UNESP - Universidade Estadual Paulista

"Júlio de Mesquita Filho"

Faculdade de Odontologia de Araraquara

FERNANDA FERREIRA JASSÉ

RESISTÊNCIA À FRATURA, GRAU DE CONVERSÃO, ADAPTAÇÃO MARGINAL E CONTRAÇÃO DE POLIMERIZAÇÃO DE UMA RESINA COMPOSTA "BULK-FILL"

Araraquara

2014

UNESP - Universidade Estadual Paulista

"Júlio de Mesquita Filho"

Faculdade de Odontologia de Araraquara

FERNANDA FERREIRA JASSÉ

RESISTÊNCIA À FRATURA, GRAU DE CONVERSÃO, ADAPTAÇÃO MARGINAL E CONTRAÇÃO DE POLIMERIZAÇÃO DE UMA RESINA COMPOSTA "BULK-FILL"

Tese apresentada ao programa de Pós-Graduação em Ciências Odontológicas, Área de concentração Dentística Restauradora, da Faculdade de Odontologia de Araraquara, da Universidade Estadual Paulista para obtenção do título de Doutor em Ciências Odontológicas.

Orientador: Prof. Dr. Edson Alves de Campos

Araraquara

2014

Jassé, Fernanda Ferreira

Resistência à fratura, grau de conversão, adaptação marginal e contração de polimerização de uma resina composta bulk-fill / Fernanda Ferreira Jassé.-- Araraquara: [s.n.], 2014.

124 f. ; 30 cm.

Tese (Doutorado) – Universidade Estadual Paulista, Faculdade de Odontologia

Orientador: Prof. Dr. Edson Alves de Campos

1. Força compressiva 2. Adaptação marginal dentária 3. Resinas compostas. 4. Preparo da cavidade dentária 5. Espectroscopia infravermelho transformada de Fourier. 6. Polimerização I. Título

Ficha catalográfica elaborada pela Bibliotecária Disleide Silvia Valério Gounella, CRB-8/5629

Diretoria Técnica de Biblioteca e Documentação da Faculdade de Odontologia de Araraquara / UNESP

UNIVERSIDADE ESTADUAL PAULISTA - UNESP
FACULDADE DE ODONTOLOGIA DE ARARAQUARA

FERNANDA FERREIRA JASSÉ

RESISTÊNCIA À FRATURA, GRAU DE CONVERSÃO, ADAPTAÇÃO MARGINAL E CONTRAÇÃO DE POLIMERIZAÇÃO DE UMA RESINA COMPOSTA “BULK-FILL”

COMISSÃO JULGADORA

TESE PARA OBTENÇÃO DO GRAU DE DOUTOR

Presidente e orientador: Prof. Dr. Edson Alves De Campos

2º Examinador: Prof. Dr. Marcelo Ferrarezi De Andrade

3º Examinador: Prof. Dr. Milton Carlos Kuga

4º Examinador: Prof. Dr. Rafael Leonardo Xediek Consani

5º Examinador: Profa. Dra. Andréia Bolzan De Paula

Araraquara, 23 de julho de 2014.

Dados Curriculares

Fernanda Ferreira Jassé

NASCIMENTO	18 de março de 1984 – Belém/PA
FILIAÇÃO	Roberto da Rocha Jassé Cleva Fernanda Ferreira Jassé
2002/2007	Graduação em Odontologia Universidade Federal do Pará – Belém/PA
2007/2009	Especialização em Prótese Dentária Faculdade de Odontologia de Araraquara Universidade Estadual Paulista "Júlio de Mesquita Filho" – UNESP
2008/2008	Aperfeiçoamento em Estética e Cosmética Associação Paulista de Cirurgiões-Dentistas – Araraquara/SP
2008/2010	Mestrado em Ciências Odontológicas, concentração em Dentística Restauradora Faculdade de Odontologia de Araraquara Universidade Estadual Paulista "Júlio de Mesquita Filho" – UNESP
2010/2011	Aperfeiçoamento em Prótese sobre Implante Associação Paulista de Cirurgiões-Dentistas – Araraquara/SP
2011/2011	Aperfeiçoamento em Implantodontia Centro de Atendimento Odontológico – Araraquara/SP

2010/2014	Doutorado em Ciências Odontológicas, concentração em Dentística Restauradora Faculdade de Odontologia de Araraquara Universidade Estadual Paulista “Júlio de Mesquita Filho” – UNESP

Dedicatória

A **Deus**, *pelo dom da vida, pela família maravilhosa e por estar presente diariamente em meus caminhos.*

À **Nossa Senhora de Nazaré**, *com especial devoção e gratidão.*

Aos meus pais, **Roberto e Cleva**, *por todo amor, cuidado, amizade, carinho e dedicação. Vos dedico este trabalho porque sem vocês ele não seria possível. Obrigada por cada palavra de apoio e auxílio nos momentos de decisão. Serei eternamente grata! Amo muito vocês!*

Ao meu esposo, **Marcelo**, *por ser meu companheiro para todas as horas, por todo apoio e sabedoria . Obrigada pelo seu amor cuidadoso, pela sua*

compreensão e toda serenidade que me transmite. Amo você!

Ao meu irmão **Ricardo**, *pelo amor e amizade e por nos ter dado um presente tão especial que é a Maria Clara, alegria de nossa família.*

À minha querida cunhada **Márcia**, *fiel escudeira. Obrigada por toda ajuda e cuidado que dedica ao meu consultório e aos meus pacientes.*

Às minhas amadas avós **Carmem e Terezinha**, *pela eterna torcida.*

Enfim, a todos que torcem por mim!

Dedico este trabalho.

Agradecimentos Especiais

Ao querido **Prof. Dr. Edson Alves de Campos**, que me aceitou como orientada, apoiou e acreditou em meu trabalho. Obrigada pelas portas que me abriu na Universidade de Genebra, pelos valiosos ensinamentos de pesquisa e didática e também pela sua amizade. A orientação termina por aqui, porém sei que posso contar sempre com sua colaboração. Meu eterno respeito e admiração.

Ao querido **Prof. Dr. Osmir Batista de Oliveira Júnior**, grande mestre, que me fez acreditar que eu poderia ser realmente professora, mesmo diante das adversidades. Obrigada pelos ensinamentos e pela torcida.

Ao querido **Prof. Dr. Marcelo Ferrarezi de Andrade**, pelo acolhimento, confiança, ensinamentos e oportunidades. Obrigada por tudo.

Ao querido **Prof. Dr. José Roberto Cury Saad**, pela amizade, ensinamentos e incentivo durante o mestrado. Meus sinceros agradecimentos.

À querida **Profª Drª Alessandra Nara de Souza Rastelli**, pela amizade e disponibilidade nesses anos de convivência. Sua contribuição foi inestimável. Muito obrigada.

Cada um de vocês teve um papel especial em minha formação.

Obrigada por tudo.

Aos Professores **Stefano Ardu**, **Dorien Lefever** e **Ivo Krejci** pelo acolhimento na Universidade de Genebra e pelo período de aprendizado intenso que me proporcionaram.

Às amigas **Carolina Santezi** e **Aliny Luizzi**, que foram verdadeiras irmãs durante esses anos de doutorado.

Aos grandes amigos que fiz na Dentística: **Andiara Ribeiro, Juliana Boaventura, Janaína Bortolatto, Mateus Tonetto, Taís Martinez, Marília Regalado, Mayra Piccioni, Fabiana Takatsui, Carlos Borges e Rafael Calixto**. E aos demais colegas que tive o prazer de conhecer nesta caminhada: **Fernando Florez, Matheus Bandéca, Esther Saad, Victor Clavijo, William Kabbach, Fábio Alvarenga, Renato Queiroz, Hélida Barud, Marília Caldonazzo, Aline Gonçalves, Cristina Presoto, Sabrina Cioffi, Juliana Saraiva e Fabrício Amaral.**

Às alunas de graduação e iniciação científica, **Natália Pollo** e **Tamiris Neves** pela colaboração nas pesquisas.

À **Hélida Barud, Hernane Barud, Alexandre Gatti, Filipe Abi Rached** e **Marie-Claude Reymond**, por toda ajuda e contribuição técnica para este trabalho.

À querida **Silvia Pupin**, que foi como uma mãe para mim nesses cinco anos de pós-graduação. E aos demais amigos da Prótese. Obrigada por tudo.

À **Creuza**, por todo carinho e amizade. E aos demais funcionários da Dentística: **Cidoca, Marinho, Wanderley** e **Priscila**. Todos vocês foram muito importantes no dia-a-dia do curso.

À **Mara**, que foi sempre incansável diante das dúvidas, problemas e solicitações. Bem como aos demais funcionários da seção de pós-graduação: **Alexandre** e **Sérgio**. Obrigada pela paciência.

Aos familiares e amigos de Belém, que apesar da distância, sempre me receberam com carinho quando eu retornava para casa. A amizade de vocês fez com que esse período de renúncias e sacrifício em busca de meu sonho se tornasse mais fácil.

Obrigada por fazerem parte dessa história.

Agradecimentos

À Faculdade de Odontologia de Araraquara, da Universidade Estadual Paulista, representada pela Senhora Diretora **Profª. Drª. Andreia Affonso Barretto Montandon** e a vice-diretora **Profª. Drª. Elaine Sgavioli Massucato**, onde me foi dada a oportunidade de crescer nos âmbitos científico e profissional.

Ao programa de Pós-Graduação em Ciências Odontológicas, da Faculdade de Odontologia de Araraquara, representado pela **Profª. Drª. Josimeri Hebling** e pelo **Prof. Dr. Osmir Batista de Oliveira Júnior**, pela inestimável contribuição para minha formação acadêmica.

Aos professores do programa de Pós-Graduação do Departamento de Dentística Restauradora, **Osmir Batista de Oliveira Júnior, Marcelo Ferrarezi de Andrade, José Roberto Cury Saad, Edson Alves de Campos, Sizenando de Toledo Porto Neto, Alessandra Nara de Souza Rastelli e Andrea Abi Rached Dantas**, pelos conhecimentos e experiência compartilhados.

Aos funcionários da biblioteca **Adriano, Maria Inês, Silvia, Eliane, Ceres, Cristina, Marley, Maria Aparecida e Odete**, pela receptividade e orientação.

À Escola de Medicina Dentária, da Universidade de Genebra, Suíça, na pessoa do **Prof. Ivo Krejci**, pela disponibilização da estrutura laboratorial e apoio técnico-científico para realização de parte desta pesquisa.

Ao Instituto de Química da UNESP de Araraquara, pelo suporte laboratorial. Em especial ao **Prof. Dr. Hernane Barud**.

Ao Departamento de Materiais Dentários e Prótese da Faculdade de Odontologia da UNESP de São José dos Campos, na pessoa do **Prof. Dr. Alexandre Luiz Souto Borges,** pela disponibilização da estrutura física e técnica para realização de um dos experimentos deste trabalho.

À empresa Dentsply DeTrey, representada pelo **Dr. Walter R. Dias**, pelo fornecimento dos materiais utilizados na pesquisa.

À Coordenação de Aperfeiçoamento de Pessoal de Nível Superior **(CAPES) e PROAP** pelo auxílio financeiro concedido durante todo o curso.

A todos aqueles que de alguma forma colaboraram para a realização deste trabalho.

O meu muito obrigada!

“Se a aparência e a essência das coisas coincidissem, a ciência seria desnecessária.”

Karl Marx

Jassé FF. Resistência à fratura, grau de conversão, adaptação marginal e contração de polimerização de uma resina composta “bulk-fill”. [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2014.

Resumo

O objetivo neste trabalho foi avaliar diferentes propriedades de uma resina composta “bulk-fill” fluida de baixo tensão de contração, por meio de três estudos: 1) avaliação da resistência à fratura de molares com cavidades classe II MOD restaurados com a resina “bulk-fill”; 2) avaliação do grau de conversão monomérica (GC%) por meio de espectroscopia FTIR (Espectroscopia Infravermelho Transformada de Fourier); e 3) avaliação da adaptação marginal cervical em esmalte e dentina, e da contração de polimerização. Para o estudo de resistência à fratura, 40 molares foram divididos em cinco grupos: 1- dentes hígidos (controle positivo); 2- dentes com cavidades classe II MOD sem restauração (controle negativo); 3- dentes com cavidades classe II MOD restaurados apenas com a resina “bulk-fill”; 4- dentes com cavidades classe II MOD restaurados com a resina “bulk-fill” recoberta por resina composta convencional nano-híbrida; e 5- dentes com cavidades classe II MOD restaurados apenas com a resina convencional nano-híbrida. Todos os espécimes foram submetidos ao teste de resistência à fratura por meio de carga compressiva axial, com o emprego de esfera metálica medindo 8 mm de diâmetro, em máquina de ensaio universal EMIC DL-2000, equipada com célula de carga de 10 kN, acionada a uma velocidade de 5 mm/min até que ocorresse a fratura do conjunto. Os dados de resistência à fratura foram submetidos à Análise de Variância (ANOVA) e teste de Tukey ($\alpha = 0,05$). Para análise do grau de conversão foram confeccionados espécimes cilíndricos (4mm x 4mm) da resina, divididos nas

profundidades de polimerização de 1mm, 2mm, 3mm e 4mm. Os espécimes foram foto-ativados pelos tempos de 20 e 40 segundos, e foram então triturados, prensados com KBr e analisados em espectrofotômetro FTIR. Os dados relativos ao GC% foram analisados estatisticamente por meio da ANOVA dois fatores. Para o terceiro estudo, no qual foi avaliada a adaptação marginal cervical e a contração volumétrica de polimerização, primeiramente 24 molares foram distribuídos em três grupos (A, B e C) de modo que receberam os mesmos tratamentos supracitados para os grupos 3, 4 e 5 do estudo de resistência à fratura, respectivamente. A adaptação marginal antes e após a ciclagem termomecânica foi avaliada por meio da técnica da réplica e de análise quantitativa (% de margens contínuas) em microscopia eletrônica de varredura (MEV). Já para a análise da contração volumétrica de polimerização, uma matriz hemi-esférica foi preenchida pelas resinas testadas e então posicionadas no aparelho AccuVol™ para que fossem obtidos os percentuais de contração volumétrica calculados após a foto-ativação. Os dados obtidos para adaptação marginal cervical foram analisados estatisticamente pela ANOVA dois fatores e teste de Sidak ao nível de significância de 5%. Já os dados de contração de polimerização foram submetidos à ANOVA e teste de Tukey ($\alpha = 0,05$). Os resultados obtidos no primeiro estudo mostraram que o grupo 3 apresentou os maiores valores de resistência à fratura (2243,1 ± 473,7N), diferindo estatisticamente dos grupos 2, 4 e 5 ($p < 0,05$). Quanto ao GC%, a resina “bulk-fill” não apresentou diferença significante ($p > 0,05$) entre as profundidades de irradiação de 1 a 4mm, enquanto que para a resina nano-híbrida houve diferença estatística significante das variáveis tempo de irradiação e profundidade, bem como da interação entre elas. No terceiro estudo, notou-se que tanto antes quanto após a ciclagem termo-mecânica os percentuais de margens contínuas na dentina cervical foram maiores ($p < 0,05$)

nos grupos A e B quando comparados com o grupo C. Enquanto que para o esmalte cervical o grupo B apresentou valores maiores aos obtidos no grupo C e o grupo A apresentou resultados intermediários entre B e C. Quanto à contração de polimerização, a resina “bulk-fill” exibiu maiores percentuais de contração em relação à resina convencional ($p<0,05$). Assim, com base nos resultados pode-se concluir que a resina testada foi capaz de devolver a resistência à fratura do dente de maneira similar ao observado para os dentes hígidos. A profundidade de polimerização em incrementos de até 4 mm de espessura, conforme anunciado pelo fabricante, pôde ser confirmada. A resina “bulk-fill” mostrou-se superior à convencional quanto à adaptação marginal cervical, entretanto apresentou maior contração de polimerização.

Palavras-chave: Força Compressiva, Adaptação Marginal Dentária, Resinas Compostas, Preparo da Cavidade Dentária, Espectroscopia Infravermelho Transformada de Fourier, Polimerização.

Jassé FF. Fracture strength, degree of conversion, marginal adaptation and polymerization shrinkage of a "bulk-fill" resin composite. [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2014.

Abstract

The aim of this study was to evaluate different properties of a low-shrinkage stress "bulk-fill" flow composite resin, through three studies: 1) evaluation of the fracture strength of molars with Class II MOD cavities restored with the "bulk-fill" resin; 2) evaluation of degree of conversion (DC%) by means of FTIR (Fourier Transform Infrared) spectroscopy; and 3) evaluation of cervical marginal adaptation in enamel and dentin, and polymerization shrinkage. For the study of fracture strength, 40 molars were divided into five groups: 1- intact teeth (positive control); 2- teeth with Class II MOD cavities without restoration (negative control); 3- teeth with Class II MOD cavities restored with only "bulk-fill" resin; 4- teeth with Class II MOD cavities restored with "bulk-fill" resin covered with conventional nano-hybrid resin; and 5- teeth with Class II MOD cavities restored with only the conventional nano-hybrid resin. All specimens were subjected to fracture strength test by means of axial compressive load, using a metal sphere measuring 8 mm in diameter, in an universal testing machine EMIC DL-2000 equipped with 10 kN load cell, which was triggered at a speed of 5 mm/min until the fracture of the specimen. Data of fracture resistance were subjected to Analysis of Variance (ANOVA) and Tukey's test ($\alpha = 0.05$). To analyze DC% cylindrical specimens (4mm x 4mm) of the resin were made and divided into the depths of polymerization of 1mm, 2mm, 3mm and 4mm. The specimens were photo-activated by the times of 20 and 40 seconds, and then they were crushed, pressed with KBr (potassium bromide) and analyzed by FTIR

spectrophotometer. Data for DC% were statistically analyzed by two-way ANOVA. For the third study, which evaluated the cervical marginal adaptation and polymerization shrinkage, at first, 24 molars were divided into three groups (A, B and C) so that they received the same treatment mentioned above for groups 3, 4 and 5 of the fracture resistance study, respectively. Cervical marginal adaptation was evaluated before and after thermomechanical loading by means of the replica technique and quantitative analysis (% of continuous margins) by scanning electron microscopy (SEM). For analysis of volumetric polymerization shrinkage, a semi-spherical mold was filled by the tested composites and then placed in the AccuVol™ device in order to obtain the percentages of polimerization shrinkage that was calculated after photo-activation. The data obtained for cervical marginal adaptation were statistically analyzed by Two-Way ANOVA and Sidak's test at a significance level of 5%, while data of polymerization shrinkage were submitted to ANOVA and Tukey's test ($\alpha = 0.05$). The results of the first study showed that group 3 presented the highest values of fracture strength (2243.1 ± 473.7 N), differing from groups 2, 4 and 5 ($p<0.05$). For DC%, the "bulk-fill" resin did not show significant difference ($p> 0.05$) between depths of irradiation from 1 to 4 mm, while for the nano-hybrid resin statistical tests revealed a significant effect of the variables irradiation time and depth as well as the interaction between them. In the third study, it was noted that both before and after thermo-mechanical loading percentages of continuous margins in the cervical dentin were higher ($p <0.05$) in groups A and B compared with group C. While for cervical enamel group B showed higher values than those obtained in group C and group A presented intermediate results between B and C. For polymerization shrinkage "bulk-fill" resin exhibited higher rates of contraction compared to conventional resin ($p<0.05$). Thus, based on the results shown in the

three studies, we concluded that the tested composite resin was able to return the fracture resistance of the tooth in a similar way to that observed for intact teeth. The depth of cure in increments of up to 4 mm-thick, as claimed by the manufacturer, could be confirmed. The "bulk-fill" resin was superior to conventional resin regarding cervical marginal adaptation, however showed higher polymerization shrinkage.

Keywords: Compressive Strength; Dental Marginal Adaptation; Composite Resins; Dental Cavity Preparation; Spectroscopy, Fourier Transform Infrared; Polymerization.

Sumário

Introdução

1 Introdução

A busca por um material restaurador direto estético, ou seja, que reproduzisse as características ópticas da estrutura dental, culminou com o surgimento da resina de Bowen por volta da década de 60 (Bowen[3], 1962). Desde então, as pesquisas acerca do aprimoramento das resinas compostas tem sido incessantes. Muitos avanços já foram obtidos quanto às mais diversas propriedades das resinas restauradoras e novas perspectivas surgiram no que diz respeito ao restabelecimento da estética e função dentárias, bem como à preservação do tecido dental sadio (Van Meerbeek et al.[41], 2003).

Os materiais e técnicas restauradoras adesivas atuais tornaram possível tratar esteticamente dentes posteriores com previsibilidade de sucesso razoável (Hirata et al.[19], 2004). Entretanto, todos os materiais resinosos ainda sofrem contração inerente à reação de polimerização e, especialmente em cavidades profundas, um alto fator de configuração cavitária (fator C) pode amplificar os problemas com restaurações em resina composta devido a esta contração (Feilzer et al.[13], 1987; Hitz et al.[20], 2010). Dois tipos de problemas relacionados à contração de polimerização dos compósitos podem ser observados: fendas podem se formar caso o compósito esteja fracamente aderido aos tecidos dentais; e caso a resistência adesiva exceda as tensões de contração, a restauração mantém uma tensão residual interna que traciona as paredes do dente (Campos et al.[5], 2009). Alguns dos efeitos desses processos que podem ser percebidos clinicamente são sensibilidade pós-operatória e/ou descolamento da interface dente-restauração, que por sua vez pode levar ao manchamento marginal, inflamação pulpar e cáries secundárias (Campos et al.[5], 2009; Lutz et al.[29], 1986).

Em restaurações adesivas diretas de classe II, métodos incrementais, emprego de “inserts” cerâmicos e aplicação de base cavitária têm sido propostos para reduzir as tensões desenvolvidas no conjunto dente-restauração devido à contração de polimerização do compósito (Lutz et al.[29], 1986; Donly et al.[11], 1989; Friedl et al.[16], 1997). Entretanto, estas técnicas ainda não são consideradas suficientemente efetivas ao serem aplicadas em restaurações de classe II amplas, considerando-se a contração de polimerização dos materiais e a cura tardia espontânea que ocorre dias após a inserção da resina (Dietschi et al.[10], 2003). Além disso, a inserção da resina torna-se mais complicada com o aumento da profundidade cavitária (Hitz et al.[20], 2010), dificultando a obtenção de adaptação marginal satisfatória (Dietschi et al.[9], 2002). A adaptação do material restaurador às margens da cavidade é fator crucial na performance a longo prazo de qualquer restauração (Stavridakis et al.[39], 2007).

Apesar dos esforços constantes das indústrias de materiais dentários em criar um material resinoso com reduzida contração de polimerização e que promova perfeito selamento marginal, este objetivo ainda não foi alcançado. Nesse sentido, uma das criações recentes foram as resinas compostas de inserção em massa, mais conhecidas como “bulk-fill”. Dentre estas resinas, foi introduzida no mercado a SureFil® SDR™ *flow* (Dentsply Caulk, Milford, Delaware), como resultado das tentativas constantes de simplificar o tratamento restaurador de cavidades profundas e de elevado fator-C, bem como a reposição de dentina.

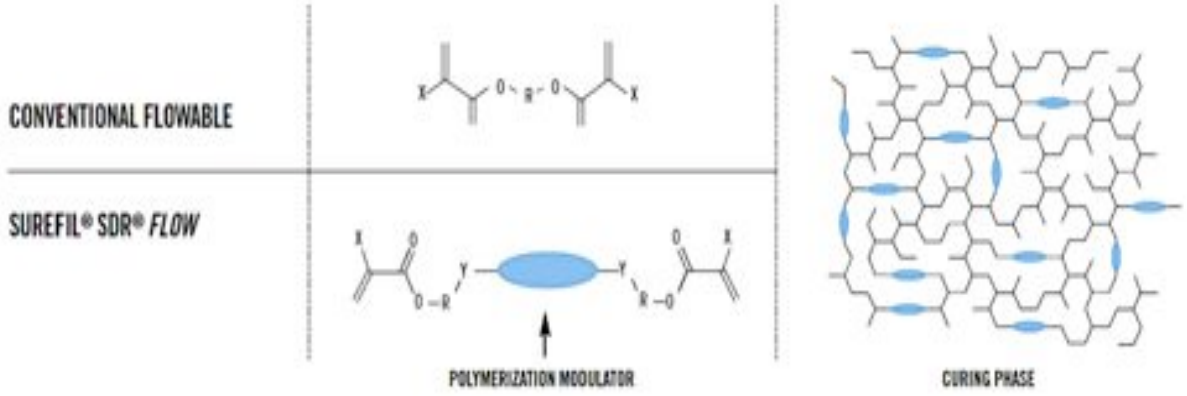
A sigla SDR™ significa “resina de estresse reduzido” (Stress Decreasing Resin) e os materiais baseados nesta tecnologia possuem a dinâmica da sua reação de polimerização modificada por meio da incorporação química de um grupo foto-ativo (modulador da polimerização – Figura 1) em uma resina à base de uretano

dimetacrilato (UDMA). De acordo com seu fabricante, esta tecnologia inovadora levou à obtenção de um compósito *flow* com polimerização residual muito baixa, transpondo a limitação provocada pela impossibilidade de inserção em massa de materiais restauradores convencionais à base de metacrilato (Koltisko et al.[25], 2010; Dentsply Caulk[8], 2010). A tecnologia empregada na SDR™ otimiza a forma com que a rede de polímero é formada durante a cura, reduzindo a tensão de polimerização causada por polímeros que se tornam muito tesos, permitindo assim a formação de uma rede polimérica mais relaxada do que na foto-ativação convencional, o que resulta em redução da tensão em até 60%. Acredita-se que o modulador de polimerização da SDR™ reduz o acúmulo de tensões em consequência da polimerização, sem uma redução na taxa de polimerização ou conversão. Ainda de acordo com o fabricante, ela foi desenvolvida para ser utilizada como base em cavidades de classe I e II, em incrementos de até 4mm de espessura, e recoberta por uma resina composta convencional para preenchimento final da cavidade (Dentsply Caulk[8], 2010).

Além dos aspectos relativos à contração de polimerização, a qualidade da polimerização e suas consequências também constituem-se como preocupações dos pesquisadores, já que a adequada conversão dos materiais resinosos em polímero é um fator essencial para a obtenção de propriedades físicas satisfatórias e bom desempenho clínico das restaurações (Amirouche-Korichi et al.[1], 2009; Jassé et al.[24], 2013). Sabe-se bem que a energia luminosa transmitida através dos compósitos é atenuada drasticamente conforme o aumento da distância entre a superfície irradiada e a fonte de luz, levando a uma diminuição no grau de conversão monomérica (Lindberg et al.[28], 2005). Por esse motivo, os incrementos das resinas compostas convencionais são limitados a 2mm de espessura (Leprince et al.[27],

2012). Um baixo grau de conversão é capaz de comprometer propriedades das resinas compostas, tais como: dureza, desgaste, resistência à compressão, tração, flexão, solubilidade, descoloração e degradação (Chung, Greener[7], 1990; Imazato et al.[22], 1995). Desta forma, como as novas resinas "bulk-fill" apresentam especificações diferenciadas quanto à espessura de seus incrementos, torna-se importante avaliar a profundidade de polimerização das mesmas.

Figura 1 - Representação gráfica da rede polimérica formada na SDR *flow* comparada àquela formada em compósito *flow* convencional (Fonte: Dentsply Caulk[8]).



A maioria dos estudos que avaliaram as propriedades físico-químicas da resina SDR, apontaram resultados satisfatórios quando a mesma era comparada às resinas compostas convencionais (Campos et al.[6], 2014; El-Damanhoury, Platt[12], 2013; Furness et al.[17], 2014; Ilie et al.[21], 2013; Moorthy et al.[30], 2012; Nazari et al.[31], 2013; Roggendorf et al.[35], 2011; Salerno et al.[38], 2011; Van Ende et al.[40], 2013). Entretanto, por se tratar de um produto relativamente novo no mercado odontológico e da carência de resultados de estudos clínicos de longo prazo, as análises in vitro se apresentam como alternativas ainda válidas, especialmente quando simulam as condições orais de carga e temperatura. Muito embora os estudos laboratoriais não

permitam a reprodução real da carga mastigatória, eles representam importante fonte de informação.

Considerando que é comum a ocorrência de situações clínicas em que lesões cariosas produzem grande perda de estrutura, é fundamental a recuperação do elemento dental do ponto de vista anatômico e funcional (Krejci et al.[26], 1993). Desse modo, justifica-se avaliar in vitro a melhor forma de recuperar o elemento dental, fundamentando assim futuras condutas clínicas dos profissionais. A escolha do material restaurador a ser empregado em uma cavidade depende da avaliação das condições do próprio dente. Com base nisto, é preciso conhecer os materiais restauradores disponíveis com relação às suas propriedades, composição e desempenho clínico de forma a possibilitar uma seleção apropriada conforme as necessidades do remanescente dental.

Dentro desse contexto, este estudo visa avaliar a resistência à fratura e adaptação marginal em preparos de classe II restaurados com a resina *flow* de reduzido *stress* de contração SureFil® SDR™, bem como o grau de conversão e contração de polimerização da mesma.

Proposição

2 Proposição

2.1 Geral

Avaliar diferentes propriedades de uma resina composta fluida “bulk fill”.

2.2 Específicas

Este estudo foi dividido em três capítulos compostos por artigos científicos de acordo com os objetivos propostos:

- Avaliar in vitro a resistência à fratura de molares humanos com preparos cavitários de classe II MOD restaurados com a resina SureFil® SDR™ *flow.*

- Avaliar o grau de conversão monomérica da resina SureFil® SDR™ *flow* em diferentes profundidades.

- Avaliar a contração volumétrica de polimerização da resina SureFil® SDR™ *flow*, bem como sua influência sobre a adaptação marginal cervical em molares humanos com preparos cavitários de classe II MOD, antes e após ensaio de ciclagem termo-mecânica em máquina de mastigação computadorizada.

Capítulos

3 Capítulos

3.1 Capítulo 1

- Fracture resistance of molars with class II cavities restored with different resin-based materials.
- A ser enviado para publicação no periódico Operative Dentistry.

3.2 Capítulo 2

- Bulk-fill *versus* conventional composite: a comparative analysis on degree of conversion.
- A ser enviado para publicação no periódico European Journal of Dentistry.

3.3 Capítulo 3

- Marginal adaptation in cervical dentin and enamel and polymerization shrinkage of a composite based on SDR technology.
- A ser enviado para publicação no periódico American Journal of Dentistry.

Capítulo 1

3.1 Capítulo 1[*] [**] [***]

FRACTURE RESISTANCE OF MOLARS WITH CLASS II CAVITIES RESTORED WITH DIFFERENT RESIN-BASED MATERIALS

FF Jassé • NF Pollo • EA Campos • I Krejci • S Ardu

Fernanda Ferreira Jassé, DDS, MSc, PhD student. UNESP Univ Estadual Paulista, Araraquara School of Dentistry, Department of Restorative Dentistry, Araraquara, SP, Brazil.

Natália Fernandes Pollo, DDS student. UNESP Univ Estadual Paulista, Araraquara School of Dentistry, Araraquara, SP, Brazil.

Edson Alves de Campos, DDS, MSc, PhD. UNESP Univ Estadual Paulista, Araraquara School of Dentistry, Department of Restorative Dentistry, Araraquara, SP, Brazil.

Ivo Krejci, Prof. Dr. Med. Dent. University of Geneva, Dental School, Division of Cariology & Endodontology, Geneva, Switzerland.

Stefano Ardu, Dr. Med. Dent., PhD. University of Geneva, Dental School, Division of Cariology & Endodontology, Geneva, Switzerland.

---

[*] De acordo com as normas estabelecidas pelo periódico Operative Dentistry.
[**] Metodologia detalhada no Apêndice 6.1.
[***] Metodologia aprovada pelo Comitê de Ética em Pesquisa conforme certificado exposto no Anexo 7.1.

Corresponding author: Fernanda Ferreira Jassé, UNESP Univ Estadual Paulista, Araraquara School of Dentistry, Department of Restorative Dentistry, Araraquara, SP, Brazil. 1680 Humaitá St., Zip Code: 14801-903, Araraquara, SP, Brazil.

Phone: +55 16 33016388

E-mail: fernandajasse@hotmail.com

Clinical Relevance

Considering the weakness of teeth caused by MOD cavity preparations, it becomes important to find a restorative material, which is able to reestablish the tooth resistance.

SUMMARY

The aim of the current study was to evaluate the fracture resistance of molars with class II MOD cavities restored with different restorative techniques and materials. Forty extracted molars were divided into 5 groups to test their fracture resistance. Group 1, the positive control, consisted of healthy teeth. Groups 2 to 5 received MOD Class II cavities with standardized dimensions. Teeth of group 2 were not restored and were considered as the negative control. In groups 3 to 5, the cavities were restored with: SDR™ *flow* resin only, SDR™ *flow* resin coated with a nano-hybrid resin and nano-hybrid resin only. All specimens were tested for resistance to fracture by means of axial compressive load, using a metallic sphere measuring 8 mm in diameter on an universal testing machine EMIC DL-2000, with a 10 kN load cell operated at a speed of 5 mm / min until the fracture of the tooth. Data were submitted to Analysis of Variance (ANOVA) and Tukey's test (α = 0.05). Group 3 showed the highest fracture resistance (2243,1 ± 473,7N) significantly different from groups 2, 4 and 5 ($p < 0,05$). The fracture strength of teeth restored with SDR™ *flow* MOD restorations is similar to intact natural teeth.

Keywords: Compressive Strength, Composite Resins, Cavity Preparation.

INTRODUCTION

Since the introduction of the acid etching technique by Buonocore in 1955 and advances obtained with composite resins, new insights have emerged with respect to restore dental function and aesthetics, as well as conservation of dental tissues.[1]

The current adhesive restorative materials and techniques makes it possible to treat aesthetically posterior teeth with a reasonable predictability of success.[2] However, all resin materials undergo an inherent shrinkage in the polymerization reaction and especially in deep cavities. Furthermore, a high cavity configuration factor (C-factor) can amplify the problems with composite resin restorations due to shrinkage.[3,4] Two types of problems related to polymerization shrinkage of composites can be observed: gaps can be formed if the composite is weakly attached to the dental tissues; if the bond strength exceeds the shrinkage stress, the restoration maintains an internal tension that pulls the walls of the tooth.[5] Some of the effects of these processes that may be perceived clinically are postoperative pain and degradation of the tooth-restoration interface, which in turn can lead to marginal staining, secondary caries and pulp inflammation.[5,6]

In direct adhesive Class II restorations, methods such as incremental techniques, the use of ceramic inserts or application of a base have been proposed to reduce the stress developed in tooth-restoration joint due to polymerization shrinkage of the composite.[7-9] However, these techniques are not considered sufficiently effective when applied to large class II restorations in view of the polymerization shrinkage of the materials and post curing effects that occur hours after insertion of the composite.[10] Moreover, insertion of the resin becomes more difficult when the cavity depth is increasing.[4] Due to these factors, it may be difficult to obtain a good marginal adaptation in broad and deep cavities.[11] The adaptation of

the restorative material to the cavity margins is a crucial factor in the long-term performance of any restoration.[12] That is why low shrinking stress flowable composites recently came into focus of research in the field of posterior restorations.[13]

A new restorative material suitable for posterior teeth with improved characteristics in respect to the polymerization shrinkage, SureFil SDR™ (Stress Decreasing Resin) *flow* was introduced into the market. According to the manufacturer, it was developed from a unique technology which led to a flow composite with very low residual polymerization shrinkage.[14,15] The technology employed in the SDR™ optimizes the way the polymer network is formed during cure, reducing the polymerization stress caused by polymers that become too stiff, thereby forming a polymer network more relaxed than in conventional photo activation, which results in reduction of contraction stress of up to 60%. This is done through a polymerization modulator that was chemically incorporated into the structure of the organic matrix of SDR™ resin monomer. According to the manufacturer, the polymerization modulator of SDR™ reduces the buildup of stress as a result of polymerization, without a reduction in the rate of polymerization or conversion.[15]

Sound teeth rarely fracture under normal masticatory function. Several studies have emphasized the importance of maintaining the tooth structure to preserve the strength of the remaining tooth.[16-18] Generally, the broader involvement by caries or cavities, the weaker the tooth becomes.[19] The weakening of the teeth due to MOD preparations and the contribution of restorative materials to improve the strength of the remaining dental tissues have been studied experimentally by several authors since 1956.[20-23] Most of these studies have in common the use of destructive

experimental systems fracturing teeth by applying an axial load, with the use of a steel sphere or cylinder in contact with the slope of the cusps.

Although in vitro studies are not a reproduction of a typical real chewing force, they represent an important source of information. Compression tests, in which a force is applied until specimens' fracture are the most used methodology to compare the fracture resistance of teeth that received various restorative material.

Within this context, this study aimed to compare the fracture resistance of healthy teeth and teeth with class II MOD cavities restored with SureFil SDR™ *flow*. The null hypothesis tested was that the fracture resistance of teeth restored with the new bulk-fill flow resin is not significantly different from healthy teeth.

MATERIALS AND METHODS

Forty sound human third-molars with similar dimensions were selected for the study. The teeth were subjected to cleaning and visual inspection with a magnifying glass, making sure the absence of cracks, cavities or any defect. The dimensions of the teeth were as follows, as verified by a digital caliper: 9.0±1.0 mm bucco-lingual and 7.0±1.0 mm mesio-distal. Teeth disinfection was performed with 0.1% thymol solution. Subsequently, teeth were stored in distilled water at 4˚C until used. The roots of the teeth were then put in the center of plastic cylindrical molds (20 mm in diameter and height), and embedded with epoxy resin until 2 mm of the cement-enamel junction. Care was taken to maintain the occlusal surface of the teeth parallel to the base of the cylinder, since in the tests of fracture resistance, the compressive force is applied parallel to the long axis of the teeth. The teeth were randomly assigned to five equal groups (table 1).

Class II box-shaped (MOD) cavities were prepared with parallel walls and bevelled enamel margins, with cervical margins located 1.0 mm below (mesial) and above (distal) the cement-enamel junction. Figure 1 illustrates the dimensions of the cavities. The cavities were prepared using cylindrical flat-end coarse-grained diamond burs under abundant water spray. Finishing procedures were performed with fine-grained burs in the same shape. Dimensions of preparations were monitored by using a caliper and a periodontal probe.

Teeth allocated to groups 3, 4, and 5 were subjected to the following restorative procedures:

Group 3: Cavities were cleaned with water spray and dried with absorbent paper, leaving dentin slightly moistened. Then, the self-etching one-bottle adhesive Xeno® V (Dentsply Caulk, Milford, Delaware) was applied according to the manufacturer's instructions and light cured for 20 s. The restorative material SureFil® SDR™ *flow* (Dentsply Caulk, Milford, Delaware) was applied directly into the cavity preparation using a Centrix® syringe (Centrix System, DFL, Jacarepaguá, Rio de Janeiro) with slow and steady pressure. The filling took place from the deepest portion of the cavity to the cavosurface edge in increments of up to 4 mm in height. The resin tip was withdrawn progressively as the cavity was filled. Increments were photo cured using the LED-based curing unit Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein), with power density of approximately 1100 mW/cm^2. Finishing procedures were performed using fine-grained burs.

Group 4: The same procedures as mentioned above for group 3 were performed until the application of the adhesive system. Then, SDR™ *flow* was put directly into the cavity using a Centrix® syringe with a slow and steady pressure. The insertion took place from the deepest portion of the cavity to 2mm below the

cavosurface edge. This increment was then light-cured, and the occlusal portion of the cavity (2mm) was filled with the nano- hybrid composite EsthetX™ HD (Dentsply Caulk, Milford, Delaware), followed by curing of this resin according to the manufacturer's instructions. Finishing procedures were performed using fine-grained burs.

Group 5: The same procedures as mentioned above for group 3 were performed until the application of the adhesive system. Then, the cavities were filled with oblique increments of approximately 2 mm thickness with Esthet-X™ HD following the manufacturer's instructions. Each increment was light-cured separately. Finishing procedures were performed using fine-grained burs.

After restoration, all groups were stored in distilled water, protected from light, at a temperature of 37˚C. After one week all the specimens were removed from the storage conditions and tested for fracture resistance by means of an axial compressive load, through the use of a metallic sphere measuring 8 mm in diameter. This sphere was attached to a universal testing machine EMIC DL-2000 (São José dos Pinhais, PR, Brazil) with a load cell of 10 kN, which was driven at a speed of 5 mm/min until fracture of the tooth.

The force necessary to fracture each tooth was recorded in Newtons (N) and the data was subjected to ANOVA and Tukey’s tests for the five experimental conditions.

RESULTS

Mean values of fracture resistance (N) and standard deviations for each of the five experimental conditions are shown in Table 2.

The fracture resistance of group 3 was significantly higher than the other groups *(p <0.05)*, except for group 1, which showed no significant difference *(p> 0.05)*. Teeth restored with SDR™, with SDR™ + nano-hybrid resin, only with nano-hybrid resin and intact teeth (groups 3, 4, 5 and 1, respectively) showed significantly higher fracture resistance when compared to the prepared/non-restored teeth (group 2) (*p <0,05*). There were no statistically significant differences between groups 1 and 3, between groups 1 and 4 and between groups 4 and 5 *(p> 0.05).*

DISCUSSION

The success of a restorative treatment depends, among other factors, of the correct selection of the restorative material and of an appropriate technique. Several studies have indicated that the fracture resistance of large cavity-prepared teeth is reduced due to loss of tooth structure.[20-23] Similarly, the results of this study also indicated that the tooth resistance was reduced after cavity preparation. Moreover, it is interesting to note that in the studies of Re *et al*.[25] and Blaser *et al*.[26] there was no significant difference between the fracture resistance of healthy teeth and those which were prepared and not restored.

The composite SureFil® SDR™ *flow* was recently marketed as a restorative material for direct use. There are no data available in the literature regarding the resistance to fracture of teeth restored with it. In the present study, we observed that teeth restored with the new resin present improved fracture resistance when compared to teeth restored with a nano-hybrid composite and similar values to healthy teeth. Therefore, the null hypothesis was accepted.

Dentin's structural strength depends on the quality and integrity of their anatomical form, so that the main problem is the small amount of remaining sound

dentin that maintains and supports the restoration.[27] Therefore, preparation of an extensive MOD cavity may cause fracture of tooth cusps if it is not restored.[28,29] The results of this study also showed that the restoration of a wide tooth cavity is important to achieve an increase of its resistance to fracture. Therefore, the reinforcement of the cavity with a suitable restorative material is needed to support the remaining tooth structure.

In a study by Yogesh and Jagadish,[30] the authors suggested that composites when used in posterior teeth show great potential as a reinforcement material of cusps. Other studies have also demonstrated improved fracture resistance of teeth after the use composite resins for MOD restoration.[19,28,31] However, the shrinkage of composites during polymerization reaction is one of the main factors adversely affecting the success of direct adhesive restorations made of composite resin. The polymerization reaction of photo-activated composites leads to the development of higher stresses when these composites are adhered to the walls of the cavity. To minimize the effects of polymerization shrinkage the incremental insertion technique of composite resins has been used, and it is widely accepted in the dental community, as it is believed that this technique results in reduced accumulation of stresses in the tooth-restoration interface.[7] As the concepts of incremental insertion have been described as mandatory when working with composite materials, in this study the 2 mm-incremental insertion technique was employed with the conventional composite. As for the teeth restored with SDR™ *flow*, 4 mm increments in height were used, by filling most of the cavity in a single insertion / photo-activation, since this is a composite based on the "bulk-fill" concept, with reduced polymerization stress. Probably due to the cusps reinforcement potential of composites associated with lower polymerization stress generated by resin SDR™ *flow*, groups of teeth

restored with the new resin showed fracture resistance values similar to those of intact teeth. While teeth restored with conventional resin showed lower resistance than healthy teeth.

Despite the fact that the group restored only with the resin SDR™ *flow* showed superior resistance when compared to other groups of restored teeth, it is not clinically possible to use it in the entire cavity, i.e. without the 2 mm occlusal coating using the conventional composite. This occurs because the resin SDR™ flow, that is a fluid-consistency resin, does not allow proper sculpture and finishing of the occlusal surface and marginal ridges. In addition to the functional aspect, the aesthetic factor must also be considered, since the new resin is marketed in a single color, which is moderately translucent. Therefore, using the resin SDR™ *flow* according to the manufacturer's recommendations as a base material coated with a conventional resin, it is possible to obtain adequate results of fracture resistance, with values similar to those obtained for intact teeth, according to the results of the present study. Using the material in this manner, it becomes possible to achieve the restorative needs of a dental element with a wide cavity for both, the functional and the aesthetic aspect.

The method of applying occlusal compressive load during the fracture test is another important factor. In this *in vitro* study, the axial forces were applied at the center of the occlusal surface. Clinically, axial forces in addition to lateral forces and fatigue must be considered. Burke *et al*.[22] concluded that the best method to measure the resistance of premolars to fracture is the use of a cylinder with a defined diameter. The use of a metal sphere of 8 mm in diameter for testing fracture resistance, as used in the study conducted by Barbosa and Piazza[24] proved to be ideal for molars because it comes in contact with the functional and non-functional

cusps, being in positions close to what is found clinically. That's why the teeth were subjected to a compressive load by means of a vertical steel sphere of 8 mm in diameter.

Regarding the clinical relevance of these findings, it should be considered that this study was conducted under *in vitro* conditions and the mechanical test was performed 7 days after the restorations placement. Ideally, more reliable test methods must be developed in order to better reproduce the *in vivo* failure mechanisms that occur clinically with teeth and restorations. Further tests as fatigues test and clinical investigations are also recommended to check the results of this study.

CONCLUSIONS

Within the limitations of this study, it can be concluded that:

1. Teeth with MOD cavity preparations have decreased fracture resistance;
2. The restorative technique involving the use of SDR™ *flow* showed the ability to reverse the fracture resistance lost after MOD cavity preparation.

ACKNOWLEDGMENTS

The authors thank DENTSPLY DeTrey (Konstanz, Germany) for kindly donating the materials used in this study. This work was supported by a grant from CAPES.

References

1. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P & Vanherle G (2003) Buonocore memorial lecture.

Adhesion to enamel and dentin: current status and future challenges *Oper Dent* **28(3)** 215-235.

2. Hirata R, Higashi C & Masotti A (2004) Simplificando o uso de resinas compostas em dentes posteriores *R Dental Press Estét* **1(1)** 18-34.
3. Feilzer AJ, De Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *J Dent Res* **66(11)** 1636-1639.
4. Hitz T, Ozcan M & Gohring TN (2010) Marginal Adaptation and Fracture Resistance of Root-canal Treated Mandibular Molars with Intracoronal Restorations: Effect of Thermocycling and Mechanical Loading. *J Adhes Dent* **12(4)** 279-286.
5. Campos EA, Andrade MF, Porto-Neto ST, Campos LA, Saad JRC, Deliberador TM & Oliveira-Júnior OB (2009) Cuspal movement related to different bonding techniques using etch-and-rinse and self-etch adhesive systems *Eur J Dent* **3(3)**: 213-218.
6. Lutz F, Krejci I & Barbakow F (1991) Quality and durability of marginal adaptation in bonded composite restorations *Dent Mater* **7(2)** 107-113.
7. Lutz E, Krejci I & Oldenburg TR (1986) Elimination of polymerization stresses at the margins of posterior composite resin restorations: a new restorative technique *Quintessence Int* **17(12)** 777-784.
8. Donly KJ, Wild TW, Bowen RL & Jensen ME (1989) An in vitro investigation of the effects of glass inserts on the effective composite resin polymerization shrinkage *J Dent Res* **68(8)** 1234-1237.
9. Friedl KH, Schmalz G, Hiller KA & Mortazavi F (1997) Marginal adaptation of composite restorations versus hybrid ionomer/composite sandwich restorations *Oper Dent* **22(1)** 21-29.

10. Dietschi D, Olsburgh S, Krejci I & Davidson C (2003) In vitro evaluation of marginal and internal adaptation after occlusal stressing of indirect class II composite restorations with different resinous bases *Eur J Oral Sci* **111(1)** 73-80.
11. Dietschi D, Monasevic M, Krejci I & Davidson C (2002) Marginal and internal adaptation of class II restorations after immediate or delayed composite placement *J Dent* **30(5-6)** 259-269.
12. Stavridakis MM, Kakaboura AI, Ardu S & Krejci I (2007) Marginal and internal adaptation of bulk-filled class I and cuspal coverage direct resin composite restorations *Oper Dent* **32(5)** 515-523.
13. Schattenberg A, Busemann I, Willershausen B & Ernst CP (2008) Determination of Polymerization Stress in Low Shrinkage Resin Composites *J Dent Res* **87** (Abstract 1818).
14. Koltisko B, Dai Q, Jin X, Bertrand S & Lu H (2010) The Polymerization Stress of Flowable Composites *J Dent Res* **89** (Abstract 321).
15. Dentsply Caulk (2010) Surefil SDR flow – Product Brochure. *Available in*: http://www.surefilsdrflow.com/sites/default/files/SureFil_Brochure.pdf.
16. Marchi GM, Mitsui FH & Cavalcanti AN (2008) Effect of remaining dentine structure and thermal-mechanical aging on the fracture resistance of bovine roots with different post and core systems *Int Endod J* **41(11)** 969-976.
17. Marchan SM, Coldero L, White D, Smith WA & Rafeek RN (2009) Cusp fracture resistance of maxillary premolars restored with the bonded amalgam technique using various luting agents *Int J Dent* **2009** 946830.
18. Ng CC, Dumbrigue HB, Al-Bayat MI, Griggs JA & Wakefield CW (2006) Influence of remaining coronal tooth structure location on the fracture resistance of restored endodontically treated anterior teeth *J Prosthet Dent* **95(4)** 290-296.

19. Mondelli RFL, Ishikiriama SK, Oliveira Filho O & Mondelli J (2009) Fracture resistance of weakened teeth restored with condensable resin with and without cusp coverage *J Appl Oral Sci* **17(3)** 161-165.

20. Vale WA (1956) Cavity preparation *Irish Dent Rev* **2** 33-41.

21. Mondelli J, Steagall L, Ishikiriama A, de Lima Navarro MF & Soares FB (1980) Fracture strength of human teeth with cavity preparations *J Prosthet Dent* **43(4)** 419-422.

22. Burke FJ, Wilson NH & Watts DC (1993) The effect of cavity wall taper on fracture resistance of teeth restored with resin composite inlays *Oper Dent* **18(6)** 230-236.

23. Mondelli J, Sene F, Ramos RP & Benetti AR (2007) Tooth structure and fracture strength of cavities *Braz Dent J* **18(2)** 134-138.

24. Barbosa AN & Piazza JL (2010) Resistência à fratura de dentes com perda estrutural restaurados com resina composta e sistema adesivo autocondicionante *Rev Sul-Bras Odontol* **7(1)** 11-18.

25. Re GJ, Draheim RN & Norling BK (1981) Fracture resistance of mandibular molars with occlusal Class I amalgam preparations *J Am Dent Assoc* **103(4)** 580-583.

26. Blaser PK, Lund MR, Cochran MA & Potter RH (1983) Effect of designs of Class II preparations on resistance of teeth to fracture *Oper Dent* **8(1)** 6-10.

27. Assif D & Gorfil C (1994) Biomechanical considerations in restoring endodontically-treated teeth *J Prosthet Dent* **71(6)** 565-567.

28. Gelb MN, Barouch E & Simonsen RJ (1986) Resistance to cusp fracture in Class II prepared and restored premolars *J Prosthet Dent* **55(2)** 184-185.

29. Pilo R, Brosh T & Chweidan H (1998) Cusp reinforcement by bonding of amalgam restorations *J Dent* **26(5-6)** 467-472.

30. Jagadish S & Yogesh BG (1990) Fracture resistance of teeth with Class II silver amalgam, posterior composite, and glass cermet restorations *Oper Dent* **15(2)** 42-47.

31. McCullock AJ & Smith BG (1986) In vitro studies of cusp reinforcement with adhesive restorative material *Br Dent J* **161(12)** 450-452.

TABLE CAPTIONS

Table 1: Experimental groups.

Table 2: Mean fracture resistance and standard deviations for each experimental condition.

FIGURE CAPTIONS

Figure 1. Dimensions of MOD preparations.

TABLES

Table 1

Group	Cavity	Type of restoration
1	Intact	Intact teeth
2	MOD	Non-restored
3	MOD	Completely filled with SDR™
4	MOD	Filled with SDR™ + Esthet-X HD
5	MOD	Completely filled with Esthet-X HD

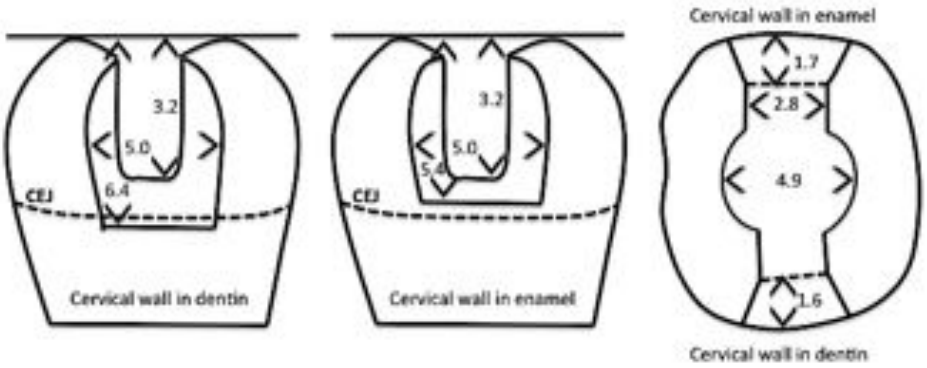
Table 2

Group	n	Type of restoration	Mean ± SD (N)
1	8	Intact teeth	$2243,1 \pm 473,7^{AB}$
2	8	Non-restored	$708,4 \pm 269,7^{D}$
3	8	Completely filled with SDR™	$2849,3 \pm 433,2^{A}$
4	8	Filled with SDR™ + Esthet-X™ HD	$1753,8 \pm 512,3^{BC}$
5	8	Completely filled with Esthet-X™ HD	$1406,8 \pm 428,5^{C}$

Same letters indicate statistically similar values – ANOVA and Tukey's tests (p > 0,05).

FIGURES

Figure 1



Capítulo 2

3.2 Capítulo 2[*] [**]

Bulk-fill *versus* conventional composite: a comparative analysis on degree of conversion

Fernanda Ferreira Jassé[a*], Hélida Gomes de Oliveira Barud[a], Juliana Maria Capelozza Boaventura[a], Hernane da Silva Barud[b], Alexandre Gatti[b], Edson Alves de Campos[a]

[a] Department of Restorative Dentistry, Araraquara School of Dentistry, UNESP Univ Estadual Paulista, Araraquara, Brazil

[b] Araraquara Chemistry Institute, UNESP Univ Estadual Paulista, Araraquara, Brazil

Running title: Monomer conversion of composites

[*]**Corresponding author**:

Fernanda Ferreira Jassé

1680 Humaitá St.

Centro 14801-903 - Araraquara, SP

Tel +55 16 33016388

fernandajasse@hotmail.com

E-mail co-authors: helida.barud@gmail.com; juliboav@hotmail.com; hernane.barud@gmail.com; alegatti@gmail.com; edson.campos@foar.unesp.br

[*] De acordo com as normas estabelecidas pelo periódico European Journal of Dentistry.
[**] Metodologia detalhada no Apêndice 6.1.

Bulk-fill *versus* conventional composite: a comparative analysis on degree of conversion

Abstract

Objective: This study aimed to determine the degree of conversion (DC) of two resin-based composites: one conventional nano-hybrid composite and one bulk-fill flowable composite (SureFil® SDR™ *flow*). **Methods:** DC was evaluated at 1, 2, 3 and 4 mm depth at varying irradiation times (20 and 40 s) using Fourier transform infrared (FTIR) spectroscopy. Disc-shaped specimens were prepared for each milimeter and photo-activated with a second-generation LED LCU. Specimens were stored for 24 h in a dark ambient and then pulverized into a fine powder that was mixed with potassium bromide (KBr). After homogenization of the mixture a pellet was done by pressing. All pellets were evaluated using an infrared spectrometer equipped with TGS detector using diffuse reflectance. The percentage of monomer conversion was determined from the ratio of absorbance intensities of aliphatic carbon-carbon double bonds (C=C) and the internal standard before and after the curing of the composite, represented by the aromatic carbon-carbon single bonds (C-C). **Results:** Two-Way ANOVA of the DC data for “bulk-fill” resin showed no significant difference ($p>0,05$) for irradiation depths ranging from 1 to 4mm, while for conventional nano-hybrid resin statistic tests revealed a significant effect of the variables time and depth, and also of the interaction between them. **Conclusion:** The results indicated that SDR™ achieves a 4mm depth of cure with both 20 and 40 second light-exposure.

Keywords: Degree of conversion, Composite Resins, Bulk-fill flowable .

Introduction

Since the introduction of composites in the early sixties, resin-based materials have presented a constantly technical progress with regard to new types of filler particles, different concentrations of these particles and their considerable size reduction.[1] However, despite all the evolution of resin-based materials, conventional composite resins of the beginning of the XXI century still present unfavorable aspects such as an insufficient degree of monomer conversion[2] and generation of stress by polymerization shrinkage upon curing.[3]

Among other properties, a resin-based material may exhibit two important features to be considered as ideal: high degree of conversion (DC) and minimal polymerization shrinkage.[4] However, these features vary generally antagonistically, because a high degree of conversion occurs when the monomer components chemical interlacing is increased, which consequently produces higher volume shrinkage.[5] The main problem caused by higher volume shrinkage is the stress concentration induced to cavity walls,[6] which goes along with cracks, gaps, secondary caries[7] and postoperative sensitivity.[8] On its turn, a lower degree of conversion may reflect in problems like release of unreacted monomer components[9] or high water sorption.[10]

New resin-based restorative materials for posterior teeth, which have improved characteristics with respect to polymerization shrinkage, were recently launched in the market. They are the so-called “bulk fill” materials. Manufacturers have claimed they reach satisfactory degree of conversion to a maximal increment thickness of 4-5 mm, depending on trademark, what can transpose the need for applying and curing of the composite resin in increments of limited thickness[11]. The “bulk filling” of cavities, besides saving clinical time, also prevents the incorporation of

air bubbles and contamination between increments, thus favoring the homogeneity of the restorative material.

It is well known that light energy transmission through the composite is attenuated drastically with increasing distance from the surface irradiated by the light source, what leads to a decrease in monomer conversion. Because of this, the increments of conventional composite resins are limited to 2 mm. A decrease in monomer conversion can compromise physical properties and clinical performance of the restorations,[5] since degree of conversion is a co-determining factor of the restorative resins' physical and chemical properties such as hardness, wear resistance, compressive strength, flexural strength, dimensional stability, solubility, discoloration and degradation reactions.[12] Some studies also found that the release of unreacted monomer that remains in the material[5] may stimulate bacteria growth around the restoration, irritate soft tissues and also cause allergic reactions in some patients.[9,13]

Several methods have been used to assess the degree of conversion. Infrared spectroscopy, calorimetry, Raman scattering[14] and even indirect techniques such as measuring top and bottom hardness have been employed. Both infrared spectroscopy and Raman spectroscopy are classified as vibrational techniques, because they are sensitive to the vibrational modes of molecules[15] while calorimetry allows to measure the metacrylic groups conversion through the exothermic polymerization reaction.[16] Among the spectroscopic techniques commonly reported in the literature, Raman technique shows the advantage of analysing the material without any sample preparation, however FTIR (Fourier transform infrared) technique has traditionally provided much useful information on the monomer conversion of dental composites.

Although there are several techniques to assess DC described in the literature, the development of "bulk-fill" resins brings with it the need to adapt the different existing techniques, especially in regard to the preparation of specimens, as manufacturers advertise their adequate photo-activating in increments up to 4mm in depth, which is beyond the specification of ISO 4049 standard for testing composites.[17]

Considering the above mentioned facts, the aim of this study was to evaluate the degree of conversion through FTIR analysis of a bulk-fill flow composite in comparison to a nano-hybrid composite resin containing conventional matrix by means of variable specimen depth and irradiation time. The following null hypotheses were tested: 1- Depth has no significant influence on DC; 2- irradiation time has no significant influence on DC; 3- The bulk-fill flow composite shows no difference in DC behavior to the conventional material.

Materials and methods

Table 1 shows the resin composites tested.

Specimen preparation

To determine degree of conversion at different depths, a set of four cylindrical molds was planned, according to the schematic drawing in figure 1. The molds (1 mm depth and 4 mm diameter) were successively filled and superposed on each other, with a polyester strip separating each mold from its neighbor. The LED light-curing unit (Bluephase G2, Ivoclar-Vivadent, Schaan, Liechtenstein) tip was then placed against the upper surface of the set of molds through a glass slide and light

activation was initiated using the high power mode (approximately 1100mW/cm^2) for either 20 or 40s. After light activation the specimens were carefully removed from the molds and identified according to the number of the sample and its depth (i.e. 1, 2, 3 and 4 mm). Five replications were performed for each composite resin (n=5). The specimens were then stored for 24h in the dark at 37± 1°C before analysis.

Degree of conversion (%) analysis

After storage, composites were pulverized into a fine powder, which was weighed (5 mg) and thoroughly mixed with bromide potassium (KBr) powder salt (100 mg). The obtained mixture was inserted into a pelleting device and pressed with a load of 10 t during 1 minute to obtain a pellet.

The analyzing of the pellets was carried out in a Fourier transform infrared spectrophotometer (Nexus-470 FT-IR, Thermo Nicolet, E.U.A) to measure the DC of the composites. DC is provided by the number of carbon-double-bonds (C=C), that are converted into carbon-single-bonds. FT-IR spectra of both uncured and cured samples were obtained through a TGS detector using diffuse reflectance coupled to a computer. The spectra were recorded in the absorbance operating mode under the following predetermined settings: 32 scans, 4 cm^{-1} resolution and 300 to 4000 cm^{-1} wavelength.

The percentage of unreacted double-carbon bonds (% C=C) is obtained by the ratio between the absorbance intensities of aliphatic C=C (peak at 1638 cm^{-1}) and the internal standard before and after the composite cure, represented by the aromatic C-C (peak at 1608 cm^{-1}). DC was determined according to the following formula:

$$DC(\%) = 1 - \frac{\left(1638cm^{-1} / 1608cm^{-1}\right)cured}{\left(1638cm^{-1} / 1608cm^{-1}\right)uncured}$$

Results

Results are listed in Table 2. The Two-Way ANOVA of the DC data for SDR™ resin showed no significant difference ($p>0,05$) for irradiation depths ranging from 1 to 4mm (in 1mm increments). The mean DC of SDR™ varied from a maximum of 78,24 (±3,46)% at an irradiation depth of 2 mm (40 s irradiated) to a minimum of 73,20 (±2,92)% at an irradiation depth of 4 mm (20 s irradiated). As for Esthet-X™ Two-Way ANOVA and Bonferroni test revealed a significant effect of the variables time and depth, and also of the interaction between them. For Esthet-X™ DC varied from a maximum of 74,79 (±2,63)% (2 mm – 40 s irradiated) to a minimum of 3,94 (±9,50)% (4 mm – 20 s irradiated).

Considering DC as a function of irradiation time, a difference could be observed only for Esthet-X™ in 4 mm depth, where 40 s of irradiation showed a significantly better DC than 20 s of irradiation. However, for Esthet-X™, only the DC of 1 mm and 2 mm (for both 20 and 40 s of irradiation) are statistically similar, so that from 3 mm onwards DC decreased linearly. Insofar SDR™ presented no difference between irradiation times for all millimeters of depth. Both composite resins performed similar until 2 mm in depth.

Discussion

It is well known that light energy emitted from a LCU decreases while transmitted through composites.[18] So the bigger the distance from the irradiated surface the lower will be DC of the resin-based material.[19] Based on this knowledge, authors have regarded incremental techniques to apply and cure resin composites with a 2mm-maximal-thickness increment.[2,19] However in contrast to these principles, the use of bulk-fill composites with a 4mm-maximal-thickness increment have been advocated nowadays, allowing a less time-consuming and safer restoration technique than before, especially for deep cavities. According to the values of monomer conversion achieved in this study, SDR™ meets the 4 mm manufacturer's assumption with 20 seconds of photo-activation. Thus, considering a molar with a Class II MOD cavity of about 6mm-deep in proximal boxes, to restore it with a bulk-fill resin can provide a saving of clinical time less than half the time it would take to restore it by the conventional incremental technique. It is possible because with a single increment of 4mm we can fill the two proximal boxes and part of the occlusal box and polymerize it with just 20 s and the other 2mm that were left we can fill them in three conventional increments (increments of no more than 2mm with a conventional resin) polymerized for at least 20 s each. Thus, it totals up 80 seconds of photoactivation, while for conventional technique we would need approximately ten increments of a conventional resin for filling the same cavity, each being cured for at least 20 s, totaling 200 s at all.

There are over 25 years the International Organization for Standardization (ISO) introduced a method for defining the depth of cure of resin composites, the well-known "ISO 4049; Depth of cure".[20] According to the method, tube-shaped

specimens of the tested resin are light-cured and the uncured part of the resin is then scraped away. Depth of cure is then given by the total length of the residual hard tube divided by the factor two. The resulting value determines the maximum increment thickness of the tested resin. Considering that resin composites are in continuous development as regards their composition and properties, studies have tested new ways to evaluate the depth of cure of new resins, especially for “bulk-fill” ones.[17,19,21] In a research published in 2012,[19] the authors verified the accuracy of the ISO 4049 method when used for “bulk-fill” materials and concluded that the method overestimates depth of cure. Based on these findings, an alternative way was used in this study to determine depth of cure through degree of conversion assessment of each millimeter of resin samples.

Many studies have assessed DC of the external surface of a restoration using simple techniques, however the evaluation of monomer conversion in the inner layers of resins is not so simple to assess.[7] In the current study, a matrix has been manufactured specifically for the assessment of the degree of conversion of the resins at different depths. Similar device was used in the study performed by Finan *et al*.[17], who assessed the degree of conversion up to 8 mm deep in specimens of 11 mm diameter. In the previously mentioned study, the authors observed for SDR™ a maximum degree of conversion of 59% at a irradiation depth of 1 mm and at least 45% to 8 mm-depth. In the present study, using a similar methodology, SDR™ resin reached maximum degree of conversion of 76% considering the same irradiation time of 20s. The difference between results of the mentioned researches can probably be due to photo-activating unit. In this study a high intensity LED was used while in that study a quartz tungsten halogen (QTH) light curing unit (operating at an output intensity of 650 ± 26 mW/cm^2) was used. Reuggeberg,[22] also reports the

variation from infrared spectroscopy technique, more specifically to the standard baseline technique that has been used when C=C peak intensities are determined. The author points out that baselines can be drawn in a number of locations, and not all researchers draw baselines in a similar manner. Thus, degree of conversion results may be imprecise when inter-laboratory results are compared.[22]

The degree of conversion evaluation permits characterization of the transformation of monomers into polymer. Several methodologies can be used to assess the degree of conversion of composite resins.[16] FTIR spectroscopy is a technique that may be applied for the evaluation of monomer conversion into polymer through calculation of the ratio of the aliphatic carbon-to-carbon (C=C) absorption at 1638 cm^{-1} to the aromatic C=C absorption at 1608 cm^{-1}.[22] This technique is well reported in the literature, though recent studies[7,17,21,23] have been used infrared spectroscopy with ATR accessory in demand for simplified sample preparation when compared to TGS detector. Considering the technique used in this study, Shin *et al.*[15] highlighted the fact that samples prepared in thin and fragile sections is one of the disadvantages of the technique. However we believe that by crushing the specimen representative of each millimeter of samples we are using a portion of the specimen's body for analysis and not just the surface of it, as occurs when the ATR crystal is used.

The efficiency of polymerization is completely dependent on the characteristics of the light produced.[16] Thus, a second generation LED LCU was chosen because its peak emission coincides with the maximum absorption of the camphoroquinone, which is the photo-initiator contained in both tested resins.[24] This kind of LED LCU was used in the present study in order to achieve the best results of the tested materials, so that LCU does not adversely influence the research results.

In this in vitro study depth had influence on degree of conversion to Esthet-X™ resin, but not to SDR™. Therefore, the first null hypothesis was partially accepted. The same happened to the second null hypothesis, since the irradiation time presented influence just on Esthet-X™ resin. However, up to 2 mm in depth (maximum increment recommended by the manufacturer) there was no influence of irradiation time, because for both times tested the DC values were statistically similar. The third null hypothesis was rejected, since the materials tested behaved differently as to degree of conversion.

This study evaluated only one bulk-fill material, which is a limitation with respect to generalizing the conclusions. More studies should be done to assess the behavior of other brands of bulk-fill composites.

Conclusions

- This study indicates that SDR™ achieves a 4mm depth of cure with both 20 and 40 seconds light-exposure.
- Depth has significant influence on DC to Esthet-X™, but not to SDR™.
- Irradiation time does not have significant influence on DC to SDR™, while to Esthet-X™ it does.

Acknowledgements

The authors would acknowledge receipt of the bulk-fill SDR™ flowable resin from Dr. Walter R. Dias (DDS, MS), DENTSPLY DeTrey GmbH, Konstanz, Germany. This work was supported by a grant from CAPES.

References

1. Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. *J Am Dent Assoc* 2003;134:1382–90.
2. Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and polymerization of composite restorative materials. *J Dent* 1992;20:183–8.
3. Davidson CL, Feilzer AJ. Polymerisation shrinkage and polymerisation shrinkage stress in polymer-based restoratives. *J Dent* 1997;25:435–40.
4. Dewaele M, Truffier-Boutry D, Devaux J, Leloup G. Volume contraction in photocured dental resins: the shrinkage- conversion relationship revisited. *Dent Mater* 2006;22:359–365.
5. Amirouche-Korichi A, Mouzali M, Watts DC. Effects of monomer ratios and highly radiopaque fillers on degree of conversion and shrinkage-strain of dental resin composites. *Dent Mater* 2009;25:1411–1418.
6. Ferracane JL, Mitchem JC. Relationship between composite contraction stress and leakage in class V cavities. *Am J Dent* 2003;16:239–243.
7. Frauscher KE, Ilie N. Degree of conversion of nano-hybrid resin-based composites with novel and conventional matrix formulation. *Clin Oral Investig* 2013 Mar;17(2):635-42.
8. Opdam NJ, Roeters FJ, Feilzer AJ, Verdonschot EH. Marginal integrity and postoperative sensitivity in class 2 resin composite restorations in vivo. *J Dent* 1998;26:555–562.
9. Tuna EB, Aktoren O, Oshida Y, Gencay K. Elution of residual monomers from dental composite materials. *Eur J Paediatr Dent* 2010;11:110–114.

10. Sideridou I, Achilias DS, Spyroudi C, Karabela M. Water sorption characteristics of light-cured dental resins and composites based on Bis-EMA/PCDMA. *Biomaterials* 2004;25:367– 376.
11. Dentsply Caulk. Surefil SDR flow – Product Brochure. 2010 Available in:http://www.surefilsdrflow.com/sites/default/files/SureFil_Brochure.pdf
12. Chung KH, Greener HE. Correlation between degree of conversion, filler concentration and mechanical properties of posterior composite resins. *J Oral Rehabil* 1990;17:487-94.
13. Moore BK, Platt JA, Borges G, Chu TM, Katsilieri I. Depth of cure of dental resin composites: ISO 4049 depth and microhardness of types of materials and shades. *Oper Dent* 2008 Jul-Aug;33:408-12.
14. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the "depth of cure" of dimethacrylate-based dental composites. *Dent Mater* 2012 May;28:512-20.
15. Shin WS, Li XF, Schwartz B, Wunder SL, Baran GR. Determination of the degree of cure of dental resins using Raman and FT-Raman spectroscopy. *Dent Mater* 1993 Sep;9:317-24.
16. Silva EH, Albuquerque RC, Lanza LD, Vieira GC, Peixoto RT, Alvim HH, Yoshida MI. Influence of different light sources on the conversion of composite resins. *Indian J Dent Res* 2011 Nov-Dec;22:790-4.
17. Finan L, Palin WM, Moskwa N, McGinley EL, Fleming GJ. The influence of irradiation potential on the degree of conversion and mechanical properties of two bulk-fill flowable RBC base materials. *Dent Mater* 2013 Aug;29:906-12.
18. Price RB, Murphy DG, Dérand T. Light energy transmission through cured resin composite and human dentin. *Quintessence Int* 2000;31:659–67.

19. Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? *Dent Mater* 2012 May;28:521-8.
20. ISO 4049:1988 (2.). Dentistry—resin-based filling materials. International Organization for Standardization; 1988.
21. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. *Clin Oral Investig* 2013 Jan;17:227-35.
22. Rueggeberg FA, Hashinger DT, Fairhurst CW. Calibration of FTIR conversion analysis of contemporary dental resin composites. *Dent Mater* 1990 Oct;6:241-9.
23. Alshali RZ, Silikas N, Satterthwaite JD. Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals. *Dent Mater* 2013 Sep;29:e213-7.
24. Uhl A, Sigusch BW, Jandt KD. Second generation LEDs for the polymerization of oral biomaterials. *Dent Mater* 2004;20:80-7.

TABLES

Table 1 - Summary of the dental resin composites investigated in this study.

Brand name	Type	Composition	Filler loading	Manufacturer	Shade
SureFil® SDR™ *flow*	Flowable bulk fill	Ba-Al-F-B silicate glass, Sr-A-F silicate glass, Modified UDMA, EBPADMA, TEGDMA. Camphorquinone photo-initiator (CQ), butylated hydroxytoluene (BHT), UV stabilizer, Titanium dioxide, Iron oxide pigments.	68 wt%	Dentsply	universal
Esthet-X® HD	Conventional nanohybrid	Bis-GMA, Bis-EMA, TEGDMA, Camphorquinone photo-initiator (CQ), UV stabilizer, pigments. Combination of particulate fluoro-barium-borosilicate glass with a mean particle size below 1µm and silica nanoparticles of 0,04 µm.	60 wt%	Dentsply	A2

UDMA: urethane dimethacrylate; EBPADMA: ethoxylated Bisphenol A dimethacrylate; TEGDMA: ttriethyleneglycol dimethacrylate; Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA: Bisphenol A dimethacrylate.

Table 2 – Means and standard deviation of degree of conversion (DC) evaluated on specimens of variable depths after different irradiation times.

Material	Time (s)	DC 1mm (%)	DC 2mm (%)	DC 3mm (%)	DC 4mm (%)
Esthet-X * α	20	70,74 aA (±4,97)	62,40 aA (±3,99)	44,46 bA (±23,90)	3,94 cB (±9,50)
	40	73,66 aA (±4,36)	74,79 aA (±2,63)	42,14 bA (±6,55)	29,81 bA (±5,23)
SDR ** α	20	76,66 dC (±3,48)	76,82 dC (±1,27)	76,72 dC (±3,69)	73,20 dC (±2,92)
	40	75,62 dC (±1,95)	78,24 dC (±3,46)	76,24 dC (±4,46)	74,64 dC (±7,07)

* The influence of effects and interaction between time and curing depth was considered significant ($p<0,05$).

** The influence of effects and interaction between time and curing depth was considered not significant ($p>0,05$).

α Equal lowercase letters in the same row represent no statistical difference; equal capital letters in the same column represent no statistical difference ($p<0,05$).

FIGURES

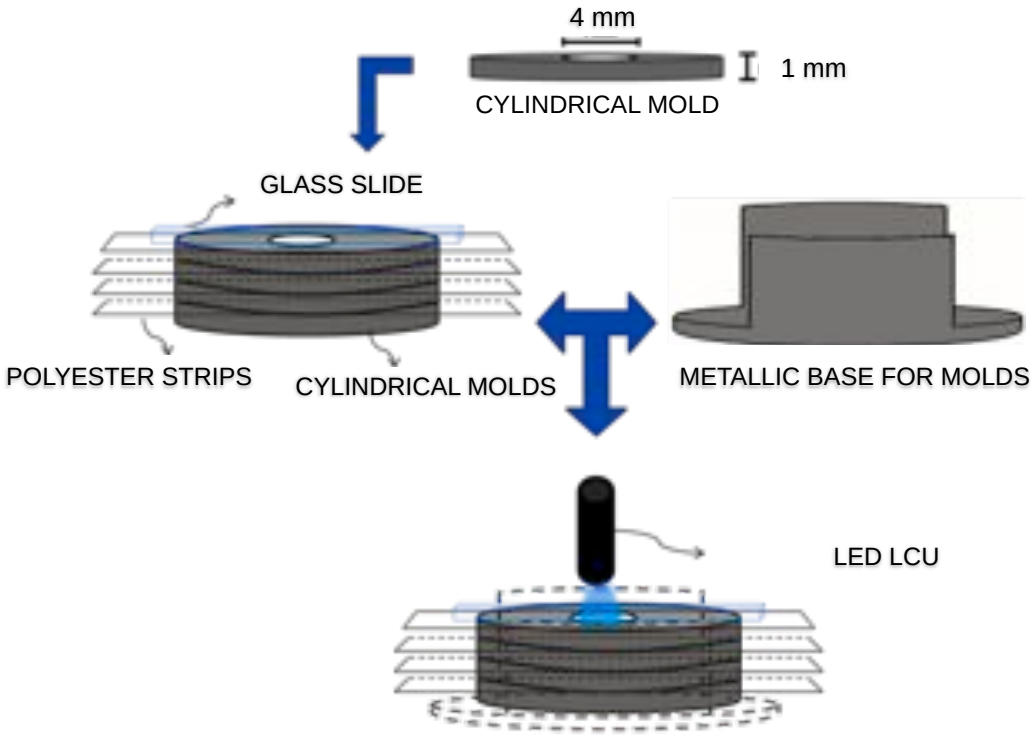


Figure 1 - Schematic representation of sample preparation.

Capítulo 3

3.3 Capítulo 3[*] [**] [***]

MARGINAL ADAPTATION IN CERVICAL DENTIN AND ENAMEL AND POLYMERIZATION SHRINKAGE OF A COMPOSITE BASED ON SDR TECHNOLOGY

Fernanda Ferreira Jassé[a*], DMD; Natália Fernandes Pollo[a]; Anna Karina Figueiredo Costa[b], DMD; Alexandre Luiz Souto Borges[b], DMD, PhD; Edson Alves de Campos[a], DMD, PhD; Dorien Lefever[c], DMD, PhD; Ivo Krejci[c], Prof. Dr. med. Dent.; Stefano Ardu[c], Dr. Med. Dent., PhD.

[a] Department of Restorative Dentistry, Araraquara School of Dentistry, UNESP Univ Estadual Paulista, Araraquara, Brazil

[b] Department of Dental Materials and Prosthesis, São José dos Campos School of Dentistry, UNESP Univ Estadual Paulista, São José dos Campos, Brazil

[c] Division of Cariology & Endodontology, Dental School, University of Geneva, Geneva, Switzerland

[*]Corresponding author: Tel +55 16 33016388

Address: Rua Humaitá, 1680 City: Araraquara – SP

E-mail: fernandajasse@hotmail.com

[*] De acordo com as normas estabelecidas pelo periódico American Journal of Dentistry.
[**] Metodologia detalhada no Apêndice 6.1.
[***] Metodologia aprovada pelo Comitê de Ética em Pesquisa conforme certificado exposto no Anexo 7.1.

MARGINAL ADAPTATION IN CERVICAL DENTIN AND ENAMEL AND POLYMERIZATION SHRINKAGE OF A COMPOSITE BASED ON SDR™ TECHNOLOGY

Abstract

PURPOSE: The aim of this study was to evaluate marginal adaptation in cervical dentin and enamel of Class II MOD restorations, before and after thermo-mechanical loading, using a composite based on SDR™ technology. Volumetric polymerization shrinkage was also assessed.

METHODS: For marginal adaptation assessment, twenty-four Class II MOD cavities with cervical margins extending 1.0mm below (distal) and 1.0mm beyond (mesial) the cement-enamel junction were prepared in extracted human molars. Teeth were filled as follows: group A – bulk-filled with only SureFil® SDR™ *flow* (first increment 4mm-thick + second increment 2mm-thick); group B - bulk-filled with SureFil® SDR™ *flow* as a base (first increment 4mm-thick) and covered with the conventional nano-hybrid composite EsthetX™ HD (second increment 2mm-thick); group C – incrementaly filled with only EsthetX™ HD. Marginal adaptation was evaluated with scanning electron microscope before and after thermo-mechanical loading (240,000 loading cycles and simultaneous 600 thermal cycles). For volumetric polymerization shrinkage evaluation, a semi-sphere mold was filled with the tested composites and then it was placed in AccuVol™ device to obtain the percent volumetric shrinkage calculated after light curing.

RESULTS: After statistical analysis, it was noted that both before and after loading, results of continuous margins in cervical dentin were superior ($p < 0.05$) for groups A and B compared to group C. While in cervical enamel group B presented superior values than group C, and group A presented intermediary results between B and C.

Concerning polymerization shrinkage, SureFil® SDR™ *flow* showed greater percentage of shrinkage compared to EsthetX™ HD ($p<0.05$).

CLINICAL SIGNIFICANCE

The development of low shrinkage stress resins is an immeasurable advance in the field of posterior teeth restorations, since the reduction of such stresses may avoid a variety of clinical problems. So it is important to evaluate the properties of new resin-based materials in order to find the best way to restore posterior teeth.

Introduction

The decrease of polymerization shrinkage stress has been the main objective of researches related to new resin-based composite (RBC) materials development. According to Kleverlaan & Feilzer,[1] volumetric shrinkage varies from 1%-3% for packable RBCs and up to 6% for flowable RBCs when polymerization of dimethacrylate-based composites takes place.[1] Shrinkage stresses are highly dependent on a combination of material properties and the cavity preparation design.[2] These stresses may affect marginal adaptation through gap formation, both internally and at the cavo-surface margins, thus generating clinical problems such as microleakage, marginal staining, recurrent caries, post-operative sensitivity, and even irritation of pulp tissue.[2,3] Several strategies to reduce shrinkage stress have been proposed in the literature. Among them, it can be found incremental layering techniques,[4] varying light-curing protocol,[5] applying a base with low-modulus resin to absorb shrinkage stress, using of ceramic inserts, and C-factor modification.[5] Unfortunally, these solutions bring with them some disadvantages such as loss of clinical time and technical sensitivity.[6]

Despite the efforts of dental materials companies to create a RBC material with low shrinkage allowing perfect marginal seal, this goal has not been achieved yet. One of the latest developments in this area was the “bulk-fill” flowable resins introduction as a result of ongoing efforts to simplify clinical restorative treatment of deep and high C-factor cavities, and dentin replacement. SureFil® SDR™ flow, as it was introduced in the American market, is recommended by its manufacturer to be used in a 4 mm-thickness increment due to its reduced polymerization stress, which needs to be veneered with 2 mm of a conventional resin composite.[7]

The acronym SDR™ means “stress decreasing resin” and materials based on this technology have the dynamics of their polymerization reaction modified by incorporating a photoactive group in a urethane-based methacrylate resin. When compared to conventional methacrylate-based resins, the modified UDMA resin presented a 60% to 70% decreasing in shrinkage stress in the unfilled resin.[8] The photoinitiator incorporated into the resin allows a slow curing rate. Jin *et al*.[8] related that this lower curing stress could be retained also in filled compositions.

Most studies that evaluated various properties of SDR™ resin have appointed satisfactory results for it when compared with conventional resins.[3,9-17] However, by the case of a still recent product on the market and the absence of long-term results, *in vitro* analyzes are valid when simulate the oral conditions of load and temperature on long-term. Thus, the aim of this study is to compare a low-shrinkage flowable resin-based composite and a conventional nano-hybrid RBC in terms of polymerization shrinkage and cervical marginal quality before and after thermo-mechanical challenges, using the same adhesive system. The tested null hypotheses were that: 1- there would be no significant difference in cervical marginal adaptation between the tested materials both before and after thermo-mechanical loading; 2- there would be no significant difference between the tested materials when measuring volumetric polymerization shrinkage.

Materials and methods

Marginal adaptation

Cavity preparation

Twenty-four extracted, intact, non-carious, unrestored human molars stored in 0.1% thymol solution were selected. Teeth were gotten from the human tooth bank of the Dental Medicine Section of the University of Geneva according to the requirements of the local ethical committee. After selection, teeth were debrided of any plaque or calculus deposits with a hand-scaler and examined to ensure that they were free of defects.

All teeth were mounted in the center of custom-made metallic holders and fixed with a light-curing composite. The root bases were then coated with a cold-polymerizing resin (Technovit 4071, Heraeus Kulzer; Wehrheim, Germany) to complete the stabilization of specimens. Standardized large class II MOD cavities were prepared with parallel walls and bevelled enamel margins. Proximal margins were prepared 1.0 mm beyond (mesial) and 1.0 mm below (distal) the cementum-enamel junction. The overall dimensions and depth of the cavities are illustrated in **Fig. 1**. Cavities were prepared using a high-speed hand-piece with copious air-water spray with 80-µm diamond burs (Komet, Lemgo, Germany) and 40-µm finishing diamond burs (Komet, Lemgo, Germany). The dimensions of the preparations were monitored using a caliper and a periodontal probe. All the teeth were randomly divided into 3 experimental groups (n = 8).

Restorative procedure

The materials used in this study are listed in **Table 1**.

All restorations were made by the same operator. A single-component self-etch dental adhesive (Xeno V™, Dentsply DeTrey, Konstanz, Germany) was applied according to manufacturer's instructions in all groups, and light-cured for 20 seconds using a LED curing light (Bluephase G2, Ivoclar Vivadent) with output irradiance of

approximately 1100 mW/cm^2. Throughout the experiment, the light output from the LCU was verified by a radiometer (Fieldmaster Power Meter, Coherentmodel, EUA). Then cavities were surrounded with a metal matrix band (Metafix, Kerr, Bioggio, Switzerland) and restored as follows (**Table 2**):

- Group A: composite resin SureFil® SDR™ *flow* was dispensed directly into the cavity preparation with the aid of a syringe under slow and constant pressure, in one 4 mm-bulk increment and light-cured for 20 s. The remainder of the cavity was filled with another bulk-increment of the same composite.
- Group B: restored according to manufacturer's orientation. Composite resin SureFil® SDR™ *flow* was dispensed directly into the cavity preparation with the aid of a syringe under slow and constant pressure, in one 4 mm-bulk-increment and light-cured for 20 s. The remainder of the cavity was filled with the nanohybrid composite EsthetX™ HD through incremental technique and also light-cured for 20 s.
- Group C: cavities were completely filled through incremental technique (oblique increments of approximately 2 mm thick) with the nano-hybrid composite EsthetX™ HD. Each increment was light-cured separately for 20 s.

Immediately after light-curing, occlusal margins were finished with fine diamond burs and proximal margins were finished and polished with flexible aluminum-oxide disks (SofLex Pop-On, 3M ESPE, St. Paul, USA).

Thermo-mechanical cycling and evaluation of marginal adaptation

After finishing procedures, epoxy resin (Epofix, Struers, Copenhagen, Denmark) replicas of each restoration were obtained by using a polyvinylsiloxane material (President light body, Coltène/Whaledent AG, Altstätten, Switzerland).

After storage for 24 hours in water at 37°C, thermo-mechanical loading was carried out in a computer-controlled chewing machine. All specimens were submitted to 240,000 mechanical loading cycles. Load was transferred to the center of the occlusal surface at max. 49 N and frequency of 1.7 Hz. Load were applied by using natural cusps taken from extracted human molars. Simultaneously, thermocycling was performed in flushing water with temperatures changing 600 times from 5°C to 50°C, with a dwell time of 2 min.

After chewing simulation cycles, a new set of epoxy resin replicas was obtained. All replicas were mounted on aluminium stubs, gold-coated and examined under a SEM (Digital SEM XL20, Philips, Eindhoven, Netherlands) at a standard 200x magnification. By means of computer-assisted quantitative margin analysis the marginal quality, before and after thermo-mechanical loading, was expressed as the percentage of continuous margin for the total margin length.[18]

Volumetric polymerization shrinkage

Volumetric Shrinkage (VS) of the tested composite resins was evaluated using a video-imaging device (AccuVol; Bisco Inc., Schaumburg, IL, USA), operating in the single-view mode at room temperature (23/24°C). Specimens (n=5) were made by placing 15 µl (± 1) of the tested resins into a semi-sphere mold that was moved to a polytetrafluoroethylene pedestal (4.2 mm diameter) in front of the camera (CCD) of the machine. The obtained image was captured and digitized by AcuVol ® Bisco software (MIOD Detection), and after the flow of the material for 2 minutes, the

perimeter of the samples was measured by a virtual dotted line. The measured size was stored in the program representing the initial sample volume. Then the composite was cured for 20 seconds (using LED LCU Bluephase G2, Ivoclar Vivadent) so that the curing tip was placed 5 mm from the top surface of the composite. After five minutes from light-curing, the volume shrinkage image was recorded. This waiting time is given in order to lower the temperature to room temperature.

Statistical Analysis

For marginal adaptation values, the dependent variables “restorative protocols” and “loading intervals” were statistically analyzed using Two-Way ANOVA and Sidak’s post hoc test at a 5% level of significance.

For polymerization shrinkage, statistical analysis was performed using ANOVA and Tukey’s post hoc test at a 5% level of significance.

Results

Marginal adaptation

The results of cervical dentin and enamel marginal adaptation, expressed as percentages of continuous margins (%CM) are shown in **Table 3**. When considering dentin margins length, the lowest values of %CM were observed in Group C (Esthet-X™ only), both before and after thermo-mechanical loading. The same occurred for enamel margins length. Non-significant differences were noted between groups A and B both for enamel or dentin margins. Most of the tested groups did not present significant differences when before and after thermo-mechanical loading values were

compared, both for enamel and dentin margins too. The only exception was observed for group A in cervical enamel.

Mean scores of cervical enamel integrity varied from 25,93 % (Group B) to 11,39 % (Group C) before thermo-mechanical loading and from 18,73% (Group B) to 0.0% (Group C) after thermo- mechanical loading. For cervical dentin, mean values varied from 65,81 % (Group A) to 5,76 % (Group C) and from 53,40% (Group A) to 0.0% (Group C) before and after thermo-mechanical loading, respectively.

Volumetric polymerization shrinkage

Table 4 presents a summary of the volumetric polymerization shrinkage of the composite resins investigated in this study. Esthet-X™ exhibited statistically significant ($p < 0.05$) lower shrinkage values in comparison to SDR™ resin.

Discussion

The results of the present study showed that the tested materials exhibited unsatisfactory cervical marginal adaptation both before and after thermo-mechanical loading, mostly in enamel. Besides the unsatisfactory results, groups that used the “bulk-fill” composite demonstrated statistically better cervical marginal adaptation than the group where only conventional composite was applied. Therefore, the first null hypothesis was rejected.

The worse results obtained in enamel were probably due to the use of one-bottle self-etching adhesive without prior application of phosphoric acid. As previously shown by other authors in the literature[15,19-22] we reaffirm the importance

of selective etching in enamel margins when self-etching adhesive systems are used in order to obtain enamel adhesion resistant to ageing.[3]

Considering the groups where SDR™ was used, the results obtained in dentine are comparable with those of Campos *et al*.[3], where a gold standard etch-and-rinse adhesive was applied. Concerning methodology, the mentioned study was performed in similar conditions to this study. Thus, the use of one-bottle self-etching adhesive Xeno V™ does not impaired the results for dentine substrate.

Clinical success of novel restorative materials needs long periods of use by general practitioners to be considered clinically reliable. Although *in vivo* tests remain the gold standard way to assess the performance of dental materials, *in vitro* tests are also valuable as a way to characterize new materials before clinical-long-term results are available in the literature[16,20,23,24]. In this study, cervical marginal integrity of the tested materials was evaluated before and after thermo-mechanical loading in order to simulate long-term fatigue in oral cavity.

Pecie *et al*.[25] affirmed through polymerization stress maps that cervical zone is the dental region of maximum stress accumulation, being the dentin-restoration interface the most problematic area for adhesion. Cervical marginal adaptation was assessed in this study considering the problems that clinicians find when need to restore this specific area in class II cavities. In contrast to the above-mentioned study, our research presented cervical enamel as the problematic area, probably due to the adhesive system selected. Even though the poor results observed for all tested groups, groups in which SDR™ was used showed significantly better marginal adaptation values both before and after thermo-mechanical loading. Our results lead us to conclude that SDR™ favors marginal adaptation due to flow in the proximal boxes.

Different than would be expected, the claimed low-shrinkage flowable composite SDR™ presented higher percentage of volumetric polymerization shrinkage than the conventional composite Esthet-X™ HD, so the second null hypothesis was also rejected. Burgess & Cakir[7] also have used Accuvol device to measure polymerization shrinkage of SDR™ and have obtained a value of 3.1% of shrinkage, somewhat lower than it was found in the present study. However, the authors pointed out that although the polymerization shrinkage, marginal stress may be reduced by increased flowing during its bulk filling. Another research is in agreement with our study, obtaining greater shrinkage values for flowable materials (including SDR™) in comparison to nano-hybrid material.[26] According to Rodriguez *et al*.[27] there exists a direct relationship between shrinkage and amount of organic matrix in the composite resin. Thus, the higher resin content of the flowable composite could explain the higher volumetric shrinkage observed. Therefore, even though SDR™ presents higher volumetric shrinkage compared to Esthet-X™ HD it can also be assumed that the slow rate of polymerization promoted by the new modulator can diminish the shrinkage stress. This assumption can be enhanced by the results obtained in a study where “bulk-fill” flowable bases (including SDR™) significantly reduced cuspal deflection in comparison with a conventional composite used through the oblique incremental filling technique.[12]

Several authors recommend the use of a flowable composite as a stress-absorbing liner or base believing that its relatively low modulus of elasticity and deformation ability can aid to decrease polymerization shrinkage stress.[2,28,29] However, conflicting results have been reported regarding the action of flowable composite materials as stress breakers or adaptation promoters.[30,31]

Although nominal values obtained for cervical dentin using only SDR™ had been higher, the manufacturer's recommendation to overlay the "bulk-fill" base material must be followed. A study that evaluated mechanical performance of "bulk-fill" composites showed that modulus of elasticity and hardness of SDR™ was considerably lower than conventional nanohybrid and microhybrid resins.[11] Thus, these features need to be compensated by the better mechanical properties of conventional composites.

Conclusions

Regarding the limitations of the present study, it was concluded that:

- When SDR™ was used, significant improvement could be found in cervical marginal adaptation compared with the conventional composite, both before and after thermo-mechanical loading.
- SDR™ presented higher percentage of volumetric polymerization shrinkage than the conventional composite, being necessary specific studies to clear if the new modulator incorporated to the flowable resin can diminish the shrinkage stress.
- The results of this study confirm that the new resin has characteristics comparable or superior to conventional resin regarding cervical marginal adaptation. However, only long-term clinical trials can confirm the clinical success of the new material.

Acknowledgements

The authors would acknowledge receipt of the bulk-fill SDR flowable resin from Dr. Walter R. Dias (DDS, MS), DENTSPLY DeTrey GmbH, Konstanz, Germany. This work was supported by a grant from CAPES.

References

1. Kleverlaan CJ, Feilzer AJ. Polymerization shrinkage and contraction stress of dental resin composites. *Dent Mater* 2005; 21: 1150-1157.
2. Davidson CL, Feilzer AJ. Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives. *J Dent* 1997; 25 :435–40.
3. Campos EA, Ardu S, Lefever D, Jassé FF, Bortolotto T, Krejci I. Marginal adaptation of class II cavities restored with bulk-fill composites. *J Dent* 2014; May; 42 (5): 575-81.
4. Lutz F, Krejci I, Oldenburg TR. Elimination of polymerization stress at the margins of posterior composite resin restorations: a new restorative technique. *Quintessence Int* 1986; 17: 777-784.
5. Cunha LG, Sinhoreti MA, Consani S, Sobrinho LC. Effect of different photoactivation methods on the polymerization depth of a light-activated composite. *Oper Dent* 2003; 28: 155-159.
6. Gregor L, Bortolotto T, Feilzer AJ, Krejci I. Shrinkage kinetics of a methacrylate- and a silorane-based resin composite: effect on marginal integrity. *J Adhes Dent* 2013 Jun; 15 (3): 245-50.
7. Burgess J, Cakir D. Comparative properties of low-shrinkage composite resins. *Compend Contin Educ Dent* 2010 May; 31 Spec No 2:10-5.

8. Jin X, Bertrand S, Hammesfahr PD. New radically polymerizable resins with remarkably low curing stress. *J Dent Res* 2009; 88 (Spec Iss A): 1651.
9. El-Damanhoury H, Platt J. Polymerization Shrinkage Stress Kinetics and Related Properties of Bulk-fill Resin Composites. *Oper Dent* 2013 Jul 18. [Epub ahead of print]
10. Furness A, Tadros MY, Looney SW, Rueggeberg FA. Effect of bulk/incremental fill on internal gap formation of bulk-fill composites. *J Dent* 2014 Apr; 42 (4): 439-49.
11. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. *Oper Dent* 2013 Nov-Dec; 38 (6): 618-25.
12. Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, Fleming GJ. Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials. *J Dent* 2012 Jun; 40 (6): 500-5.
13. Nazari A, Sadr A, Saghiri MA, Campillo-Funollet M, Hamba H, Shimada Y, Tagami J, Sumi Y. Non-destructive characterization of voids in six flowable composites using swept-source optical coherence tomography. *Dent Mater* 2013 Mar; 29 (3): 278-86.
14. Nazari A, Sadr A, Shimada Y, Tagami J, Sumi Y. 3D assessment of void and gap formation in flowable resin composites using optical coherence tomography. *J Adhes Dent* 2013 Jun; 15 (3): 237-43.
15. Roggendorf MJ, Krämer N, Appelt A, Naumann M, Frankenberger R. Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. *J Dent* 2011 Oct; 39 (10): 643-7.

16. Salerno M, Derchi G, Thorat S, Ceseracciu L, Ruffilli R, Barone AC. Surface morphology and mechanical properties of new-generation flowable resin composites for dental restoration. *Dent Mater* 2011 Dec; 27 (12): 1221-8.
17. Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, Van Meerbeek B. Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity-bottom dentin. *Dent Mater* 2013 Mar; 29 (3): 269-77.
18. Krejci I, Duc O, Dietschi D, de Campos E. Marginal adaptation, retention and fracture resistance of adhesive composite restorations on devital teeth with and without posts. *Oper Dent* 2003 Mar-Apr; 28 (2): 127-35.
19. Van Meerbeek B, De Munck J, Yoshida Y, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 2003; 28: 215–35.
20. Peumans M, De Munck J, Van Landuyt KL, Poitevin A, Lambrechts P, Van Meerbeek B. A 13-year clinical evaluation of two three-step etch-and-rinse adhesives in non-carious class-V lesions. *Clin Oral Investig* 2012 Feb; 16 (1): 129-37.
21. Frankenberger R, Tay FR. Self-etch vs etch-and-rinse adhesives: effect of thermo-mechanical fatigue loading on marginal quality of bonded resin composite restorations. *Dent Mater* 2005 May; 21 (5): 397-412.
22. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. *Dent Mater* 2011 Jan; 27 (1): 17-28.
23. Garcia-Godoy F, Kramer N, Feilzer AJ, Frankenberger R. Long-term degradation of enamel and dentin bonds: 6-year results in vitro vs. in vivo. *Dent Mater* 2010; 26: 1113–8.

24. Kramer N, Garcia-Godoy F, Reinelt C, Feilzer AJ, Frankenberger R. Nanohybrid vs. fine hybrid composite in extended Class II cavities after six years. *Dent Mater* 2011; 27: 455–64.
25. Pecie R, Onisor I, Krejci I, Bortolotto T. Marginal adaptation of direct class II composite restorations with different cavity liners. *Oper Dent* 2013 Nov-Dec; 38 (6): E210-20.
26. Garcia D, Yaman P, Dennison J, Neiva G. Polymerization Shrinkage and Depth of Cure of Bulk Fill Flowable Composite Resins. *Oper Dent* 2013 Dec 4. [Epub ahead of print]
27. Rodriguez VI, Abate PF, Macchi RL. Immediate polymerization shrinkage in light cured restorative resins. *Acta Odontol Latinoam* 2006 19 (1): 3-7.
28. Braga RR, Ferracane JL. Alternatives in polymerization contraction stress management. *Crit Rev Oral Biol Med* 2004; 15: 176–84.
29. Ferracane JL. Placing dental composites – a stressful experience. *Oper Dent* 2008; 33 (3): 247–57.
30. Cara RR, Fleming GJP, Palin WM, Walmsley AD, Burke FJT. Cuspal deflection and microleakage in premolar teeth restored with resin-based composites with and without an intermediary flowable layer. *J Dent* 2007; 35: 482–9.
31. Oliveira LCA, Duarte Jr S, Araujo CA, Abrahão A. Effect of low-elastic modulus liner and base as stress-absorbing layer in composite resin restorations. *Dent Mater* 2010; 26: e159–69.

TABLE CAPTIONS

Table 1: Summary of the materials used in this study.

Table 2: Experimental groups divided according to restorative technique.

Table 3: Cervical marginal adaptation in enamel and dentin expressed as percentages of continuous margins [(mean%) (± standard deviation)], using different restoring protocols, before and after thermo-mechanical loading.

Table 4: Mean values (%) (± standard deviation) of volumetric polymerization shrinkage of the tested composites (ANOVA and Tukey test, $p<0,05$).

FIGURE CAPTIONS

Figure 1. Dimensions of MOD preparations.

TABLES

Table 1. Summary of the materials used in this study.

Brand name	Type	Composition	Filler Wt% / Vol%	Manufacturer
SureFil™ SDR™ *flow*	Flowable bulk fill composite resin	Ba-Al-F-B silicate glass, Sr-A-F silicate glass, Modified UDMA, EBPADMA, TEGDMA. Camphorquinone photo-initiator (CQ), butylated hydroxytoluene (BHT), UV stabilizer, Titanium dioxide, Iron oxide pigments.	68/44 %	Dentsply DeTrey, Konstanz, Germany
Esthet-X™ HD	Conventional nanohybrid composite resin	Bis-GMA, Bis-EMA, TEGDMA, Camphorquinone photo-initiator (CQ), UV stabilizer, pigments. Combination of particulate fluoro-barium-borosilicate glass with a mean particle size below 1µm and silica nanoparticles of 0,04 µm.	77/60 %	Dentsply Caulk, Milford, USA
Xeno™ V	One-component self-etching adhesive	Bifunctional acrylic amides, Acrylamido alkylsulfonic acid, "inverse" functionalized phosphoric acid ester, Acrylic acid, Camphorquinone, Coinitiator, Butylated benzenediol, Water, Tert-butanol.	----	Dentsply DeTrey, Konstanz, Germany

UDMA: urethane dimethacrylate; EBPADMA: ethoxylated Bisphenol A dimethacrylate; TEGDMA: ttriethyleneglycol dimethacrylate; Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA: Bisphenol A dimethacrylate.

Table 2. Experimental groups divided according to restorative technique.

Group	First increment (4mm thick)	Second increment (2mm thick)
A	SureFil® SDR™ *flow*	SureFil® SDR™ *flow*
B	SureFil® SDR™ *flow*	EsthetX™ HD
C	EsthetX™ HD	EsthetX™ HD

Table 3. Cervical marginal adaptation in enamel and dentin expressed as percentages of continuous margins [(mean%) (± standard deviation)], using different restoring protocols, before and after thermo-mechanical loading.

GROUP	CERVICAL ENAMEL		CERVICAL DENTIN	
	Before loading	After loading	Before loading	After loading
A	21,71 (±11,56) ABa	7,86 (±4,70) ABb	65,81 (±15,73) Aa	53,40 (±22,72) Aa
B	25,93 (±15,22) Aa	18,73 (±11,54) Aa	52,27 (±23,30) Aa	39,28 (±16,71) Aa
C	11,39 (±4,87) Ba	0 (±0) Ba	5,76 (±5,60) Ba	0 (±0) Ba

*Means with same capital letters in columns are not statistically different at p = 0.05. Means with same lower case letters in lines mean p > 0.05.

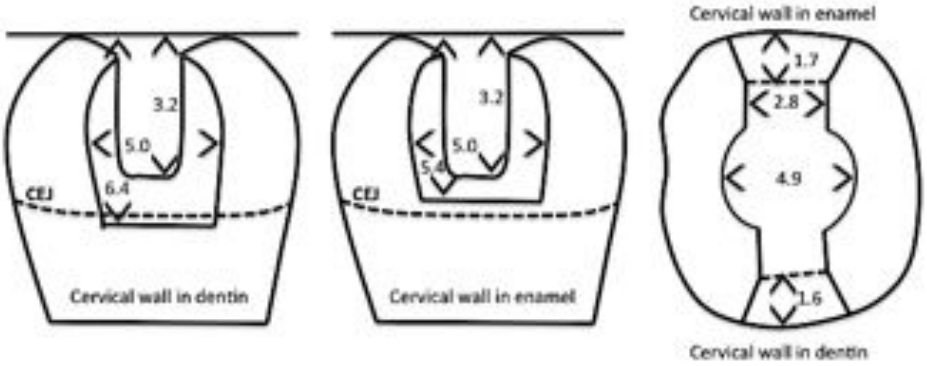
Table 4. Mean values (%) (± standard deviation) of volumetric polymerization shrinkage of the tested composites (ANOVA and Tukey test, p<0,05).

	Esthet-X	SDR
Mean	2,710 ^{A}	4,942 ^{B}
SD	(±0,287)	(±0,660)

*Different superscript letters within line mean p < 0.05.

FIGURES

Figure 1. Dimensions of MOD preparations.



Considerações Finais

4 Considerações finais

A resina composta SDR™ *flow* passou a ser recentemente comercializada no Brasil como um material restaurador de uso direto para dentes posteriores. Poucos estudos estão disponíveis na literatura avaliando suas propriedades, especialmente no que diz respeito à resistência à fratura. No primeiro estudo deste trabalho, observou-se uma tendência dos dentes com cavidades MOD restaurados com a nova resina apresentarem resistência à fratura melhorada em relação aos dentes restaurados com a resina convencional e similar aos dentes hígidos.

A dentina é a base sólida necessária para a restauração do dente. A sua resistência estrutural depende da qualidade e integridade da sua forma anatômica, de modo que o problema fundamental é a quantidade reduzida de dentina sadia remanescente que mantém e apoia a restauração (Assif, Gorfil[2], 1994). Por conseguinte, a preparação extensa de uma cavidade MOD pode provocar fratura de cúspides se o dente não é restaurado (Gelb et al.[18], 1986; Pilo et al.[33], 1998). Os resultados do presente estudo mostraram que a recuperação de um dente com cavidade ampla é importante para se alcançar um aumento da sua resistência à fratura. Portanto, o reforço da cavidade com um material restaurador adequado é necessário para dar suporte à estrutura dentária remanescente. Em um estudo realizado por Jagadish, Yogesh[23] (1990), os autores sugeriram que as resinas compostas quando utilizadas em dentes posteriores apresentam um grande potencial como material de reforço de cúspide.

Apesar do grupo restaurado apenas com a resina SDR™ *flow* ter apresentado resistência superior aos demais grupos de dentes restaurados, não é clinicamente possível sua utilização na cavidade inteira, isto é, sem o recobrimento oclusal de 2

mm com resina composta convencional. Isto ocorre pelo fato de que a resina SDR™ *flow*, por ser de consistência fluida, não permite escultura e acabamento adequados da superfície oclusal e cristas marginais. Além do aspecto funcional, também deve ser considerado o fator estético, já que a nova resina é comercializada em uma única cor, a qual é moderadamente translúcida. Portanto, utilizando-se a resina SDR™ *flow* de acordo com as recomendações do fabricante, ou seja, como material de base recoberto por uma resina convencional, é possível se obter resultados de resistência à fratura adequados e semelhantes aos obtidos para os dentes hígidos, de acordo com os resultados deste estudo. Utilizando-se o material desta forma, torna-se possível atender às necessidades restauradoras de um elemento dental com cavidade ampla tanto no aspecto funcional quanto estético.

Os conceitos de inserção incremental têm sido descritos como obrigatórios quando se trabalha com resinas compostas (Sakaguchi et al.[37], 1992; Flury et al.[14], 2012). Entretanto, contrariando estes princípios, o uso das resinas “bulk-fill” em incrementos mais espessos que os convencionais tem sido defendido, permitindo uma técnica restauradora mais rápida e, consequentemente, mais segura por reduzir as chances de contaminação pela saliva, já que o tempo de isolamento torna-se mais curto, bem como por favorecer a inserção da resina nas caixas proximais, especialmente para cavidades classe II profundas em que a inserção incremental fica dificultada. No segundo estudo foi analisado o grau de conversão das resinas em diferentes profundidades, com o intuito de se obter informações acerca da extensão de polimerização da resina SDR™ no quarto e último milímetro do incremento recomendado pelo fabricante, pois sabe-se que uma polimerização insuficiente da resina é capaz de alterar suas propriedades mecânicas, podendo contribuir para o surgimento de cáries secundárias e danos à polpa dental

(Rueggeberg et al.[36], 1990). De acordo com os valores de grau de conversão observados neste estudo, a resina SDR™ alcança os 4mm de polimerização satisfatória com 20 segundos de foto-ativação para um único incremento. Desta forma, considerando-se a restauração de uma cavidade classe II MOD profunda é possível uma economia de tempo clínico de aproximadamente 50% do tempo que seria dispendido para realização da técnica incremental convencional no preenchimento da cavidade.

Os resultados do terceiro estudo mostraram adaptação marginal cervical insatisfatória, tanto antes quanto após a ciclagem termo-mecânica, principalmente quando o substrato avaliado foi o esmalte. Porém, apesar dos resultados insatisfatórios, os grupos nos quais foi usada a resina SDR™, apresentaram adaptação marginal cervical consideravelmente superior ao grupo em que apenas a resina convencional foi usada. Acredita-se que os péssimos resultados obtidos em esmalte se devam ao uso do sistema adesivo auto-condicionante de frasco único sem a aplicação prévia de ácido fosfórico. Assim, como já demonstrado por outros autores (Roggendorf et al.[35], 2011; Van Meerbeek et al.[41], 2003; Peumans et al.[32], 2012; Frankenberger, Tay[15], 2005; Van Meerbeek et al.[42], 2011), reafirma-se aqui a importância do condicionamento ácido seletivo nas margens de esmalte quando sistemas adesivos auto-condicionantes são usados, para que se obtenha adesão em esmalte e dentina resistente ao envelhecimento (Campos et al.[6], 2014).

Diferentemente do esperado, a resina de reduzido *stress* de contração, apresentou maiores percentuais de contração de polimerização volumétrica quando comparada à resina convencional, dado este confirmado por estudo prévio realizado (Burgess, Cakir[4], 2010), no qual os autores ressaltam que apesar da contração de polimerização o *stress* marginal pode ser reduzido pela fluidez da resina. Segundo

Rodriguez et al.[34] (2006), existe relação direta entre contração de polimerização e percentual de matriz orgânica nas resinas compostas. Desta forma, o elevado teor orgânico do compósito *flow*, poderia explicar a contração volumétrica observada. Entretanto, mesmo que a SDR™ apresente maior contração volumétrica em relação à resina convencional, também pode-se supor que a polimerização mais lenta promovida pelo novo modulador pode ser capaz de diminuir o *stress* de contração. Esta suposição pode ser fortalecida pelos resultados obtidos em um estudo em que bases “bulk-fill” fluidas (incluindo a SDR™) reduziram consideravelmente a deflexão de cúspides em comparação à resina convencional inserida por meio da técnica incremental oblíqua (Moorthy et al.[30], 2012).

De maneira geral, os resultados obtidos neste trabalho mostraram que a nova resina fluida permite a reconstrução do elemento dental com propriedades vantajosas em relação à resina convencional, funcionando bem como dentina artificial. Entretanto, apesar das análises realizadas nos três estudos aqui apresentados caracterizarem propriedades dos materiais que estão diretamente ligadas à qualidade e longevidade das restaurações com eles realizadas, deve-se considerar que esta pesquisa foi realizado em condições in vitro. Idealmente, estudos clínicos devem ser desenvolvidos de modo que se evidencie melhor o comportamento das restaurações em situações clínicas reais.

Referências

5 Referências[*]

1. Amirouche-Korichi A, Mouzali M, Watts DC. Effects of monomer ratios and highly radiopaque fillers on degree of conversion and shrinkage-strain of dental resin composites. Dent Mater. 2009; 25(11): 1411-8. doi: 10.1016/j.dental.2009.06.009.
2. Assif D, Gorfil C. Biomechanical considerations in restoring endodontically treated teeth. J Prosthet Dent. 1994; 71(6): 565-7.
3. Bowen RL, inventor. Dental filling material comprising vinyl silane treated fused silica and a binder consisting of the reaction product of bisphenol and glycidyl acrylate. United States patent US 3066112A. 1962 Nov 27.
4. Burgess J, Cakir D. Comparative properties of low-shrinkage composite resins. Compend Contin Educ Dent. 2010; 31 Spec No 2:10-5.
5. Campos EA, Andrade MF, Porto-Neto ST, Campos LA, Saad JRC, Deliberador TM, Oliveira-Júnior OB. Cuspal movement related to different bonding techniques using etch-and-rinse and self-etch adhesive systems. Eur J Dent. 2009; 3(3): 213-8.
6. Campos EA, Ardu S, Lefever D, Jassé FF, Bortolotto T, Krejci I. Marginal adaptation of class II cavities restored with bulk-fill composites. J Dent. 2014; 42(5): 575-81.
7. Chung KH, Greener EH. Correlation between degree of conversion, filler concentration and mechanical properties of posterior composite resins. J Oral Rehabil. 1990; 17(5): 487-94.

[*] De acordo com o manual da FOAr/UNESP, adaptadas das normas Vancouver. Disponível no site: http://www.foar.unesp.br/#!/biblioteca/manual

8. Dentsply Caulk. Surefil SDR flow – Product Brochure [Internet]. Milford: DENSTPLY International; 2010 [acesso em 2012 Jul 27]. Disponível em: http://www.surefilsdrflow.com/sites/default/files/SureFil_Brochure.pdf.
9. Dietschi D, Monasevic M, Krejci I, Davidson C. Marginal and internal adaptation of class II restorations after immediate or delayed composite placement. J Dent. 2002; 30(5-6): 259-69.
10. Dietschi D, Olsburgh S, Krejci I, Davidson C. In vitro evaluation of marginal and internal adaptation after occlusal stressing of indirect class II composite restorations with different resinous bases. Eur J Oral Sci. 2003; 111(1): 73-80.
11. Donly KJ, Wild TW, Bowen RL, Jensen ME. An in vitro investigation of the effects of glass inserts on the effective composite resin polymerization shrinkage. J Dent Res. 1989; 68(8): 1234-7.
12. El-Damanhoury H, Platt J. Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. Oper Dent. 2014; 39(4): 374-82. Epub 2013 Jul 18.
13. Feilzer AJ, De Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. J Dent Res. 1987; 66(11): 1636-9.
14. Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? Dent Mater. 2012; 28(5): 521-8.
15. Frankenberger R, Tay FR. Self-etch vs etch-and-rinse adhesives: effect of thermo-mechanical fatigue loading on marginal quality of bonded resin composite restorations. Dent Mater. 2005; 21(5): 397-412.

16. Friedl KH, Schmalz G, Hiller KA, Mortazavi F. Marginal adaptation of composite restorations versus hybrid ionomer/composite sandwich restorations. Oper Dent. 1997; 22(1): 21-9.
17. Furness A, Tadros MY, Looney SW, Rueggeberg FA. Effect of bulk/incremental fill on internal gap formation of bulk-fill composites. J Dent. 2014; 42(4): 439-49.
18. Gelb MN, Barouch E, Simonsen RJ. Resistance to cusp fracture in Class II prepared and restored premolars. J Prosthet Dent. 1986; 55(2): 184-5.
19. Hirata R, Higashi C, Masotti A. Simplificando o uso de resinas compostas em dentes posteriores. R Dental Press Estét. 2004; 1(1): 18-34.
20. Hitz T, Ozcan M, Gohring TN. Marginal adaptation and fracture resistance of root-canal treated mandibular molars with intracoronal restorations: effect of thermocycling and mechanical loading. J Adhes Dent. 2010; 12(4): 279-86.
21. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. Oper Dent. 2013; 38(6): 618-25.
22. Imazato S, Tarumi H, Kobayashi K, Hiraguri H, Oda K, Tsuchitani Y. Relationship between the degree of conversion and internal discoloration of light-activated composite. Dent Mater J. 1995; 14(1): 23-30.
23. Jagadish S, Yogesh BG. Fracture resistance of teeth with Class II silver amalgam, posterior composite, and glass cermet restorations. Oper Dent. 1990; 15(2): 42-7.
24. Jassé FF, Braga Borges CH, Tonetto MR, Rastelli ANS, Bagnato VS, Campos EA, Santos RSS, Bandeca MC, Saad JRC. Long-term surface hardness and monomer conversion of a nanoflled and a microhybrid composite resin. J Contemp Dent Pract. 2013; 14(5): 876-82.

25. Koltisko B, Dai Q, Jin X, Bertrand S, Lu H. The polymerization stress of flowable composites. J Dent Res. 2010; 89(Spec Iss B): abstract 321.
26. Krejci I, Kuster M, Lutz F. Influence of dentinal fluid and stress on marginal adaptation of resin composites. J Dent Res. 1993; 72(2): 490-4.
27. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the "depth of cure" of dimethacrylate-based dental composites. Dent Mater. 2012; 28(5): 512-20. doi: 10.1016/j.dental.2011.12.004.
28. Lindberg A, Peutzfeldt A, van Dijken JW. Effect of power density of curing unit, exposure duration, and light guide distance on composite depth of cure. Clin Oral Investig. 2005; 9(2): 71-6.
29. Lutz E, Krejci I, Oldenburg TR. Elimination of polymerization stresses at the margins of posterior composite resin restorations: a new restorative technique. Quintessence Int. 1986; 17(12): 777-84.
30. Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, Fleming GJ. Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials. J Dent. 2012; 40(6): 500-5.
31. Nazari A, Sadr A, Saghiri MA, Campillo-Funollet M, Hamba H, Shimada Y, Tagami J, Sumi Y. Non-destructive characterization of voids in six flowable composites using swept-source optical coherence tomography. Dent Mater. 2013; 29(3): 278-86.
32. Peumans M, De Munck J, Van Landuyt KL, Poitevin A, Lambrechts P, Van Meerbeek B. A 13-year clinical evaluation of two three-step etch-and-rinse adhesives in non-carious class-V lesions. Clin Oral Investig. 2012; 16(1): 129-37.
33. Pilo R, Brosh T, Chweidan H. Cusp reinforcement by bonding of amalgam restorations. J Dent. 1998; 26(5-6): 467-72.

34. Rodriguez VI, Abate PF, Macchi RL. Immediate polymerization shrinkage in light cured restorative resins. Acta Odontol Latinoam. 2006; 19(1): 3-7.
35. Roggendorf MJ, Krämer N, Appelt A, Naumann M, Frankenberger R. Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. J Dent. 2011; 39(10): 643-7.
36. Rueggeberg FA, Hashinger DT, Fairhurst CW. Calibration of FTIR conversion analysis of contemporary dental resin composites. Dent Mater. 1990; 6(4): 241-9.
37. Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and polymerization of composite restorative materials. J Dent. 1992; 20(3): 183–8.
38. Salerno M, Derchi G, Thorat S, Ceseracciu L, Ruffilli R, Barone AC. Surface morphology and mechanical properties of new-generation flowable resin composites for dental restoration. Dent Mater. 2011; 27(12): 1221-8.
39. Stavridakis MM, Kakaboura AI, Ardu S, Krejci, I. Marginal and internal adaptation of bulk-filled class I and cuspal coverage direct resin composite restorations. Oper Dent. 2007; 32(5): 515-23.
40. Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, Van Meerbeek B. Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity-bottom dentin. Dent Mater. 2013; 29(3): 269-77.
41. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent. 2003; 28(3): 215-35.
42. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. Dent Mater. 2011; 27(1): 17-28.

Apêndice

6 Apêndice

6.1 Metodologia

As resinas compostas testadas estão especificadas na Tabela A1.

Tabela A1 - Informacões dos materiais testados

NOME COMERCIAL	TIPO	COMPOSIÇÃO QUÍMICA	FABRICANTE	COR
SureFil® SDR™ *flow*	"bulk fill" fluida	Vidro de bário-alumínio-fluor-borosilicato, Vidro de estrôncio-alumínio-fluor-silicato, Resina modificada de dimetacrilato de uretano, Etoxilato bisfenol A dimetacrilato (EBPADMA), Trietilenoglicol dimetacrilato (TEGDMA), Foto-iniciador canforquinona (CQ), Hidroxitolueno butilado (BHT), Estabilizador UV, Dióxido de titânio, Pigmentos de óxido de ferro.	Dentsply	---
Esthet-X® HD	Nano-híbrida convencional	Bis-GMA, Bis-EMA, Trietilenglicol-dimetacrilato, foto-iniciador canforquinona, estabilizador UV, pigmentos. Combinação de partículas de Vidro de bário-fluor-borosilicato com tamanho médio de partículas abaixo de 1µm e nanopartículas de silica de 0,04 µm.	Dentsply	A2

Seleção e preparo dos dentes para os ensaios de resistência à fratura (Capítulo 1) e adaptação marginal (Capítulo 3)

Foram selecionados 64 molares humanos hígidos. Os dentes foram submetidos à limpeza e exame visual com lupa, certificando-se da ausência de trincas, cáries ou quaisquer alterações que pudessem interferir nos resultados. As dimensões V-L (9,0 mm) e M-D (7,0 mm) dos dentes foram padronizadas por meio de um paquímetro digital, aceitando-se variações de até 1,0 mm em ambas as dimensões. A seguir, foi efetuada a desinfecção dos dentes com solução de timol a 0,1%. Posteriormente foram armazenados em água destilada a 4˚C, até o momento de prepará-los para a obtenção dos espécimes.

Para possibilitar a confecção dos espécimes para o ensaio de resistência à fratura, 40 dentes foram incluídos pela raiz, até aproximadamente 2 mm da junção cemento/esmalte, em resina epóxi no interior de uma cilindro plástico com diâmetro e altura de 20 mm, de modo que a superfície oclusal dos dentes ficasse paralela à base do cilindro, uma vez que, na realização dos testes de resistência à fratura, a força compressiva deveria ser aplicada paralelamente ao longo eixo dos dentes.

Em seguida, os 40 dentes foram divididos aleatoriamente com a finalidade de se obter cinco grupos experimentais (n=8) (Tabela A2).

Tabela A2 - Grupos experimentais do ensaio de resistência à fratura

GRUPOS	CONDIÇÃO DOS DENTES
1F	Hígidos
2F	Preparados sem restauração
3F	Restaurados com resina SDR™
4F	Restaurados com resina SDR™ + resina nano-híbrida
5F	Restaurados com resina nano-híbrida

Para o ensaio de mastigação, 24 dentes foram selecionados e o comprimento radicular de cada um foi ajustado de modo a permitir que se encaixassem na câmara de teste do equipamento de carga mecânica (Department of Cariology, Endodontics and Pedodontics, University of Geneva). Os espécimes foram então devidamente posicionados e fixados com resina foto-ativada em um suporte metálico (Figura A1), e a base das raízes recoberta com resina acrílica de auto-polimerização para completar a estabilização dos espécimes (Figura A2). Na

seqüência, eles foram distribuídos aleatoriamente em três grupos experimentais (n=8), os quais estão descritos na Tabela A3.

Figura A1 - Suporte metálico para posicionamento dos dentes.

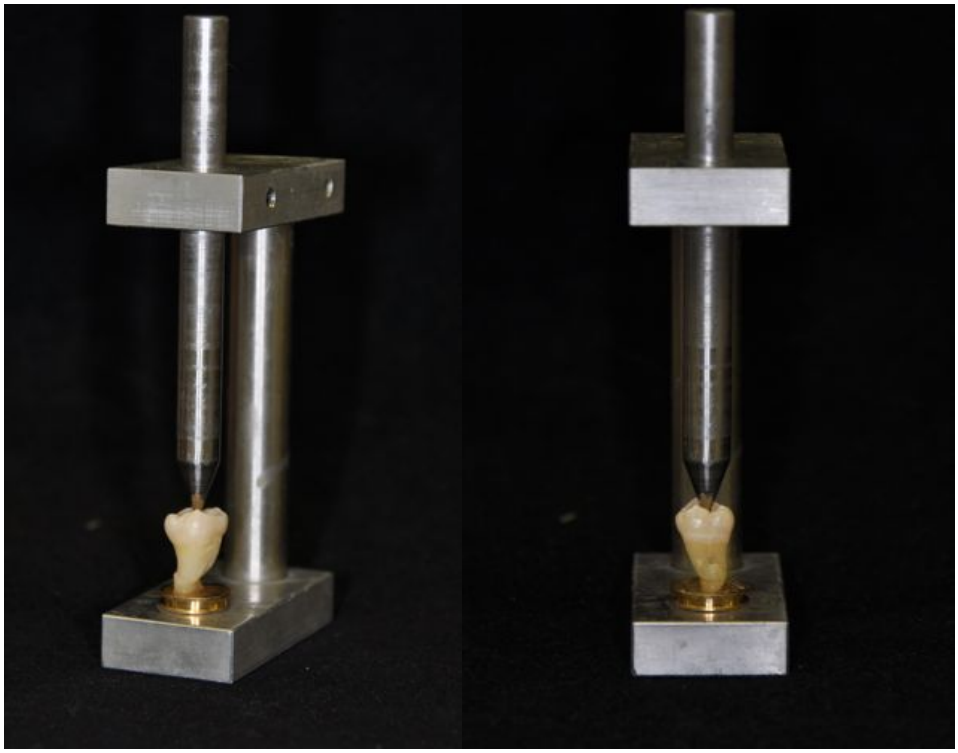


Figura A2 – Matriz plástica sendo preenchida com resina acrílica.

Tabela A3 - Grupos experimentais do ensaio de mastigação/adaptação marginal.

GRUPOS	CONDIÇÃO DOS DENTES
1A	Restaurados com resina SDR™
2A	Restaurados com resina SDR™ + resina nano-híbrida
3A	Restaurados com resina nano-híbrida

Os preparos cavitários foram confeccionados de maneira padronizada. Foram preparadas cavidades de classe II MOD com paredes paralelas e margens de esmalte biseladas, com margens proximais localizadas 1,0 mm além (mesial) e aquém (distal) da junção cemento-esmalte. A Figura A3 ilustra as dimensões do preparo. As cavidades foram preparadas utilizando-se pontas diamantadas cilíndricas de extremidade plana de granulação grossa, sob spray de água abundante, e tiveram acabamento realizado com pontas de granulação fina com o mesmo formato. As dimensões dos preparos foram monitoradas por meio da utilização de paquímetro e sonda periodontal. Os preparos foram realizados com o auxílio de um estereomicroscópio com magnificação de 6.3:1 (Leica MZ6, Leica Microsystems, Wetzlar, Germany – Figura 4).

Figura A3 – Dimensões dos preparos classe II.

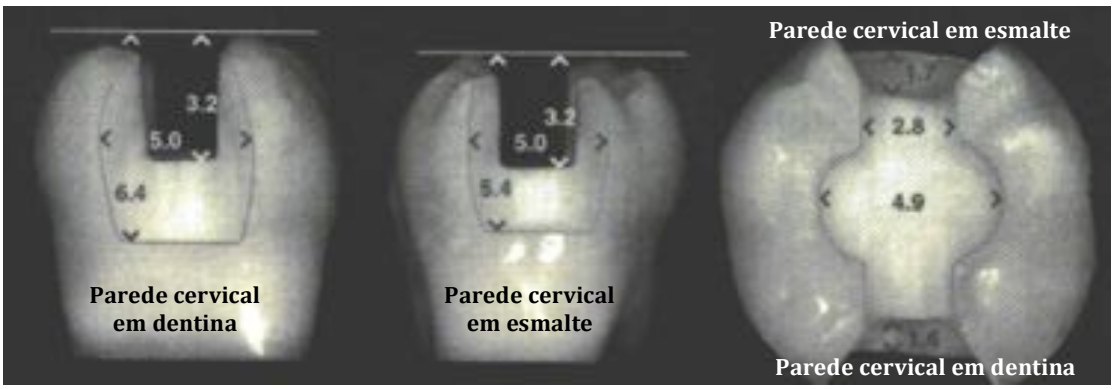


Figura A4 – Preparo sendo realizado com o auxílio de um estereomicroscópio.

Procedimentos restauradores

Os dentes alocados nos grupos 3F, 4F, 5F, 1A, 2A e 3A foram submetidos aos seguintes procedimentos restauradores:

- Grupos 3F e 1A: após terminados os preparos de classe II, as cavidades foram lavadas e secas com papel absorvente. Em seguida, o adesivo auto-condicionante de frasco único Xeno® V (Dentsply Caulk, Milford, Delaware) foi aplicado de acordo com as instruções do fabricante e fotoativado por 20 s. O material restaurador SureFil® SDR™ *flow* foi dispensado diretamente no preparo cavitário com o auxílio de uma seringa Centrix (Sistema Centrix, DFL, Jacarepaguá, Rio de Janeiro) sob pressão lenta e constante. O preenchimento foi realizado a partir da porção mais profunda da cavidade até a margem cavo-superficial, em incrementos de até 4 mm de altura. Os incrementos foram fotoativados por meio de um aparelho fotoativador à base

de LEDs Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein), com irradiância de aproximadamente 1100 mW/cm^2. Durante todo o experimento, a potência de saída do aparelho fotoativador foi medida através de um potenciômetro calibrado (Fieldmaster Power Meter, Coherentmodel, EUA). O acabamento imediato das restaurações foi realizado com pontas de granulação fina e extra-fina.

- Grupos 4F e 2A: foram realizados os mesmos procedimentos já mencionados para o grupo 3 até a aplicação do sistema adesivo. Em seguida, o material restaurador SureFil® SDR™ *flow* foi dispensado diretamente no preparo cavitário com o auxílio de uma seringa Centrix sob pressão lenta e constante. A cavidade foi preenchida a partir de sua porção mais profunda até 2 mm aquém da margem cavo-superficial. Este incremento foi então fotoativado por meio de um aparelho fotoativador à base de LEDs Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein), e a porção oclusal da cavidade foi preenchida com uma resina composta nano-híbrida à base de metacrilato EsthetX™ HD (Dentsply Caulk, Milford, Delaware) de acordo com as instruções do fabricante, seguido pela fotoativação desta resina também de acordo com o fabricante da mesma. O acabamento imediato das restaurações foi realizado com pontas de granulação fina e extra-fina.

- Grupos 5F e 3A: foram realizados os mesmos procedimentos já mencionados para os demais grupos até a aplicação do sistema adesivo. Em seguida, a cavidade foi preenchida com incrementos oblíquos de aproximadamente 2 mm de espessura, com a resina composta nano-híbrida EsthetX™ HD, seguindo-se as instruções do fabricante. Após a fotoativação de cada

incremento separadamente, foi realizado acabamento das restaurações com pontas de granulação fina e extra-fina.

Após a etapa restauradora, a qual foi realizada por um mesmo operador, todos os grupos experimentais foram armazenados em água destilada, ao abrigo de luz, a uma temperatura de 37˚C.

Ensaio de resistência à fratura (Capítulo 1)

Após uma semana, os espécimes dos cinco grupos foram removidos das condições de armazenamento e submetidos ao teste de resistência à fratura por meio de uma carga compressiva axial, através do emprego de uma esfera metálica medindo 8 mm de diâmetro. Esta esfera foi adaptada a uma máquina de ensaio universal EMIC DL-2000 (São José dos Pinhais, PR, Brasil – Figura A5), com célula de carga de 10 kN, acionada a uma velocidade de 5 mm/min até que ocorresse a fratura do dente (Figuras A6 e A7).

Figura A5 – Máquina de ensaio universal EMIC DL-2000 (São José dos Pinhais, PR, Brasil).

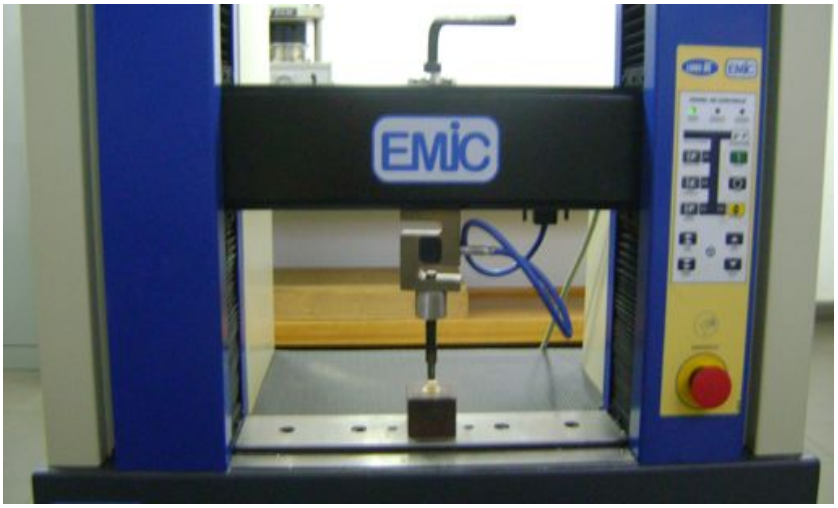


Figura A6 – Esfera metálica em contato com as cúspides do dente.

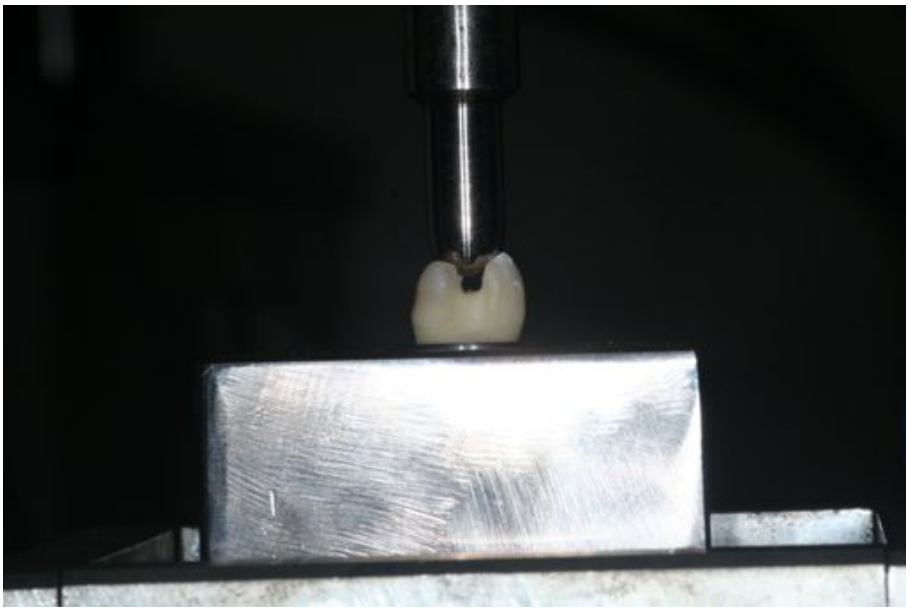
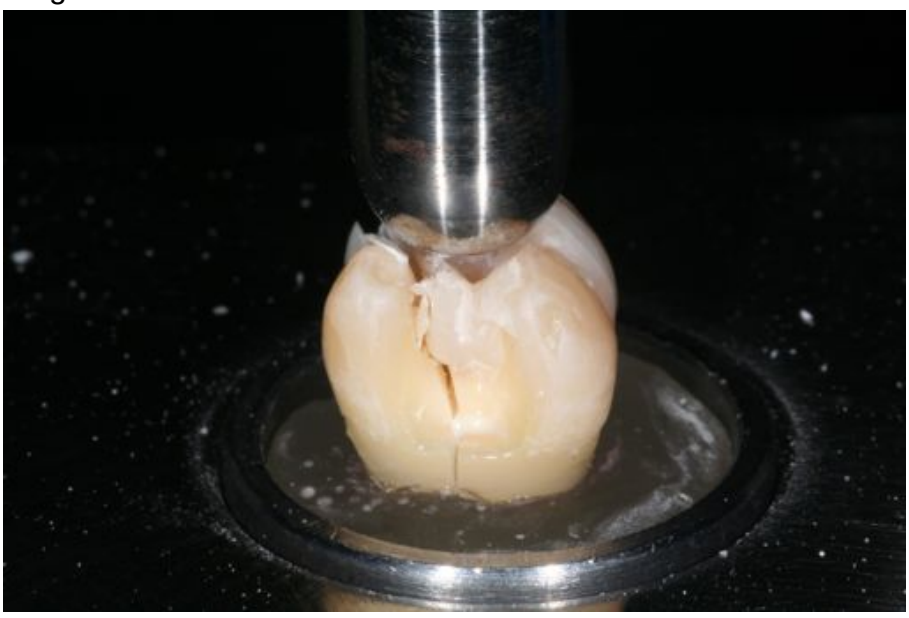


Figura A7 – Momento da fratura do dente.



Ensaios de ciclagem termo-mecânica e adaptação marginal (Capítulo 3)

Os espécimes foram submetidos simultaneamente a desafios termo-mecânicos em uma máquina de mastigação controlada por computador. A termociclagem foi executada em banhos de água em duas diferentes temperaturas (5 e 50˚C) por 600 vezes, com um intervalo de permanência de dois minutos para

cada temperatura. Simultaneamente, os espécimes foram submetidos à carga mecânica que consistiu de 240.000 ciclos a uma frequência de 1,7 Hz, com carga máxima de 49N transferida ao centro da superfície oclusal por meio de cúspides antagonistas naturais (Figuras 8 e 9).

Figura A8 – Espécimes sendo submetidos a desafios termo-mecânicos em máquina de simulação mastigatória.

Figura A9 – Computador servidor da máquina de mastigação.

A adaptação marginal antes e após a ciclagem termo-mecânica foi avaliada por meio da técnica da réplica e análise quantitativa em microscopia eletrônica de varredura (MEV) com ampliação de 200x, de acordo com o protocolo previamente estabelecido por Krejci[26] et al. (1993). Para isso, antes e após a ciclagem as margens das restaurações foram limpas com escova e pedra-pomes. Após a limpeza, foram realizadas impressões utilizando silicona por adição (Aquasil Ultra, Dentsply Caulk, Milford, Delaware), as quais foram posteriormente preenchidas com resina epóxi (Epofix, Struers, Copenhagen, Denmark) para a obtenção de réplicas. Após cobertura áurica, as réplicas foram observadas quantitativamente com relação à integridade marginal. A micromorfologia marginal foi avaliada com relação às seguintes qualidades: “margem contínua” e “margem não-contínua” ao longo da periferia das restaurações. As diferentes qualidades marginais foram quantificadas como um percentual do perímetro total de margens analisadas.

Análise da contração volumétrica de polimerização (Capítulo 3)

A mensuração da contração volumétrica das resinas SureFil® SDR™ *flow* e EsthetX™ HD foi realizada por meio do dispositivo de vídeo-imagem AcuVol® (Bisco, Schaumburg, EUA) do Laboratório de Bioengenharia do Departamento de Materiais Odontológicos e Prótese da Faculdade de Odontologia da UNESP (São José dos Campos) e o aparelho de fotoativação utilizado foi o Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein).

O AcuVol® captura e analisa imagens das amostras utilizando uma câmera de vídeo CCD em preto e branco equipada com macrolentes de 45 mm com abertura de 1,0 cm. A amostra de resina iluminada por LEDs vermelhos tem sua imagem capturada a uma distância de 10 mm. Em seguida, um processador de vídeo digitaliza a imagem a uma resolução de 512 x 486 pixels e 8 bits/pixel. Um *software* específico (MIOD Detection Technologies) para análise de imagem é então utilizado para calcular o volume da amostra através da divisão da imagem capturada em centenas de fatias (slices) horizontais, cada uma com altura de 1 pixel. O *software* emprega um método de limite de detecção no qual o computador determina a localização dos limites esquerdo e direito da amostra, através da comparação do brilho do pixel contra um valor padrão recomendado pelo fabricante que é significativamente mais brilhante do que os níveis de intensidade dos pixels do background. Os valores limite são então subtraídos para se obter a largura dos slices em pixels. O cálculo do volume da amostra é realizado pelo software de análise de imagem pela adição dos valores de volume de todos os slices horizontais.

Para mensuração da contração, foram confeccionados cinco corpos-de-prova, cada um com cerca de 15 µl (± 1) das resinas compostas, que foram manualmente moldados em uma semi-esfera e colocados em um pedestal de politetrafluoretileno com 4,2 mm de diâmetro na frente da câmera (CCD) da máquina. A imagem obtida foi capturada e digitalizada pelo *software* AcuVol® e depois do escoamento do material por 2 minutos, o perímetro das amostras foi mensurado por uma linha pontilhada virtual.

O tamanho mensurado foi armazenado no programa, representando o volume inicial da amostra. Em seguida, o compósito foi foto-ativado por 20 s de

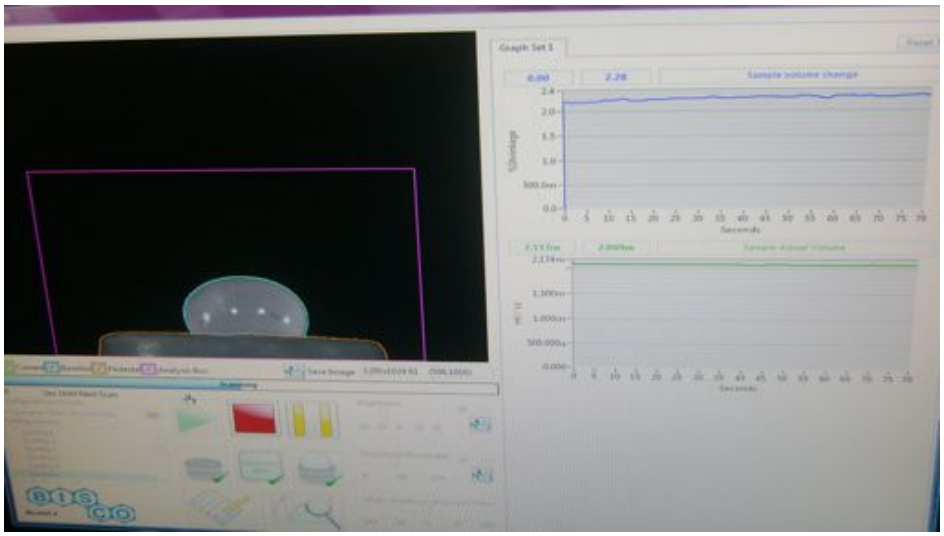
modo que a ponteira do foto-polimerizador ficou posicionada a 5 mm da superfície de topo da resina composta (Figura A10).

Figura A10 – Fotoativação da resina dentro do aparelho AcuVol®.

Antes dos procedimentos de fotoativaçao, a potência de saída do aparelho fotoativador foi medida através de um potenciômetro calibrado (Fieldmaster Power Meter, Coherentmodel, EUA) e o diâmetro da ponteira do aparelho fotoativador foi medido com um paquímetro digital (Mitutoyo, Tóquio, Japão). A densidade de potência (mW/cm^2) foi calculada como a razão entre a potência de saída pela área da ponteira através da seguinte fórmula: I = P / A, onde P é a potência em miliwatts e A é a área da ponteira em centímetros quadrados.

Ao final do período de polimerização, outra linha pontilhada contornou o perímetro do volume final apresentado pelo material. Como as linhas (inicial e final) são representadas em uma mesma imagem, torna-se possível a visualização da contração do corpo-de-prova por meio da diferença entre as linhas virtuais pontilhadas inicial e final. O *software* calcula então, de forma automatizada, o percentual de contração volumétrica (Figura A11).

Figura A11 – Cálculo da contração volumétrica de polimerização pelo *software*.



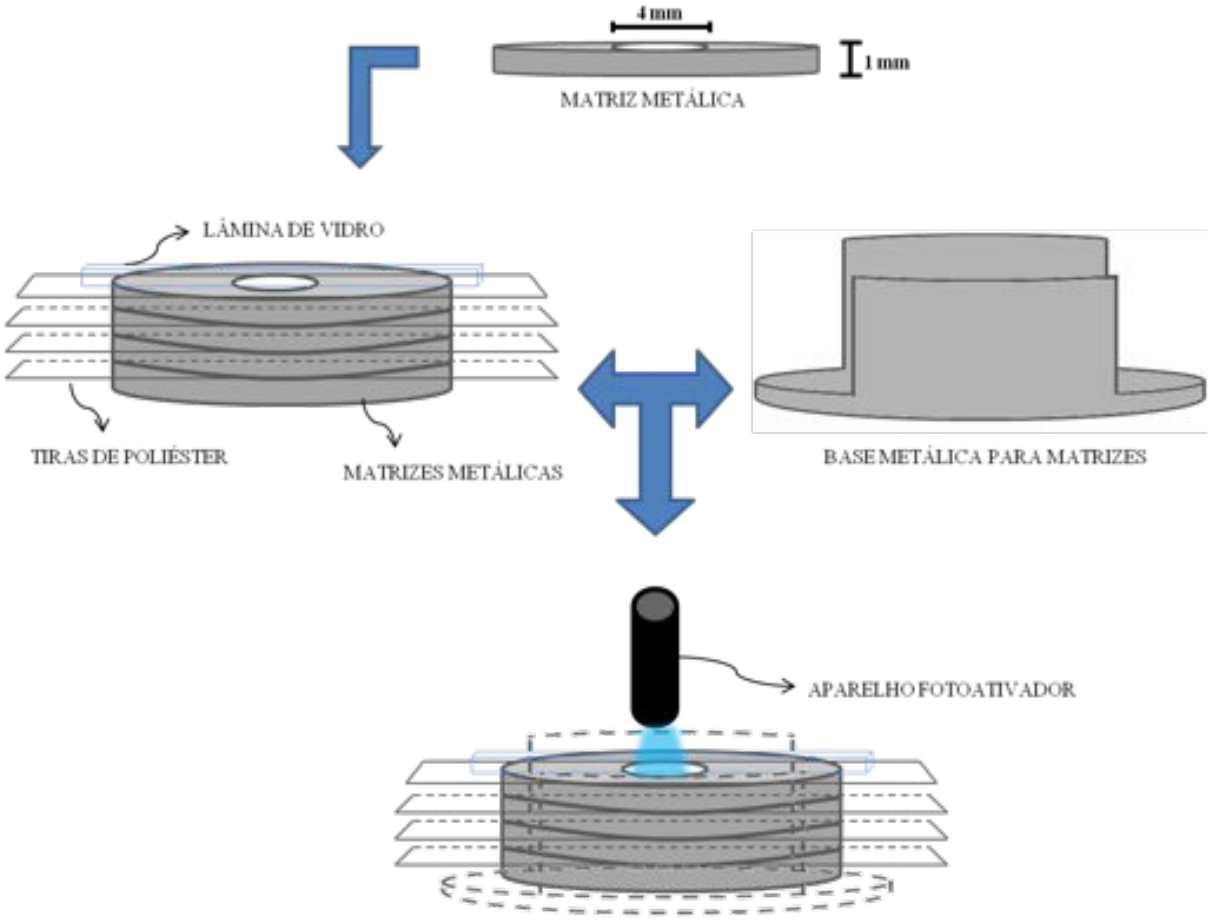
Análise do grau de conversão (Capítulo 2)

Confecção dos espécimes

De acordo com o fabricante da resina SureFil® SDR™ flow, ela pode ser dispensada em uma cavidade em incrementos de até 4mm de espessura, com efetiva polimerização. Portanto, com o intuito de analisar a qualidade da polimerização foi avaliado o grau de conversão da resina em diferentes profundidades. Foram confeccionados oito espécimes cilíndricos (4mm de diâmetro x 4mm de espessura) da resina SDR™. Cada espécime foi dividido em quatro discos que representaram as profundidades de polimerização de 1mm, 2mm, 3mm e 4mm, a partir da superfície de topo do espécime (superfície voltada para a fonte de luz). Os espécimes foram confeccionados de acordo com o esquema ilustrado na Figura A12. Quatro matrizes metálicas com orifício central medindo 4mm de diâmetro por 1mm de espessura foram posicionadas em uma base metálica, de modo que cada uma foi preenchida com a resina testada e ficaram interpostas por tiras de poliéster. Após as matrizes estarem totalmente preenchidas pela resina, foi

posicionada sobre a superfície de topo outra tira de poliéster, e sobre esta uma lâmina de vidro. Realizou-se pressão digital sobre a lâmina, com o intuito de promover adequado escoamento do material e padronizar a superfície dos corpos-de-prova. A ponta do aparelho fotoativador Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein) foi posicionada no centro da matriz, em contato com a lâmina de vidro. A foto-ativação foi realizada nos tempos de 20 e 40 segundos.

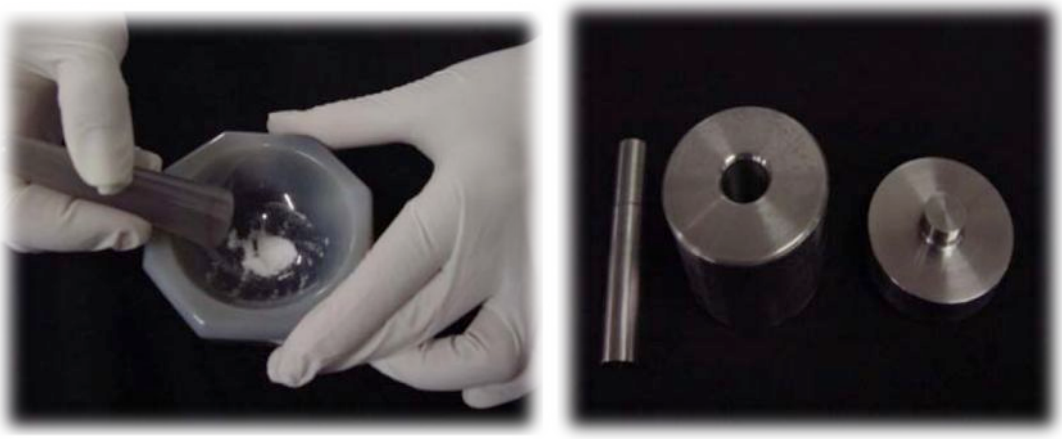
Figura A12 - Representação esquemática da confecção dos espécimes.



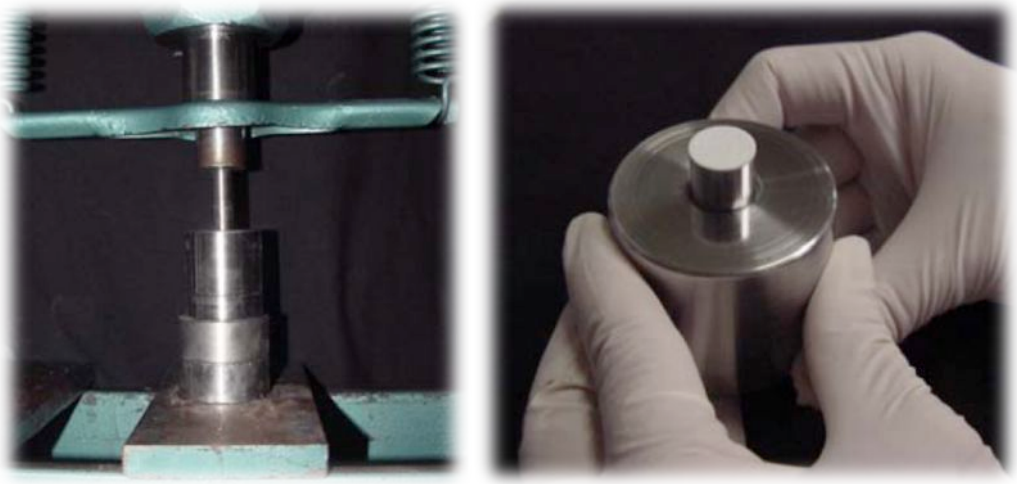
Os espécimes foram armazenados em recipientes ao abrigo da luz devidamente identificados (número do espécime / profundidade), em estufa à

temperatura de 37ºC. Vinte e quatro horas após a confecção dos espécimes, estes foram triturados em grau e pistilo de ágata até a obtenção de um fino pó (Figura A13). Em balança de precisão foram pesados 5 mg da resina testada. A quantidade pesada foi misturada à 100 mg da substância translúcida brometo de potássio (KBr - Merk). Após homogeneização, o pó obtido foi colocado em pastilhador metálico (Figura A14), e em seguida levado à prensa (Figura A15) sob pressão de 8 toneladas durante 1 minuto para obtenção de uma pastilha (Figura A16) a qual foi analisada em espectrofotômetro no infravermelho (FT-IR). Foram também confeccionados oito espécimes da resina não curada, misturando-se uma pequena porção da mesma com 100mg de KBr para posterior avaliação.

Figuras A13 e A14 – Trituração do espécime em grau e pistilo de ágata e pastilhador metálico.



Figuras A15 e A16 - Pastilhador metálico levado à prensa e pastilha obtida.



Para a obtenção dos espectros na região do infravermelho, foi empregado o espectrofotômetro FT-IR (Nexus-470, Figura A17) equipado com detector TGS na faixa de 4000 – 300 cm^{-1}, utilizando acessório de refletância difusa operando com 32 scans, resolução de 4 cm^{-1} acoplado a microcomputador servidor. Os espectros foram obtidos pela técnica de transmissão observando-se os picos de absorbância.

Figura A17 - Espectrofotômetro FT-IR (Nexus-470).

Após a obtenção dos picos de absorbância, o percentual de duplas ligações carbônicas não convertidas (%C=C) foi determinado pela taxa de intensidade de absorção entre ligações C=C em 1.637cm^{-1} e ligações C-C em 1.610 cm^{-1}, antes e após a polimerização. O grau de conversão (GC) correspondente foi calculado pela fórmula:

$$GC(\%) = 1 - \frac{\left(1637cm^{-1} / 1610cm^{-1}\right)curada}{\left(1637cm^{-1} / 1610cm^{-1}\right)não - curada}$$

Anexo

UNIVERSIDADE ESTADUAL PAULISTA " JÚLIO DE MESQUITA FILHO"

FACULDADE DE ODONTOLOGIA DE ARARAQUARA

Comitê de Ética em Pesquisa

Certificado

Certificamos que o projeto de pesquisa intitulado *"RESISTÊNCIA À FRATURA E ADAPTAÇÃO MARGINAL EM PREPAROS CAVITÁRIOS DE CLASSE II MOD RESTAURADOS COM COMPÓSITO FLOW DE BAIXA CONTRAÇÃO"* sob o protocolo nº 36/11, de responsabilidade do Pesquisador (a) *EDSON ALVES DE CAMPOS* está de acordo com a Resolução 196/96 do Conselho Nacional de Saúde/MS, de 10/10/96, tendo sido aprovado pelo Comitê de Ética em Pesquisa–FOAr, com validade de 01 (um) ano, quando será avaliado o relatório final da pesquisa.

Certify that the research project titled *"FRACTURE RESISTANCE AND EXTERNAL ADAPTATION OF CLASS II CAVITIES RESTORED WITH A BULK-FILL FLOWABLE COMPOSITE"*, protocol number 36/11, under Dr *EDSON ALVES DE CAMPOS* responsability, is under the terms of Conselho Nacional de Saúde/MS resolution # 196/96, published on May 10, 1996. This research has been approved by Research Ethic Committee, FOAr-UNESP. Approval is granted for 01 (one) year when the final review of this study will occur.

Araraquara, 08 de julho de 2011.

Prof Dr Maurício Meirelles Nagle

Coordenador

Araraquara, 23 de julho de 2014.

FERNANDA FERREIRA JASSÉ