



**Universidade Norte do Paraná**

**UNOPAR**

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CENTRO DE CIÊNCIAS BIOLÓGICAS E DA SAÚDE  
MESTRADO EM ODONTOLOGIA

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**INFLUÊNCIA DE DESAFIOS QUÍMICOS NAS  
PROPRIEDADES DE RESINAS COMPOSTAS**

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Londrina  
2011

VIVIAN CRISTIANE BUENO RIBEIRO DE JESUS

## **INFLUÊNCIA DE DESAFIOS QUÍMICOS NAS PROPRIEDADES DE RESINAS COMPOSTAS**

Dissertação apresentada à Universidade Norte do Paraná - UNOPAR, como requisito parcial para a obtenção do título de Mestre em Odontologia, área de concentração Dentística Preventiva e Restauradora.

Orientadora: Profa. Dra. Ana Raquel Benetti  
Co-orientadora: Profa. Dra. Regina Célia Poli-Frederico

Londrina  
2011

AUTORIZO A REPRODUÇÃO TOTAL OU PARCIAL DESTE TRABALHO, POR QUALQUER MEIO CONVENCIONAL OU ELETRÔNICO, PARA FINS DE ESTUDO E PESQUISA, DESDE QUE CITADA A FONTE.

## Dados Internacionais de catalogação na publicação

Universidade Norte do Paraná

Biblioteca Central

Setor de Tratamento da Informação

J56i

Jesus, Vivian Cristiane Bueno Ribeiro de.

Influência de desafios químicos nas propriedades de resinas compostas / Vivian Cristiane Bueno Ribeiro de Jesus . Londrina: [s.n], 2011. lli; 52p.

Dissertação (Mestrado). Odontologia. Dentística Preventiva e Restauradora. Universidade Norte do Paraná.

Orientador: Prof<sup>a</sup>. Dr<sup>a</sup>. Ana Raquel Benetti

1- Odontologia - dissertação de mestrado - UNOPAR 2- Resinas compostas 3- Cor 4- Descoloração 5- Embebição 6- Solubilidade 7- Rugosidade I- Benetti, Ana Raquel, orient. II- Universidade Norte do Paraná.

CDU 616.314-089.27/.28



# Universidade Norte do Paraná

## Pró-Reitoria de Pesquisa e Pós-Graduação

### Programa do Mestrado em Odontologia – Dentística Preventiva e Restauradora

#### ATA DE DEFESA DE DISSERTAÇÃO

Aos dezanove dias do mês de dezembro do ano de dois mil e onze, no Centro de Ciências Biológicas e da Saúde, desta Universidade, às nove horas, reuniu-se a Banca Examinadora indicada pelo Programa de Pós-Graduação e homologada pelo Colegiado dos Programas Pós-Graduação *Stricto Sensu*, composta por 1. Prof<sup>ª</sup>. Dr<sup>ª</sup>. Ana Raquel Benetti, presidente da banca. 2. Prof<sup>ª</sup>. Dr<sup>ª</sup>. Raquel Sano Suga Terada. 3. Prof<sup>ª</sup>. Dr<sup>ª</sup>. Sandrine Bittencourt Berger. A reunião tem por objetivo julgar o trabalho da aluna *Vivian Cristiane Bueno Ribeiro de Jesus Gomes*, sob o título “*Influência de desafios químicos nas propriedades de resinas compostas*”. Os trabalhos foram abertos pela presidente da banca. A seguir foi dada a palavra à estudante para apresentação do trabalho. Cada examinadora arguiu a mestranda, com tempos iguais de arguição e resposta. Terminadas as arguições, procedeu-se o julgamento do trabalho, concluindo a Banca Examinadora de Dissertação por sua **APROVAÇÃO** e com a recomendação de envio dos exemplares no prazo de 60 dias, para homologação pelo Colegiado de Pós-Graduação. Nada mais havendo a tratar, foi lavrada a presente ata, que vai assinada pelos membros da Banca Examinadora.


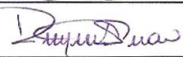
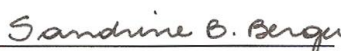
Londrina, 19 de dezembro de 2011

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## O sonho

Sonhe com aquilo que você quer ser,  
porque você possui apenas uma vida  
e nela só se tem uma chance  
de fazer aquilo que quer.

Tenha felicidade bastante para fazê-la doce.  
Dificuldades para fazê-la forte.  
Tristeza para fazê-la humana.  
E esperança suficiente para fazê-la feliz.

As pessoas mais felizes não têm as melhores coisas.  
Elas sabem fazer o melhor das oportunidades  
que aparecem em seus caminhos.

A felicidade aparece para aqueles que choram.  
Para aqueles que se machucam.  
Para aqueles que buscam e tentam sempre.  
E para aqueles que reconhecem  
a importância das pessoas que passaram por suas vidas.

Clarice Lispector

À Deus,

Aos meus pais Waldomiro e Edna, que me ensinaram o valor do amor e da dignidade e me deram muito apoio nos momentos mais difíceis da minha vida, aos meus irmãos Rodrigo e Aline, meu esposo Pedro, que esteve ao meu lado não medindo esforços para me ajudar, e pela compreensão do meu filhinho Matheus, pelas horas ausentes para realização deste trabalho.

## AGRADECIMENTOS

À Universidade Norte do Paraná, UNOPAR, representada pela Chanceler, Prof<sup>a</sup>. Elisabeth Bueno Laffranchi e pela Reitora, Profa. Wilma Jandre Mello;

À Pró-Reitoria de Pesquisa e Pós-Graduação, representada pelo Prof. Dr. Hélio Hiroshi Suguimoto;

Ao Centro de Ciências Biológicas e da Saúde, representado pelo Prof. Ruy Moreira da Costa Filho;

Ao Programa de Pós-graduação em Odontologia, representado pelo Prof. Dr. Alcides Gonini Júnior;

Agradeço em especial à professora Ana Raquel Benetti pela amizade e orientação, pelas suas observações, por seu apoio e inspiração no amadurecimento dos meus conhecimentos e conceitos que me levaram à execução e conclusão deste trabalho.

A todos os professores do Mestrado em Odontologia da Unopar que foram tão importantes na minha vida acadêmica e no desenvolvimento desta dissertação.

Agradeço também, as professoras Raquel Terada e Sandrine Berger, que gentilmente aceitaram o convite para participação desta banca.

À professora Terezinha Barata pelas observações, sugestões e também pelo incentivo.

Ao Natan Luiz Martinelli, que colaborou com os experimentos.

Aos amigos e colegas, em especial, Karla Ulinski e Renata Carreira pela grande amizade, pelo incentivo e pelo apoio constantes.

Agradeço também à Míria Cristina Bernini e ao Jonatas Renan G. Montanucci, pela atenção e colaboração na utilização do laboratório da Biodinâmica.

A todos os funcionários da UNOPAR.

Agradeço a todas as pessoas que contribuíram para a conclusão deste trabalho, a todas elas registro minha imensa gratidão.

JESUS, Vivian Cristiane Bueno Ribeiro de. Influência de desafios químicos nas propriedades de resinas compostas. 2011. 52p. Dissertação (Mestrado em Odontologia). Universidade Norte do Paraná, Londrina.

## RESUMO

O objetivo desse estudo foi investigar o efeito de desafios químicos prolongados na estabilidade de cor, susceptibilidade ao manchamento, sorção de água, solubilidade e rugosidade de uma resina composta à base de silorano, quando comparada a resinas à base de metacrilatos. Corpos-de-prova cilíndricos foram fabricados a partir de quatro resinas compostas para os testes de estabilidade de cor (n=12), rugosidade, sorção e solubilidade (n=24). Inicialmente foram registradas a cor, a massa e a rugosidade de todos os corpos-de-prova das resinas à base de silorano (Filtek P90, 3M ESPE) ou metacrilatos (Filtek Z250, 3M ESPE; Filtek Z350XT, 3M ESPE; Master Fill, Biodinâmica). Os corpos-de-prova foram armazenados individualmente a 37°C em soluções de ácido cítrico a 0,02N, ácido fosfórico a 0,02N, etanol a 75% ou água durante 7, 14, 21 e 180 dias, quando novas medidas foram conduzidas. Um teste de manchamento foi realizado após os desafios químicos através da imersão dos corpos-de-prova em café durante 3 semanas a 37°C. As mudanças de cor foram caracterizadas pelo sistema CIEL\*a\*b\*. Os dados foram submetidos à análise de variância a dois critérios ou para medidas repetidas, seguido do teste de comparações múltiplas de Tukey ( $\alpha = 0,05$ ). Embora estatisticamente significantes, as alterações de cor nas resinas à base de metacrilatos não foram visivelmente perceptíveis após a imersão em água, ácido cítrico, ácido fosfórico ou etanol, mas mostraram-se visíveis para a resina à base de silorano imersa em etanol por 180 dias. Após o teste de manchamento, as resinas à base de metacrilatos sofreram maior pigmentação pelo café após o armazenamento em etanol, quando comparadas à imersão em outras soluções. A resina à base de silorano não apresentou manchamento, além de demonstrar baixa sorção de água e solubilidade, porém mostrou-se mais rugosa do que as resinas à base de metacrilatos investigadas nesse estudo. Não se observou efeito prejudicial das soluções na rugosidade ou solubilidade dos materiais investigados. A partir das limitações desse estudo, concluiu-se que o contato com o etanol afetou a estabilidade de cor, a sorção de água e o grau de manchamento das resinas à base de silorano ou metacrilatos de modo distinto.

**Palavras-chave:** Resinas compostas; Cor; Descoloração; Embebição; Solubilidade; Rugosidade.



JESUS, Vivian Cristiane Bueno Ribeiro de. Influence of chemical challenges on the properties of resin composites. 2011. 52p. Dissertation (Master in Dentistry). University North of Paraná, Londrina.

## ABSTRACT

The purpose of this study was to investigate the effect of prolonged chemical challenges on color stability, staining susceptibility, water sorption, solubility, and roughness of a silorane-based resin composite when compared to methacrylate-based composites. Cylindrical specimens were fabricated from four resin composites for the different tests. Initial color, roughness and mass were registered for specimens fabricated from methacrylate (Filtek Z250, 3M ESPE; Filtek Z350XT, 3M ESPE; Master Fill, Biodinâmica) or silorane-based (Filtek P90, 3M ESPE) resins. Specimens were individually stored at 37°C in 0.02N citric acid, 0.02N phosphoric acid, 75% ethanol or distilled water for 7, 14, 21, and 180 days, when new measurements were performed. A staining test was performed after the chemical challenge by immersion in coffee during 3 weeks at 37°C. Color changes were characterized using the CIEL\*a\*b\* color system. Data were submitted to two-way analysis of variance or analysis of variance for repeated measures, followed by Tukey's multiple comparison test. Color changes on the methacrylate-based resins were not visually perceptible after immersion in water, citric acid, phosphoric acid or ethanol, but were visible for the silorane-based resin immersed in ethanol for 180 days. The methacrylate-based resins stored in ethanol were significantly more stained by coffee than those stored in other media. The silorane-based resin demonstrated no staining, low water sorption and solubility, but increased roughness, when compared to the methacrylate-based resins. No effect of the immersion solution was noticed on roughness or solubility of the investigated materials. Considering the limitations from this study, it was concluded that the contact with ethanol influenced color stability, water sorption and staining susceptibility differently for the methacrylate or silorane-based resins.

**Keywords:** Composite resins; Color; Discoloration; Absorption; Solubility; Roughness

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## 1 INTRODUÇÃO

A odontologia restauradora atual se depara com alguns desafios, dentre eles o desenvolvimento de materiais restauradores que apresentem durabilidade e estabilidade na cavidade bucal, um ambiente rico em patógenos e umidade, sujeito à ação de soluções químicas e alimentos. O desgaste químico superficial que afeta a estrutura dentária, sem o envolvimento de microorganismos, mas provocado por produtos ácidos e químicos, acomete também os materiais restauradores e modifica suas propriedades (YAP et al., 2005). Quando em contato com essas substâncias, diferentes propriedades das resinas compostas podem ser prejudicadas, dentre elas a perda de massa e aumento da rugosidade superficial (CHAN et al., 1980; WILTSHIRE; LABUSCHAGNE, 1990).

Nos dias atuais, os avanços na composição das resinas compostas têm trazido vantagens consideráveis. Novas resinas compostas têm sido desenvolvidas com a finalidade de superar as limitações desses materiais restauradores. Dentre os principais avanços conseguidos nas resinas compostas, destaca-se a silanização e redução no tamanho das partículas de carga, o que trouxe melhorias em características como estética, lisura superficial, e desgaste superficial das resinas compostas. Adicionalmente, desenvolveu-se uma nova matriz resinosa, o silorano, com o propósito de superar alguns inconvenientes relacionados às resinas compostas à base de metacrilato, como a inibição da polimerização provocada por radicais de oxigênio, a formação de tensões e a contração volumétrica resultantes da reação de polimerização. O material à base de silorano se polimeriza através da abertura de anéis oxirano (EICK et al., 2006). Nesse sentido, este material pode reduzir a deflexão das cúspides em direção ao centro do dente em preparos cavitários extensos (PALIN et al., 2005b).

Outra característica da matriz orgânica à base de silorano é sua maior hidrofobicidade, e conseqüente maior estabilidade, devido à presença de radicais siloxano. A maior hidrofobicidade é responsável pela sua estabilidade de cor, manutenção do polimento (FURUSE et al., 2008), menor adesão de placa bacteriana (BUERGERS et al., 2009), além de menor sorção de água, solubilidade e coeficiente de difusão (ILIE; HICKEL, 2006). A associação entre a menor sorção de água, solubilidade e coeficiente de difusão características do silorano (PALIN et al.

2005a), podem potencialmente melhorar a estabilidade hidrolítica das restaurações de resina composta (ILIE; HICKEL, 2006). A estrutura do siloxano, sendo mais hidrofóbica, reduz a absorção de água do meio bucal (EICK et al., 2006), melhorando suas propriedades físicas e possivelmente resultando em menor pigmentação exógena.

Entretanto, no meio bucal as resinas compostas podem ser expostas de forma intermitente ou contínua a agentes químicos como água, heptano (solução que simula o efeito de gorduras como manteiga, carnes gordas e óleos vegetais), ácido cítrico e solução de etanol (que simulam determinadas bebidas, incluindo álcool, legumes, frutas, doces e xaropes). A exposição intermitente ocorre durante o comer ou beber até que os dentes sejam limpos. Por outro lado, a exposição contínua pode ocorrer quando os agentes químicos são absorvidos por detritos aderentes (como o cálculo ou partículas de alimentos) nas margens de restaurações ou em resíduos resultantes da decomposição bacteriana (YAP et al., 2001). De acordo com YESILYURT et al. (2009), a resina à base de silorano mantém-se estável e insolúvel em simulados de fluidos biológicos quando imersa em soluções aquosas contendo enzimas, como a hidrolase ou esterase, ou ainda ácido clorídrico diluído. Supõe-se que a maior estabilidade hidrolítica desse material resulte em menor rugosidade e menor manchamento com o decorrer do tempo frente aos desafios químicos. No entanto, não se observam trabalhos na literatura que tenham comprovado esse fato. Embora os efeitos de líquidos e alimentos tenham sido amplamente investigados em resinas compostas à base de metacrilato, isso ainda não foi conduzido para as resinas compostas à base de silorano. Portanto, o objetivo desse estudo foi investigar o efeito de soluções químicas em propriedades como alteração de cor e capacidade de manchamento, sorção de água, solubilidade e rugosidade da resina à base de silorano, comparando-a a outras resinas compostas à base de metacrilatos.

## 2 REVISÃO DE LITERATURA

### 2.1 EVOLUÇÃO DAS RESINAS COMPOSTAS

As resinas compostas têm sofrido melhorias contínuas em suas propriedades, o que resultou no emprego desses materiais como principal opção restauradora não somente em dentes anteriores, mas também em dentes posteriores (American Dental Association, 2001). A origem das resinas compostas atuais se iniciou a partir dos trabalhos de Bowen (1956), inicialmente com o uso de resinas epóxicas em materiais restauradores, seguido do desenvolvimento do bisfenol A glicidil dimetacrilato (BisGMA). Desde então, as resinas compostas têm sofrido modificações em sua composição através da incorporação de outros monômeros (YESILYURT et al., 2009), como trietileno glicol dimetacrilato (TEGDMA) ou uretano dimetacrilato (UDMA), o que influencia diretamente as propriedades desses materiais. De maneira geral, as resinas à base de metacrilatos são compostas de uma matriz orgânica constituída de monômeros resinosos (cerca de 25 a 30%) e uma matriz inorgânica, composta de partículas de carga de vidro ou cerâmica (cerca de 70 a 75%) tratadas com um agente responsável pelo acoplamento entre as fases, o silano. Essas resinas compostas apresentam como denominador comum a polimerização por radicais de metacrilatos (WEINMANN et al., 2005).

O sistema de polimerização dos metacrilatos é formado em geral por dois componentes: a canforoquinona, que é um agente fotossensível e o principal fotoiniciador, e uma amina terciária, responsável pela transferência de hidrogênio. Este sistema decompõe-se imediatamente frente à exposição à luz com comprimentos de onda entre 430 e 490nm, gerando radicais livres que iniciam o processo de polimerização (WEINMANN et al., 2005). Durante a reação de polimerização ocorre a aproximação entre os monômeros constituintes do material, o que resulta em contração volumétrica e geração de estresses durante sua polimerização (WEINMANN et al., 2005). A contração de polimerização das resinas compostas à base de metacrilato é da ordem de 1,5 a 5%, o que é suficiente para

resultar no desenvolvimento de tensões internas que podem ameaçar a durabilidade das restaurações (FERRACANE, 2005). Esses fenômenos são responsáveis pelas principais limitações das resinas compostas à base de metacrilatos, pois podem trazer como conseqüências: fraturas do esmalte, comprometimento da integridade marginal, descoloração nas margens da restauração, microinfiltração, desenvolvimento de cáries secundárias, sensibilidade pós-operatória e conseqüente falha da restauração (WEINMANN et al., 2005; DUARTE et al., 2009; AL-BONI; RAJA, 2010).

Várias tentativas têm sido propostas para se reduzir a contração de polimerização, incluindo o desenvolvimento de monômeros resinosos à base de metacrilatos que resultem em menor contração volumétrica (WEINMANN et al., 2005). Entretanto, o uso de monômeros de alto peso molecular é limitado pela sua alta viscosidade, indesejável para boas características de manipulação das resinas compostas (WEINMANN et al., 2005). Dessa forma, outras propostas visam minimizar clinicamente os efeitos adversos resultantes da contração de polimerização, como por exemplo, a inserção e fotoativação do material pela técnica incremental (FERRACANE, 2005), embora essas técnicas não possam eliminar completamente os estresses gerados no material (VAN ENDE et al., 2010). Além da técnica incremental, outras alternativas incluem ainda a redução da razão de polimerização da resina composta através de diferentes métodos de fotoativação, a aplicação de camadas mais espessas de adesivo sob a restauração ou o emprego de materiais de preenchimento com baixo módulo de elasticidade, com o intuito de minimizar as tensões de contração na interface dente-restauração (AL-BONI; RAJA, 2010).

Uma alternativa proposta mais recentemente para contornar os efeitos da contração de polimerização, dentre outros problemas, foi a modificação na natureza da matriz orgânica das resinas compostas, através do desenvolvimento do silorano. O silorano foi desenvolvido com o objetivo de minimizar alguns inconvenientes constatados para as resinas à base de metacrilato: principalmente o estresse de contração gerado durante a polimerização, mas também a inibição da polimerização superficial quando em contato com radicais de oxigênio e absorção de água em contato com o ambiente bucal (WEINMANN et al., 2005; ILIE; HICKEL, 2006; DUARTE et al., 2009; AL-BONI; RAJA, 2010), inerentes às resinas à base de

metacrilato.

Durante reação de polimerização do silorano ocorre à abertura do anel oxirano, o que compensa parcialmente a sua contração de polimerização. A contração do silorano foi determinada em 0,99% em volume (WEINMANN et al., 2005; AL-BONI; RAJA, 2010). O sistema catiônico de iniciação da reação de polimerização do silorano é constituído por três componentes: a canforoquinona, um sal de iodônio e um doador de elétrons (WEINMANN et al., 2005; ILIE; HICKEL, 2006). No processo de oxi-redução, o doador de elétrons promove a decomposição do sal de iodônio em um cátion ácido, que por sua vez inicia a abertura do anel oxirano e gera um novo centro ácido. Esse processo resulta em uma reação de polimerização de início mais lento, porque é necessário mais tempo para formar cátions suficientes para iniciar a polimerização (PALIN et al., 2005a). Após a adição de um radical oxirano, o anel epóxi é aberto para formar uma cadeia ou uma rede polimérica (WEINMANN et al., 2005). A reação de polimerização catiônica é insensível ao contato com o oxigênio e o silorano apresenta maior hidrofobicidade devido à presença de radicais siloxano (WEINMANN et al., 2005; ILIE; HICKEL, 2006; AL-BONI; RAJA, 2010). Além de resultar em menor contração de polimerização, a reação catiônica também otimiza o grau de conversão do material (ILIE; HICKEL, 2006; LIEN; VANDEWALLE, 2010).

Por causa da reduzida contração de polimerização do silorano em comparação a uma resina convencional à base de metacrilato, a interface dente-restauração é exposta de forma significativa a menos estresses (PALIN et al., 2005b; FERRACANE, 2005). Isso justifica a menor deflexão das cúspides e reduzida microinfiltração resultantes da contração de polimerização para a resina composta à base de silorano, quando comparada às resinas compostas à base de metacrilato (PALIN et al., 2005b; AL-BONI; RAJA, 2010). A menor microinfiltração demonstrada pelo silorano em comparação a outras resinas compostas à base de metacrilato sugere melhor desempenho clínico e maior durabilidade da restauração (AL-BONI; RAJA, 2010). Entretanto, vale ressaltar que é importante estar atento para a configuração da cavidade, a polimerização adequada do material e a técnica de inserção incremental, que são importantes para a diminuição das tensões de contração geradas durante a polimerização de qualquer resina composta, até mesmo aquela à base de silorano (VAN ENDE et al., 2010).

Outras propriedades desejáveis têm sido também relatadas para a resina à base de silorano (WEINMANN et al., 2005), tais como a melhor profundidade de cura, menor contração de polimerização, maior resistência mecânica, bem como a dureza equivalente e temperatura de transição vítrea aceitável quando comparadas à resina convencional à base de metacrilato (ILIE; HICKEL, 2006). O material à base de silorano tem apresentado ainda maior resistência à flexão e tenacidade à fratura, mas relativamente menor resistência à compressão e dureza do que os materiais restauradores à base de metacrilato (LIEN; VANDEWALLE, 2010). Entretanto, por apresentar matriz orgânica de natureza distinta, a resina composta à base de silorano requer a utilização de um sistema adesivo específico a fim de proporcionar uma boa qualidade de ligação desse material à dentina (DUARTE et al., 2009). As boas propriedades mecânicas e a baixa contração de polimerização fazem do silorano uma alternativa restauradora promissora. No entanto, são necessárias investigações quanto a outros aspectos não menos importantes, em especial quando esses materiais estão sujeitos ao contato com substâncias químicas presentes na cavidade bucal.

## 2.2 DESAFIOS PARA AS RESINAS COMPOSTAS NA CAVIDADE BUCAL

Diversos desafios estão presentes na cavidade bucal tanto para as estruturas dentárias, como para os materiais restauradores utilizados para substituir as estruturas dentárias perdidas. Em primeiro lugar, a cavidade bucal é um ambiente úmido, o que por si só representa um grande desafio para a longevidade dos materiais restauradores. Sabe-se que as resinas compostas, apesar de sua relativa hidrofobicidade, absorvem água e solubilizam-se no ambiente bucal. A sorção de água, bem como as propriedades mecânicas e o grau de conversão do material, dependem diretamente da composição monomérica das resinas compostas (YESILYURT et al., 2009). Resinas com maior conteúdo de TEGDMA na matriz orgânica apresentam maior sorção de água, uma vez que esse monômero é mais hidrofílico do que o BisGMA e o UDMA (YESILYURT et al., 2009).

As resinas à base de metacrilato, por sua vez, apresentam maior



solubilidade e sorção de água quando comparadas à resina à base de silorano, pela menor polaridade do silorano (PALIN et al., 2005a) e sua maior hidrofobicidade devido à presença de radicais siloxano (WEINMANN et al., 2005; ILIE; HICKEL, 2006; AL-BONI; RAJA, 2010). O silorano tem demonstrado menor expansão higroscópica e maior estabilidade dimensional em solução aquosa, quando comparado a uma resina à base de metacrilato (WEI et al., 2011). Uma possível explicação para essas diferenças se dá pela forte ligação da água com as ligações de hidrogênio na molécula do polímero de metacrilato, seguida da ligação progressiva de moléculas de água com as ligações de hidrogênio intermoleculares, o que induz a plastificação e expansão do polímero de metacrilato (WEI et al., 2011). A menor sorção de água, solubilidade e coeficiente de difusão para a resina à base de silorano, quando comparada às resinas convencionais à base de metacrilato (PALIN et al., 2005a; ILIE; HICKEL, 2006; AL-BONI; RAJA, 2010), são características importantes para melhorar a estabilidade hidrolítica das restaurações de resina composta (ILIE; HICKEL, 2006).

Além da química do monômero resinoso, a sorção de água e solubilidade da resina composta são influenciadas por uma série de fatores como: o grau de polimerização do polímero, o tamanho das partículas de carga, o formato e distribuição dessas partículas e a qualidade de ligação interfacial entre as partículas de carga e a matriz resinosa (YESILYURT et al., 2009). Uma pequena expansão higroscópica seria interessante para aliviar as tensões entre as partículas de carga e a matriz resinosa, favorecendo a manutenção das ligações moleculares por períodos mais prolongados (WEINMANN et al., 2005; ILIE; HICKEL, 2006). Entretanto, deve-se considerar a influência do processo de sorção de água pelas resinas com a conseqüente retenção de corantes. Por essa razão, a sorção de água se torna preocupante a partir do momento em que possa interferir com outras propriedades, principalmente a estabilidade de cor e a resistência ao manchamento. Durante a contração polimerização, ainda, a difusão da umidade nas resinas compostas à base de metacrilato pode resultar na propagação de microtrincas na matriz resinosa, o que abre caminho para a penetração de agentes químicos e pigmentos, assim acelerando a degradação do material (YESILYURT et al., 2009).

A qualidade hidrofóbica é, portanto, interessante não somente para garantir a resistência mecânica da resina composta no ambiente bucal, mas também

para minimizar a absorção de corantes provenientes da dieta, o que conseqüentemente torna-a menos sensível ao manchamento exógeno (WEINMANN et al., 2005). Em geral, o café tem sido empregado como elemento indicador da coloração das resinas compostas, pois além de ser consumido com freqüência em nosso dia-a-dia, apresenta resultados bem definidos de manchamento frente às variáveis impostas ao material, como por exemplo: quanto maior a quantidade de matriz orgânica, maior será o manchamento superficial da resina composta provocado pelo café (CHAN et al., 1980; SOUZA et al., 2006).

Na cavidade bucal as resinas compostas podem ainda ser expostas, de forma intermitente ou contínua, aos agentes químicos encontrados na saliva, alimentos e bebidas (YAP et al., 2001; YESILYURT et al., 2009). A exposição intermitente ocorre durante o ato de comer ou beber, enquanto a exposição contínua pode acontecer quando os agentes químicos são absorvidos por detritos aderentes (como o cálculo ou partículas de alimentos) na superfície dos dentes ou restaurações (YESILYURT et al., 2009). Essas substâncias podem ser de origem exógena ou endógena (YU et al., 2009). As soluções ácidas têm efeito erosivo no esmalte e na dentina, mas possíveis efeitos nos materiais restauradores são limitados (YU et al., 2009). Dentre os materiais restauradores, as resinas compostas têm apresentado melhor resistência à erosão, sendo mais resistentes também do que o esmalte humano (YU et al., 2009).

Mesmo assim, as resinas compostas podem sofrer dissolução por solventes orgânicos (YAP et al., 2001). A água destilada é geralmente empregada em pesquisas para simular o ambiente bucal úmido fornecido pela saliva. O ácido cítrico e solução de etanol são adotados para simular determinadas bebidas, incluindo o álcool e a ação de legumes, frutas, doces e xaropes (YESILYURT et al., 2009), sendo alguns desses simuladores de líquidos salivares utilizados em estudos laboratoriais determinados de acordo com as diretrizes da Federação de Alimentos e Medicamentos dos Estados Unidos (Food and Drug Administration, 1976).

O ácido cítrico têm sido empregado para simular o efeito de frutas cítricas, sucos e bebidas carbonadas, cujo pH se assemelha ao da solução de ácido cítrico a 0,02N (YESILYURT et al., 2009). Os ácidos: acético, propiônico e láctico têm provocado um amolecimento superficial nas resinas compostas, influenciando a sua dureza, resistência à abrasão e capacidade de manchamento (SOUZA et al., 2006).

A matriz orgânica das resinas compostas pode ainda ser potencialmente danificada por soluções orgânicas de heptano e etanol, enquanto as partículas de carga podem sofrer degradação pela água e pelo ácido cítrico (YESILYURT et al., 2009). Entretanto, o efeito dessas soluções nas propriedades das resinas compostas depende do tempo de imersão, da temperatura e do pH da solução (YAP et al., 2001; HERBSTTRITH SEGUNDO et al., 2007; YESILYURT et al., 2009).

Os efeitos deletérios dos ácidos são pH-dependente, e podem produzir diferenças significativas na resistência à flexão e dureza das resinas compostas à base de metacrilatos, dependendo do meio acondicionado (YESILYURT et al., 2009). As resinas compostas que apresentam UDMA em sua composição são mais susceptíveis à dissolução por fluidos simuladores da dieta, quando comparadas às resinas à base de BisGMA (YESILYURT et al., 2009). Entretanto, a partir de todas as soluções utilizadas para simular os fluidos orais, as soluções de etanol sabidamente provocam amolecimento da matriz resinosa e, conseqüentemente, resultam no comprometimento das propriedades mecânicas das resinas compostas (BENETTI et al. 2009; DEWAELE et al., 2009). Adicionalmente, as soluções aquosas de etanol penetram na matriz de resina e promovem a liberação de monômeros não reagidos, que podem apresentar efeitos tóxicos (ILIE; HICKEL, 2006).

A resina composta à base de silorano, por outro lado, mostra-se mais resistente a alguns dos fluidos biológicos simulados. Estudos têm mostrado que o silorano é mais estável e insolúvel do que as resinas à base de metacrilato quando imerso em ácido cítrico, ácido clorídrico, heptano, ou soluções enzimáticas (WEIMANN, 2005; EICK et al., 2006; YESILYURT et al., 2009). Mesmo quando imerso em etanol, uma solução sabidamente plastificadora de resinas compostas, o silorano apresentou dureza superior do que as resinas à base de metacrilato (YESILYURT et al., 2009). As diferenças na dureza e resistência à flexão do silorano imerso em fluidos biológicos simulados, quando comparado à resina à base de metacrilato, podem resultar principalmente das discrepâncias quanto à sorção de água apresentada pelos polímeros distintos (YESILYURT et al., 2009).

Sabe-se que as características físicas e mecânicas das resinas compostas podem sofrer influência do meio de imersão, da composição do material e da qualidade de polimerização, o que pode refletir no desempenho clínico das

restaurações (HERBSTRITH SEGUNDO et al., 2007). Assim, torna-se interessante verificar a influência de diferentes soluções biológicas nas propriedades físicas como rugosidade e perda de massa das resinas compostas e, principalmente, investigar a influência que essas soluções apresentam na estabilidade de cor e capacidade de manchamento das resinas compostas à base de metacrilato ou silorano.

### 3 ARTIGO

Esta dissertação é composta de um estudo em formato de artigo intitulado: “**Effect of prolonged chemical challenges on selected properties of a silorane-based resin composite**”. Este artigo será submetido à publicação no periódico ***Journal of Esthetic and Restorative Dentistry***, cujo fator de impacto em 2010 foi 0.736. O artigo foi formulado de acordo com as orientações gerais para elaboração de manuscritos a serem considerados para publicação na revista (ANEXO 1).

## **Effect of prolonged chemical challenges on selected properties of a silorane-based resin composite**

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## Abstract

**Statement of the problem:** Although silorane has been shown to be hydrophobic and stable in simulated biological fluids, the effect of prolonged chemical challenges on this material's properties required investigation. **Purpose:** To investigate the effect of prolonged chemical challenges on color stability, staining susceptibility, water sorption, solubility, and roughness of a silorane-based resin composite when compared to methacrylate-based composites. **Materials and Methods:** Initial color, roughness and mass were registered for specimens fabricated from methacrylate or silorane-based resins. Specimens were individually stored at 37°C in 0.02N citric acid, 0.02N phosphoric acid, 75% ethanol or distilled water for 7, 14, 21, and 180 days, when new measurements were performed. A staining test was performed after the chemical challenge by immersion in coffee during 3 weeks at 37°C. Color changes were characterized using the CIEL\*a\*b\* color system. Data were submitted to two-way analysis of variance or analysis of variance for repeated measures, and Tukey's multiple comparison test. **Results:** Although significantly different, color changes on the methacrylate-based resins were not visually perceptible after immersion in water, citric acid, phosphoric acid or ethanol, but were visible for the silorane-based resin immersed in ethanol for 180 days. The methacrylate-based resins stored in ethanol were significantly more stained by coffee than the ones stored in other media. The silorane-based resin demonstrated no staining, low water sorption and solubility, but increased roughness, when compared to the methacrylate-based resins. **Conclusions:** No effect of the immersion solution was noticed on roughness or solubility of the investigated materials. Ethanol influenced color stability, water sorption and staining susceptibility differently for the methacrylate or silorane-based resins.

**Keywords:** Composite resins; Color; Discoloration; Absorption; Solubility; Roughness

### 3.1 INTRODUCTION

One of the challenges in modern Dentistry is the development of materials that can resist in the oral environment, an environment rich in pathogens, moisture and prone to chemical challenges, which may influence the properties of restorative materials.<sup>1</sup> Dental materials are constantly exposed to different solutions, oils, acids or alcohol from drinks and food<sup>2,3</sup> that may hold prolonged contact with the dental surfaces when adhered to restoration margins or the biofilm.<sup>2</sup> The effect of such substances has been investigated for established dental materials, but are limited for more recently developed restorative materials, such as the silorane.

In that scenario, it is expected that hydrophobic materials can undergo less surface changes. Amongst the current resin-based restorative materials, silorane is known to be hydrophobic due to the presence of siloxane radicals.<sup>4,5,6</sup> Consequently, silorane has demonstrated lower hygroscopic expansion and higher dimensional stability in water,<sup>7</sup> decreased water sorption, solubility and diffusion coefficient,<sup>5,6,8,9</sup> features which can potentially increase the hydrolytic stability of composite restorations.<sup>5</sup> Although a slight water uptake could beneficially alleviate the stresses between the organic matrix and the filler content and favor prolonged molecular bonds,<sup>4,5</sup> on the other hand water sorption may facilitate staining. Therefore, the hydrophobic attribute of siloxane favors color stability and maintenance of glaze<sup>10</sup> and minimizes staining<sup>11</sup> in the silorane resin.

Additionally, it has been shown that silorane is stable and insoluble in simulated biological fluids containing citric acid, hydrochloric acid, heptane, or enzymes such as hydrolase or esterase.<sup>3,4,9</sup> However, further investigation is required to explore the behavior of silorane under prolonged chemical challenges, particularly focusing on the substances that are commonly present in the oral environment. Citric acid, for instance, is present in fruits, juices and carbonated beverages<sup>3</sup> and phosphoric acid may be found in soft drinks, frequently consumed in the modern societies. It has been shown that the organic matrix in methacrylate-based resin composites may be potentially damaged by organic solutions of



ethanol<sup>2,3</sup> while the filler content may degrade in contact with water and citric acid.<sup>3</sup> The effect of these substances on the properties of resin composites depends on the length of immersion, the temperature, and the pH of the immersion solution.<sup>2,3</sup>

Although the effect of chemical solutions has been investigated for methacrylate-based resin composites, their effect on silorane-based composites after prolonged contact has not been thoroughly explored. Therefore, the purpose of this study was to investigate the effect of prolonged immersion of a silorane-based composite in citric acid, phosphoric acid, and ethanol on selected properties (color stability, staining susceptibility, roughness, water sorption, and solubility) of this material, when compared to methacrylate-based composites. As null hypothesis, it was stated that the chemical solutions would not interfere on the investigated properties.

## 3.2 MATERIALS AND METHODS

Specimens were fabricated from silorane-based and methacrylate-based resin composites (Table 1) at room temperature ( $23 \pm 1$  °C) for the different tests: color stability, staining susceptibility, roughness, water sorption, and solubility.

### 3.2.1 Color stability test

Twelve specimens (15 mm diameter, 1 mm height) were fabricated from each investigated composite (Table 1). Each specimen was light-activated from both sides during 40 seconds ( $1200\text{mW}/\text{cm}^2$ , BlueLEX GT-1200, Xianyang Holy Medical Co. Ltd., China) and the final thickness of each specimen was confirmed with a digital caliper (Mitutoyo, Aurora, Illinois, United States). Specimens were marked with a blade so the color assessments were performed always on the same side. Initial color assessments were carried out in the spectrophotometer (X-Rite Color Master, model SP60, Grand Rapids, Michigan, United States) and respective software (X-Rite Color Master, Grand Rapids, Michigan, United States), which registered  $L^*$ ,  $a^*$ , and  $b^*$  values. According to the CIE (Comission International de l'Éclairage)  $L^*a^*b^*$  method, color is analyzed tri-dimensionally in three coordinates, which represent:

- $L^*$ : lightness, determined by variations between black and white;
- $a^*$ : variations between red (positive values) and green (negative values);
- $b^*$ : variations between yellow (positive values) and blue (negative values).

Subsequently, specimens were divided in 4 subgroups ( $n = 3$ ) and were immersed in 5 mL of one of the following solutions: water (control), 0.02N citric acid, 0.02N phosphoric acid, or 75% ethanol at 37°C during 7, 14, 21 and 180 days. Solutions were not changed throughout the experiment, but periodical checks were performed to make sure that specimens were immersed during all the investigated

period. Evaporated ethanol was completed, when necessary. The chemical challenge was adapted from the methodology proposed by Yesilyurt et al.<sup>3</sup> New color assessments were registered and the initial  $L^*$ ,  $a^*$ , and  $b^*$  values were compared to  $L^*$ ,  $a^*$ , and  $b^*$  values obtained after immersion in the investigated solutions at the different time intervals. Color change ( $\Delta E$ ) was calculated according to Hunter's equation:

$$\Delta E = [(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2]^{1/2}$$

where:

$L_2^* - L_1^*$  = variations in lightness, in distinct periods;

$a_2^* - a_1^*$  = variation in  $a^*$  axis, in distinct periods;

$b_2^* - b_1^*$  = variation in  $b^*$  axis, in distinct periods.

### 3.2.2 Staining test

After 21 days of immersion in the chemical solutions, specimens were immersed in coffee in order to check the susceptibility to staining. Coffee infusion was prepared by adding 24 grams of coffee (Café do Ponto, São Paulo, Brazil) to 300 mL boiling water during 5 minutes. Coffee was filtered and cooled, prior to receiving the composite specimens. Each specimen was immersed in 5 mL coffee during 3 weeks at 37°C. Then, specimens were rinsed, dried, and new color assessments were taken in the spectrophotometer. The  $L^*$ ,  $a^*$ , and  $b^*$  values were compared to the initial color assessments, using the same equation described previously.

### 3.2.3 Roughness

Twenty-four disk-shaped specimens (6 mm diameter, 4 mm height) were fabricated from each investigated resin composite (Table 1) between polyester strips in a cylindrical mould. Specimens were light-activated (1200mW/cm<sup>2</sup>, LED Radii-cal, Southern Dental Industries, Bayswater, Victoria, Australia) from both sides during 40 seconds. Specimens were then polished using sequential disks (Super

Snap, Shofu Inc., Kyoto, Japan) mounted in a low-speed handpiece followed by felt (Super Buff, Shofu Inc.) impregnated with polishing paste (Enamelize, Cosmedent Inc., Chicago, Illinois, United States). Specimens were cleaned in ultrasound bath for 10 minutes, dried and initial roughness was conducted.

Specimens were divided in 4 groups ( $n = 6$ ) to be immersed in: water (control), 0.02N citric acid, 0.02N phosphoric acid, or 75% ethanol at 37°C. Subsequent roughness (Ra) was registered (Hommel Tester T 1000, Hommelwerke GmbH, Schwenningen, Germany) after 7, 14, 21, and 180 days after immersion in the investigated solutions. Three random readings were performed on each specimen for each evaluation period and a mean value was calculated. The device was adjusted according to the parameters: minimum tolerance 0.01 $\mu\text{m}$ ; maximum tolerance 8.00 $\mu\text{m}$ ; total length 1.5mm; sampling length 1.25mm; cut-off 0.25mm.

### **3.2.4 Water sorption and solubility**

All specimens were weighed prior to immersion in the solutions ( $m_1$ ) and were re-weighed afterwards at different time intervals: 7, 14, 21 and 180 days, in order to investigate changes in mass. The volume ( $v$ ) of all specimens was also calculated.

Subsequently, specimens were dehydrated in a desiccator containing silica until a constant mass was obtained ( $m_3$ ). Solubility of the specimens immersed within 180 days in the different solutions was calculated using the formula:  $(m_1 - m_3)/v$ . The mass registered after 180 days of immersion in the solutions ( $m_2$ ) was used to calculate water sorption with the formula:  $(m_2 - m_3)/v$ .

### **3.2.5 Statistical methods**

Data were submitted to two-way analysis of variance or analysis of variance with repeated measures, followed by Tukey's multiple comparison test. Level of significance was defined at 0.05.

### 3.3 RESULTS

#### 3.3.1 Color stability

Analysis of variance with repeated measures identified significant differences in color change for the different materials ( $p < 0.001$ ) and storage solutions ( $p < 0.001$ ), as well as an interaction effect ( $p < 0.001$ ). The silorane-based resin presented initially higher color change amongst the investigated materials up to 21 days of immersion in the different solutions, followed by Filtek Z250, Filtek Z350XT and Master Fill, which presented equivalent lower results (Graph 1). At 180 days, a slight increase in  $\Delta E$  was noticed for all methacrylate-based materials, while the silorane-based resin remained stable, except when immersed in ethanol (Graph 2). In addition, the immersion solution had a significant effect on color change after 21 days: Ethanol was responsible for higher color changes in the investigated materials. Storage in phosphoric acid or citric acid did not influence the color change of the materials, resulting in  $\Delta E$  similar to the control (water). After 180 days of immersion in the different solutions, however, color change was similar amongst all investigated materials, except for the silorane-based resin when immersed in ethanol, which presented the highest color change (Graph 2).

#### 3.3.2 Staining

Two-way analysis of variance demonstrated that all methacrylate-based resins presented significant staining after immersion in coffee ( $p < 0.001$ ): the lowest staining was noticed for Master Fill, followed by Filtek Z350XT and Filtek Z250, the latest with comparable behavior. The silorane-based resin composite did not suffer staining (Graph 3). Immersion in ethanol resulted in significantly higher staining for the methacrylate-based resins ( $p < 0.001$ ). Storage in phosphoric acid or citric acid did not influence staining and resulted in similar  $\Delta E$  values as obtained with the immersion in water (Graph 3).

### 3.3.3 Roughness

Analysis of variance with repeated measures identified significant differences amongst the investigated resin composites ( $p < 0.001$ ). The investigated methacrylate-based resins demonstrated lower surface roughness: Filtek Z350XT and Master Fill presented the lowest values, followed by Filtek Z250 with intermediate values. The silorane-based resin demonstrated the highest surface roughness amongst the investigated materials (Graph 4). No significant effect of the immersion solution ( $p = 0.569$ ) was detected on roughness of the materials for the different intervals, nor interaction effect ( $p = 0.305$ ).

### 3.3.4 Water sorption and solubility

Two-way analysis of variance identified significant differences on water sorption amongst the investigated resin composites ( $p < 0.001$ ). The silorane-based resin and two of the methacrylate-based resins (Filtek Z250 and Filtek Z350XT) demonstrated equivalent behavior and the lowest water sorption values, while Master Fill revealed the highest water sorption amongst the investigated materials (Graph 5). The type of immersion solution also had an effect on water sorption of the investigated materials ( $p < 0.001$ ). Specimens stored in citric acid or phosphoric acid presented a similar degree of water sorption when compared to specimens stored in water (control). However, specimens stored in ethanol resulted in significantly higher water sorption (Graph 6).

Significant differences on solubility were registered for the investigated materials ( $p < 0.001$ ). The silorane-based resin and Filtek Z250 demonstrated significantly lower solubility, followed by the other methacrylate-based resins, Filtek Z350XT and Master Fill (Graph 7). No effect of the storage solutions ( $p = 0.766$ ) was noticed on the solubility of the materials.

### 3.4 DISCUSSION

The CIE L\*a\*b\* method is interesting to investigate chromaticity because it detects small differences in color change.<sup>11</sup> The first set of color measurements in this study was carried out to check possible intrinsic changes in the material, and how the chemical challenges could influence these changes. Although the silorane-based resin resulted in initially higher color changes during the first 21 days, these changes in  $\Delta E$  were smaller than 3.3, which mean that they were not visually perceptible and considered acceptable.<sup>12</sup> It is speculated that the initial color changes observed in the silorane-based resin were a result of intrinsic modification in the chemical structure of the material. In any case, all investigated materials revealed  $\Delta E$  smaller than 3.3 up to 21 days of immersion in the solutions. These results are in accordance to those found by Furuse et al.,<sup>10</sup> who also observed  $\Delta E$  smaller than 3.3 for the silorane-based resin after 192 hours of accelerated aging, but are much inferior to the results from Pires-de-Souza et al.,<sup>13</sup> who identified significantly higher color changes ( $\Delta E=18.6$ ) for this material when accelerated aging was performed for a longer period (in total 384 hours). It is valid to point out, though, that both of the previously mentioned articles applied photo-aging to induce color changes, differently than the chemical challenges conducted in this study, but nevertheless used different methodologies.

The methacrylate-based resins were initially more stable (Graph 1), but also presented an increase in  $\Delta E$  after prolonged contact with the solutions at 180 days (Graph 2). This can be explained by the fact that the organic matrix of methacrylate-based resin composites can be affected by the different solutions, which reflects also on their mechanical properties.<sup>3</sup> An interesting fact was noticed for the color evaluation at 180 days. The silorane-based resin was at that moment significantly more sensitive to the prolonged immersion in ethanol and presented  $\Delta E$  or approximately 4, i.e. visually perceptible differences (Graph 2). A possible explanation is that ethanol may induce changes in the organic matrix of the silorane. Ethanol has been shown to result in elution of monomers and initiator from the silorane-based resin, whereas no elution from this material was observed in water.<sup>14</sup> The possible effect of ethanol on the organic matrix of silorane-based resins has been noticed as well on mechanical tests conducted previously by Yesilyurt et al.,<sup>3</sup>

although these authors reported a smaller effect of ethanol on silorane when compared to the effect of ethanol on methacrylate-based resins. The same authors<sup>3</sup> observed that simulated biological fluids did not affect the mechanical properties of silorane; likewise, color stability in this study was not influenced by immersion in citric or phosphoric acid (Graph 2).

The second set of color measurements was conducted in this study in order to investigate the susceptibility to staining. The silorane-based resin did not undergo any staining when in contact with coffee (Graph 3). Coffee has been considered one of the preferential pigments for staining tests, because it produces significantly more staining than other beverages or liquids.<sup>15,16</sup> It is suggested that the pigments present in the coffee may be absorbed by the material, thus producing color changes. Therefore, color changes due to extrinsic factors may be directly related to the organic matrix specifically present in the particular resin composite, and staining could thus be reduced in more hydrophobic materials. Similar  $\Delta E$  values for the silorane-based resin to those found in this study were registered by Güler et al.,<sup>11</sup> who additionally detected minimal staining for methacrylate-based composites. In the present study, significantly higher staining was observed for the methacrylate-based resins, which may be due to the period of immersion in coffee during the staining test: in the study of Güler et al.<sup>11</sup> it was limited to 24 hours while in this study it was 3 weeks. Ertas et al.,<sup>16</sup> on the other hand, found  $\Delta E$  values higher than 3.3, i.e. visual differences, for methacrylate-based resins already after 24 hours of immersion in coffee, which suggest that the concentration of the beverage may also play an important role in the staining susceptibility of materials.

The reduced staining for the silorane-based resin registered in this study can be justified by the chemical<sup>9</sup> and dimensional stability in aqueous environments,<sup>7</sup> lower water sorption, solubility, and diffusion coefficient<sup>5,6,8</sup> of silorane when compared to methacrylate-based resins. In this study, low water sorption and solubility was also observed for the silorane-based resin composite, which corroborates with the results from previous authors.<sup>5,6,8</sup> The lower water sorption and diffusion coefficient of the silorane-based resin has been attributed primarily to its organic matrix, but may be possibly explained as well by the good synergism between the organic matrix and the filler content, which can improve hydrophobicity.<sup>8</sup>

A possible explanation for the differences between silorane and methacrylate-based resins is the strong bond between water and the hydrogen



present in the methacrylate polymers, followed by the progressive linking of water with the intermolecular hydrogen bonds, which induces plasticization and expansion of the methacrylate polymer.<sup>7</sup> It has also been suggested previously that the methacrylate-based resins may present microcracks as a result of their polymerization contraction and diffusion of fluids inside the organic matrix, which can be responsible for more rapid changes within the material.<sup>3</sup> Within the methacrylate-based resins, the ones containing TEGDMA have been reported to higher staining, possibly due to the higher hydrophilicity of this monomer.<sup>16</sup> Other factors may also influence the physical properties of methacrylate-based composites, such as the composition of the filler content, the nature of the organic network, and the physics of polymerization.<sup>17</sup> In any case, all investigated materials complied with the minimum requisites for water sorption and solubility specified by ISO 4049,<sup>18</sup> regardless of the prolonged immersion in the different solutions conducted in this study (Graphs 5, 6, 7), although in this study the tests were conducted differently than the recommendation from the standard.

In addition, more increased staining was noticed in the present study when the materials were in contact with ethanol (Graph 3). Ethanol is frequently used to investigate elution of substances from resin composites<sup>14</sup> and has been shown to affect the mechanical properties of resin composites.<sup>2,3</sup> As an organic solvent, ethanol has the potential to penetrate and affect the polymeric structure.<sup>2,3</sup> The effect of ethanol is expected to vary according to the composition of the organic matrix on the resin composite, and is usually more significant than the prolonged contact with water.<sup>3</sup> This justifies why ethanol was included in this investigation, and the effects of ethanol with the materials in the present study were particularly interesting: It induced higher color changes for the silorane after immersion for 180 days, and produced higher staining for the methacrylate-based resins after immersion in coffee. However, the influence of ethanol on the silorane-based resin composite depends on the investigated property, and can be less<sup>3</sup> or more evident<sup>14</sup> when compared to the methacrylate-based resins. Nonetheless, the results from this study suggest that the frequent contact with ethanol may be prejudicial to the longevity of silorane restorations, which should be further investigated.

Since ethanol affected some of the properties investigated in this study, the null hypothesis was partially rejected. Amongst the limitations from this study, it should be considered that the specimens were fabricated under ideal

conditions and received a homogeneous finishing and polishing, which is not always possible in the clinical practice due to the concavities or convexities in the restoration profile. It has been shown that the particle size, surface roughness and the quality of polishing will also influence the degree of staining of a material,<sup>11,19</sup> although these factors do not stand alone. In this study, the silorane-based resin was significantly rougher while Filtek Z350XT was significantly smoother, which was somehow expected due to the type and size of the filler particles. The silorane-based resin holds quartz in the micrometer scale, while Filtek Z350XT contains silica and zirconia in the nanometer scale. Regardless of being the roughest material, the silorane-based resin was the least affected by staining, which supports the idea that the organic matrix plays an important role on the staining susceptibility of the material. It is also important to emphasize that the specimens were polished prior to the roughness test using sequential paper discs, but not prior to the staining test, in which the organic matrix of the material had the possibility to flow to the surface in contact with the polyester strip.

It is also valid to highlight the difficulty on estimating how much the chemical challenges are equivalent to the clinical expectation. Some authors calculate an estimate of how different tests correspond to the clinical reality, but that is always based on an average and may of course vary according to individual habits. If we consider that a person comes in contact with one of the investigated substances 2-3 times a day during a few minutes, than 180 days in continuous contact with the solutions would correspond to several years of consumption of the most varied beverages, which might contain one of the investigated ingredients.

Despite the good properties observed for the silorane in the present study, some problems were also identified. The silorane material was not as stable as the methacrylate-based resins. Throughout the experiment, a shorter shelf-life was identified for the silorane, despite the expiry date. Another curious fact was the presence of visual undulations on the surface of silorane specimens of 15mm diameter, which might render future investigations on this material.

### **3.5 CONCLUSIONS**

Citric acid and phosphoric acid did not have a significant effect on the investigated properties. However, prolonged contact with ethanol was responsible for color changes in the silorane-based resin. Ethanol was also responsible for significantly higher water sorption and staining of the methacrylate-based resins. No effect of the investigated solutions was observed on roughness or solubility of both silorane-based and methacrylate-based resin composites. Considering the limitations from this study, it was concluded that the contact with ethanol influenced color stability, water sorption and staining susceptibility differently for the methacrylate or silorane-based resins.

#### **Clinical Significance**

The knowledge of how acids and solvents interact with the properties of restorative materials is important for the selection of a material with the desired characteristics for each specific situation. An estimate of the overall performance in the oral cavity gives support for care and improvement of the longevity of restorations.

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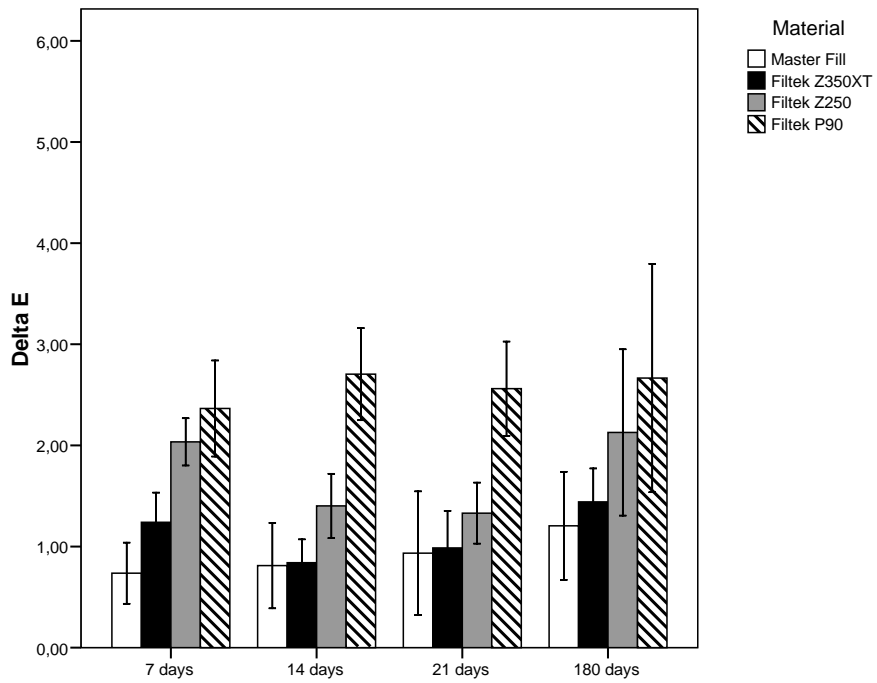
### 3.7 TABLE

Table 1 – Details from the resin composites investigated in this study.

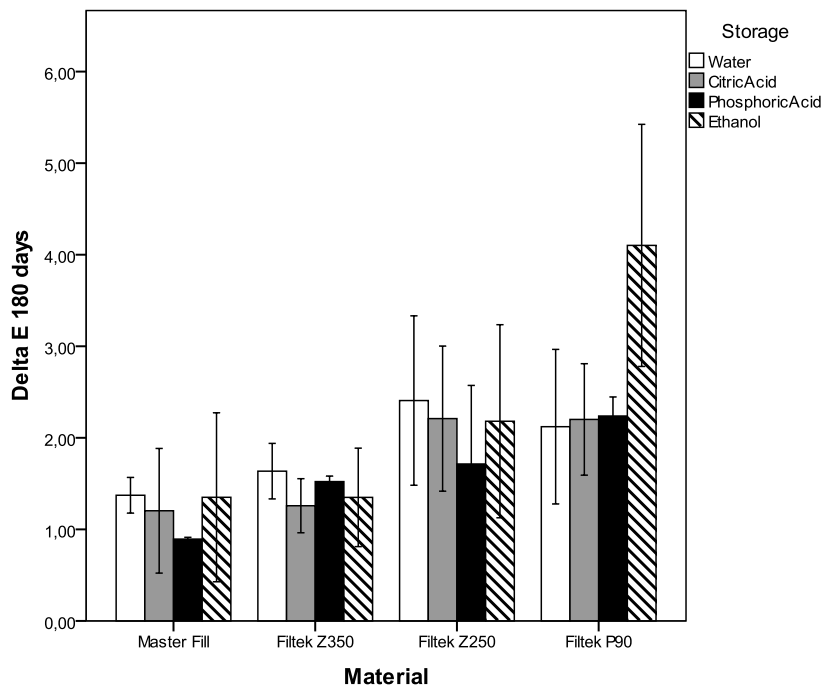
<b>Resin composite</b>	<b>Classification</b>	<b>Composition</b>
Filtek P90 3M ESPE St. Louis, United States Lot# N130928	Silorane-based microhybrid composite	Silorane resin Quartz Yttrium fluoride Pigments Initiating system
Filtek Z250 3M ESPE St. Louis, United States Lot# N164102BR	Methacrylate-based microhybrid composite	Bisphenol A polyethylene glycol diether dimethacrylate (Bis-EMA) Urethane dimethacrylate (UDMA) Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) Triethylene glycol dimethacrylate (TEGDMA) Silica Zirconia Pigments Initiating system
Master Fill Biodinâmica Ibiporã, Brazil Lot#667-10	Methacrylate-based microhybrid composite	Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) Urethane dimethacrylate (UDMA) Silica Barium borosilicate glass Pigments Initiating system
Filtek Z350XT 3M ESPE St. Louis, United States Lot# N122413	Methacrylate-based nanohybrid composite	Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) Bisphenol A polyethylene glycol diether dimethacrylate (Bis-EMA) Urethane dimethacrylate (UDMA) Triethylene glycol dimethacrylate (TEGDMA) Silica Zirconia Pigments Initiating system

### 3.8 ILLUSTRATIONS

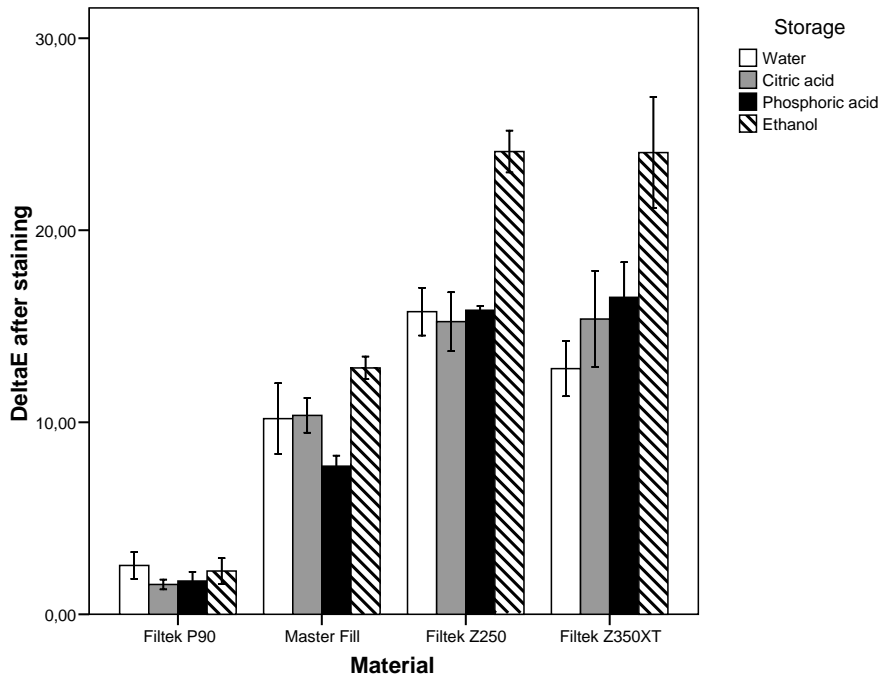
Graph 1



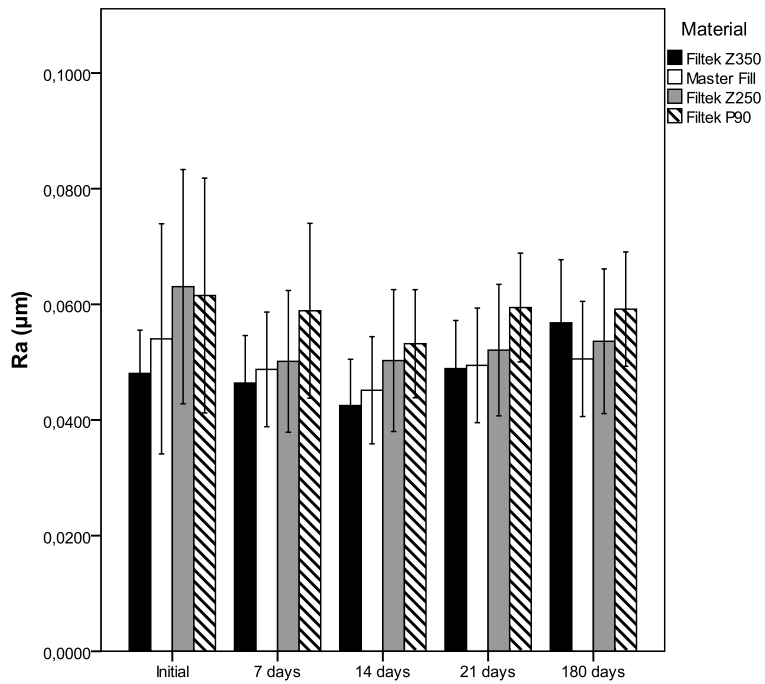
Graph 2



Graph 3

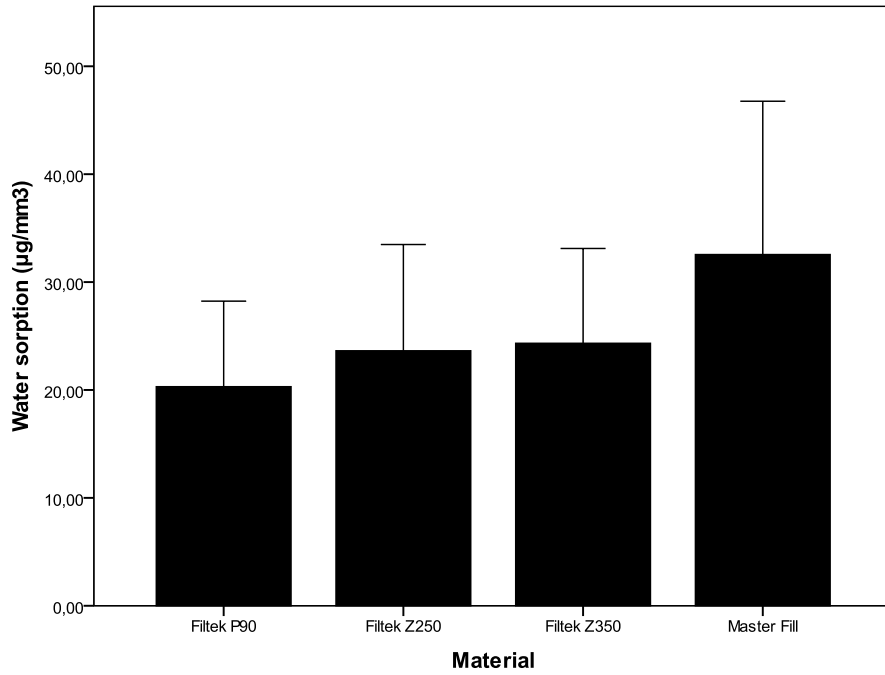


Graph 4

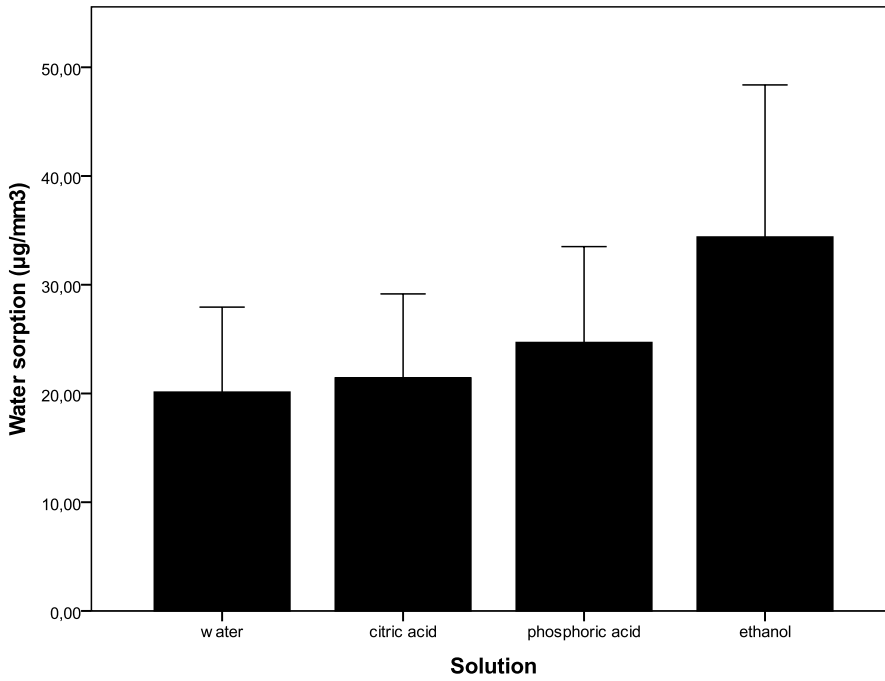




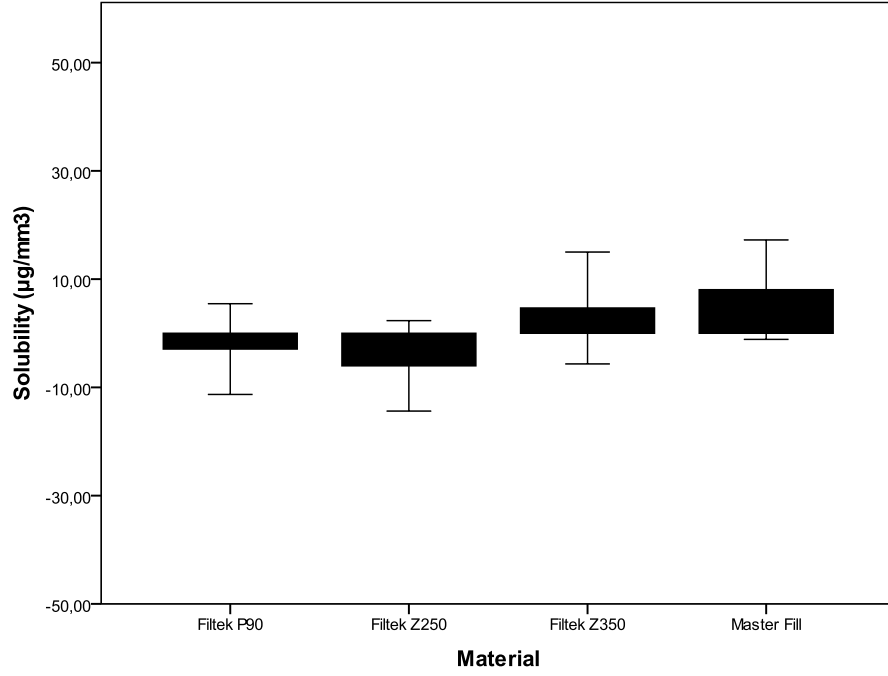
Graph 5



Graph 6



Graph 7



### 3.9 LEGENDS

Graph 1 – Color change ( $\Delta E$ ) and standard deviation for each of the investigated materials in function of time.

Graph 2 – Color change ( $\Delta E$ ) and standard deviation of the investigated materials after 180 days, in function of the different immersion solutions.

Graph 3 – Color change ( $\Delta E$ ) and standard deviation of the investigated materials after the staining test in coffee, in function of the immersion solution.

Graph 4 – Roughness ( $R_a$ ) and standard deviation of the investigated materials in function of time of immersion.

Graph 5 – Water sorption ( $\mu\text{g}/\text{mm}^3$ ) and standard deviation of the investigated resin composites after 180 days of immersion in the solutions.

Graph 6 – Water sorption ( $\mu\text{g}/\text{mm}^3$ ) and standard deviation of the materials after 180 days, in function of the immersion solution.

Graph 7 – Solubility ( $\mu\text{g}/\text{mm}^3$ ) and standard deviation of the investigated materials after 180 days of immersion in the solutions.

### **3.10 DISCLOSURE AND ACKNOWLEDGMENTS**

This manuscript results from the dissertation of Vivian Cristiane Bueno Ribeiro de Jesus, University North of Paraná, Londrina, PR, Brazil. University North of Paraná and private funds supported the study. Authors wish to acknowledge 3M ESPE and Biodinâmica, Brazil, for the donation of the restorative materials used in this research. Special acknowledgements are expressed to Biodinâmica for the use of the spectrophotometer, in particular to Míria Cristina Bernini and Jonatas Renan G. Montanucci for help and technical support. The authors do not have any financial interest in the companies whose materials are included in this article.

## 4 CONCLUSÕES

O conhecimento sobre a interação de substâncias ácidas e solventes com os materiais restauradores oferece base para estimar o seu comportamento geral na cavidade bucal, além de orientar quais cuidados são importantes para favorecer a longevidade das restaurações. De acordo com os achados desse estudo, pode-se concluir que o ácido fosfórico e o ácido cítrico não afetaram as propriedades dos materiais investigados. O contato prolongado com etanol, entretanto, produziu alterações de cor na resina à base de silorano. Mesmo em curto prazo, o etanol provocou maior sorção de água e manchamento nas resinas à base de metacrilatos. Nenhum efeito significativo dos desafios químicos foi constatado com relação à rugosidade e à solubilidade dos materiais estudados. A partir das limitações desse estudo, concluiu-se que o contato com o etanol afetou a estabilidade de cor, a sorção de água e o grau de manchamento de modo distinto para as resinas à base de silorano ou metacrilatos.

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## ANEXO 1

### NORMAS PARA ELABORAÇÃO DE MANUSCRITOS

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The research report (1) states the problem and the objective of the research; (2) describes clearly the materials and methods so they can be duplicated and their validity judged; (3) reports the results accurately and concisely; (4) provides discussion of the findings; (5) offers the conclusions that may be drawn from the research; and (6) provides, under a separate heading, a statement of the research's

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In a few sentences, please indicate the clinical importance and implications of the research or clinical technique discussed, and if applicable, its relevance to esthetic dentistry.

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Donnelly PV, Miller C, Ciardullo T, et al. Occlusion and its role in esthetics. *J Esthet Dent* 1996;8:111-8.

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Hickey JC, Zarb GA. *Boucher's prosthodontic treatment for edentulous patients*. 9th ed. St. Louis (MO): CV Mosby; 1985.

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