal (WANG et al., 2014). Actually, even after performing these procedures carefully, the complete elimination of these bacteria is very difficult to achieve in a lot of cases. The root canal treatment reduces but does not necessarily eliminate microbes and viable bacteria often remain in the dentinal tubules and system of canals, which makes complete disinfection impossible (SALEH et al., 2004; WANG et al., 2014). For this reason, an adequate filling of the root canal is considered important and beneficial in the effort to further reduce the number of remaining microorganisms, or by the antimicrobial activity of the sealer, or by their confinement and privation of nutrient supply and space to multiply (SALEH et al., 2004; WANG et al., 2014).

The necessity of a tridimensional sealing is unquestionable (SOUSA-NETO et al., 2018), in view of to eliminate not only residual bacteria and bacterial toxins but also fill the empty space, which has the potential to be infected or re-infected (SIQUEIRA-JÚNIOR et al., 2000), promoting the apical and periapical repair (TANOMARU-FILHO, 2009). A deficient filling may lead to endodontic treatment failure (SCHILDER, 1967; SPANGBERG, 1998).

Under those circumstances, consequently, the root canal obturation plays an important role in both the prevention and control of endodontic infections (SIQUEIRA-JÚNIOR et al., 2000). According to Cohen (1994), Leonardo (1998) and De Deus (1992), an adequate filling must be performed with antiseptic or inert materials, capable of assuring good sealing, to the end that it would prevent percolation and leakage of exudate into the canal, and consequently, the possibility of re-infection, creating an environment favorable to the repair process. However, the root canal filling presents several challenges, mainly regarding the difficulty of adhesion between the filling materials and the canal walls, causing spaces at the material/dentin interface and allowing the entry or migration of microorganisms (VIVAN et al., 2013).

Sousa-Neto et al. (2018) relate Goldberg in 1982, that classified the root canal filling materials into two types: those taken to the root canal in the solid state (gutta-percha) and those taken to the root canal in a plastic state (sealers). In a root canal filling, the use of only the gutta-percha is not enough to fully fill the root canal system, since it does not have the ability to adhere to dentin walls. For this reason, it becomes necessary to associate them with plastic state materials. The adhesion between solid and plastic materials and between them with the dentin walls is desirable to perform and maintain a better sealing (NAJAR et al., 2003; KONTAKIOTIS et al., 2007).

Solid materials associated with endodontic sealers are used in most of root canal filling techniques. Core obturating materials, such as gutta percha, usually occupy space, whereas the endodontic sealers enhance the possible attainment of an impervious seal by serving as a filler for canal irregularities and minor discrepancies between the root canal wall and the solid material (SIQUEIRA-JÚNIOR et al., 2000), filling areas of difficult access, such as ramifications, apical deltas, accessory canals and spaces into which gutta-percha is not able to adapt (KONTAKIOTIS et al., 2007; FLORES et al., 2011).

The endodontic sealers must have ideal physical-chemical and biological properties to obtain the best result, a condition that can influence the repair (DUARTE et al., 2000). Root canal sealers must not affect negatively or, if possible, should stimulate the apical repair and favor the biological seal (OLIVEIRA et al., 2010). It is desirable that the obturation of the root canals be performed with homogeneous, dimensionally stable, inert, physiologically acceptable materials, and which can be manipulated with sufficient plasticity to shape the internal configuration of the root canal system (SCHILDER, 1967).

There are a number of features that root canal sealers should have, as already listed by authors Prinz in 1912, Grossman in 1958, Branstetter and Fraunhofer in 1982, and widely cited in the literature, such as: have satisfactory consistency; be easily introduced and removed from the interior of the canals, if necessary; not to be porous and to remain dimensionally stable; have permanent antiseptic qualities; are not putrefactive agents; exhibit radiopacity; do not discolor dental structures; have good adhesion to the walls of the root canal; should hermetically seal the dentin tubules and apical foramen against bacterial invasion (VIVAN et al., 2013; SOUSA-NETO et al., 2018). According to Grossman (1976), a root canal sealer should have: a slow settling time that allows sufficient working time and excellent sealing, insolubility to the tissue fluids, adequate adhesion to the root canal walls and biocompatibility.

At the same time, it is observed that it is impossible for a single material to meet all these requirements and what usually occurs is the predominance of some of these characteristics. In general, the correct choice of root canal sealers should consider not only adequate physical and chemical properties but also the joint evaluation of other parameters, such as antimicrobial activity, biological behavior and tissue tolerance of material, providing a suitable sealing and low cytotoxicity, favoring or not interfering on the periapical healing (BIN et al., 2012). In endodontics, there has been continuous search for a root canal sealer that combines adequate physico-chemical properties and acceptable biocompatibility, in other words, has been continuous search for quality materials (OLIVEIRA et al., 2010).

Sousa-Neto et al. (2018) affirm that quality of materials is very important for successful dental treatment and in order to manufacture products that meet the quality standards and satisfy the technical and biological requirements, these new or existing

materials in the market must have tested their properties. In fact, the materials used for root canal filling must have evaluated their physico-chemical properties, and to determine this, there are requisites and standardized evaluation tests, defined by the International Organization for Standardization 6876 (ISO, 2012) and Specification 57 to Endodontic Filling and Sealing Materials: Laboratory Testing Methods by American National Standards / American Dental Association (ANSI/ADA, 2000), that established guidelines for the evaluation of sealers, such as flow, setting time, radiopacity, solubility, for example.

Radiopacity is an essential physical property which is necessary to distinguish the filling material from the surrounding anatomical structure such as tooth and bone, allowing to be visualized by means of radiographic exam (VIVAN et al., 2009). Also, radiopacity is necessary to check the correct obturation of the cavity, the quality of the filling, and to facilitate follow-up of the case (DUARTE et al., 2010). To this end, radiopacifying agents are added to the composition of the materials used in endodontic treatment. The standards stipulate that the radiopacity of a root canal filling material minimum is 3mm/AL (ANSI/ADA, 2000).

It is also important that the endodontic sealer had a good flow, so that it can penetrate and fill areas of anatomical complexity, occupying the space not filled by gutta-percha, forming a very compact and complete tridimensional obturator mass. Conversely, if the flow is excessive, the risk of extravasation materials to the periapex is increased, which could damage periodontal tissues and impair periapical healing (SIQUEIRA-JÚNIOR et al., 1995; VERSIANI et al., 2006; JOHNSON; GUTTMANN, 2007; BERNARDES et al., 2010). The standards stipulate that the flow must be greater than 17 mm (ANSI/ADA, 2000; ISO, 2012).

The setting time is the time required for the material to achieve its definitive properties and that, although there is no standard time, its clinical use requires that it must be long enough to ensure a longer filling, such as multi-root tooth, and to allow placement and adjustment of root canal filling if necessary (VERSIANI et al., 2006; RESENDE et al., 2009). In their study, Grossman (1976) affirmed that no ideal setting time has yet been determined, however, this should not be very long, because contact of the material with the periapical tissues may cause irritation in this region. Moreover,

a long setting time may favor the solubility of the material (CARVALHO-JÚNIOR et al., 2007).

Among the desirable physico-chemical properties of endodontic filling materials are insolubility, or low solubility in organic fluids for adequate sealing of root canal systems, required for achieving tridimensional sealing. Dissolution of the material may allow leakage and compromise the success of endodontic treatment, creating gaps that could be colonized by microorganisms and lead to re-infection (CARVALHO-JÚNIOR et al., 2007). The standards stipulate that the solubility must be below 3% (ISO, 2012).

The pH and the release of ions calcium have been pointed as very important chemical properties of a root canal sealers, since they act the tissue level stimulating and accelerating the tissue repair process (SEUX et al., 1991; HOLLAND et al., 2002). This question is intimately related to the setting time and solubility of the material as well as to the area of exposure of the material to the medium in which it is present (VIVAN et al., 2010).

Some authors acknowledge that the endodontics has shown a fast evolution in relation the materials and the clinical protocols, but it is important to note despite exists different endodontic sealers, an ideal endodontic sealer was not developed until this moment. Consequently, endodontic researchers continue to search for better materials as well as more clinically attractive (VERSIANI et al., 2006; RESENDE et al., 2009; FLORES et al., 2011).

At present, the endodontic sealer considered the gold standard by some authors is AH Plus® (Dentsply, DeTrey GmbH, Konstanz, Alemanha), an epoxy resin-based sealer widely used in endodontics and frequently used as a comparison material in endodontic research (SILVA et al., 2017). This cement has excellent physico-chemical (DE ALMEIDA et al., 2000; VERSIANI et al., 2006; SCHÄFER et al., 2015), biological (LEONARDO et al., 1999; TANOMARU-FILHO et al., 2009; MUTOH et al., 2013) and antimicrobial (SALEH et al., 2004; HEYDER et al., 2013; WANG et al., 2014). Data from the literature show that AH Plus presents good sealing, visible radiopacity, easy handling, good resistance, dimensional stability, high flow and low solubility, besides

good adhesion to root canal walls and satisfactory biocompatibility (LEONARDO et al., 1999; BIN et al., 2012).

Currently, new types of endodontic sealers have been proposed trying to improve the physico-chemical and biological properties, thus a better clinical performance. For this, these materials need to be tested to evaluate their quality, safety and properties, ensuring a suitable clinical indication and use (ØRSTAVIK, 2005). Recently, new root canal filling materials were proposed and introduced on the market, such as Sealer Plus cement (MKLife, Porto Alegre, Rio Grande do Sul, Brazil). This is an epoxy resin-based sealer presented in two-pastes. However, until this moment, there are no studies about the physico-chemical properties of this new endodontic sealer in the specific literature.

In this context, the aim of this study was to evaluate some of the physico-chemical properties of this new filling cement in comparison with AH Plus cement, which was selected because it is widely used in endodontics, frequently used as a comparison material in endodontic research and, even though it is considered the gold standard of the endodontic sealers (SILVA et al., 2017), Baldi et al. (2012) showed that the physico-chemical properties can be vary when taken from different parts of the tube. In this way, the evaluate of this new root canal sealer in our study could present better properties, would contribute to literature and endodontic clinical performance. The null hypothesis was: there would be no difference between these two cements in relation to their physico-chemical properties.

Evaluation of Physicochemical Properties of a New Root Canal Sealer

Gislene Cristina Vertuan, DDS,* Marco Antonio Hungaro Duarte, PhD,*
Ivaldo Gomes de Moraes, PhD,* Bruno Piazza, MSc,* Bruno de Carvalho Vasconcelos, PhD,[†]
Murilo Priori Alcalde, MSc,* and Rodrigo Ricci Vivan, PhD*

Abstract

Introduction: The aim of this study was to evaluate some of the physicochemical properties of a new root canal sealer. **Methods:** The sealers tested were Sealer Plus compared with AH Plus. For the radiopacity, flow, solubility, and fabrication of test specimens relative to setting times, the American National Standards Institute/American Dental Association No. 57 (2000) and International Organization for Standardization 6876 (2012) specifications were followed. To measure the initial and final setting times, the ASTM C266/2008 standard was used. pH was evaluated in the time intervals of 3, 24, 72, and 168 hours. Statistical tests were applied to the results obtained at a level of significance of 5%. **Results:** The results demonstrated that the Sealer Plus sealer showed a lower radiopacity value than AH Plus sealer ($P < .05$); however, this was higher than the minimum value recommended by the specifications (ie, 3 mm Al). Relative to flow, the value for Sealer Plus was 19.19 mm and for AH Plus, 19.81 mm ($P > .05$). Sealer Plus presented initial and final setting times of 138 minutes and 210 minutes, respectively, whereas the values for AH Plus were 437 minutes and 849 minutes, respectively ($P < .05$). Relative to solubility, Sealer Plus presented 0.21% and AH Plus, 0.27% ($P > .05$). None of the sealers showed a significant increase in pH ($P > .05$). **Conclusions:** Sealer Plus sealer presented physicochemical properties in accordance with American National Standards Institute/American Dental Association (2000) No. 57 and International Organization for Standardization 6876 (2012) specifications. (*J Endod* 2018;44:501–505)

Key Words

Endodontic sealers, materials testing, physical and chemical properties, resin epoxy, root canal obturation

Successful endodontic treatment depends on root canal cleaning, shaping, and filling (1). The filling stage comprises a set of operative procedures performed in an orderly sequence with a view to achieving tridimensional sealing. Therefore, its purpose is to prevent colonization and reinfection of the root canal by pathogenic microorganisms and their communication with the periapical tissues (2).

A deficient filling may lead to endodontic treatment failure (3, 4). According to Cohen and Burns (5), Leonardo and Leal (6), and De Deus (7), an adequate filling must be performed with antiseptic or inert materials, capable of ensuring good sealing. This would prevent percolation and leakage of exudate into the canal, and consequently, the possibility of reinfection, creating an environment favorable to the repair process. The endodontic sealer performs an important function of filling areas that are difficult to access, such as ramifications, apical deltas, accessory canals, and spaces into which gutta-percha is incapable of being adapted (2, 8).

At present, the filling sealer considered the gold standard is AH Plus (Dentsply, DeTrey GmbH, Konstanz, Germany), frequently used as a comparison material in endodontic research (9). This epoxy resin–based sealer has excellent physicochemical (10–12), biological (13–15), and antimicrobial (16–18) properties.

Recently, new root canal filling materials were proposed and introduced to the market, such as epoxy resin–based Sealer Plus sealer (MKLife, Porto Alegre, Rio Grande do Sul, Brazil). However, up to now, there are no studies in the specific literature about the physicochemical properties of the cited sealer.

The materials used for root canal filling must have adequate physicochemical and biological properties, such as antimicrobial activity and tissue tolerance (19), providing a suitable sealing and low cytotoxicity, favoring or not interfering with the periapical healing (20). To determine the physicochemical properties, there are requisites and standardized evaluation tests, defined by the International Organization for Standardization (ISO) 6876 (21) and American Dental Association (ADA) No. 57 (22). These properties are closely related and justify the biological properties of root canal sealers.

In this context, the aim of this study was to evaluate some of the physicochemical properties of this new filling sealer in comparison with AH Plus sealer. The null hypothesis was as follows: there would be no difference between these 2 sealers in relation to their physicochemical properties.

Significance

The endodontic sealer performs an important function of filling areas that are difficult to access, such as ramifications, apical deltas, accessory canals, and spaces into which gutta-percha is incapable of being adapted.

From the *Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Bauru, SP; and [†]School of Dentistry of Sobral, Federal University of Ceará, Campus Sobral, Sobral, Ceará, Brazil.

Address requests for reprints to Dr Rodrigo Ricci Vivan, Al. Octávio Pinheiro Brisolla, 9-75, 17012-901, Bauru, SP, Brazil. E-mail address: rodrigo.vivan@fob.usp.br 0099-2399/\$ - see front matter

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<https://doi.org/10.1016/j.joen.2017.09.017>

VERTUAN, G. C. et al. Evaluation of physicochemical properties of a new root canal sealer. *J Endod*, New York, v. 44, n. 3, p. 501-505, Mar. 2018. Disponível em: <https://www.ncbi.nlm.nih.gov.ez67.periodicos.capes.gov.br/pubmed/29254816>. Acesso em: 18 abr. 2018.

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Material and Methods

The sealers tested (Table 1) were manipulated in accordance with the manufacturers' instructions. For the radiopacity, solubility, and flow tests, as well as the initial and final setting times, the American National Standards Institute (ANSI)/ADA (22) No. 57 and ISO 6876 (21), specifications that specify the requisites and test methods for root canal filling materials, were followed. The ASTM C266/08 standard was used to determine the final setting time. The analyses were performed by a single, duly calibrated operator.

Radiopacity Analysis

The sealers were carefully poured into metal rings measuring 10 mm in diameter and 1-mm thick that were placed on flat, smooth glass plates. The set was stored in an oven at 37°C. After the sealers had set, the plates were removed and the thicknesses of the test specimens were checked with a pachymeter (Mitutoyo Corp, Tokyo, Japan). Those approved were placed on Kodak insight occlusal film (Kodak Comp, Rochester, NY), together with an aluminum penetrometer (graded from 2 to 16 mm Al). The films were sensitized with an X-ray appliance (Gnatus XR 6010; Gnatus, Ribeirão Preto, SP, Brazil), with 60 kV, 10 mA, with exposure times of 0.3 seconds and focus/film distance of 30 cm.

Radiopacity was analyzed by digital image, by means of the ImageJ 1.48v program (National Institutes of Health, Bethesda, MD). The radiopacity value was determined according to the radiographic density, converted into millimeters of aluminum (mm Al), according to the formula proposed by Duarte et al (23):

$A \times 2/B + \text{mm/AL}$ immediately below RDm
Being:

A = radiographic density of the material (RDm) – radiographic density of the pa aluminum step immediately below RDm;

B = radiographic density of the aluminum step immediately above RDm – radiographic density of the pa aluminum step immediately below RDm;

2 = 2-mm increment between one aluminum step and the other.

Flow Analysis

The recommendations of the ISO 6876/2012 and ANSI/ADA 2000 standards were followed. After manipulation, a volume of 0.05 mL of each sealer was deposited on a glass plate (P1) with the aid of a 1-mL insulin syringe. Within 180 ± 5 seconds after mixing the sealers, a second glass plate (P2), weighing approximately 20 g, was carefully placed on the material. After this, a 100-g weight was placed on this set (P1 + sealer + P2), totaling a mass of 120 g on the endodontic sealer between the 2 plates. After 10 minutes, the weight was removed and the largest and smallest diameters of the discs formed by the compressed sealers were measured with the aid of a digital pachymeter (Mitutoyo MTI Corp., Huntersville, NC). The maximum and minimum diameters were obtained and evaluated 3 times for each specimen, with 5 specimens being used for each experimental sealer.

Setting Time Analysis

Specifications number 57 of the ADA and 6876 of ISO were used for fabrication of the test specimens, and the ASTM C266/08 standard, for determining the setting times. After being duly proportioned and manipulated, the sealers were placed in metal rings measuring 10 mm in internal diameter and 2-mm thick ($n = 5$). The test was performed under controlled temperature and humidity conditions: $37^\circ\text{C} \pm 1^\circ\text{C}$ and $95\% \pm 5\%$, respectively. After 180 seconds had elapsed from the beginning of spatulation, the specimens were marked with vertical pressure, initially using a 113.5-g Gilmore needle to determine the initial setting time, and subsequently, a 453.5-g needle for the final setting time. The initial and final setting time values were computed in minutes for the purpose of statistical analysis.

Solubility Analysis

Three test specimens were fabricated in accordance with the ISO 6876 (21) specification. Teflon rings 20 mm in diameter and 1.5 mm high were used. These were filled with the sealers in a room at a temperature of approximately 25°C. To fill the rings, they were placed on glass plates protected with cellophane paper. After filling, a nylon thread was inserted into the sealer mass. The duly filled rings were protected with cellophane paper, and another glass plate was seated on them to regulate the test specimen surfaces. The set was taken to an oven, where it remained at 37°C and relative humidity of 95% for 3 times the length of the material setting time. After this period had elapsed, the test specimens were removed from the rings, and weighed on an analytical balance with a precision of up to thousandths of a gram. Subsequently they were immersed in receptacles containing 50 mL distilled water, sealed, and maintained for 7 days. The nylon thread that had been inserted into the test specimens allowed them to be suspended in the distilled water without touching the walls of the flasks. After the experimental period, the test specimens were removed, dried with absorbent paper, and taken to a dehumidifier for 24 hours, and were then weighed again. The solubility value was determined by the loss of mass of the test specimen that occurred during the immersion period; that is, by the difference in mass of the test specimen before and after immersion in distilled water.

TABLE 1. Root Canal Sealers, Composition, and Manufacturers

Cement	Composition	Manufacturer
AH Plus	Paste A: Bisphenol epoxy resin-A, Bisphenol epoxy resin-F, calcium tungstate, zirconium oxide, silica, iron oxide pigments. Paste B: Dibenzyl diamine, aminodiamantana, tricyclodecane-diamine, calcium tungstate, zirconium oxide, silica, silicone oil.	Dentsply, DeTrey GmbH, Konstanz, Germany
Sealer Plus	Basic Paste 6.5 g, yellow color: 40% Bisphenol A-co-epichlorohydrin 30; Bisphenol F epoxy resin (formaldehyde, oligomeric product with 40% 1-chloro-2,3-epoxypropanol and phenol) 30; 17% zirconium oxide 13; 4% silicone and siloxanes 2; 0.5% iron oxide (pigment) 0.1; 15% calcium hydroxide 10. Catalyzer Paste 9.5 g, white color: 32% Hexamethylenetetramine 28; 20% zirconium oxide 18; 4% silicone and siloxanes 2; 15% calcium hydroxide 10; 40% calcium tungstate 30.	MK Life, Porto Alegre, RS, Brazil

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pH Analysis

Ten polyethylene tubes measuring 10 mm long and 1 mm in diameter were filled with filling sealer, by using a disposable syringe and hypodermic needle for veterinary use. The specimens were individually placed in test tubes containing 10 mL of ultrapure water (Purelab Option Q, Elga, Brazil), sealed, and taken to an oven at 37°C, where they remained throughout the experimental period. pH was evaluated in the time intervals of 3, 24, 72, and 168 hours. After each experimental time interval, the specimen was removed from the tube and immersed in a new receptacle containing 10 mL of ultrapure water. pH was measured with a pH meter (Orion Star Plus pH meter; Thermo Scientific Electron Corporation, San Jose, CA), previously calibrated with control solutions with known pH values of 4 and 7. The readout temperature was 25°C. As control, the pH value of the ultrapure water (Purelab Option Q) was used.

Statistical Analysis

The data obtained by means of the different evaluations were submitted to the D'Agostino and Pearson tests to verify normal distribution. In the case of normality among 3 or more variables, the 1-way analysis of variance parametric test was used, followed by the Tukey posttest. The Mann-Whitney was used when 2 variables were compared. In case of the absence of normality, the Kruskal-Wallis nonparametric and Dunn tests for individualized analyses were used. In each test performed, a level of significance of 5% was considered. To perform the statistical tests, the GraphPad Prisma 5 program (GraphPad Software, San Diego, CA) was used.

Results

The results obtained are presented in the form of tables. Table 2 shows the means and standard deviations of radiopacity (mm Al), flow (mm) and solubility (%); Table 3 shows the initial and final setting times (minutes), and Table 4, the pH values, according to the sealers tested.

Discussion

The aim of the present study was to evaluate some of the physicochemical properties of a new sealer (Sealer Plus). The null hypothesis was partially rejected. For all the tests with AH Plus sealer, the half and final portions of the tube were used, with the purpose of standardization and avoiding variations in the results (24).

Standardization of the research methodology for the study of physicochemical properties of root canal sealers allows the evaluation of products existent on the market; development of new, and reproduction of methods and results; and more reliable comparisons to be made of data obtained by different studies (25). This standardization was established after the publication of Standard 57 of the ADA (22) and ISO 6876 (21).

Among the physical properties, in general, the standards stipulate that the radiopacity of a root canal filling material must be greater than that of dentin, minimum of 3 mm/Al; flow must be greater than 17 mm; setting time must not be long, and solubility must be below 3%.

Alkalinization capacity (increase in pH) may be considered an important chemical property, because it may induce repair by stimu-

TABLE 3. Means and Standard Deviations of Initial and Final Setting Times (Minutes) for Each of the Cements Tested

Cement	Initial setting time	Final setting time
Sealer Plus	138 (10) ^a	210 (18) ^a
AH Plus	437 (7) ^b	849 (15) ^b

Different letters in each column indicate statistically significant differences ($P < .05$).

lating the mineralization process (26). This question is closely related to the setting time and solubility of the material (27). This study showed that the AH Plus and Sealer Plus did not promote a significant pH increase. Probably, the low solubility presented for both endodontic sealers could explain our results. In addition, the Sealer Plus presented the shortest setting time, which could did not allow the calcium hydroxide release and, consequently, did not promote the pH increase. Our results are in agreement with Vivan et al (27), who reported that the filling materials that have calcium hydroxide in the composition depend on the setting time and solubility to allow a suitable hydroxyl ion release.

In relation to setting time and solubility, it is permissible to affirm that the release of hydroxyl ions by an insoluble material would occur only in the period preceding its final setting. However, this release may occur irrespective of setting, if the solubility of the material were a constant. Therefore, solubility and the release of hydroxyl ions, or even calcium ions, go together (27).

Radiopacity is an essential physical property because it allows the filling material to be visualized by means of radiographic examination, to check the quality of the filling (28). The standardization recommends the minimum radiopacity value of 3.00 mm/Al. In the results found by this study, both sealers presented values higher than the minimum recommended. In spite of the radiopacifying agents present in the constitution of these materials being the same; that is, calcium tungstate and zirconium oxide, the radiographic density of the AH Plus sealer was higher than that of Sealer Plus, with statistical significance ($P < .05$). This result allowed the authors to deduce that the proportion of the radiopacifying agents was certainly lower in the composition of Sealer Plus sealer. Comparison of the quantity of substances present in the composition of the sealers tested showed that the 2 pastes (A and B) of AH Plus contained the 2 radiopacifying agents (calcium tungstate and zirconium oxide), whereas Sealer Plus contained calcium hydroxide in the 2 pastes, but this substance had minimal radiopacity, and it contained only 1 of the radiopacifying agents (zirconium oxide) in the basic paste.

A higher flow value was observed for AH Plus sealer. These values corroborated the findings about this sealer in other studies (10, 29–32). Siqueira Jr et al (33) reported that flow plays an important role in filling sealer, because it allows the sealer to penetrate into the irregularities, isthmus, and ramifications of root canal systems. Duarte et al (34) added that this was an important property enabling filling to occur in irregular areas of a root canal.

The most frequently used method for determining the setting time is with the use of Gilmore needles that are placed on the sealer surface, and when the action of their own weight leaves no marks, the material is considered to have set (35). In this study, Gilmore needles of 113.5 g were used to evaluate initial setting, 453.5 g to evaluate final setting of the sealers. The study by Grosman (35) affirmed that no ideal setting time has yet been determined; however, this should not be very long, because contact of the material with the periapical tissues may cause irritation in this region.

Moreover, a long setting time may favor the solubility of the material in contact with oral fluids and cause loss of substance of the

TABLE 2. Means and Standard Deviations of Radiopacity (mm Al), Flow (mm), and Solubility (%) of the Cements Tested

Cement	Radiopacity	Flow	Solubility
Sealer Plus	5.42 (0.20) ^a	19.19 (0.52) ^a	0.266 (0.027) ^a
AH Plus	7.58 (0.14) ^b	19.81 (1.58) ^a	0.212 (0.046) ^a

Different letters in each column indicate statistically significant differences ($P < .05$).

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TABLE 4. Means and Standard Deviations of pH Values Found for Each Cement, in the Different Time Intervals Analyzed

Cement	3 h	24 h	72 h	168 h
Sealer Plus	5.65 (0.28) ^{a,A,B}	5.43 (0.19) ^{a,C}	5.47 (0.13) ^{a,B,C}	5.69 (0.17) ^{a,A}
AH Plus	5.51 (0.21) ^{a,A}	5.05 (0.06) ^{b,C}	5.23 (0.13) ^{b,B}	5.14 (0.06) ^{b,B,C}

The control, ultrapure water (Purelab Option Q, Elga, Brazil), presented pH value of 5.6. Values followed by different lower-case letters in each line indicate statistically significant differences according to the Student *t* test ($P < .05$). Values followed by different upper-case letters in each column indicate statistically significant differences according to analysis of variance and Tukey test ($P < .05$).

material, creating gaps that could be colonized by microorganisms and lead to reinfection (25). Nevertheless, the chemical characteristics of the material have to be considered, particularly their area in contact with tissue fluids. Filling sealers with solubility proven by routine solubility tests, and that promoted high pH values, showed no increase in pH when used under conditions representative of clinical use.

The solubility test was conducted in accordance with the ISO 6876 (21) standard, and in accordance with this standard, the solubility of an endodontic sealer must not exceed 3%. In this study, both the sealers tested were found to be within the patterns demanded by the standards, because they obtained mean values of 0.212% (AH Plus) and 0.266% (Sealer Plus). No significant differences were detected between them.

Among the desirable physicochemical properties of a filling sealer are insolubility, or low solubility in organic fluids for adequate sealing of root canal systems, required for achieving tridimensional sealing. Dissolution of the material may allow leakage and compromise the success of endodontic treatment (25). This is because the high solubility of a filling sealer may lead to loss of its structure and its disintegration, causing gaps at the sealer/dentin and sealer/gutta-percha interface, thereby creating access pathways for microorganisms and their toxic products, causing damage to the periapical tissue (25).

The pH together with calcium ion release, a property not evaluated in this study, have been pointed out as the most important properties of a filling sealer, because they act at the tissue level, stimulating and accelerating the repair process. Sealers that contain calcium oxide or hydroxide have the property of dissociating into calcium and hydroxyl ions, resulting in an increase in pH in the medium, and mineralized tissue formation. The calcium ions react with the carbon dioxide present in the tissue, and form calcite crystals, important for initiating the calcification process (26).

As pointed out previously, the release of hydroxyl ions as a consequence of increase in pH, or even the release of calcium ions, are dependent on the area of contact of the material with tissue fluids, chemical characteristics (hydrophilic or hydrophobic), presence of calcium-containing substances, setting time, and solubility. When analyzing the results, particularly those relative to Sealer Plus, the authors observed that although it had calcium hydroxide in its composition, it was not capable of alkalizing the medium to a significant extent, because the initial pH of the water was 5.6. The pH values of the 2 sealers tested were slightly below those of the control. This may perhaps have occurred as a result of the pH of the other substances of which the sealers were composed, mainly the resins (24). Therefore, the presence of an alkaline material in the Sealer Plus sealer had no action on the increase in pH. Its extremely short setting time associated with its low solubility certainly did not allow any release of hydroxyl ions.

As this is a new sealer, Sealer Plus presented no parameters for comparison. Further tests are necessary to enable indication of this sealer for clinical use. In view of the methodologies used and results obtained, the authors could conclude that Sealer Plus presented physicochemical properties in accordance with specification No. 57 of ANSI/ADA (22) and ISO 6876 (21).

Acknowledgment

The authors deny any conflicts of interest related to this study.

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3 DISCUSSION

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In endodontics there is a concern about the prevention and control of the root canal infection. Although a thorough chemomechanical prepare has been carried out, is essential a careful obturation of root canal system to avoid reinfection either by infiltration or by bacterial recolonization (SALEH et al., 2004). The survival of bacteria from the infected root canal system isn't desirable. Reducing of these microorganisms is achieved through mechanical cleaning, irrigation with antibacterial solutions, as well as temporary sealing between appointments and adequate tridimensional filling of the root canal (WANG et al., 2014).

Endodontic treatment reduces but does not necessarily eliminate microbes and viable bacteria often remain in the dentinal tubules and system of canals. Therefore, an adequate filling of the root canal is considered important and beneficial in the effort to further reduce the number of remaining microorganisms by their confinement or denying a nutrient supply (WANG et al., 2014). In summary, after cleaning and shaping an adequate filling can prevent and control bacterial infiltration, together with a satisfactory crown sealing, have been accepted as factors contributing to the success of endodontic treatment (RAY; TROPE, 1995; SANTOS et al., 2010; MARCIANO et al., 2011).

Sousa-Neto et al. (2018) affirm that in order to obtain successful dental treatment is very important the quality of materials. In most of root canal filling techniques are used solid materials associated with endodontic sealers, in a plastic state, for the purpose of obtain a better sealing, as a result of the adhesion between solid and plastic materials and between them with the dentin walls (SALEH et al., 2002; NAJAR et al., 2003; KONTAKIOTIS et al., 2007), filling areas of difficult access, such as ramifications, apical deltas, accessory canals and spaces into which gutta-percha is not able to adapt (KONTAKIOTIS et al., 2007; FLORES et al., 2011). It is fundamental the use materials that ensure satisfactory sealing and tissue tolerance, favoring the periapical healing. The endodontic filling materials should present suitable physico-chemical and biological properties (BIN et al., 2012).

The epoxy resin-based sealer named AH Plus is considered the gold standard of endodontic sealers by same authors, showing satisfactory physico-chemical and biological properties, had been widely used in endodontics and frequently used as a comparison material in endodontic research (SILVA et al., 2017). However its physico-chemical properties are be variable and presented instability, when taken from different parts of the tube (BALDI et al., 2012). AH Plus is not uniform and changes its consistency in the tubes; it was very fluid at the beginning of the tube and thicker at the end. This assumption was supported by the flow and radiopacity values in a Baldi et al. (2012) study, which were statistically different depending on the portion of the tubes studied.

There has been continuous endodontics search for a root canal sealer that combines adequate properties (OLIVEIRA et al., 2010). Some authors acknowledge that although exists different endodontic sealers, an ideal endodontic sealer was not developed until this moment (FLORES et al., 2011). Therefore, endodontic researchers continue to search for better materials as well as more clinically attractive.

Recently, a new resin epoxy based sealer were proposed and introduced on the market, named as Sealer Plus, which there are no report in the literature and could present better results and would contribute to endodontic clinical performance. Thus, it was relevant to accomplish this in vitro study, which was based on the current regulations and recommended for evaluation of physico-chemical properties of an endodontic sealer (ANSI/ADA no. 57/2000 and ISO 6876/2012).

The aim of this study was to evaluate some of the physico-chemical properties of this new root canal sealer Sealer Plus. The null hypothesis was partially rejected.

Endodontic sealers presented in two pastes should have their portions proportioned and homogenized. However, the complete miscibility is assumed to be lacking between the organic and inorganic components, contribute to segregation between them, which certainly influence the monomer conversion ratio, causing a variation in the physicochemical and mechanical properties, presenting instability when taken from different parts of the tube and resulting in materials that can be varied from study to study (BALDI et al., 2012). For this reason, with the purpose of standardization and avoiding variations in the results, for all tests with AH Plus, employee as a

comparative sealer, the half and final portions of the tube were used (BALDI et al., 2012).

Standardization of the research methodology for the study of physico-chemical properties of root canal filling cements allows the evaluation of products existent on the market; development of new, and reproduction of methods and results; and more reliable comparisons to be made of data obtained by different studies (CARVALHO-JÚNIOR et al., 2007). This standardization was established after the publication of Standard 57 of the American Dental Association (2000) and International Organization for Standardization 6876 (2012). The standards stipulate, in general, that the radiopacity of a root canal filling material must be greater than that of dentin, minimum of 3 mm/AL; flow must be greater than 17 mm; setting time must not be long, and solubility must be below 3%.

Radiopacity is an essential physical property because it allows the filling material to be visualized by means of radiographic exam, to check the quality of the filling (VIVAN et al., 2009). The standardization recommends the minimum radiopacity value of 3.00 mm/Al. In the results found by this study, both cements presented values higher than the minimum recommended, 5.42 mm/Al for Sealer Plus and 7.58 mm/Al for AH Plus. In spite of the radiopacifying agents present in the constitution of these materials being the same; that is, calcium tungstate and zirconium oxide, the radiographic density of the AH Plus cement was higher than that of Sealer Plus, with statistical significance ($p < 0.05$), which could be explained by the amount of the substance in the composition of each of the materials.

This result allowed the authors to deduce that the proportion of the radiopacifying agents was certainly lower in the composition of Sealer Plus cement. Comparison of the quantity of substances present in the composition of the cements tested showed that the two pastes (A and B) of AH Plus contained the two radiopacifying agents (calcium tungstate and zirconium oxide). Whereas, Sealer Plus contained calcium hydroxide in the two pastes, but this substance had minimal radiopacity; and it contained only one of the radiopacifying agents (zirconium oxide) in the basic paste.

In the same way, the results obtained in the present study for AH Plus corroborate to those of the other studies, which were found to be higher than of other cements, with variations ranging from 6 to 14.50 mm/Al (RESENDE et al., 2009; FLORES et al., 2011; MARCIANO et al., 2011; SCHÄFER et al., 2015). This variability found in the studies could also be explained by Baldi et al. (2012), depending on the portion of the tube where the material was being dispensed, from the beginning, half or final of the tube.

The flow plays an important role in a root canal sealer, because it allows the material to penetrate into accessory canals, irregularities, isthmus, and ramifications of root canal system, which increases the likelihood of obtaining an adequate seal (SIQUEIRA-JÚNIOR et al., 1995). Several factors may influence the penetration of endodontic sealers within confined areas of root canal system, such as the obturation technique used, the contact area, the dimensional irregularities, accessibility to the complexities and the sealer's flow rate. Ideally, the material flow cannot be excessive, because the risk of extravasation materials to the periapex is increased, which could damage periodontal tissues and impair periapical healing (SIQUEIRA-JÚNIOR et al., 1995; VERSIANI et al., 2006; JOHNSON; GUTTMANN, 2007; BERNARDES et al., 2010).

A higher flow value was observed for AH Plus cement showing 19.81mm against 19.19mm for Sealer Plus, with no statistically significance difference. These values for AH Plus corroborated the findings about this cement in other studies (SIQUEIRA-JÚNIOR et al., 1995; MCMICHEN et al., 2003; VERSIANI et al., 2006; RESENDE et al., 2009; BERNARDES et al., 2010).

The setting time is the time required for the material to achieve its definitive properties and that is dependent on its components, particle size, ambient temperature and relative humidity (ØRSTAVIK, 2005; VERSIANI et al., 2006). A short setting time may difficult a longer filling, mainly if necessary its adjustment. In the other hand, a long setting time may favor the contact of the material with the periapical tissues causing irritation in this region (GROSMAN, 1976). Moreover, a long setting time may favor the solubility of the material in contact with oral fluids (CARVALHO-JÚNIOR et al., 2007).

The most frequently used method for determining the setting time is with the use of Gilmore needles that are placed on the cement surface, and when the action of their own weight leaves no marks, the material is considered to have set (GROSMAN, 1976). In this study, Gilmore needles of 113.5 g were used to evaluate initial setting, 453.5 g to evaluate final setting of the cements, carried out in accordance to the American Society for Testing Materials Specifications ASTM C266/2008, but the samples were made following the ISO 6876 (2012) standard.

In this study, the setting time of AH Plus (849 minutes) was 4 times greater than that of Sealer Plus (210 minutes) and significant differences were detected between them. The setting time of AH Plus finding in other studies range from 494 minutes (RESENDE et al., 2009), 500 minutes (VERSIANI et al., 2006), 580 minutes (FLORES et al., 2011), 711 minutes (MARCIANO et al., 2011) and 1440 minutes (SONNTAG et al., 2015).

Endodontic sealers presented in two pastes, a base and another catalyst, after being proportioned and homogenized, have a slow reaction of polymerization of their epoxy resin amines, so that the conversion of monomers into polymers occurs gradually, which can explain the longer setting time of these materials (BALDI et al., 2012).

The low solubility in organic fluids is a desirable physico-chemical property of a sealer in order to obtain an adequate sealing of root canal systems, required for achieving tridimensional sealing. The high solubility of a root canal sealer might result in loss of structure to the oral environment and create lack of integrity in the sealer, causing gaps at the cement/dentin and cement/gutta-percha interface, and in that case these spaces might provide a pathway for microorganisms and their toxic products into periapical tissues (CARVALHO-JÚNIOR et al., 2007).

The solubility test was conducted in accordance as the ISO 6876 (2012) standard, and in accordance with this, the solubility of an endodontic cement must not exceed 3%. In this study, both the cements tested were found to be within the patterns demanded by the standards, because they obtained mean values of 0.212% (AH Plus) and 0.266% (Sealer Plus). No significant differences were detected between them. Data found in this study about AH Plus corroborate with others, including Versiani et

al. (2006) which find value of 0.21% solubility, in the same way others that show its low solubility (RESENDE et al., 2009; FLORES et al., 2011; MARCIANO et al., 2011; SONNTAG et al., 2015).

Although this may be true the chemical characteristics of the material have to be considered, particularly their area in contact with tissue fluids. Root canal sealers with solubility proven by routine solubility tests, and which promoted high pH values, showed no increase in pH when used under conditions representative of clinical use (VIVAN et al., 2010).

The pH together with calcium ion release - a property not evaluated in this study - have been pointed out as most important properties of a filling cement, because they act at tissue level, stimulating and accelerating the repair process. Cements that contain calcium oxide or hydroxide have the property of dissociating into calcium and hydroxyl ions, resulting in an increase in pH in the medium, and mineralized tissue formation. The calcium ions react with the carbon dioxide present in the tissue, and form calcite crystals, important for initiating the calcification process (ESTRELA et al., 1995; HOLLAND et al., 2002).

At the same time, the presence of calcium hydroxide in a material does not ensure its therapeutically efficiency, because the release of hydroxyl ions from the sealer depends on the hydrophilic or hydrophobic nature of its matrix and the other substances added to its formulation (SIQUEIRA-JÚNIOR et al., 1995). The release of hydroxyl ions as a consequence of increase in pH, or even the release of calcium ions, are dependent on the area of contact of the material with tissue fluids; chemical characteristics (hydrophilic or hydrophobic); presence of calcium-containing substances; setting time and solubility (VIVAN et al., 2010). In relation to setting time and solubility, it is permissible to affirm that the release of hydroxyl ions by an insoluble material would only occur in the period preceding its final setting. However, this release may occur irrespective of setting, if the solubility of the material were a constant. Therefore, solubility and the release of hydroxyl ions, or even calcium ions, go together (VIVAN et al., 2010).

When analyzing the results, particularly those relative to Sealer Plus, the authors observed that although it had calcium hydroxide in its composition, it was not

capable of alkalizing the medium to a significant extent, because the initial pH of the water was 5.6. The pH values of the two cements tested were slightly below those of the control. This may perhaps have occurred as a result of the pH of the other substances of which the cements were composed, mainly the resins. Therefore, the presence of an alkaline material in the Sealer Plus cement had no action on the increase in pH. It's extremely short setting time associated with its low solubility certainly did not allow any release of hydroxyl ions.

The ultrapure water used in this study was processed by the Purelab (Purelab Option Q, Elga, Brazil) equipment, which is located on the Biochemistry Department of University of São Paulo, Bauru Dental School. The pH measurement was performed three times by pH meter (Orion Star Plus pH meter, Thermo Scientific Electron Corporation, San Jose, California, United States of America). After the first measurement, another sample of ultrapure water was taken of other Purelab equipment (Purelab Option Q, Elga, Brazil), located in the same laboratory, and repeated the pH measurements. The second results had no difference with the first pH measurement. Although two different equipment of ultrapure water were used, the results were similar. Probably, the pH measurement had no difference because the water supply of the University was the same it is from a public source, which is previously treated before the distribution. Maybe the treatment could induce a low pH values even when filtered in the ultrapure water equipment.

Under those circumstances, was chosen to perform the tests with the propose of submit the sealers to the same situation, an acid challenge test, allowing to verify the alkalinization capacity of the endodontic sealers. In addition, during a periapical inflammation or infection, the periapical tissues are an acid ambient with pH 5.5 or 5.6 from pus (MALAMED, 2004), similar pH of the ultrapure water used in this study.

The results of this studies showed that the sealers tested did not promoted a significant pH increase. It can be observed that the pH of the medium in which the specimens were had little change, both for alkalinization and for acidification. The tendency of alteration of hydroxyl ion release is due to the chemical reaction between the epoxy resin and the hardener, which decrease the sealer solubility, and consequently the release of the ions presents in the composition to decrease (VIVAN et al., 2010; BALDI et al., 2012). The possible explanation could be related to the short

setting time and low solubility, which can difficulty the calcium hydroxide dissolution and hydroxyl ions release (VIVAN et al., 2010).

As this is a new cement, Sealer Plus presented no parameters for comparison. Further tests are necessary to enable indication of this cement for clinical use. Given these points, the results showed that Sealer Plus has a suitable performance as good as AH Plus and can be used for an adequate endodontic obturation.

4 CONCLUSION

4 CONCLUSION

According to standardization and methodologies applied in this study, in view of the results obtained, it could be concluded that Sealer Plus presented physico-chemical properties in accordance with the specifications No.57 of ANSI/ADA (2000) and ISO 6876 (2012).

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APPENDIX

DECLARATION OF EXCLUSIVE USE OF THE ARTICLE IN DISSERTATION

We hereby declare that we are aware of the article (EVALUATION OF PHYSICOCHEMICAL PROPERTIES OF A NEW ROOT CANAL SEALER) will be included in Dissertation of the student Gislene Cristina Vertuan was not used and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, 14 de maio de 2018.

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Publication: Journal of Endodontics

Publisher: Elsevier

Date: March 2018

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