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Ana Paula Piovezan Fugolin

Análise das Propriedades Físico-Químicas de Compósitos Auto-Adesivos e Bulk-Fill

Analysis of Physical-Chemical Properties of Self-Adhering and Bulk-Fill Composites

Piracicaba

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Universidade Estadual de Campinas
Faculdade de Odontologia de Piracicaba

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Tese apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Doutora em Materiais Dentários.

Thesis presented to Piracicaba Dental School, State University of Campinas in partial fulfillment of the requirements for the degree of PhD in Dental Materials.

Orientador: Prof. Dr. Simonides Consani

Este exemplar corresponde à versão final da tese defendida por Ana Paula Piovezan Fugolin e orientada pelo Prof. Dr. Simonides Consani.

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Prof. Dr. SIMONIDES CONSANI

Prof. Dr. WILLIAM CUNHA BRANDT

Prof. Dr. RICARDO DANIL GUIRALDO

Prof. Dr. LOURENÇO CORRER SOBRINHO

Profa. Dra. DEBORA ALVES NUNES LEITE LIMA

RESUMO

Novos compósitos *bulk-fill*, autoadesivos e de menor contração foram desenvolvidos propondo mudanças na técnica restauradora incremental preconizada. Os objetivos desta pesquisa foram: 1) analisar os compósitos Tetric EvoCeram Bulk Fill (TEC), Surefil SDR (SDR), Vertise Flow (VF), Filtek Low Shrinkage (SIL) e compará-los com o compósito convencional Z100 (Z100) quanto à contração volumétrica, tensão de contração, grau de conversão, cinética, resistência à flexão e módulo de elasticidade; 2) avaliar a profundidade de polimerização e a resistência da união à tração de cavidades de Classe II restauradas com diferentes técnicas e diversas associações de materiais submetidas à ciclagem mecânica; e 3) analisar a adaptação marginal por microscopia eletrônica de varredura de restaurações de Classe II antes e após ciclagem mecânica e resistência coesiva. No capitulo 1, a contração volumétrica foi avaliada por dilatômetro de mercúrio e *bonded disc* (n=5) e a tensão de contração com Bioman (n=5). O grau de conversão foi analisado com espectroscopia de infravermelho próximo (NIR) (n=5) e a cinética por meio de *optical bench* (n=5). Resistência à flexão e módulo de elasticidade foram mensurados em ensaio com três pontos de apoio após 10 e 60 minutos da fotoativação. Os dados foram analisados por ANOVA e teste de Tukey (5%). VF apresentou os maiores valores de contração volumétrica e tensão de contração, enquanto que SIL obteve os menores. SDR apresentou a maior taxa de polimerização e os maiores valores de grau de conversão. VF apresentou os maiores valores de resistência à flexão após 10 e 60 minutos e Z100 os maiores valores de módulo de elasticidade. No capítulo 2, cavidades de Classe II ocluso-distais em terceiros molares humanos extraídos foram restauradas usando sistema adesivo convencional – XP Bond (XP) ou à base de silorano (SSA) associado aos compósitos TEC, SDR, VF, SIL e Z100 inseridos por meio da técnica incremental (I) e *bulk* (B). Os grupos experimentais testados foram: XP-Z-B, XP-Z-I, SSA-Z-B, SSA-Z-I, XP-TEC-B, XP-TEC-I, XP-SDR-Z100, SSA-SDR-Z100, VF-Z-B e SSA-SIL-B (n=8). Metade do total das amostras foi preparada para obtenção de palitos e submetida ao teste de resistência da união à microtração após sete dias de armazenagem, enquanto outra metade foi submetida à ciclagem mecânica antes do ensaio de resistência da união. A profundidade de polimerização foi mensurada em restaurações com 4,0 mm de profundidade removidas da cavidade e submetidas ao ensaio de dureza Knoop (n=3). Os resultados foram analisados por ANOVA e teste de Tukey (5%). Quanto à resistência de união o grupo XP-SDR-Z apresentou os maiores valores em ambas as superfícies (oclusal e cervical) nos grupos ciclados ou não. Os menores valores foram exibidos por VF-Z-B em ambas as superfícies para os grupos não ciclados e SSA-SDR-Z para os ciclados. Em relação à profundidade de polimerização, VF apresentou a maior redução da dureza, enquanto os demais compósitos apresentaram valores de redução menores do que 20%. No capítulo 3, as cavidades de Classe II restauradas seguiram as mesmas etapas dos grupos experimentais apresentados no capítulo 2 (n=5) e foram submetidas ao ensaio de ciclagem mecânica; porém, foram previamente moldadas para obtenção de replicas para análise da integridade marginal em microscopia eletrônica de varredura. As imagens foram analisadas pelo *software* Image J para verificar a porcentagem de fendas. Os compósitos utilizados para restaurar as cavidades foram submetidos ao teste de resistência coesiva (n=5). Os resultados foram analisados por ANOVA e teste de Tukey (5%). A análise da adaptação marginal qualitativa e quantitativa mostrou alteração significativa antes e após a ciclagem mecânica apenas para todos o grupo SSA-Z-B. Os resultados de resistência coesiva mostraram que Z100, SDR e SIL apresentaram os maiores valores, seguido de TEC. VF apresentou os menores valores. Concluiu-se que os novos compósitos apresentam propriedades comparáveis e, em algumas situações, melhores quando comparado ao compósito convencional.

Palavras-Chave: compósitos bulk fill, materiais auto-adesivos, materiais a base de silorano, propriedades físico-químicas.

ABSTRACT

New bulk-fill, self-adhering and low shrinkage materials were developed and purposed significant changes in the preconized incremental restorative technique. The aims of this research were: 1) analyze the new composites represented by Tetric EvoCeram Bulk Fill (TEC), Surefil (SDR), Vertise Flow (VF), Filtek Low Shrinkage (SIL) and compare them with the conventional composite Z100 (Z100) in relation to volumetric shrinkage, stress of polymerization, degree of conversion, kinetics, flexural strength and modulus; 2) evaluate depth of cure of different composites and microtensile bond strength of Class II cavities filled by different restorative techniques and materials association submitted to mechanical fatigue-cycling test; and 3) analyze marginal adaptation by scanning electron microscopy (SEM) of Class II restoration before and after mechanical cycling, and ultimate tensile strength. In the charter 1, the volumetric shrinkage was evaluated by mercury dilatometer and bonded-disc techniques (n=5) and stress of polymerization by Bioman instrument (n=5). Degree of conversion was analyzed with NIR-spectroscopy (n=5) and the kinetics by the optical bench (n=5). Flexural strength and modulus were carried out using a three-point bending test after 10 and 60 minutes after photocuring. Results were analyzed by ANOVA and Tukey's test (5%). VF showed the highest values of volumetric shrinkage and stresses of polymerization and SIL the lowest ones. SDR obtained the highest rate of polymerization and the highest degree of conversion values. VF presented the highest values of flexural strength in both tested times, and Z100 the highest values of modulus. In the charter 2, Class II occluso-distal cavities (6 x 2 x 4 mm) in extracted human molars were restored using a etch-and-rinse adhesive system – XP Bond (XP) or silorane-based (SSA) associated to TEC, SDR, VF, SIL and Z100 composites placed by incremental (I) or bulk (B) technique. The tested experimental groups were: XP-Z-B, XP-Z-I, SSA-Z-B, SSA-Z-I, XP-TEC-B, XP-TEC-I, XP-SDR-Z100, SSA-SDR-Z100, VF-Z-B e SSA-SIL-B (n=8). Half of the samples were prepared to obtain sticks and submitted to the microtensile bond strength test after 7 days of storage. The other samples were submitted to the mechanical fatigue-cycling test before the microtensile bond strength test. Depth of cure was carried out in restorations with 4.0 mm of depth, removed and submitted to Knoop hardness test (n=3). Results were statistically analyzed by ANOVA and Tukey's test (5%).

In relation to microtensile bond strength, in overall, XP-SDR-Z showed the highest values in both analyzed surfaces (occlusal and cervical) in cycling and no-cycling groups. The lowest values were exhibited by VF-Z-B in both analyzed surfaces in no-cycling groups and SSA-SDR-Z in cycling groups. In relation to depth of cure, VF obtained the lowest top-to-bottom ratio, while the other tested composites exhibited less than 20% of reduction. In the charter 3, Class II cavities were prepared following the same steps described in charter 2 (n=5) and were carried out to mechanical fatigue-cycling test. However, impressions were made before and after to obtain replicas to SEM analysis of the marginal integrity. The micrographs were analyzed by Image J software to measure the discontinuity percentage. The composites used in the cavities were submitted to the ultimate tensile strength (UTS) (n=5). Results were statistically analyzed by ANOVA and Tukey's test (5%). Marginal adaptation analysis did not show significant alteration before and after cycling for all groups except to SSA-Z-B where cracks and gaps were found in the adhesive interface. In relation to UTS results Z100, SDR and SIL showed the highest results, followed by TEC. VF exhibited the lowest values. It is possible to conclude that new composites show comparable properties and in some situation better than conventional material.

Key words: bulk fill composites, self-adhering materials, silorane-based materials, physico-chemical properties

SUMÁRIO

DEDICATÓRIA

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Se você quer um pedacinho do Paraíso, acredite em Deus.

Mas se você quer conquistar o mundo, acredite em você

porque Deus já te deu tudo o que você precisa

para você vencer.

Augusto Branco

INTRODUÇÃO

A formulação dos compósitos tem evoluído consideravelmente desde que esses materiais foram introduzidos na Odontologia na década de 1960 [1]. No início da evolução, as mudanças mais importantes estavam vinculadas as partículas de carga; porém, atualmente o foco tem sido a matriz orgânica, visando, principalmente, o desenvolvimento de sistemas com reduzida contração de polimerização que, consequentemente, podem gerar menor tensão de contração nos compósitos e materiais autoadesivos [1].

Os compósitos autoadesivos representam nova categoria de material recentemente introduzida no mercado e que, de acordo com instruções dos fabricantes, não requerem tratamento prévio do substrato[2]. Esses materiais são compostos por monômeros mais ácidos que reagem com o substrato e se infiltram na estrutura do dente, resultando em retenções micromecânicas potencialmente reforçadas por interação química adicional [2,3]. Portanto, trata de materiais que visam a facilitar e simplificar a técnica restauradora; porém, espera-se desses materiais menor desempenho no que diz respeito à adesão quando comparado aos sistemas convencionais [2]. Esse fato ocorre porque o material é mais viscoso, enquanto que os adesivos convencionais são mais fluidos e conseguem promover melhor interação com o substrato [2]. Nessa nova categoria de compósitos encontra-se disponível no mercado o Vertise Flow (Kerr Dental, Orange, CA, USA) que, segundo o fabricante, pode ser utilizado para forramento de cavidades de Classes I ou II, para selamento de fóssulas e fissuras, reparos em cerâmica e restauração de cavidades de Classes I ou II pequenas [4]. Porém, existem poucas informações na literatura que permitam caracterizar esse material e predizer o desempenho clínico.

No que diz respeito às estratégias para reduzir a contração volumétrica e minimizar as tensões geradas durante a contração de polimerização foram desenvolvidos os compósitos à base de silorano [5]. Esses materiais são compostos pela combinação de siloxano que confere características hidrófobas ao material e por anéis oxirano cicloalifáticos que se abrem durante a polimerização causando expansão [5]. Teoricamente, esses materiais apresentam menores contração volumétrica e tensão de contração, sem comprometer as propriedades mecânicas [5,6]. Porém, alguns dados apresentados na literatura são contraditórios e inconclusivos. Assim, um trabalho mostra que esse material apresenta menor contração volumétrica sem interferir na quantidade de tensões geradas [7] e outro defende que materiais à base de silorano geram menor quantidade de tensões quando comparado aos materiais à base de metacrilato [8].

Entretanto, compósitos à base de silorano requerem a utilização de sistema adesivo específico também à base de silorano. Por se tratar de categorias diferentes de polímeros, acredita-se existir incompatibilidade desses adesivos com os compósitos à base de metacrilato. Entretanto, os resultados mostraram que essa combinação poderia ser promissora [9].

Mesmo com a evolução dos compósitos, a técnica restauradora foi pouco modificada. A inserção em pequenos incrementos tem sido ainda largamente preconizada com a intenção de minimizar as tensões de contração[10], promover maior grau de conversão e obter adequada adaptação marginal [11]. Na tentativa de facilitar o procedimento clínico, diminuir o tempo restaurador e reduzir as tensões de contração foram desenvolvidos compósitos que, segundo os fabricantes, podem ser inseridos em incrementos mais espessos. Um deles é o SDR Posterior Bulk Fill Flowable Base (Dentsply, Konstanz, Germany), introduzido no mercado como compósito de menor viscosidade, podendo ser aplicado em incrementos de até 4 mm e recoberto por camada de aproximadamente 2,0 mm de compósito convencional [11]. Outro compósito similar é o Tetric EvoCeram Bulk Fill (Ivoclar-Vivadent, Schaan, Liechtenstein) que, conforme alegado pelo fabricante, pode ser utilizado em bloco na técnica restauradora e também inserido em camadas de até 4 mm, gerando menor quantidade de tensões de contração e propriedades mecânicas satisfatórias [12]. Entretanto, são ainda escassas as informações encontradas na literatura sobre esses materiais.

As tensões geradas durante a contração de polimerização e a contração volumétrica são apontadas na literatura como uma das principais causas de falha da adesão [13], sendo parâmetros importantes para a avaliação do desempenho clínico desses materiais poliméricos. Contudo, parece existir relação entre as tensões geradas durante a contração de polimerização e a taxa de polimerização; porém, os resultados mostrados na literatura são controvertidos [13], havendo trabalhos que mostram que reduções significativas nas taxas de polimerização não correspondem necessariamente à redução significativa das tensões de contração [7,14]. Isso provavelmente ocorre pelo fato de que o desenvolvimento de tensões estaria relacionado não somente à contração de polimerização, mas também à taxa de polimerização e ao módulo de elasticidade do material[14]. Dessa forma, taxa de polimerização e módulo de elasticidade parecem ser fatores importantes para o completo entendimento da cinética do desenvolvimento de tensões durante a polimerização dos compósitos.

A resistência à flexão pode ser indicativa do desempenho e da longevidade dos compósitos quando submetidos às forças mastigatórias, estabelecendo o perfil do material em relação às propriedades mecânicas [15]. Esse fato ocorre da mesma maneira do que no teste de resistência da união por microtração, frequentemente utilizado para avaliar a adesão entre substrato e sistema adesivo e entre sistema adesivo e material restaurador [16]. A análise do grau de conversão permite avaliar a eficiência da polimerização e valores reduzidos de grau de conversão podem estar associados às propriedades mecânicas deficientes dos compósitos, menor resistência à fratura e ao desgaste e também ao aumento da citotoxicidade [17].

Adequada adaptação interna é apontada na literatura como um dos principais desafios para a Odontologia restauradora [18]. Esta premissa ocorre porque muitos fatores contribuem para o desenvolvimento de fendas entre substrato e material restaurador. Dentre eles estão: diferenças no coeficiente de expansão térmica do dente e do material restaurador, adesão inadequada à dentina e contração de polimerização do material restaurador [18]. Além desses fatores, as restaurações estão sujeitas às tensões mecânicas e alterações térmicas ocorridas na cavidade bucal, condições que contribuem significativamente para a deterioração da interface dente-material [17]. Falhas na adaptação estão também vinculadas aos fenômenos como degradação, descoloração e pigmentação, cáries recorrentes, microinfiltração e hipersensibilidade, associação que reduz o tempo de vida útil da restauração [18].

Dessa forma, considerando a disponibilidade de novos compósitos que propõem inovações significativas nas técnicas restauradoras e a falta de evidências científicas na literatura, seria conveniente o estudo desses materiais e técnicas para avaliar a efetividade no desempenho clínico, assim como a combinação entre técnicas restauradoras e diferentes materiais.

Considerando esse contexto, o objetivo neste estudo foi avaliar:

1. Os compósitos Z100, Tetric Evo Ceram Bulk Fill, SDR, Filtek Silorane e Vertise Flow quanto à contração volumétrica, tensão de contração, cinética, grau de conversão, resistência à flexão e módulo de elasticidade.
2. A profundidade de polimerização e a resistência da união resina-dentina por microtração em cavidades de Classe II restauradas com diferentes compósitos (Z100, Tetric Evo Ceram Bulk Fill, SDR, Filtek Silorane e Vertise Flow), utilizando sistemas diferentes adesivos (XP Bond e Silorane System Adhesive), diferentes técnicas restauradoras e ciclagem mecânica das restaurações;
3. A adaptação marginal de restaurações de Classe II antes e após a ciclagem mecânica por meio da microscopia eletrônica de varredura e a resistência coesiva dos compósitos Z100, Tetric Evo Ceram Bulk Fill, SDR, Filtek Silorane e Vertise flow.

CAPÍTULO 1

Analysis of physical-chemical properties of bulk-fill, self-adhering and low-shrinkage dental composites

Abstract

Objective: To determine flexural strength, *modulus*, kinetics, volumetric shrinkage, degree of conversion and stress of polymerization of bulk-fill, self-adhering and low-shrinkage dental composite materials.

Materials and methods: The materials used for this study were Z100 (Z100), Tetric EvoCeram Bulk Fill (TEC), Surefil SDR flow (SDR), Vertise Flow (VF), and Filtek Silorane (SIL). Light curing was carried out using a led unit with 1170mW/cm$^{2.}$ Degree of conversion (DC) was measured in real time with FT-NIR spectroscopy (n=5). Stress of polymerization (SP) was monitored using Bioman instrument. (n=5). Flexural strength (FS) and Modulus (M) were determined after 10 and 60 minutes using a three point bending test. Volumetric shrinkage (VS) was investigated using bonded-disc technique and mercury dilatometer (n=3), and kinetics reaction was monitored by optical bench (n=5). Data were analyzed using ANOVA and Tukey's test (α=0.05).

Results: DC showed the highest results for VF (76.04%) and SDR (65.92%). SIL, Z100 and TEC exhibited the lowest results (41.98, 45.76 and 46.74%, respectively). In relation to SP, VF obtained the highest values (20.04 MPa) and SIL the lowest ones (5.11 MPa). The highest values of FS were showed by VF in 10 and 60 minutes (78.80 and 101.00 MPa, respectively), and the other materials presented statistically similar results. Z100 exhibited the highest values of M in 10 and 60 minutes (9.59 and 11.24 GPa, respectively) while SDR the lowest ones (3.70 GPa). VS measured by mercury dilatometer and bonded-disc showed statistical similarity ($p>0.05$). VF was the material with the highest VS (3.65%)

and LS with the lowest values (0.66%). Analyzing reaction kinetics, SDR (8.90 $\%.s^{-1}$) showed the best results, followed by VF (5.53 $\%.s^{-1}$).

Conclusion: The tested materials showed different behaviors in relation to physic-chemical properties. SIL showed the lowest values for volumetric shrinkage and stress of polymerization. The highest flexural strength was showed by VF.

1. Introduction

Resin composite have been used in dentistry for nearly decades[1]. Since these materials were introduced to dentistry their composition has evolved significantly. But until recently, the most important changes have involved the particles filler, which has been reduced in size to produce materials with better mechanical properties[2]. Beside the important changes in filler amount, shape and surface treatment, changes in monomer structure or chemistry and modifications of dynamics of the polymerization reaction seem to be the most promising approaches[3]. Current changes are more focused on the polymeric matrix of the material, principally to develop systems with reduced polymerization shrinkages[2] and to simplify the clinical application steps needed to bond the composite restoration to substrate[4].

The most common resin composites are methacrylate-based. The predominant base monomer used in these dental composites has been Bis-GMA, which is mixed with other dimethacrylates, such as TEGDMA and UDMA[5]. During polymerization of this material, the formation of a polymer network results in a denser structure, leading to a volumetric shrinkage[6]. These composites require an enamel and dentin surface pretreatment using either an etch-and-rise or self-etch adhesive making rather complex and often very technique sensitive[7]. To simplify the clinical application and technique sensitivity, self-adhering composites were introduced in the market and the first product of this new generation to be launched was the Vertise Flow (Kerr Corporation), a flowable resin composite[8]. According to the manufacturer's instructions, self-adhering composites do not require any pretreatment of the substrate[4] and its self-adhesiveness would be based upon the use of acidic monomers that demineralize and simultaneously infiltrate the tooth substrate, resulting in micro-mechanical retention enhanced by additional chemical interaction[4].

Few years ago, a new category of flowable resin composites called bulk-fill flowable composites was launched[9]. Surefil® SDR™ flow (Dentsply) is one of this composite category, and according to mansfacture's intructions this material is designed to be used as base in class I and II restorations placed in 4 mm increments with minimal polymerization stress[10].

Tetric Evo-Ceram Bulk Fill (Ivoclar-Vivadent) is another bulk-fill material that according to manufacturer takes the effort out of posterior tooth restorations, can be used in increments of up to 4 mm, and shows low shrinkage stress[11].

In order to reduce the rate of shrinkage stress manufacturers have invested their resources in the development of low-shrinkage restorative composites and, recently, a number of examples of these new materials is available for clinical use[12]. Filtek^{TM} LS – Low Shrink Posterior (3M ESPE) is a low-shrinkage composite based on a silorane resin consisting of siloxane and oxirane functional molecules. However, shrinkage stress involves more than how much a composite shrinks in volume. Thus, other factors such as elastic *modulus*, volumetric shrinkage, filling and curing protocol, degree of conversion, cavity shape, remaining tooth and restorative technique can also be included in shrinkage stress considerations[12].

Therefore, this study was intended to investigate these bulk-fill, self-adhering and low-shrinkage materials with respect to flexural strength, *modulus*, kinetics, volumetric shrinkage, degree of conversion and stress of polymerization of bulk-fill, self-adhering and low-shrinkage. The null hypothesis is that there would be no statistically differences in performance between the five resin composites tested.

2. Materials and Methods

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

Table 1: Investigated Restorative Materials and Their Composition According to Information Provided by the Respective Manufacturers

Composite (Abbreviation)	**Manufactures and Classification**	**Components**	**Lot**
Z100 (Z100)	3M ESPE Conventional	Organic matrix: Bis-GMA, TEGDMA Filler Particles: 85 wt%, 66 vol% - silica, zirconia	N427925
Tetric EvoCeram Bulk Fill (TEC)	Ivoclar Vivadent Bulk-Fill	Organic matrix: Bis-GMA, Bis-EMA, UDMA Filler Particles: 80 wt%, 60 vol% - Barium glass, YbF_3, mixed oxide, PPF	R49602
Surefil SDR flow (SDR)	Dentsply Bulk-Fill	Organic matrix: Modified UDMA, EBPADMA, TEGDMA Filler Particles: 68 wt%, 44 vol% - Ba-glass, Sr-glass	100506
Filtek Silorane Low Shrink Posterior Restorative (SIL)	3M ESPE Low-Shrink	Organic matrix: 3,4-epoxycyclohexyl-ethyl-cyclo-poly-methylsiloxane, bis-3,4-epoxycyclohexyl-ethyl-phenyl-methylsilane Filler Particles: 76 wt%, 55 vol% - SiO_2, YbF_3	N436469
Vertise Flow (VF)	Kerr Corporation Self-Adhering	Organic matrix: GPDMA, HEMA, Bis-GMA Filler Particles: 70 wt%, 48 vol% - prepolymers, silaneted Ba-glass, SiO_2, YbF_3	4675010

EBPDADMA ethoxylated bisphenol-A-dimethacrylate, *TEGDMA* triethylene glycol dimethacrylate, *UDMA* urethane dimethacrylate, *Bis-GMA* bisphenol-A-glycidyldimethacrylate, *GPDMA* glycerolprosphoric acid dimethacrylate, *HEMA* hydroxyethyl methacrylate, *Bis-EMA* bisphenol-polyethylene glycol dimethacrylate, *YbF_3* ytterbium trifluoride, *SiO_2* silicium oxide, *Ba-glass* barium glass, *Sr-glass* Strontium glass, *PPF prepolymerized filler*.

2.1 Flexural Strength Test and *Modulus* of Elasticity

The measurement of flexural strength and *modulus* of the resin composites was carried out by the 3-point bending method. Bipartite matrix was used to made bars with 2x2x20 mm. Matrix was filled with only one increment of the resin composite and light-cured for 40 seconds using DEMI^{TM} Plus LED Curing Light (Kerr Corporation). The intensity of the light was checked periodically with a potentiometer to ensure that 1170 mW/cm^2 was always delivered during the experiments. Ten samples were made for each composite. The surface of the sample was polished with 1,200 grit paper to create a glossy and flat surface. Five samples were tested after 10 minutes of the photocuring process and the other five after 1 hour. The measurements were performed using an universal testing machine (Model TT-B Universal Testing Instrument, Instron Engineering Corporation, Canton, MA) at a crosshead speed of 1 mm/s.

The flexural strength (FS) in MPa was calculated using the formula:

$$FS = \frac{3 \times F \times l}{2 \times b \times h^2}$$

Where F is the maximum load in Newtons exerted on the sample at the point of the fracture; l is the distance in mm between the supports (20 mm); b and h are, respectively, the width and thickness in mm of the sample. The *modulus* was given by the software.

2.2 Stress of Polymerization

A Bioman instrument was used to analyze the shrinkage-stress kinetics.

As previously described by Gonçalves *et al,* 2012[13], the system consisted of a cantilever load cell whose extremity is fitted to a rigid integral clamp on its free end. The clamp holds a 10 mm diameter and 22 mm tall steel rod vertically and perpendicular to the load cell axis. A 5-mm diameter, 1-mm tall steel rod was fixed at the center of the lower face of the standard rod with a cyanoacrylate adhesive to produce a rod substrate with a reduced surface are to be consistent with that used in the other test systems. The opposite surface was a rigid fused silica glass plate of 3 mm thickness.

The surface of the silica glass plate was treated with a thin layer of silane ceramic primer (3M ESPE, St. Paul, MN, USA) and the surface of the piston with Z-Prime Plus (Bisco Inc., IL, USA).

The composite was then inserted into the 1-mm gap between the upper rod and the lower glass slide and shaped into a cylinder. The samples were light-cured through the glass using a DEMI LCU in 46.8 J/cm^2 (1170 mW/cm^2 for 40 s) with a special tip (n=5). Data were registered for 10 min by a computer and the final shrinkage-stress calculated.

2.3 Volumetric shrinkage

The volumetric shrinkage was measured by two methods, Mercury Dilatometer and Bonded-Disc technique. This occurred because the two composites tested, SDR and VF, are flowable materials making impossible the test in the Mercury Dilatometer.

2.3.1 Mercury Dilatomer

Composite volumetric shrinkage was carried out in a mercury dilatometer (ADA Health Foundation, Gaithersburg, MD, USA). For this, approximately 0.1 g of composite was placed on a glass slide previously sandblasted and treated with silane ceramic primer (3M ESPE, St. Paul, MN, USA). A glass column was clamped to the glass slide, filled with mercury and a LVDT (linear variable differential transducer) probe was placed on top of the mercury column.

The composite was light-cured from underneath, through the glass slide using a QTH unit (QHL75, Dentsply) with a radiant exposure of 18 J/cm2. The data was recorded during a total period of 60 min. The volumetric shrinkage was calculated using the LVDT probe readings and previously recorded mass and density values. Five samples were tested for each composite (n=5).

2.3.2 Bonded-Disc Technique

This analysis was made according to Bryant & Mahler, 2007[14].

The bonded-disc technique consisted of a 3 mm-thick glass plate, a thin circular metal ring (16.6 mm inner diameter, 18.9 mm outer diameter, and 0.20 to 0.60 mm thick) and a thin round glass microscope coverslip (0.22 mm diameter and 0.16 mm thick). The surfaces of the glass components that contacted the samples were sandblasted.

The metal ring was placed onto the glass plate, the resin composite was inserted on the central region of the glass plate inside the metal ring, the coverslip was placed, and a force was applied make contact between coverslip and metal ring.

The components were placed beneath an LVDT device with the transducer core placed on the flexible coverslip. The output of the LVDT was connected to a mV recorder which recorded the coverslip deflection in µm vs. time. The samples were light-cured for 40 seconds with the DEMI light-curing unit (n=5).

Volumetric shrinkage was determined to be the deflection of the glass coverslip and the variation of the voltage according to the formula:

$$\% \text{ Shrinkage} = \frac{\Delta v \ x \ 2}{\Delta h}$$

Where Δv is the difference between initial and final voltage, and Δh is the difference between initial and final height of the sample.

2.4 Degree of Conversion

Degree of conversion of the resin composites was carried out with a Fourier Transformed Near-Infrared (NIR) Spectroscopy (Nicolet 6700 FTIR, Thermo Scientific, Pittsburgh, PA, USA). Disc-shaped samples were made using silicone rubber molds (n = 5; Ø = 6.5 mm; h = 0.8 mm) sandwiched between glass slides and photoactivated for 40 seconds. After 24 hours, NIR spectra were recorded in the absorbance mode. Degree of conversion was determined by calculation the variation in intensity of the methacrylate peak at 6165cm^{-1} and 4625 cm^{-1} to Z100, TEC, SDR and VF, and peaks near the epoxy region at 4581 cm^{-1} and 4071 cm^{-1} were selected to SIL. Two spectra per sec were collected with 4 cm^{-1} resolution and three spectra were obtained before of the light-curing process and used as a reference. The degree of conversion was calculated according to the formula:

$$DC = \left(1 - \frac{cured}{incured}\right) \times 100$$

2.5 Reaction Kinetics

Reaction Kinetics was monitored by an optical bench for 10 minutes. According to Howard B *et al,* 2010[15] in this technique stable and repeatable orientation and alignment of the specimen, curing light and analytical instrumentation were facilitated with an optical bench fitted with a collimating lens on the NIR output fiber, which focused the NIR signal through the mounted sample to a condensing lens that maximized signal collection for the NIR return fiber. The visible light intensity transmitted through the sample was monitored after passing through a calibrated neutral density filter, an aperture to exclude stray light and finally into the UV–Vis fiber optic inlet. With this test, it is possible to determine the vitrification point and maximum rate of polymerization (n=5).

2.6 Statistical Analysis

For stress polymerization, volumetric shrinkage, degree of conversion and kinetics, a statistical evaluation was performed with One-Way ANOVA and Tukey's test at a 5% level of significance. For flexural strength, *modulus* and volumetric shrinkage, the statistical analysis was performed with Two-Way ANOVA and Tukey’s test at a 5% level of significance.

3. Results

Descriptive statistics for flexural strength and *modulus* are presented in Table 2. All composites showed statistically significant increase of flexural strength after 60 minutes. VF recorded the highest ($p<0.05$) mean flexural strength in both tested times.

In relation to *modulus*, only TEC and SDR exhibited statistically significant difference ($p<0.05$) between 10 and 60 minutes. Z100 recorded the highest mean in both times and SDR the lowest ones in both tested times.

Table 2. Average of flexural strength (MPa) and *Modulus* (GPa) with respective standard deviations, after 10 and 60 minutes

Composites	Flexural Strength		*Modulus*	
	10 min	60 min	10 min	60 min
Z100	65.30 (10.39) Bb	68.91 (15.29) Ab	9.59 (0.87) Aa	11.24 (2.74) Aa
TEC	52.22 (10.60) Bb	66.83 (12.27) Ab	4.46 (0.62) Bc	6.52 (0.58) Abc
SDR	54.29 (9.57) Bb	68.99 (9.73) Ab	1.75 (0.52) Bd	3.70 (0.27) Ad
VF	78.80 (14.75) Ba	101.00 (9.09) Aa	4.12 (0.40) Ac	5.49 (0.65) Ac
SIL	61.24 (12.10) Bb	72.49 (9.31) Ab	6.09 (0.53) Ab	8.11 (0.44) Ab

Means followed by different letters (upper in row and lower in column) showed statistically significant difference ($p\leq0.05$).

The results of polymerization stress, volumetric shrinkage and degree of conversion are described in the Table 3. VF showed the highest means of polymerization stress (20.04 ± 0.54) followed by Z100 (16.69 ± 0.31), TEC (10.54 ± 0.40) and SDR (8.54 ± 0.55). SIL recorded the lowest results (5.11 ± 0.50).

VF showed the highest volumetric shrinkage results (3.65 ± 0.23), followed by Z100 (2.41 ± 0.03), TEC (2.44 ± 0.07) and SDR (2.34 ± 0.07) that recorded similar results. The lowest results were presented by SIL (0.66 ± 0.21).

The results of degree of conversion indicated a significant difference among tested resin composites. SDR presented the highest values (76.04 ± 4.36) followed by VF (65.92 ± 3.11). SIL (41.98 ± 1.84), Z100 (45.76 ± 2.36) and TEC (46.74 ± 1.89) recorded the lowest similar results.

Table 3. Average of stress of polymerization (MPa), volumetric shrinkage (%) and degree of conversion (%) with respective standard deviations

Composites	Polymerization Stress	Volumetric Shrinkage		Degree of conversion
		Mercury Dilatometer	Bonded Disc	
Z100	16.69 (0.31) b	2.54 (0.02) aA	2.41 (0.03) bA	45.76 (2.36) c
TEC	10.54 (0.40) c	2.33 (0.03) aA	2.44 (0.07) bA	46.74 (1.89) c
SDR	8.54 (0.55) d	NA	2.34 (0.07) b	76.04 (4.36) a
VF	20.04 (0.54) a	NA	3.65 (0.23) a	65.92 (3.11) b
SIL	5.11(0.50) e	0.72 (0.04) aA	0.66 (0.21) cA	41.98 (1.84) c

Means followed by different letters (upper in row and lower in column) show statistically significant difference ($p \leq 0.05$)

Kinetics reaction results are described in the Table 4. SDR recorded the highest values (8.9 ± 0.8) of maximum rate of polymerization followed by VF (5.5 ± 0.9). Z100 and TEC showed the lowest and similar results (2.8 ± 0.2 and 2.5 ± 0.9, respectively).

Table 4. Maximum rate of polymerization (%.s^{-1}) and vitrification point (%) average with respective standard deviations

Composites	Maximum rate of polymerization	Vitrification Point
Z100	2.8 (0.2) c	7.2 (1.6) c
TEC	2.5 (0.9) c	10.1 (1.1) bc
SDR	8.9 (0.8) a	26.0 (3.0) a
VF	5.5 (0.9) b	14.1 (2.2) b
SIL	NA	NA

Means followed by different letters in each column show statistically significant difference ($p \leq 0.05$).

4. Discussion

Large and significant differences ($p<0.001$) were observed for all considered physical-mechanical properties for the tested composite, which led to the rejection of the null hypothesis.

Flexural Strength and *Modulus*

According to this work, the flexural strength of the TEC and SIL bulk-fill composites are closer to those of the SDR flowable materials and Z100 conventional composite. This result is in agreement with previous study[16].

The positive correlation between flexural strength and *modulus* and filler mass fraction is also in accordance with previous works[3,17] but not in another where the influence of the organic matrix was more prominent[18]. Differences in flexural strength and *modulus* may be due to specificities of the organic matrix, such as variations of filler size and morphologies, monomer type and ratio or photoinitiation chemistries[18]. The VF self-adhering flowable composite suffered the greatest of the flexural strength values. This can be related to the inorganic content. According to manufacturer, VF consists of 4 filler types: barium glass filler (10 µm), pre-polymerized filler (20 µm), nano-sized colloidal silica (10-40 nm) and nano-sized Ytterbium fluoride filler (40 nm) totaling 70 wt% and resulting in high flexural strength[19]. On the other hand, pre-polymerized filler (PPF) would be responsible for smooth and flexibility characteristics of this material, explaining the low modulus.

Z100, SDR, TEC and SIL showed similar values of flexural strength. Although previous studies[20,21] the bulk-fill materials exhibited lower mechanical properties compared with the conventional composites, it should be noted that certain properties of the bulk-fill may be equivalent or very similar to conventional materials. This performance of low-shrinkage and bulk-fill materials would be attributed to modifications of monomers and filler contents, and addition of polymerization modulators and initiation boosters resulting in materials with improved properties[22].

The lowest *modulus* showed by SDR can be related to composition. The manufacturer renounced to bisphenol-A-dimethacrylate (Bis-GMA) and formed the organic matrix out of other dimethacrylates[10, 23]. This way, the SDR is supposed to be less viscous because UDMA, TEGDMA and ethoxylated EBPDMA form more flexible polymers than Bis-GMA[21]. On the other hand, Z100 showed the highest *modulus*. This result may be due to the combination of Bis-GMA and TEGDMA in its matrix as well as its higher filler content. Asmussen and Peutzfeldt[24] found that the combination of 50% Bis-GMA, 50% TEGDMA in organic matrix, and 0% UDMA is responsible for the highest elastic modulus.

Tested composites showed higher results of flexural strength and *modulus* after 60 minutes time. This can be explained by a continuous polymerization reaction even after the photocuring process. On the bottom, a slower polymerization reaction could be noted. This way, during the next minutes after the stopping of the photocuring occurs the activation of the camphorquinone, induction of polymerization nucleus, and formation of longer polymer chains, resulting in better properties[25]. And according to Burstcher[26], even a small increase in the extent of degree of conversion near the end of the polymerization process can largely affect the density of cross-linking in the polymer network, and thus the mechanical properties of resin composites.

Degree of conversion

SDR showed the highest values of DC (76.06%). This value is in agreement with previous study[27]. The curing efficiency of SDR was found to be overall satisfactory and this result is in according to the findings of previous investigations[23,27,28]. The peculiar photoinitiating system may have contributed to such outcome[21]. SDR features a photoactive group embedded in urethane-based methacrylate monomers and capable of interacting with camphorquinone[10]. Such interaction, claimed to modulate curing for stress control purposes, might also have resulted in deeper polymerization[25]. Beside the photoinitiating system, also the optical properties might have had a significant role in this regard. Specifically, the translucency of SDR is expected to favor light penetration, thus enabling increased degree of conversion[27].

Comparing out results of previous study[29], VF showed similar values of degree of conversion. Since no definitive information is available about its chemical composition, it is difficult to draw an accurate explanation. However, a reasonable reason is based on the viscosity of this composite. VF shows flow consistency that is probably due to the high ratio of low molecular weight monomers with high mobility and, than, high degree of conversion.

Z100, TEC and SIL obtained similar results. This is according to previous study, where TEC showed similar degree of conversion when compared to the tested conventional composite[30]. Filler inorganic particles of these materials might be one reason for this result. All of them show very similar percentage and sizes of filler content and according to literature the inorganic particles can interfere in the mobility of reactive sites and actively participate in the scattering phenomena of the light, defining the degree of conversion[30].

Due to different organic and filler compositions of the composites, mechanical superiority cannot be predicted by degree of conversion. Together with a different filler loading and filler type, each monomer associated to additional group can imply in different properties in different molecular architecture[29]. Thus, a high DC in the case of VF and SDR does not necessarily mean similar mechanical values as reflected in the mechanical properties measured in this study.

Kinetics

Important characteristics of the composites, as vitrification point and rate of polymerization can be studied by analyzing the polymerization reaction. Vitrification point is the degree of conversion at the maximum rate of polymerization. Defines the point in conversion at which diffusion limitations dramatically decrease rate of polymerization and ultimately influences final degree of conversion and stress development within the material. The vitrification point marks the point in conversion at which diffusional limitations preclude propagation of reactive agents. After the vitrification point, the reaction undergoes deceleration. Past that point, the material is no longer capable of relieving polymerization stress[31].

The higher the conversion at vitrification, the better the material can accommodate polymerization stress. SDR presented the highest DC@Rp_{max} among all materials. Interestingly, SDR was also the material with the higher value of Rp_{max}, showing that higher rates of polymerization do not always translate into higher polymerization stress.

Considering that there was too much "noise" in the silorane reaction kinetics, it was not possible to obtain data about Rpmax and vitrification point. But analyzing these reactions it is possible to see that some change in monomer area starts 40 seconds after the photocuring process. This way, we can suppose that this material shows a slow polymerization reaction.

Volumetric Shrinkage and Stress of Polymerization

SIL showed the lowest volumetric shrinkage than the tested methacrylate-based composite resins. The type of the polymerization reaction might be responsible for the reduction of shrinkage[30]. Cationic ring opening polymerization of the cycloaliphatic oxirane moieties would be the reason for silorane-based composites to show this result[27]. This result is in agreement with previous studies[32,33].

VF showed the highest values of volumetric shrinkage and stress of polymerization. It could be related to the composition. VF is a self-adhering flowable composite that, by manufacturer, combines the resin technology of composites and adhesives into one step, needing neither etching nor a bonding agent[21]. The composition of VF is not totally known but to perform the function of adhesive this material contains glycerol phosphate dimethacrylate (GPDM) and low molecular weight methacrylate co-monomers, explicating high values to shrinkage and stress. Even presenting 70 wt% and 48 vol% of filler particles[19], the influence of organic matrix looks to be stronger in the volumetric shrinkage and stress[34].

In this instance, TEC and SDR bulk-fill composites presented similar results to Z100, a conventional composite. This is not in agreement with previous study to evaluate restorations in natural teeth and the TEC and SDR showed a significantly less volumetric shrinkage than the tested conventional composite[35]. The discrepancy between results might be related that in the previous study the materials were placed in situ in a class I cavity preparation, using bonding agents and the evaluation was performed by micro-CT.

However, TEC, SDR and, Z100 showed different values of polymerization stress. These composites contain different filler particles volume (60 vol%, 44 vol% and, 66 vol%, respectively) and SDR, with the lowest volume, obtained the highest stress of polymerization. It can suggest that high filler particles levels are not efficient to reduce polymerization stress, what it is in agreement with other studies[36,37]. The lower values of stress of SDR compared with Z100 and TEC could be explained by its composition. SDR has a polymerization modulator chemically embedded in the resin monomer and it would be able to reduce stress build-up upon polymerization without a reduction in the polymerization rate or conversion[10]. According to the manufacturer, this component is responsible for more linear/branching chain propagation without much cross-linking, and hence slower modulus development. This modulating effect allows extended polymerization reaction without a sudden increase in cross-link density. The extended curing reaction maximizes the overall degree of conversion, what is in agreement with ours degree of conversion results, and minimizes the polymerization stress[10].

5. Conclusion

The tested materials showed different behaviors in relation to physic-chemical properties:

1) SIL showed the lowest values for volumetric shrinkage and stress of polymerization;
2) The best flexural strength was showed by VF;
3) SDR obtained satisfactory performance in all tested properties.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

References

1. Ferracane JL. Buonocore Lecture. Placing dental composites - a stressful experience. Oper Dent. 2008; 33(3): 243-53.
2. Ferracane JL. Resin Composite - State of the art. Dent Mater. 2011; 27(1): 29-38.
3. Ilie N, Hickel R. Investigations on a methacrylate-based flowable composite based on the SDR™ technology. Dent Mater. 201; 27(4): 348-55.
4. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Landuyt KL, Van Meerbeek B, De Munck J. TEM interfacial characterization of an experimental self-adhesive filling material bonded to enamel/dentin. Dent Mater. 2011; 27(8): 818-24.
5. Peutzfeldt A. Resin composites in dentistry: the monomer systems. Eur J Oral Sci. 1997; 105(2): 97-116.
6. Bowen RL. Properties of a silica-reinforced polymer for dental restorations. J Am Dent Assoc. 1963; 66: 57-64.
7. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent. 2003; 28(3): 215-35.
8. Rengo C, Goracci C, Juloski J, Chieffi N, Giovannetti A, Vichi A, Ferrari M. Influence of phosphoric acid etching on microleakage of a self-etch adhesive and a self-adhering composite. Aust Dent J. 2012; 57(2): 220-6.
9. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Investig, 2012 Mar 14. [Epub ahead of print].
10. SureFil SDR Flow Technical Profile. [access 2014 dec 2]. Available in: http://dentsplymea.com/sites/default/files/SDR_OPL_factfile_3p.pdf
11. Technical Profile de Tetric EvoCeram Bulk Fill. [acess 2014 dec 2]. Available in: http://www.ivoclarvivadent.com/zoolu-website/media/document/12247/Tetric+EvoCeram+Bulk+Fill.

12. Boaro LC, Gonçalves F, Guimarães TC, Ferracane JL, Versluis A, Braga RR. Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites. Dent Mater. 2010; 26(12): 1144-50.

13. Gonçalves F, Boaro LC, Ferracane JL, Braga RR. A comparative evaluation of polymerization stress data obtained with four different mechanical testing systems. Dent Mater. 2012; 28(6): 680-6. doi: 10.1016/j.dental.2012.03.004.

14. Bryant RW, Mahler DB. Volumetric contraction in some tooth-coloured restorative materials. Aust Dent J. 2007; 52(2): 112-7.

15. Howard B, Wilson ND, Newman SM, Pfeifer CS, Stansbury JW. Relationships between conversion, temperature and optical properties during composite photopolymerization. Acta Biomater. 2010; 6(6): 2053-9. doi: 10.1016/j.actbio.2009.11.006.

16. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, Leloup G. Physico-mechanical characteristics of commercially available bulk-fill composites. J Dent. 2014; 42(8): 993-1000. doi: 10.1016/j.jdent.2014.05.009.

17. Sabbagh J, Vreven J, Leloup G. Dynamic and static moduli of elasticity of resin-based materials. Dent Mater. 2002; 18(1): 64–71.

18. Leprince J, Palin WM, Mullier T, Devaux J, Vreven J, Leloup G. Investigating filler morphology and mechanical properties of new low-shrinkage resin composite types. J Oral Rehabil. 2010; 37(5): 364–76.

19. Vertise Flow Technical Bulletin. [acess 2014 dec 2]. Available in: http://www.kerrdental.com/cms-filesystem-action/KerrDental-products-techinfo/vertiseflow_techbulletin_34929b.pdf

20. El-Damanhoury H, Platt J. Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. Oper Dent. 2014; 39(4): 374-82. doi: 10.2341/13-017-L.

21. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, Leloup G. Physico-mechanical characteristics of commercially available bulk-fill composites. J Dent. 2014; 42(8): 993-1000. doi: 10.1016/j.jdent.2014.05.009.

22. Flury S, Peutzfeldt A, Lussi A. Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites. Dent Mater. 2014; 30(10):

1104-12. doi: 10.1016/j.dental.2014.07.001.

23. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Investig. 2013; 17(1): 227-35. doi: 10.1007/s00784-012-0702-8.

24. E Asmussen, A. Peutzfeldt. Influence of UEDMA, BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. Dent Mater. 1998; 14(1): 51–6.

25. Ilie N, Durner J. Polymerization kinetic calculations in dental composites: a method comparison analysis. Clin Oral Investig. 2014; 18(6): 1587-96. doi: 10.1007/s00784-013-1128-7.

26. Burtscher P. Stability of radicals in cured composite materials. Dent Mater. 1993; 9(4): 218-21.

27. Goracci C, Cadenaro M, Fontanive L, Giangrosso G, Juloski J, Vichi A, Ferrari M. Polymerization efficiency and flexural strength of low-stress restorative composites. Dent Mater. 2014; 30(6): 688-94. doi: 10.1016/j.dental.2014.03.006.

28. 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; 29(8): 906-12. doi: 10.1016/j.dental.2013.05.008.

29. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of a self-adhesive and four novel flowable composites. J Adhes Dent. 2013; 15(3): 229-36. doi: 10.3290/j.jad.a29530.

30. Par M, Gamulin O, Marovic D, Klaric E, Tarle Z. Raman Spectroscopic Assessment of Degree of Conversion of Bulk-Fill Resin Composites - Changes at 24 Hours Post Cure. Oper Dent. 2014 Oct 2. [Epub ahead of print].

31. Odian, G. Principles of polymerization. 4th ed. New York: John Wiley & Sons; 2004.

32. Yamasaki LC, De Vito Moraes AG, Barros M, Lewis S, Francci C, Stansbury JW, Pfeifer CS. Polymerization development of "low-shrink" resin composites: Reaction kinetics, polymerization stress and quality of network. Dent Mater. 2013; 29(9):

e169-79. doi: 10.1016/j.dental.2013.04.021.

33. Karaman E, Ozgunaltay G. Polymerization shrinkage of different types of composite resins and microleakage with and without liner in class II cavities. Oper Dent. 2014; 39(3): 325-31. doi: 10.2341/11-479-L.

34. Ellakwa A, Cho N, Lee IB. The effect of resin matrix composition on the polymerization shrinkage and rheological properties of experimental dental composites. Dent Mater. 2007; 23(10): 1229-35.

35. Hirata R, Clozza E, Giannini M, Farrokhmanesh E, Janal M, Tovar N, Bonfante EA, Coelho PG. Shrinkage assessment of low shrinkage composites using micro-computed tomography. J Biomed Mater Res B Appl Biomater. 2014 Aug 13. doi: 10.1002/jbm.b.33258. [Epub ahead of print]

36. El-Damanhoury H, Platt J. Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. Oper Dent. 2014; 39(4): 374-82. doi: 10.2341/13-017-L.

37. Ferracane JL. Developing a more complete understanding of stresses produced in dental composites during polymerization. Dent Mater. 2005; 21(1): 36-42.

CAPÍTULO 2

Microtensile bond strength and hardness in depth of materials and restorative technique combinations

Abstract

Objective: To evaluate hardness in depth of different composites classes and microtensile bond strength (µTBS) of Class II cavities filled by different restorative techniques and diverse materials association before and after mechanical fatigue-cycling test.

Materials and methods: Class II occluso-distal cavities (6 x 2 x 4 mm) in extracted human molars were restored using XP Bond (XP) etch-and-rinse adhesive system or silorane-based (SSA) associated to Z100 (Z100), Tetric EvoCeram Bulk Fill (TEC), Surefil SDR flow (SDR), Vertise Flow (VF), and Filtek Silorane (SIL) composites placed by incremental (I) or bulk (B) technique. The tested experimental groups were: XP-Z-B, XP-Z-I, SSA-Z-B, SSA-Z-I, XP-TEC-B, XP-TEC-I, XP-SDR-Z100, SSA-SDR-Z100, VF-Z-B and SSA-SIL-B (n=16). Half of the samples were prepared to obtain sticks and submitted to the µTBS test after 7 days of storage. The other samples were submitted to the mechanical fatigue-cycling test before the µTBS test. Hardness in depth was carried out in restorations with 4.0 mm of depth, removed, included in epoxy resin and submitted to Knoop hardness test (n=3). Results were statically analyzed by ANOVA and Tukey's test ($p<0.05$).

Results: In relation to µTBS results in no-cycled groups, VF-Z-B exhibited the lowest values in occlusal and cervical surfaces (11.44 and 9.80 MPa, respectively), and XP-SDR-Z obtained the highest values in both tested surfaces (28.23 and 38.25 MPa). In cycled groups for the occlusal surface, the group SSA-SDR-Z obtained the lowest results (7.81 MPa), and XP-Z-I and XP-SDR-Z the highest results (23.24 and 21.48 MPa, respectively). To cervical surface, XP-TEC-B exhibited the lowest values (14.29 MPa) and XP-TEC-I and XP-SDR-Z the highest ones (33.15 and 31.90 MPa, respectively). The mechanical fatigue-cycling test affected significantly only the groups XP-TEC-I and SSA-SDR-Z. VF

obtained the lowest top-to-bottom ratio depth of cure results (0.66), while the other composites exhibited less than 20% of reduction.

Conclusion: Incremental technique not allow better bonding strength than bulk technique. The bulk-fill tested materials showed different performances. The combination between silorane-based and methacrylate-based materials did not appear to be promising. The self-adhering material (VF) showed unsatisfactory adhesion and depth of cure.

1. Introduction

During the polymerization of composite resins, the formation of a polymer network results in a denser structure, increasing the volumetric shrinkage[1] and, consequently the shrinkage stress that can cause adhesive failures, cracks in the tooth structure, secondary caries and premature failure of restorations[2]. Furthermore, these composites require a surface pretreatment of enamel and dentin using either an etch-and-rise or self-etch adhesive making the placement of the materials more complex in a technique more sensitive[3].

In order to simplify the clinical application and to reduce the technique sensitivity, the Vertise Flow[4] (Kerr, Orange, USA) self-adhesive composite was introduced to the market as the first flowable resin composite. According to the manufacturer's instructions, self-adhesive composites do not require any pretreatment of the substrate[5] and its self-adhesiveness is based upon the use of acidic monomers that de-mineralizes and simultaneously infiltrates the tooth substrate, resulting in micro-mechanical retention, potential enhanced by additional chemical interaction[5].

To minimize volumetric shrinkage and subsequent shrinkage stress, manufacturers have invested in the development of low-shrinkage restorative composites. Recently, an example of this new material (Filtek^{TM} LS – Low Shrink Posterior) is available for clinical use[6]. According to manufacturer's instructions, Filtek LS requires a specific self-etch adhesive system and can be placed in a bulk increment reducing the working time, volumetric shrinkage and stress[7].

Bulk-fill composites, such as Tetric Evo-Ceram Bulk Fill and SureFill SDR Flow, have also been recently developed to reduce placement time and simplify the procedure. These materials are designed to be placed in 4 mm thick increments, without negatively affecting the mechanical and physical properties[8].

The introduction of these new resin composites allows for an alteration in the restorative technique. Incremental layering has long been accepted as a standard technique for placement of resin-composite in cavity preparations[9]. This technique consists of placing increments of resin-composite material in thickness of 2 mm or less followed by exposure to light curing from an occlusal direction and then repeating increments until the preparation is filled[9,10]. The advantages for this technique are adequate light penetration and subsequent polymerization resulting in enhanced physical and mechanical properties, decreased cytotoxicity[11,12], and reduction of polymerization shrinkage stress[13]. But the incremental technique has disadvantages as the possibility of incorporating voids and contamination between composite layers, bond failures between increments, difficulty in placement because of limited access in conservative preparations, and the long time required to fill the cavity[9, 14]. Thus, the use of bulk-fill techniques is being encouraged because this would substantially simplify restorative procedures and reduce chair time[15].

However, when incremental and bulk-fill techniques are compared, the results are conflicting. A study has reported that the incremental technique produces higher shrinkage stress and cuspal deflection[14]. In contrast, other studies have shown reduced cuspal deflection and higher resin-dentin micro-tensile bond strength with an incremental technique compared to bulk-fill technique in large cavities[16,17]. It should be noted that some of the difference in outcomes might be due to difference in curing protocols employed in these various studies.

The purpose of this study was to evaluate depth of cure and microtensile dentin bond strength of different combinations of materials and restorative techniques, including bulk fill and self-adhesive flowable composites. The hypotheses tested were:

(1) Microtensile bonding strength values would not be affect by the bonding agent;
(2) Different restorative protocols would be similar in microtensile bond strength performance;
(3) Different materials would not be affecting the bonding strength values;
(4) Tested resin composites would show similar results for hardness in depth.

2. Materials and methods

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

Table 1: Materials used in this study.

Resin Composites (Abbreviation)	Organic Matrix	Inorganic Filler	Manufacturer and Batch No.
Z100 (Z100)	Bis-GMA, TEGDMA, 2-benzotriazolylmethylphenol.	Zirconia/sílica: 0.01-3.5μm 85%(wt) and 66% (vol).	3M ESPE N427925
Tetric EvoCeram Bulk Fill (TEC)	Dimethacrylate.	Ba-glass, YbF_3, mixoxide, PPF 81%(wt) and 61%(vol).	Ivoclar Vivadent R49602
Surefil SDR flow (SDR)	Mod UDMA, EBPADMA, TEGDMA.	Ba-Al-F-B-Si glass, Sr-F-Si glass 68%(wt) and 45%(vol).	Dentsply 100506
Filtek Silorane Low Shrink Posterior Restorative (SIL)	Silorane (3,4-epoxycyclohexylethylcyclo-polymethylsiloxane, bis3,4-epoxycyclohexylethyl-phenylmethylsilane).	Quartz radiopaque yttrium fluoride 76%(wt) and 50%(vol).	3M ESPE N436469
Vertise Flow (VF)	GPDM and HEMA	Prepolymerized filler, barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride 70%(wt) and 50%(vol).	Kerr Corporation 4675010

Adhesives (Abbreviation)	Composition	Instructions for Use	Manufacturer and Batch No.
XP Bond Universal Total Etch Adhesive (XP)	Etchant: Caulk 34% Tooth Conditioner Gel (34% phosphoric acid) Primer/Bond: TCB resin, PENTA, UDMA, TEGDMA, BHT, CQ, amorphous silica (0503004020), mixed with SCA (self-cure activator 041203)	Acid etch: 15 seconds, rinse for 15 seconds, blot excesso water using a cotton pellet, do not air-dry Adhesive: Apply uniformly, wait for 20 seconds, dry air for 5 seconds, and 10 seconds of light curing	Dentsply 120113

Silorane System Adhesive (SSA)	Self-Etch Primer: Phosphorylated methacrylates, Bis-GMA, HEMA, water, ethanol, silane- treated silica filler. Bond: Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators and stabilizers.	Primer: application for 15 seconds with black microbrush, followed by gentle air dispersion and 10 seconds of light curing Adhesive: application with green microbrush followed by air dispersion and 10 seconds of light curing	3M ESPE N439118

Abbreviations: Bis-GMA, bisphenol A glycol dimethacrylate; bis-EMA, bisphenol A ethoxylated dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

2.1 Restorative Procedures

One hundred and sixty non-restored, caries free human third molars extracted were used within 3 months after extraction. This study was approved by The Ethics Committee in Research (Register # 029/2012). The teeth were washed and stored in 0.1% thymol solution at 37°C until they will be used. The root surface was embedded in dental stone leaving the crown exposed. After this, the teeth were sectioned horizontally using a diamond saw (Accutom-50; Strues A/S. Ballerup, Denmark) under constant water-cooling to obtain flat and sound dentin surfaces. The sectioned dentin surfaces were then hand-polished with a 600-grit silicon carbide abrasive paper for 30 seconds under running water to obtain a uniform surface.

Class II cavities (6.0 mm wide x 2.0mm deep x 4.0 mm tall) were prepared in the mesial and distal surfaces of each tooth. All of the preparations were done using FG 1016HL spherical diamond burs (KG Sorensen, Cotia, SP, Brazil) with a high-speed hand piece with copious air water spray and finished with a FG 1092F finishing Diamond bur (KG Sorensen). Each bur was replaced after every six preparations. Inner angles of the cavities were rounded and the margins were not beveled. The molars were randomly divided into ten groups (n = 16), as outlined in Table 2.

Table 2: Experimental Groups

Group	Materials and Restorative Technique		
Code	Adhesive	Composite	Technique
XP-Z-B	XP Bond	Z100	Bulk fill
XP-Z-I	XP	Z100	Incremental
SSA-Z-B	SSA	Z100	Bulk fill
SSA-Z-I	SSA	Z100	Incremental
XP-TEC-B	XP Bond	Tetric Evo Ceram Bulk Fill	Bulk fill
XP-TEC-I	XP Bond	Tetric Evo Ceram Bulk Fill	Incremental
XP-SDR-Z	XP Bond	SDR Flow (3 mm) + Z100 (1 mm)	Bulk/Capping Layer
SSA-SDR-Z	SSA	SDR Flow (3 mm) + Z100 (1 mm)	Bulk/Capping Layer
VF-Z-B	Vertise Flow as liner (< 0.5mm)	Z100 (3.5 mm)	Liner/Bulk
SSA-Sil-B	SSA	Filtek Silorane	Bulk

XP Bond (Dentsply) and Silorane System Adhesive (3M ESPE) were applied according to manufacturers' instructions (Table 1).

Light curing was performed using a DEMI^{TM} Plus LED Curing Light (Kerr Corporation, Orange, CA, USA). The irradiance (1170 mW/cm^2) from the light was checked throughout the experiments with a radiometer (Demetron Research; Danbury, CT, USA).

Cavities were surrounded with a metal matrix band, bonded with the respective adhesives, and restored according to each experimental group. In groups XP-Z-B, SSA-Z-B, XP-TEC-B and SSA-SIL-B, the cavities were filled with only one increment and light-cured for 40 seconds. In groups XP-Z-I, SSA-Z-I and XP-TEC-I, the cavities were filled with 4 oblique increments and each increment was light-cured for 10 seconds. In groups XP-SDR-X and SSA-SDR-Z, the cavities were filled with the SDR resin composite first with a 3 mm layer which was light-cured for 30 seconds, and then Z100 composite was applied for the residual height of the cavity in only one increment of 1 mm thickness and light-cured for 10 seconds. For VF-Z-B Vertise Flow group, the self-adhering resin composite was used as a liner. A thin layer (<0.5mm) was brushed onto the entire cavity wall and beveled area with a moderate pressure for 20 seconds and light-cured for 20 seconds. The restorations were built up with a bulk increment of Z100 composite resin and light-cured for 40 seconds. In this way, all restorations received the same total curing time of 40 seconds for a radiant exposure of 46.8 J/cm^2 (measured from the light tip).

Finishing and polishing of all the restorations was done using flexible discs (SofLex Pop-on; 3M ESPE, St. Paul, USA). One operator performed all restorations. After finishing and polishing, the samples were randomly separated into two subgroups (n=8). One group was stored in distilled water for 7 days at 37°C and then submitted to a mechanical fatigue-cycling test. The other group was stored in distilled water for 7 days at 37°C and then submitted to microtensile bond strength testing to dentin.

2.2 Mechanical Fatigue-Cycling Test

After the storage time, the specimens were cyclically loaded in a mechanical fatigue-cycling machine developed at Oregon Health & Science University. This machine employs an eccentric cam driven by a DC motor and loading is applied with dead weights. A 1.5 mm diameter stainless steel ball was attached to the occlusal surface of the restoration with a flowable resin composite that was light-cured for 10 seconds. The specimens were then placed in the fatigue-cycler and loaded with a stainless steel bolt contacting the ball for a total of 200,000 cycles under a cyclic load of 18–85 N at 1.25 Hz. The specimens were constantly bathed in re-circulated 37°C water and, after 200,000 cycles, the specimens were removed from the fatigue machine and prepared to the microtensile bond strength test.

2.3 Microtensile bond strength dentin test

Before the microtensile bond strength test, an additional 2.0 mm resin layer was added on the restorations to increase the length of the sticks for securing to the steel-testing device with cyanoacrylate. To accomplish this, the surface of the restoration was abraded with a finishing diamond bur FG 1092F, cleaned with 37% phosphoric acid and treated with XP Bond adhesive system. The composite used was Venus Diamond (Heraeus Kulzer, GmbH, Hanau, Germany) shade HKOM, light-cured for 20 seconds.

Specimens were sectioned perpendicular to the interface using an automated precision water-cooled diamond saw (Accutom-50) to obtain rectangular sticks. The occlusal and gingival slabs were then further sectioned perpendicularly to produce bonded sticks approximately 1.0 mm^2 in cross section. The sticks were immersed in distilled water at 37ºC and tested after 24 hours.

The tensile testing was performed in an universal testing machine (Model TT-B; Instron, Canton, MA) at a crosshead speed of 0.05 mm/min until failure. The bonded surface area was calculated by using a digital caliper (Mitutoyo, Tokyo, Japan). Each stick was attached to the grips of a microtensile device with a cyanoacrylate resin (Super Glue; Henkel/Loctite, Westlake, Ohio, USA). The failure loads were recorded in Newtons (N), and the bond strength values were calculated in Mega Pascal (MPa) by dividing the failure load by the adhesive surface area (mm^2).

2.4 Hardness in depth test

The resin composites tested were: Z100, Tetric Evo-Ceram Bulk Fill, SDR Flow, Filtek Silorane and Vertise Flow.

One occlusal-distal class II cavity (2.0-mm deep x 4.0-mm tall x 6.0-mm width) was prepared in a third molar. The cavity was lubricated with petroleum jelly and filled with only one increment of the composite and light-cured for 40 seconds. After this, the restoration was removed using a dental probe (n=3). The same cavity was used for all materials to standardize the volume of material and photocuring conditions.

The samples were mounted in epoxy resin and cut through the middle to expose the central region of the restoration. Indentations were sequentially made using a hardness testing machine (Accutom-50). Eight readings were taken from the top to bottom surfaces (at levels of 0.5 mm) under a load of 100 g for 20 s dwell time. The KHN for each depth was recorded as the average of 3 indentations made at the same depth.

2.5 Statistical Analysis

For depth of cure test statistical evaluation was performed with one-way ANOVA and Tukey's test at a 5% level of significance. For microtensile bond strength the statistical analysis was performed with multiple-way ANOVA and Tukey’s test at a 5% level of significance.

3. Results

Results of microtensile bond strength are described in the Table 3. In relation to non-mechanical fatigue cycled groups there was not statistical difference between occlusal and cervical surfaces, except for XP-SDR-Z. Analyzing occlusal surface of these groups, XP-Z-B and XP-SDR-Z showed the highest values while VF-Z-B the lowest ones. On the cervical surfaces similar situation was found, XP-SDR-Z presented the highest values and VF-Z-B the lowest ones.

In mechanical-fatigue cycled groups, only XP-TEC-I and XP-SDR-Z presented difference between cervical and occlusal surfaces. Considering occlusal surface, XP-Z-I and XP-SDR-Z showed the highest values and SSA-SDR-Z the lowest ones. The other groups presented intermediate results. In relation to cervical surface, XP-TEC-I and XP-SDR-Z showed the highest values while XP-TEC-B the lowest ones.

Table 3. Average (standard deviation) of microtensile bond strength (MPa) for tested groups

Cycling	Group	Surface	
		Occlusal	Cervical
No	XP-Z-B	27.67(3.30)Aa	31.71 (4.21) Aab
	XP-Z-I	21.73 (4.54) Aabc	23.01 (3.58) Abcd
	SSA-Z-B	16.51 (3.68) Acd	17.96 (5.55) Ade
	SSA-Z-I	18.11 (5.64) Abcd	20.31 (3.25) Acd
	XP-TEC-B	21.26 (2.91) Aabc	20.03 (2.62) Ad
	XP-TEC-I	*26.99 (5.36) Aab	29.69 (5.61) Aabc
	XP-SDR-Z	28.23 (5.51) Ba	38.25 (5.67) Aa
	SSA-SDR-Z	*21.04 (7.25) Aabc	*22.71 (3.27) Abcd
	VF-Z-B	11.44 (2.39) Ad	9.80 (2.06) Ae
	SSA-SIL-B	15.02 (4.58) Acd	15.74 (2.37) Ade
Yes	XP-Z-B	18.10 (4.02) Aab	22.70 (4.55) Aabc
	XP-Z-I	23.24 (3.91) Aa	26.76 (2.17) Aab
	SSA-Z-B	13.64 (3.25) Aab	17.73 (2.33) Abc
	SSA-Z-I	14.25 (3.57) Aab	14.85 (4.41) Abc
	XP-TEC-B	20.21 (4.03) Aab	14.29 (4.33) Ac
	XP-TEC-I	14.28 (3.52) Bab	33.15 (3.46) Aa
	XP-SDR-Z	21.48 (2.19) Ba	31.90 (4.66) Aa
	SSA-SDR-Z	7.81 (2.46) Ab	16.70 (9.00) Abc
	VF-Z-B	10.94 (4.58) Aab	15.11 (5.02) Abc
	SSA-SIL-B	11.98 (3.57) Aab	14.48 (4.16) Abc

Means followed by different letters (capital in row and lower case in column) showed statistically significant difference ($p \leq 0.05$). *Difference between cycling and no cycling in the same surface and group.

Depth of cure results (KNH) and bottom-to-top ratio (%) are described in the Table 4. The hardness obtained on the bottom surface for Z100, TEC, SDR, and SIL were corresponding to more than 80% of the top. Only VF showed reduction of 34% on the bottom surface. SDR showed the greatest depth of cure (reduction of 11.0 %). In all depths Z100 showed the highest values of Knoop hardness ($p \leq 0.05$).

Table 4. Knoop hardness (KNH) of composites in different depths and bottom-to-top ratio (%)

Depth (mm)	Composites				
	Z100	TEC	SDR	VF	SIL
Top	104.04 (6.95)Aa	65.77 (8.67)Ba	40.88 (0.96)Ca	45.26 (2.32)Ca	54.01 (4.86)BCa
0.5	103.44 (1.41)Aa	63.47 (4.06)Ba	40.12 (1.00)Da	43.37 (2.47)CDab	53.90 (5.69)BCa
1.0	102.48 (1.46)Aab	63.01 (1.15)Ba	39.39 (2.71)Ca	42.08 (2.32)CDab	52.60 (2.43)BCa
1.5	95.38 (1.21)Aab	62.81 (1.90)Ba	39.12 (0.60)Da	41.26 (5.03)Cab	51.50 (2.06)BCa
2.0	93.72 (3.45)Aab	62.13 (1.22)Ba	38.78 (1.20)Da	40.01 (8.54)CDa	51.20 (2.74)BCa
2.5	92.81 (7.55)Aab	60.57 (3.20)Ba	38.07 (1.04)Ca	40.48 (0.67)Cab	50.95 (2.87)BCa
3.0	90.38 (1.14)Aab	59.59 (1.33)Ba	37.62 (1.68)Ca	39.39 (1.97)Cab	48.61 (2.61)BCa
Bottom	89.30 (7.45)Ab	56.73 (1.46)Ba	36.75 (1.35)CDa	30.15 (1.97)Db	48.83 (2.48)BCa
Bottom-to-Top Ratio*	**0.85A**	**0.86A**	**0.89A**	**0.66B**	**0.90A**

Means followed by different letters (capital in row and lower case in column) showed statistically significant difference ($p \leq 0.05$). *A ratio of bottom-to-top surface microhardness over 0.80 indicates adequate depth of cure.

4. Discussion

Microhardness has been suggested as adequate method to evaluate depth of cure of resin composites by hardness in depth test. According previous study, a ratio over 0.80 of bottom-to-top microhardness indicates appropriate depth of cure[18].

Interestingly the depth of cure for bulk-fill materials tested (SDR, TEC and SIL) was improved when compared with the conventional resin composite (Z100) and it is in agreement with previous study[19]. This result might be explained by the improvements in initiator system and increased translucency of these materials[20]. SDR and TEC contains a translucent filler and matrix that allow the light to pass through the material[21,22].

Additionally, the unique combination of filler with SDR patented urethane di-methacrylate resin has been indicated with responsible for providing high depth of cure[23]. Inorganic filler particles of SDR consists in large filler size with polygonal shaped features in comparison with other conventional flowable resin composites. The filler load was increased, but the filler-matrix interface was assumed to be decreased, due to the bigger size of the filler particle, allowing more curing light to transmit through the composite and improving the depth of cure[21].

On the other hand, TEC included Ivocerin (Ivoclar Vivadent), a germanium-based photoinitiator, has a higher photo-curing activity than camphorquinone, due to its higher absorption in the region between 400 and 550 nm. Additionally, this photoinitiator can be used without the addition of an amine as co-initiator and forms at least two radicals able to initiate the radical polymerization. This way, it seems to be more efficient than camphorquinone/amine systems with only one radical having that capability[21]. However, in this study, TEC presented no difference in the microhardness value of the bottom-to-top ratio compared with the conventional nonflowable composite (Table 4).

The VF auto-adhering flowable composite showed the lowest values of depth of cure. There is not another study about this material, and the fact that its composition is not totally known makes difficult to draw a possible explanation. One possible reason for this result is the filler particle size. VF inorganic content consists of nano-sized particles[24] and this might affect directly the properties of the material. First of all, nano-sized particles show a specific surface area bigger than micro-sized particles and, then these particles require a greater amount of the silane to coat them[25]. The additional silane required would be able to decrease the physical-mechanical properties of the resin composites[25]. Another important factor is that the increased content of nano-sized particles could increase the light reflection and scattering inside the bulk of the material, reducing the monomer conversion[25,26]. In addition, the amount of the light that reaches the bottom surface would be insufficient to keep the ratio of bottom-to-top over 0.80. In relation to organic content, VF consists of GPDM and HEMA. About GPDM no data chemical analytic are available what becomes difficult to elucidate fully the behavior of this monomer during photocuring reaction. On the other hand, previous study demonstrated that the addition of HEMA decreased the degree of conversion and showed different polymerization behaviors, due to lower polymer reactivity[27].

The results to dentin microtensile bond strength, when the groups without mechanical cycling were compared in occlusal and cervical surfaces, XP-Z-B and XP-SDR-Z showed the highest values and VF-Z-B the lowest ones (Table 3). VF results were consistent with a previous study[28]. The more reasonable reason for this panorama is based to the composition of VF. Even though there are not available chemical analytical data of VF, GPDM (glycerol phosphate dimethacrylate), the mainly component of organic matrix, is used in a conventional adhesive system (OptiBond FL, Kerr) and some performance data can be found. It is speculated that etching potential of GPDM is rather than its penetrate capacity, what would be responsible for your weak bonding to the substrate[28]. The high inorganic filler content of VF (70 wt%) is also pointed like a complicating factor to the infiltration. VF is highly viscous and its wetting ability looks to be not so satisfactory producing a relatively superficial interaction, which probably contributes to the low microtensile bond strength results.

The great SDR results are in agreement with previous study[29]. The high depth of cure and the good interaction between SDR and XP Bond can be considered to explain this result. Both SDR and XP Bond are manufactured by Dentsply, thus it was expected the good interaction between them.

The great performance of the combination between XP and Z100 was expected due to the fact that the composition of these materials to be compatible. However, the bulk technique showed greater results than incremental technique what was unexpected. Recent study[30] tested two low-shrinkage composite and one conventional found better results for bulk technique to cuspal deformation; however, microtensile bond strength and ultimate tensile strength results were significantly lower in comparison with incremental technique. Assuming the great results of depth of cure to Z100 resin composite, we can speculate that the current study performs the microtensile bond strength test after 7 days of the storage and the specimens was not exposed to any adverse situation, these conditions were not sufficient to microtensile bond strength results have been affected by the increasing of polymerization shrinkage stress associated to bulk fill technique.

Statistically significant differences between occlusal and cervical surfaces were only found to XP-SDR-Z and XP-TEC-I groups. Surprisingly it occurred for the two bulk-fill tested materials and that showed high bottom-to-top ratio. Considering these findings we can speculate that high microhardness values do not always translate in homogeneous bond strength.

Analyzing the groups submitted to the mechanical fatigue-cycling, on occlusal surface XP-Z-I and XP-SDR-Z groups showed the highest values of microtensile bond strength and SSA-SDR-Z the lowest ones. On cervical surface the highest values were found to XP-TEC-I and XP-SDR-Z and the lowest ones to XP-TEC-B.

The method adopted for the fatigue test was chosen because the cycling loading would simulate the clinical mastication on dental material more accurately than other methods[31].

The same way as occurred with no mechanical fatigue-cycling groups, XP-SDR-Z exhibited the highest values followed by XP-Z-I. The combination between XP Bond adhesive system and SDR even after the mechanical fatigue-cycling test continued to show the highest results what can be related to the high degree of conversion of this material. Recent study[32] performed successive measurements at various depths to assess the influence of increasing depth of cure efficiency and SDR registered 74.7% of degree of conversion in 4.0 mm deep. This result was associated to a great light transmittance[33] and to the peculiar photoinitiating system resulting in deeper polymerization.

Even knowing that other properties must be analyzed, the similarity between XP-SDR-Z bulk-fill composite and XP-Z-I conventional material associated to the incremental technique, might mean a significant improvement in the bulk-fill materials and maybe, in the near future, a remarkable evolution of the restorative technique.

Overall, the association between SSA and methacrylate-based composites did not result in positive results. SSA adhesive system consists of self-etch primer and bond, and this system has been specially designed to provide bonding between Filtek Silorane Low Shrink Posterior Restorative and dental substrates[7]. Until this moment, only one study analyzed the effect of the matching between methacylated-based materials and SSA[34] and the results showed bond strength results significantly low. The authors attributed these adverse results to the different chemical compositions of the materials the incompatibility between them[34].

However, the silorane system (SSA-SIL-B) did not register high values of microtensile bond strength. Following the manufacturer instructions, the self-etch primer should be applied for 15 s, dried by gentle air and photocured for 10 s, and then, the bond should be applied and photocured[7]. With a pH of 2.7, the self-etch primer looks to promote a mild etching and demineralization of the dental substrates and additionally, the bond is highly viscous what it can be speculated to be responsible for a weak interaction with the substrates.

In relation to the cervical surface of the mechanical-cycled groups, the bulk-fill materials exhibited the best and the worst results. The problem the polymerization in the bottom seems to be persistent in TEC. When it was applied in incremental technique, the results were really good, but when it was placed in bulk technique the values of microtensile bond strength were low.

An important factor to be highlighted was that the group 9 (VF + Z100) exhibited the highest quantify of pre-test failures after mechanical cycling test. However, even though this technical condition, VF did not present the lowest values for microtensile bond strength, being compared to SIL performance.

5. Conclusions

Incremental technique not allow better bonding strength than bulk technique. The bulk-fill tested materials showed different performances. The combination between silorane-based and methacrylate-based materials did not appear to be promising. The self-adhering material (VF) showed unsatisfactory adhesion and depth of cure.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

References

1. Bowen RL. Properties of a silica-reinforced polymer for dental restorations. J Am Dent Assoc, 1963; 66: 57-64.
2. Jianying L, Fok A, Satterthwaite J and Watts D. Measurement of the full-field polymerization shrinkage and depth of cure of dental composites using digital image correlation. Dent Mater. 2009; 25(5): 582-88. doi: 10.1016/j.dental.2008.11.001.
3. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent. 2003; 28(3): 215-35.
4. Rengo C, Goracci C, Juloski J, Chieffi N, Giovannetti A, Vichi A, Ferrari M. Influence of phosphoric acid etching on microleakage of a self-etch adhesive and a self-adhering composite. Aust Dent J. 2012; 57(2): 220-6. doi: 10.1111/j.1834-7819.2012.01689.x.
5. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Landuyt KL, Van Meerbeek B, De Munck J. TEM interfacial characterization of an experimental self-adhesive filling material bonded to enamel/dentin. Dent Mater. 2011; 27(8): 818-24. doi: 10.1016/j.dental.2011.04.007
6. Boaro LC, Gonçalves F, Guimarães TC, Ferracane JL, Versluis A, Braga RR. Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites. Dent Mater. 2010; 26(12): 1144-50. doi: 10.1016/j.dental.2010.08.003
7. Filtek Silorane Low Shrink Restorative System Technical Profile. [acess 2014 dec 2]. Available in: http://multimedia.3m.com/mws/media/598061O/filtek-silorane-tpp-ebu.pdf?fn=Filtek_Silorane_TPP_EBU.pdf
8. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Investig. 2013; 17(1): 227-35. doi: 10.1007/s00784-012-0702-8.

9. El-Safty S, Silikas N, Watts DC. Creep deformation of restorative resin-composites intended for bulk-fill placement. Dent Mater. 2012; 28(8): 928-35. doi: 10.1016/j.dental.2012.04.038.
10. Pilo R, Oelgiesser D, Cardash HS. A survey of output intensity and potential for depth of cure among light-curing units in clinical use. J Dent. 1999; 27(3): 235-41.
11. Poskus LT, Placido E, Cardoso PE. Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations. Dent Mater. 2004; 20(8): 726-32.
12. Kovarik RE, Ergle JW. Fracture toughness of posterior composite resins fabricated by incremental layering. J Prosthet Dent. 1993; 69(6): 557-60.
13. Lutz F, Krejci I, Barbakow F. Quality and durability of marginal adaptation in bonded composite restorations. Dent Mater. 1991; 7(2): 107-13.
14. Abbas G, Fleming GJ, Harrington E, Shortall AC, Burke FJ. Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments. J Dent. 2003; 31(6): 437-44.
15. Van Ende A, Mine A, De Munck J, Poitevin A, Van Meerbeek B. Bonding of low-shrinking composites in high C-factor cavities. J Dent. 2012; 40(4): 295-303. doi: 10.1016/j.jdent.2012.01.004.
16. Lee MR, Cho BH, Son HH, Um CM, Lee IB. Influence of cavity dimension and restoration methods on the cusp deflection of premolars in composite restoration. Dent Mater. 2007; 23(3): 288-95.
17. Park J, Chang J, Ferracane J, Lee IB. How should composite be layered to reduce shrinkage stress: incremental or bulk filling? Dent Mater. 2008; 24(11): 1501-5. doi: 10.1016/j.dental.2008.03.013.
18. Bouschlicher MR, Rueggeberg FA, Wilson BM. Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. Oper Dent. 2004; 29(6): 698-704.
19. Benetti A, Havndrup-Pedersen C, Honoré D, Pedersen M, Pallesen U. Bulk-Fill Resin Composites: Polymerization Contraction, Depth of Cure, and Gap Formation. Oper Dent. 2014 Sep 11. [Epub ahead of print]

20. 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. doi: 10.1016/j.dental.2012.02.002.
21. Jang JH, Park SH, Hwang IN. Polymerization Shrinkage and Depth of Cure of Bulk-Fill Resin Composites and Highly Filled Flowable Resin. Oper Dent. 2014 Aug 19. [Epub ahead of print]
22. Lassila LV, Nagas E, Vallittu PK, Garoushi S. Translucency of flowable bulk-filling composites of various thicknesses. Chin J Dent Res. 2012; 15(1): 31-5.
23. SureFil SDR Flow Technical Manual. [acess 2014 dec 2]. Available in: http://www.surefilsdrflow.com/sites/default/files/SureFil_Technical_Manual.pdf
24. Vertise Flow Technical Bulletin. [acess 2014 dec 2]. Available in: http://www.kerrdental.com/cms-filesystem-action/KerrDental-products-techinfo/vertiseflow_techbulletin_34929b.pdf
25. Valente LL, Peralta SL, Ogliari FA, Cavalcante LM, Moraes RR. Comparative evaluation of dental resin composites based on micron- and submicron-sized monomodal glass filler particles. Dent Mater. 2013; 29(11): 1182-7. doi: 10.1016/j.dental.2013.09.006.
26. de Moraes RR, Gonçalves Lde S, Lancellotti AC, Consani S, Correr-Sobrinho L, Sinhoreti MA. Nanohybrid resin composites: nanofiller loaded materials or traditional microhybrid resins? Oper Dent. 2009; 34(5): 551-7. doi: 10.2341/08-043-L.
27. Collares FM, Ogliari FA, Zanchi CH, Petzhold CL, Piva E, Samuel SM. Influence of 2-hydroxyethyl methacrylate concentration on polymer network of adhesive resin. J Adhes Dent. 2011; 13(2): 125-9. doi: 10.3290/j.jad.a18781.
28. Poitevin A, De Munck J, Van Ende A, Suyama Y, Mine A, Peumans M, Van Meerbeek B. Bonding effectiveness of self-adhesive composites to dentin and enamel. Dent Mater. 2013; 29(2): 221-30. doi: 10.1016/j.dental.2012.10.001.
29. Juloski J, Carrabba M, Aragoneses JM, Forner L, Vichi A, Ferrari M. Microleakage of Class II restorations and microtensile bond strength to dentin of low-shrinkage composites. Am J Dent. 2013; 26(5): 271-7.

30. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, Soares CJ. Incremental filling technique and composite material--part I: cuspal deformation, bond strength, and physical properties. Oper Dent. 2014; 39(2): E71-82. doi: 10.2341/12-441-L.
31. Lohbauer U1, Belli R, Ferracane JL. Factors involved in mechanical fatigue degradation of dental resin composites. J Dent Res. 2013; 92(7): 584-91. doi: 10.1177/0022034513490734.
32. Par M, Gamulin O, Marovic D, Klaric E, Tarle Z. Raman Spectroscopic Assessment of Degree of Conversion of Bulk-Fill Resin Composites - Changes at 24 Hours Post Cure. Oper Dent. 2014 Oct 2. [Epub ahead of print]
33. Bucuta S, Ilie N. Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites. Clin Oral Investig. 2014; 18(8): 1991-2000. doi: 10.1007/s00784-013-1177-y.
34. Musa AA, Nahedh HA. Incremental Layer Shear Bond Strength of Low-shrinkage Resin Composites Under Different Bonding Conditions. Oper Dent. 2014; 39(6):603-11. doi: 10.2341/13-104-L.

CAPÍTULO 3

Effect of different restorative techniques and materials association on the marginal adaptation of class II cavities

Abstract

Objective: To analyze marginal adaptation of bulk-fill, self-adhering and low-shrinkage composites in Class II cavities before and after mechanical cycling, and to evaluate ultimate tensile strength (UTS) of these materials.

Materials and methods: Fifty Class II cavities (6 x 4x 2 mm) were prepared in extracted human molars and restored using XP Bond (XP) methacrylate-based adhesive or Silorane System Adhesive (SSA) silorane-based (SSA). These adhesive systems were associated to Z100 (Z100), Tetric EvoCeram Bulk Fill (TEC), Surefil SDR flow (SDR), Vertise Flow (VF) or and Filtek Silorane (SIL) composites placed by incremental (I) or bulk (B) technique. The tested experimental groups were: G1: XP-Z-B, G2: XP-Z-I, G3: SSA-Z-B, G4: SSA-Z-I, G5: XP-TEC-B, G6: XP-TEC-I, G7: XP-SDR-Z100, G8: SSA-SDR-Z100, G9: VF-Z-B, and G10: SSA-SIL-B (n=5). After the restorative procedures, the samples were impressed and submitted to 200,000 cycles in mechanical fatigue-cycling test, and new impressions were made to obtain epoxy resin replicas. Scanning electron microscopy evaluation was carried out in cervical, vestibular and lingual margins at 30, 100 and 500x magnification. The replicas were compared and analyzed by the software Image J to measure the discontinuity percentage. To develop the UTS test hourglass (2 mm x 8 mm x 4 mm x 1.5 mm) were made and stressed to failure after 24 hours. Results were statically analyzed by ANOVA and Tukey's test ($p<0.05$).

Results: Marginal adaptation analysis (%) did not show significant alteration before and after cycling for all groups, except to G3 where cracks and gaps were found in the adhesive interface. In relation to UTS test, Z100, FS and SDR showed the highest results followed by TEC. VF showed the lowest values.

Conclusion: The restorative protocols did no affect the marginal adaptation and all materials showed ultimate tensile strength satisfactory to support the stress generated by the mechanical fatigue-cycling test.

1. Introduction

Recently, several new restorative materials have been introduced in the market. Among them there are self-adhering and bulk-fill resin composites that emerged with the promise of reducing the number of the clinical steps and simplify the restorative technique. The self-adhering composites combined the rheological properties of the flowable composite with the adhesive potential of a bonding agent system dispensing any pre-treatment on the substrate[1]. Bulk-fill composites, according to the manufacturer's instructions, can be placed in up 4 mm and would be able to ensure superior margins due to lower volumetric shrinkage and stress of polymerization[2].

Considering that the polymerization shrinkage and stress generated are the main drawback of the methacrylate-based materials[3], silorane-based restorative system was introduced few years ago. According to the manufacturer, the system consists of a self-etch primer bond and silorane resin composite, and this material shows less than 1% of volumetric shrinkage due to the ring-opening silorane chemistry[4].

The real interest in developing lower shrinkage materials is based on the fact that polymerization shrinkage and the stress generated at the interface between restoration and tooth are the main responsible for the cracks on the enamel, cuspal deflection and interfacial debonding[5], factors involved on the marginal adaptation. The marginal quality unsatisfactory is associated to microleakage and recurrent caries, important factors for occurrence of the failure and replacement of the restorations[6].

Noting the current scenario composed of the introduction of several new materials and the suggestion of significant changes on the restorative technique, the purpose of this study was to examine the marginal adaptation of Class II cavities restored with different associations of materials and techniques, and submitted to occlusal forces simulated by mechanical fatigue-cycling test, and to evaluate the ultimate tensile strength of these new materials. The null hypothesis tested was that there would be no differences in marginal adaptation in cavities restored with different techniques before and after mechanical fatigue-cycling test.

2. Materials and Methods

All materials tested in this study were described on the Table 1.

Table 1: Materials used in the study

Material **(Abbreviation)**	Manufacturer	Component	Lot
Z100 **(Z100)**	3M ESPE	Matrix: Bis-GMA, TEGDMA, 2-benzotriazolylmethylphenol. Filler: zirconia/sílica: 0.01-3.5µm 85%(wt) and 66% (vol).	N427925
Tetric EvoCeram Bulk Fill **(TEC)**	Ivoclar Vivadent	Matrix: Dimethacrylate. Filler: Ba-glass, YbF_3, mixoxide, PPF 81%(wt) and 61%(vol).	R49602
Surefil SDR flow **(SDR)**	Dentsply	Matrix: Mod UDMA, EBPADMA, TEGDMA. Filler: Ba-Al-F-B-Si glass, Sr-F-Si glass 68%(wt) and 45%(vol).	100506
Filtek Silorane Low Shrink Posterior Restorative **(SIL)**	3M ESPE	Matrix: silorane (3,4-epoxycyclohexylethylcyclo-polymethylsiloxane, bis3,4-epoxycyclohexylethyl-phenylmethylsilane). Filler: quartz radiopaque yttrium fluoride 76%(wt) and 50%(vol).	N436469
Vertise Flow **(VF)**	Kerr Corporation	Matrix: GPDM and HEMA Filler: prepolymerized filler, barium glass filler, nano-sized colloidal silica, nano-sized Ytterbium fluoride 70%(wt) and 50%(vol).	4675010
XP Bond Universal Total Etch Adhesive **(XP Bond)**	Dentsply	Etchant: 36% phosphoric acid (0506000765) Primer/Bond: TCB resin, PENTA, UDMA, TEGDMA, BHT, CQ, amorphous silica (0503004020), mixed with SCA (self-cure activator 041203)	120113
Silorane System Adhesive **(SSA)**	3M ESPE	Self-Etch Primer: Phosphorylated methacrylates, Bis-GMA, HEMA, water, ethanol, silane- treated silica filler. Bond: Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators and stabilizers.	N439118

Abbreviation: Bis-GMA, bisphenol A glycol dimethacrylate; bis-EMA, bisphenol-polyethylene glycol dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate, EBPDADMA ethoxylated bisphenol-A-dimethacrylate.

2.1 Teeth Preparation

Fifty extracted caries-free third human molars stored in 0.1% thymol solution were used in this study. This study was approved by The Ethics Committee in Research under register # 029/2012). The root teeth were embedded in dental stone and the teeth were sectioned horizontally using a diamond saw (Accutom-50, Strues A/S. Ballerup, Denmark) under constant water-cooling to obtain flat and sound dentin surfaces. The sectioned dentin surfaces were hand-polished with a 600-grit silicon carbide abrasive paper for 30 seconds under running water and analyzed with stereomicroscopic to ensure absence of residual enamel.

Class II cavities (6.0 mm wide x 2.0mm deep x 4.0 mm height) were prepared in the mesial and distal surfaces of the teeth, with 1 mm-cervical margin established below the enamel-cement junction. All preparations were done using FG 1016HL spherical diamond burs (KG Sorensen, Cotia, SP, Brazil) adapted to a high-speed hand piece and water-air spray cooling. The finishing was performed with a FG 1092F finishing Diamond bur (KG Sorensen). Each bur was replaced after six cavity preparations and the inner angles of the cavities were rounded. The dimensions of each preparation were measured using a digital caliper (Mitutoyo Corporation USA, Aurora, IL, USA). The same operator performed all procedures.

2.2 Teeth Restoration

The molars were randomly divided into ten groups (n=5), as outlined in Table 2. Except for the VF-Z-B group, the adhesive procedures were performed with XP Bond and Silorane System Adhesive followed by the manufacturers' instructions. An individual metallic matrix was used to build up the proximal wall. The groups were restored with: four oblique increments (XP-Z-I, SSA-Z-I, XP-TEC-I) or one increment (XP-Z-B, SSA-Z-B, XP-TEC-B, SSA-SIL-B). In the XP-SDR-Z and SSA-SDR-Z groups, 3 mm of the cavities were filled with SDR in one increment and the Z100 was used for complete the cavity. In VF-Z-B group, the cavities were washed and dried with air for 5 seconds, and a thin layer (<0.5 mm) of VF was applied in the walls and beveled area of the cavity for 20 seconds using a brush, and light-cured for 20 seconds. For all restorations, a DEMI^{TM} Plus LED Curing Light (Kerr Corporation, Orange, CA, USA) was used with an irradiance of 1170 mW/cm^2. The light irradiance was checked during the experiments with a radiometer (Demetron Research Corp, Danbury, CT, USA).

All margins were finished with a FG 1092F finishing Diamond bur (KG Sorensen) and flexible disks (SofLex Pop-On, 3M ESPE, St. Paul, USA). After the finishing procedures, impressions of the teeth were made using polyether material (Impregum Soft Polyether Impression Material; 3M ESPE, St. Paul, USA) and epoxy resin replicas were obtained.

Table 2. Experimental groups

Group Code	Adhesive	Composite	Technique (Light-Curing time - second)
G1	XP Bond	Z100	Bulk fill (40 s)
G2	XP	Z100	Incremental (10 s each increment)
G3	SSA	Z100	Bulk fill (40 s)
G4	SSA	Z100	Incremental (10 s each increment)
G5	XP Bond	TEC	Bulk fill (40 s)
G6	XP Bond	TEC	Incremental (10 s each increment)
G7	XP Bond	SDR Flow (3 mm) + Z100 (1 mm)	Bulk (30 s)/Capping Layer (10 s)
G8	SSA	SDR Flow (3 mm) + Z100 (1 mm)	Bulk (30 s)/Capping Layer (10 s)
G9	Vertise Flow as liner (< 0.5mm)	Z100 (3.5 mm)	Liner (20 s)/Bulk (40 s)
G10	SSA	SIL	Bulk (40 s)

2.3 Mechanical Fatigue-Cycling Test

The mechanical fatigue-cycling test was performed 24 h after the restorative procedures. During the storage period, the teeth were kept in distilled water in 37 °C. All specimens were submitted to 200,000 cycles under load of 18–85 N at 1.25 Hz and bathed in re-circulated 37 °C water in a mechanical fatigue-cycling machine (Oregon Health & Science University). This machine employs an eccentric cam drive by a DC motor and loading to be applied with dead weights. A 1.5 mm diameter stainless steel sphere was attached to the occlusal surface of the restoration with a flowable resin composite light-cured for 10 seconds. The specimens were placed in the mechanical fatigue-cycling machine and loaded with a stainless steel bolt contacting the sphere.

After loading, a new set of epoxy resin replicas was obtained, mounted on aluminum stubs and sputter-coated with 50 nm of gold–palladium (Hummer VII, Anatech Ltd, Alexandria, VA, USA). Cervical, mesial and distal margins were evaluated by scanning electron microscopy (Quanta 200, FEI, Hillsboro, OR, USA) performed at 100x magnification for before and after mechanical fatigue-cycling comparison.

To measure the discontinuity percentage of the gaps the software Image J (Research Services Branch, National Institute of Mental Health, Bethesda, Maryland, USA, 2009) was used. The SEM micrographs were loaded in the software and the perimeter of the restoration and the gaps length were measured. The discontinuity percentage (%) was obtained for each sample by the formula:

$$\text{Discontinuity Percentage (\%)} = \frac{\text{Gaps length x 100}}{\text{Perimeter of the restoration}}$$

2.4 Ultimate Tensile Strength Test (UTS)

The materials tested Z100, TEC, SIL, SDR and VF are described on the Table 1.

Hourglass resin composite specimens (2 mm thickness × 8 mm length × 4 mm width × 1.5 mm constriction) were prepared using silicone rubber molds (n=5) sandwiched between polyester strip and photoactivated for 40 seconds. The surface of the sample was polished with 1,200 grit paper to remove any irregularity and was storage for 24 hours at 37 °C in dark and dry containers.

After the storage time, the specimens were attached to a metal jig using a cyanoacrylate gel (Super Bonder, Loctite, Henkel Corp., Rocky Hill, CT, USA) and stressed to failure in an universal testing machine (Instron 4411, Instron Corp, Canton, MA, EUA) with a 500 N load cell and a cross-head speed of 1.0 mm/min. The cross-sectional area of each tested specimen was measured after failure using a digital caliper (Mitutuyo, Tokyo, Japan). The UTS results were calculated by dividing the fracture load values by the surface area and expressed in MPa.

2.5 Statistical Analysis

For ultimate tensile strength and discontinuity percentage statistical evaluation was performed with one-way ANOVA and Tukey's test at a 5% level of significance.

3. Results

Ultimate tensile strength results are presented in the Table 3. Z100, FS and SDR showed the highest results followed by TBF. VF showed the lowest values.

Table 3: Average (standard deviation) of ultimate tensile strength (MPa) for tested composites

Composites	Cohesive Strength
Z100	151.67 (28.67) A
TEC	103.16 (5.20) B
SIL	141.04 (16.54) A
SDR	146.40 (15.64) A
VF	79.43 (12.87) C

Means followed by different letters showed statistically significant difference ($p \leq 0.05$).

The discontinuity percentage results are described in the Table 4. Group 3 exhibited the highest values (12.25%). The other tested groups showed statistically similar results.

Table 4: Average (standard deviation) of discontinuity percentage (%) for the tested groups.

Experimental Groups	Discontinuity Percentage (%)
G1	1.28 (0.18) B
G2	1.38 (0.29) B
G3	12.25 (2.08) A
G4	0.98 (0.12) B
G5	0.76 (0.23) B
G6	0.85 (0.15) B
G7	0.75 (0.08) B
G8	1.18 (0.32) B
G9	1.25 (0.24) B
G10	0.92 (0.14) B

Means followed by different letters showed statistically significant difference ($p \leq 0.05$).

SEM representative micrographs are showed in Figure 1 and 2. Except to G3 all groups showed continuous margins before and after mechanical fatigue-cycling test in the 3 analyzed faces (cervical, lingual and vestibular). In the Figure 1 margin patterns before and after cycling considered satisfactory.

For SSA-Z-B, some gaps in the adhesive interface and cracks could be seen. Figure 2 represents non-continuous margins in the cervical surface.

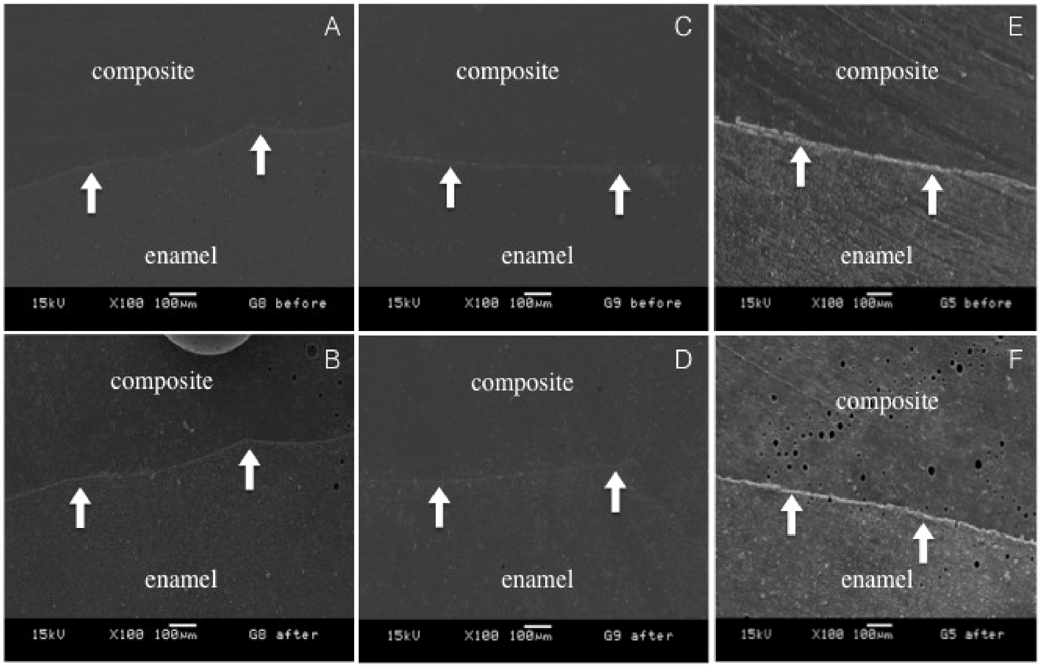


Figure 1. Representative micrographs of the replicas. Satisfactory marginal adaptations before and after mechanical fatigue-cycling test are observed. Arrows indicate the interface between restoration and substrate. It is possible to observe the absence of cracks and gaps.

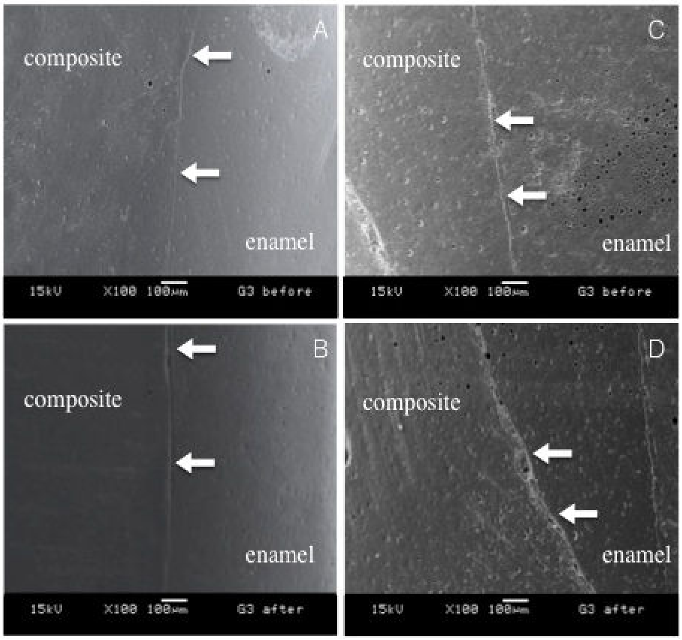


Figure 2. Representative micrographs of the replicas. Marginal discontinuity after mechanical fatigue-cycling test is observed. Comparing 2A and 2B, and 2C and 2D it is possible to note significant gap between restoration and substrate (arrows).

4. Discussion

The results of this research showed that almost all groups demonstrated satisfactory marginal adaptation before and after mechanical fatigue-cycling test. Therefore, the wok hypothesis that no differences in marginal adaptation in cavities restored with different techniques before and after mechanical fatigue-cycling test would be noted was rejected.

These results are not in agreement with previous study where significant differences on margins were detected after thermo-mechanical loading[7]. This fact can be attributed to the difference on the thermo-mechanical loading carried out and adhesive system used.

The results of the current study can be explained by the criterion of rupture established on the Griffith's theory. According this theory, one system can change from intact to broken condition due to a process of continuous decreasing made by a potential energy[8]. In other words, the fracture resistance of a system would be in function of the cohesive force between the atoms. However, the discrepancy between the theoretical cohesive strength and the fracture resistance could be explained by the microscopic defects or cracks that always are presented in the material body. Under a thermo-mechanical tension applied, these defects or cracks would be able to concentrate the tension and increase the surface defect. Therefore, the fracture under tension occurs when the cohesive strength of the material is exceeded where is concentrated the stress. By analogy, it can be speculate that the specimens submitted to the mechanical fatigue-cycling test showed cohesive strength sufficient to avoid the propagations of the cracks leading to marginal gaps. Another important aspect might to be influenced this result would be in relation to defect size. In other words, the surface failures cited by Griffith's theory are smaller than the gaps observed in the current study. This way would be possible to understand that the stress concentration was hampered by the gap size not exceeding the cohesive strength of the interface.

The analysis about ultimate tensile strength showed the conventional (Z100), silorane-based (SIL) and the bulk-fill flowable material (SDR) presented similar results. SIL showed UTS results comparable to a methacrylate-based conventional material what is in agreement to the previous study[9]. The similar performance was attributed only to the lower shrinkage stress of the SIL[9] but it is possible to speculate that both materials present satisfactory degree of polymerization and mechanical properties. The fact of SDR had exhibited high UTS values might to be correlated with the filler particles combination, incorporation of another photoactive group in the methacrylate resin, and higher translucency[10].

Interestingly, TEC obtained only intermediate values of UTS. Recent study evaluated shear bond strength of SDR and TEC[11]. Regardless of substrate and adhesive system, SDR obtained higher results than TEC what was attributed to better wettability and lower viscosity[11].

The satisfactory performance of SDR is in accordance with previous clinical study that evaluated Class I and Class II restorations during three years[12]. The results showed SDR clinical effectiveness comparable with an established conventional nanohybrid material associated to incremental technique. The authors attributed this performance mainly to higher depth of cure[12]. On the other hand, VF obtained the lowest results but this did not result in unsatisfactory marginal adaptation. It is possible to speculate that even though exhibiting lowest values, they were sufficient to support the generated stress.

The results of the present study showed that bulk fill materials presented satisfactory polymerization which is in harmony with previous studies[13,14,15]. These studies found absence of decrease in microhardness values for the bulk fill materials toward increment thickness and improved depth of cure when compared with conventional composites. The authors attribute to modifications of monomer, filler components, polymerization modulators and/or addition of initiation boosters the results presented by bulk-fill materials[13, 14]. These findings added to the results of the present study show the bulk fill restorative technique, in the future, may become to preconized. This alteration would represent economy of time for clinicians and more comfort for the patients. However, other studies should be done to evaluate other important properties of the bulk fill materials.

Bonding agents play crucial role among the all factors involved in gap formation the[14, 16]. It is assumed that stress of polymerization would impose tensile stress to the adhesive interface between the cavity and the composite affecting the marginal integrity. If the tensile stress was superior to bond strength of adhesive system, cracks and gaps will be observed on the interface[16]. In the current research, XP Bond and Silorane Adhesive System showed satisfactory performance except to group SSA-Z-B showed marginal discontinuity in some samples. In a previous study, the etch-and-rinse XP Bond adhesive system was investigated[17] in relation to the degree of conversion, microtensile bond strength and silver nitrate uptake. The results showed higher values of microtensile bond strength and degree of conversion and lower values of nitrate uptake characterizing a really satisfactory performance of this material[17]. These findings support the results of the present research.

In relation to the SSA performance, the current results are in agreement with previous clinical studies that evaluated during two and five years the clinical performance of restorations performed with Filtek Silorane Restorative System and the analysis demonstrated similar clinical performance between this system and other two conventional with respect to marginal quality[3,18].

However, the combination between SSA and Z100 bulk restorative technique did not show a high marginal integrity quality after the mechanical fatigue-cycling test in comparison the other groups. It is possible to speculate that the weak interaction between Z100 (a methacrylate-based composite) and the silorane adhesive system would permit that induced stress on the adhesive interface promotes adhesion failure. In other words, these tensions concentrated in the adhesive interface would result in cracks and gaps. This fact associated to the attenuation of the curing light on the bulk bottom would cause a decreasing of the G3 marginal quality.

The G9 group showed completely restoration debonding after the mechanical fatigue-cycling test. Previous study analyzed adaptation, microleakage and fissure penetration of Vertise Flow when used as a pit and fissure sealant and the results showed unsatisfactory penetration when this material was used following manufacturer's instructions, i.e. any pre-treatment was not carried out on the substrate[19]. This behavior could be attributed to low potential of demineralization of the acid monomers and the difficulty of material penetration due to its high viscosity leading to, probably, low values of bond strength. Previous study[20] tested the microtensile bond strength of VF and found

64% of pre-testing failure and the lowest values of bonding effectiveness in comparison to any adhesive system. In spite of the marginal adaptation of the others 4 samples had been satisfactory, this completely debonding added to low microtensile bond strength results would be able to indicate this material should need some improvements.

5. Conclusion

Within the limitations of this study, it can be concluded that the improvements carried out on bulk-fill materials resulted in comparable properties to the conventional composite what can represent significant alterations in the preconized restorative technique.

Acknowledgments

The authors thank the funding agency CAPES (Process # 1577-12-3) Brazil and the dental companies listed in Table 1 are gratefully acknowledged for the generous donation of materials used for this study.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

References

1. Vertise Flow Technical Bulletin. [access 2014 dec 03]. Available on: http://www.kerrdental.com/cms-filesystem-action/KerrDental-products-techinfo/vertiseflow_techbulletin_34929b.pdf
2. Tetric EvoCeram Bulk Fill Technical Profile. [access 2014 dec 03]. Available on:http://downloads.ivoclarvivadent.com/zoolu-website/media/document/22053/Tetric+EvoCeram+Bulk+Fill?_ga=1.30383551.1668449058.1416252276
3. Baracco B, Perdigão J, Cabrera E, Ceballos L. Two-year clinical performance of a low-shrinkage composite in posterior restorations. Oper Dent. 2013; 38(6): 591-600. doi: 10.2341/12-364-C

4. Filtek Silorane Low Shrinkage Restorative System Technical Profile. [acess on 2014 dec 03]. Available on: http://multimedia.3m.com/mws/media/598061O/filtek-silorane-tpp-ebu.pdf?fn=Filtek_Silorane_TPP_EBU.pdf
5. Ferracane JL. Buonocore Lecture. Placing dental composites--a stressful experience. Oper Dent. 2008; 33(3): 247-57. doi: 10.2341/07-BL2.
6. de Andrade A, Duarte R, Silva FM, Batista A, Lima K, Monteiro G, Montes M. Resin Composite Class I Restorations: A 54-month Randomized Clinical Trial. Oper Dent. 2014; 39(6): 588-94. doi: 10.2341/14-067-C.
7. 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. doi: 10.1016/j.jdent.2014.02.007.
8. Phylosophical Transactions Of the Royal Society of London. Series A, Containing Papers of a Mathematical or Physical Character, vol. 221 (1921), 163-198. [access 2014 nov 27]. Available on: http://www.cmse.ed.ac.uk/AdvMat45/Griffith20.pdf
9. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, Soares CJ. Incremental filling technique and composite material - Part I: cuspal deformation, bond strength, and physical properties. Oper Dent. 2014; 39(2): E71-82. doi: 10.2341/12-441-L.
10. Surefil SDR Technical Profile. [access on 2015 jan 19]. Available on: http://www.surefilsdrflow.com/sites/default/files/SureFil_Technical_Manual.pdf
11. Ilie N, Schöner C, Bücher K, Hickel R. An in-vitro assessment of the shear bond strength of bulk-fill resin composites to permanent and deciduous teeth. J Dent. 2014; 42(7): 850-5. doi: 10.1016/j.jdent.2014.03.013.
12. van Dijken JW, Pallesen U. A randomized controlled three year evaluation of "bulk-filled" posterior resin restorations based on stress decreasing resin technology. Dent Mater. 2014; 30(9): e245-51. doi: 10.1016/j.dental.2014.05.028.
13. Flury S, Peutzfeldt A, Lussi A. Influence of increment thickness on microhardness and dentin bond strength of bulk fill resin composites. Dent Mater. 2014; 30(10): 1104-12. doi: 10.1016/j.dental.2014.07.001.

14. Benetti A, Havndrup-Pedersen C, Honoré D, Pedersen M, Pallesen U. Bulk-Fill Resin Composites: Polymerization Contraction, Depth of Cure, and Gap Formation. Oper Dent. 2014 Sep 11. [Epub ahead of print]
15. El-Damanhoury H, Platt J. Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites. Oper Dent. 2014; 39(4): 374-82. doi: 10.2341/13-017-L.
16. 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. doi: 10.1016/j.dental.2012.11.002.
17. Hass V, Dobrovolski M, Zander-Grande C, Martins GC, Gordillo LA, Rodrigues Accorinte Mde L, Gomes OM, Loguercio AD, Reis A. Correlation between degree of conversion, resin-dentin bond strength and nanoleakage of simplified etch-and-rinse adhesives. Dent Mater. 2013; 29(9): 921-8. doi: 10.1016/j.dental.2013.05.001.
18. Schmidt M, Dige I, Kirkevang LL, Vaeth M, Hørsted-Bindslev P. Five-year evaluation of a low-shrinkage Silorane resin composite material: A randomized clinical trial. Clin Oral Investig. 2014 Apr 1. [Epub ahead of print]
19. Eliades A, Birpou E, Eliades T, Eliades G. Self-adhesive restoratives as pit and fissure sealants: a comparative laboratory study. Dent Mater. 2013; 29(7): 752-62. doi: 10.1016/j.dental.2013.04.005.
20. De Munck J, Luehrs AK, Poitevin A, Van Ende A, Van Meerbeek B. Fracture toughness versus micro-tensile bond strength testing of adhesive-dentin interfaces. Dent Mater. 2013; 29(6): 635-44. doi: 10.1016/j.dental.2013.03.010.

CONCLUSÃO

Considerando os resultados obtidos foi possível concluir que:

Capítulo 1

- Os compósitos *bulk-fill* e o auto-adesivo apresentaram propriedades comparáveis ao material convencional. Porém, em relação ao tensão de contração e contração volumétrica, o material de baixa contração exibiu desempenho significativamente melhor que os demais materiais testados.

Capítulo 2

- A combinação de compósitos a base de metacrilato com sistema adesivo a base de silorano resultou em valores de resistência de união à tração significativamente menores;
- A técnica restauradora *bulk* não representou em todos os casos redução nos valores de resistência de união;
- O compósito Surefil SDR associado ao sistema adesivo XP Bond apresentou resultados consideravelmente superiores aos demais grupos;
- Apenas os grupos XP-SDR-Z e XP-TEC-I apresentaram diferença de valores de resistência de união entre superfície oclusal e cervical;
- O compósito Vertise Flow teve a maior redução de profundidade de polimerização.

Capítulo 3

- Adaptação marginal de todos os grupos testados foi satisfatória antes do teste de ciclagem mecânica;
- Após a ciclagem, apenas o grupo restaurado com sistema adesivo a base de silorano combinado com o compósito Z100 inserido em único bloco apresentou evidentes alterações nas margens;
- Os demais grupos, independente da técnica restauradora e a combinação de materiais testada, apresentaram satisfatória qualidade marginal após a ciclagem.

REFERÊNCIAS

1. Ferracane JL. Resin composite – State of the art. Dent Mater 2011; 27(1): 28-39.

2. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Landuyt KL, Van Meerbeek B, De Munck J. TEM interfacial characterization of an experimental self-adhesive filling material bonded to enamel/dentin. Dent Mater. 2011; 27(8): 818-24.

3. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, Van Meerbeek B. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials. 2007; 28(26): 3757-85.

4. Boletim Técnico de Vertise Flow. [acesso 2014 dez 2]. Disponível em: http://www.kerrdental.com/cms-filesystem-action/KerrDental-products-techinfo/vertiseflow_techbulletin_34929b.pdf

5. Schneider LF, Cavalcante LM, Silikas N, Watts DC. Degradation resistance of silorane, experimental ormocer and dimethacrylate resin-based dental composites. J Oral Sci. 2011; 53(4): 413-19.

6. Ilie N, Hickel R. Silorane-based dental composite: behavior and abilities. Dent Mater. J 2006; 25(3): 445-54.

7. Marchesi G, Breschi L, Antoniolli F, Di Lenarda R, Ferracane J, Cadenaro M. Contraction stress of low-shrinkage composite materials assessed with different testing systems. Dent Mater. 2010; 26(10): 947-53.

8. Naoum SJ, Ellakwa A, Morgan L, White K, Martin FE, Lee IB. Polymerization profile analysis of resin composite dental restorative materials in real time. J Dent. 2012; 40(1): 64-70.

9. Giachetti L, Russo DS, Baldini M, Goracci C, Ferrari M. Reparability of aged silorane with methacrylate-based resin composite: micro-shear bond strength and scanning electron microscopy evaluation. Oper Dent. 2012; 37(1): 28-36.

10. Ferracane JL. Buonocore Lecture. Placing dental composites - a stressful experience. Oper Dent. 2008; 33(3): 247-57.

11. 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): 643-47.

12. Perfil Técnico de Tetric EvoCeram Bulk Fill. [acesso 2014 dez 2]. Disponível em: http://www.ivoclarvivadent.com/zoolu-website/media/document/12247/Tetric+EvoCeram+Bulk+Fill.

13. Gonçalves F, Azevedo CL, Ferracane JL, Braga RR. BisGMA/TEGDMA ratio and filler content effects on shrinkage stress. Dent Mater. 2011; 27(6): 520-26.

14. Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resin-composites: a systematic review. Dent Mater. 2005; 21(10): 962-70.

15. Papadogiannis D, Tolidis K, Lakes R, Papadogiannis Y. Viscoelastic properties of low-shrinking composite resins compared to packable composite resins. Dent Mater J. 2011; 30(3): 350-357.

16. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: A critical literature review. Dent Mater. 2010; 26(2): e78-e93.

17. Satterthwaite JD, Vogel K, Watts DC. Effect of resin-composite filler size and shape on shrinkage-strain. Dent Mater. 2009; 25(12): 1612-15.

18. Sharma RD, Sharma J, Rani A. Comparative evaluation of marginal adaptation between nanocomposites and microhybrid composites exposed to two light cure units. Indian J Dent Res 2011; 22(3): 495.

19. Howard B, Wilson ND, Newman SM, Pfeifer CS, Stansbury JW. Relationships between conversion, temperature and optical properties during composite photopolymerization. Acta Biomater. 2010; 6(6): 2053-9. doi: 10.1016/j.actbio.2009.11.006.

APÊNDICE

Apêndice 1: Materiais e Métodos

Capítulo 1:

Determinação da Tensão de Polimerização por meio do Bioman

O Bioman é composto por célula de carga fixada a uma barra metálica em *cantilever*, sendo o compósito inserido entre o pistão e a placa de vidro em posição perpendicular à célula de carga. O sistema apresenta um orifício onde é encaixado o aparelho fotoativador acoplado a uma ponta de acrílico transparente que permite total reflexão da luz. Uma sonda LVDT é usada para monitorar o deslocamento da extremidade livre da barra e, dessa forma, permitir o cálculo da deformação do sistema (Figura 1).

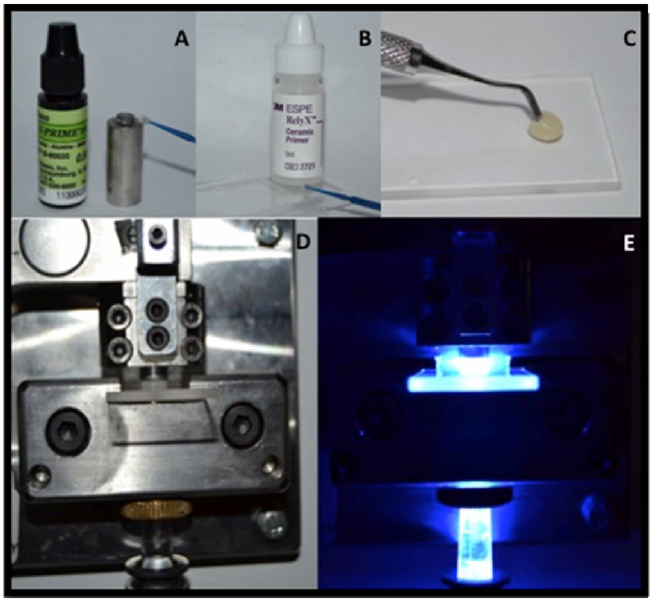
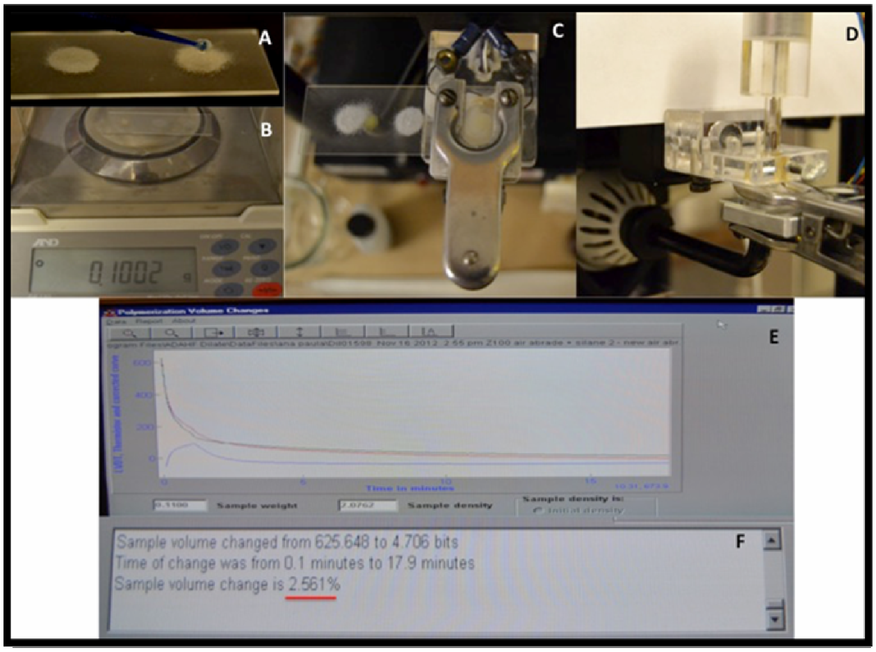


Figura 1. (A) Condicionamento do pistão com Z-Prime Plus (Bisco, Inc). (B) Condicionamento da placa de vidro com RelyX Ceramic Primer (3M ESPE). (C) Inserção do compósito na placa circular de vidro (8,0 mm de diâmetro por 1,0 mm de espessura). (D) Dispositivo *Bioman* em posição, pistão inserido e parafusado, e placa de vidro com a amostra acoplada. (E) Aparelho fotoativador com ponta especial encaixado no dispositivo Bioman.

Determinação da contração volumétrica

O método mais utilizado para determinação volumétrica é por meio do dilatômetro de mercúrio (Figura 2); porém, esse método só pode ser utilizado para mensurar a contração volumétrica de compósitos com consistência regular, sendo necessária prévia determinação da densidade do compósito por meio do método de Arquimedes. Compósitos de consistência fluida escoam quanto a coluna de mercúrio for posicionada, impossibilitando a leitura. Dessa forma, esse método foi realizado apenas para validar os resultados da técnica do *bonded-disc* (Figura 3) que permite que todos os compósitos, independente da consistência, sejam avaliados.



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Sample volume changed from 625.648 to 4.706 bits
Time of change was from 0.1 minutes to 17.9 minutes
Sample volume change is 2.561%
```

Figura 2. (A) Condicionamento da placa de vidro com RelyX Ceramic Primer (3M ESPE). (B) Amostras com massa mensurada em balança analítica. (C) Amostra acoplada ao dilatômetro com presilha metálica. (D) Coluna de mercúrio sendo posicionada em contato com a amostra e aparelho fotoativador acoplado em baixo da placa de vidro. O modelo do aparelho não permite que o fotoativador fosse substituído por aquele utilizado para os demais experimentos. O grau de conversão dessas amostras foi mensurado e comparado com o grau de conversão das amostras fotoativadas com Led Demi Plus (Kerr Corporation) para verificar a qualidade da fotoativação. (E) Gráfico fornecido pelo *software* em função do tempo após o final da monitoramento. (F) Valor da contração volumétrica (%) fornecida pelo *software*.

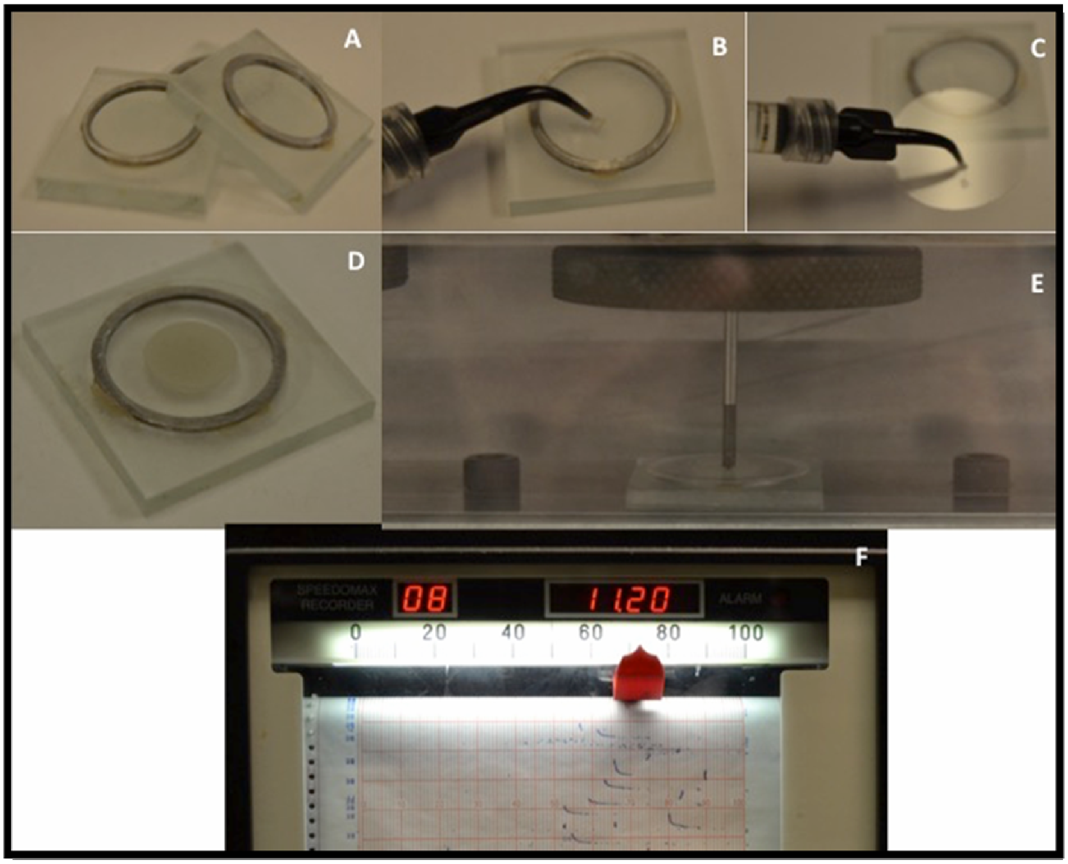

Figura 3. (A) Anel metálico fixado com cera pegajosa na placa de vidro. (B) Superfície da placa de vidro sendo tratada com silano (C) *Coverslip* tratada com silano. (D) Compósito inserido e *coverslip* em posição. (E) Amostra inserida no dispositivo LVDT, com sonda transdutora posicionada no centro da superfície da *coverslip* . (F) *Display* do dispositivo mostrando a voltagem. O valor da contração volumétrica foi calculado em função da diferença das voltagens inicial e final e da espessura da amostras.

Reação cinética

A cinética da polimerização de cada compósito foi mensurada por meio de espectrofotometria infravermelho próximo (NIR) conectado por meio de cabos de fibra óptica a uma *optical bench* (Figura 4) constituída por uma série de lentes colimadoras e condensadoras capazes de captar os raios infravermelhos e promover o monitoramento da intensidade da luz visível transmitida através da amostra.

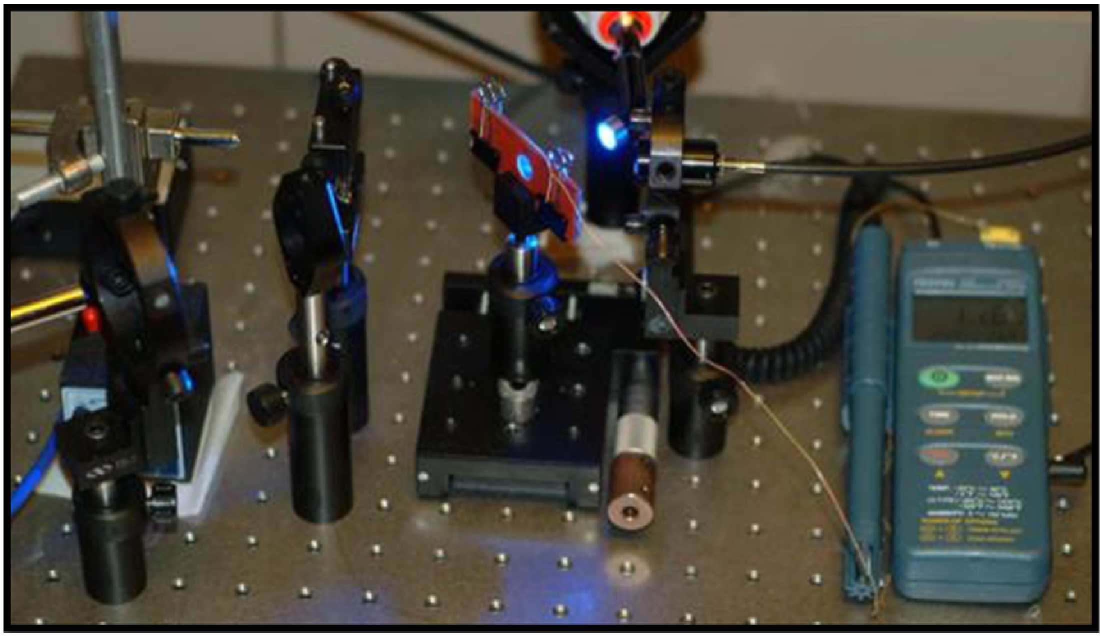

Figura 4. Imagem representativa mostrando os componentes básicos da *Optical Bench* (Howard *et al.*, 2010)[19]*;* aparelho fotoativador, amostra posicionada entre duas placas de vidro, saída da fibra óptica do espectrofotômetro NIR direcionada através de uma lente colimadora, lentes condensadoras, fibra óptica de retorno do NIR, filtro de densidade neutra, entrada da fibra óptica UV/Vis e termômetro digital.

Capítulo 2:

Confecção das amostras para o teste de profundidade de polimerização

Para a análise da profundidade de polimerização, uma cavidade de Classe II ocluso-distal (5,0 x 6,0 x 2,0 mm) foi confeccionada com ponta diamantada esférica em alta rotação. Um porta-matriz com matriz metálica foi adaptado e a cavidade isolada com vaselina em pasta, sendo o excesso removido com película de papel absorvente. Os compósitos foram inseridos em único incremento e fotoativados por 40 segundos. A restauração foi removida da cavidade, o topo identificado com caneta esferográfica preta e incluída em *stub* de resina epóxica que, posteriormente, foi cortado com cortadeira metalográfica para expor a região central interna da restauração (Figura 5).

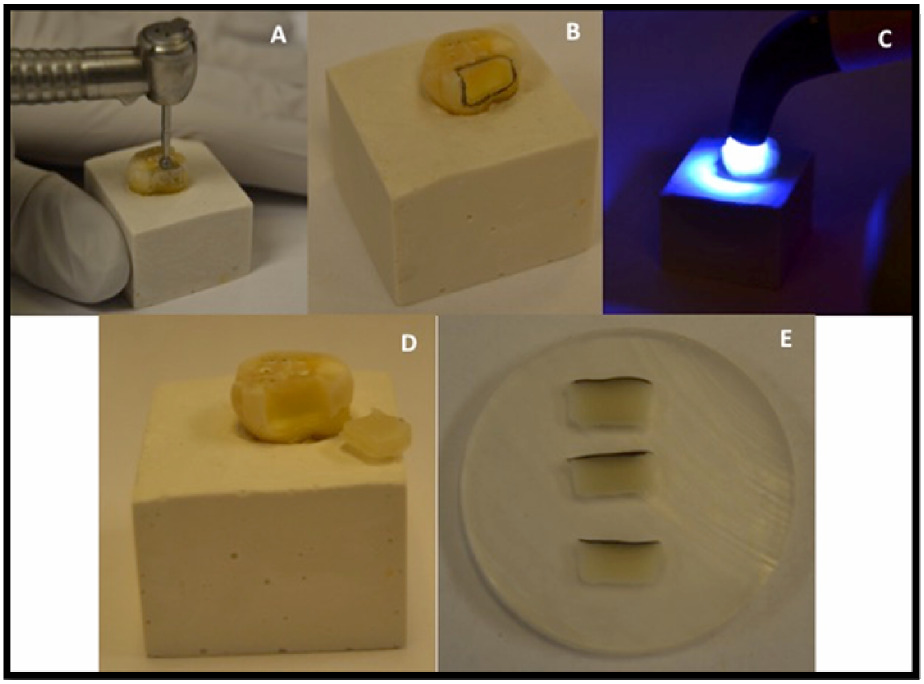

Figura 5. (A) Ponta diamantada utilizada para confecção das cavidades. (B) Cavidade confeccionada. (C) Compósito sendo fotoativado. (D) Restauração removida da cavidade. (E) Aspecto dos *stubs* com as restaurações.

Teste de ciclagem mecânica

Para simulação da mastigação foi utilizada a máquina para teste de ciclagem mecânica desenvolvida pela Oregon Health & Science University (Figura 6).

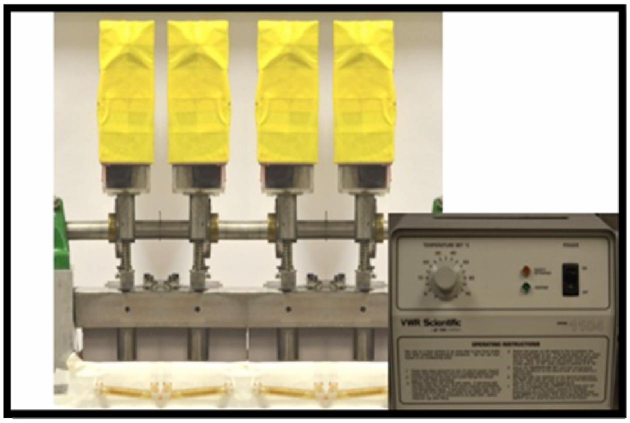

Figura 6. Máquina de ensaio para ciclagem mecânica desenvolvida pela OHSU.

As amostras são constantemente banhadas por água mantida a 37°C. Essa máquina funciona com um motor de corrente continua rotativa, sendo os pesos suportados por um conjunto de molas (Figura 7). O parafuso de aço conectado ao sistema é colocado de maneira cíclica em contato com a esfera acoplada à restauração (Figura 8).

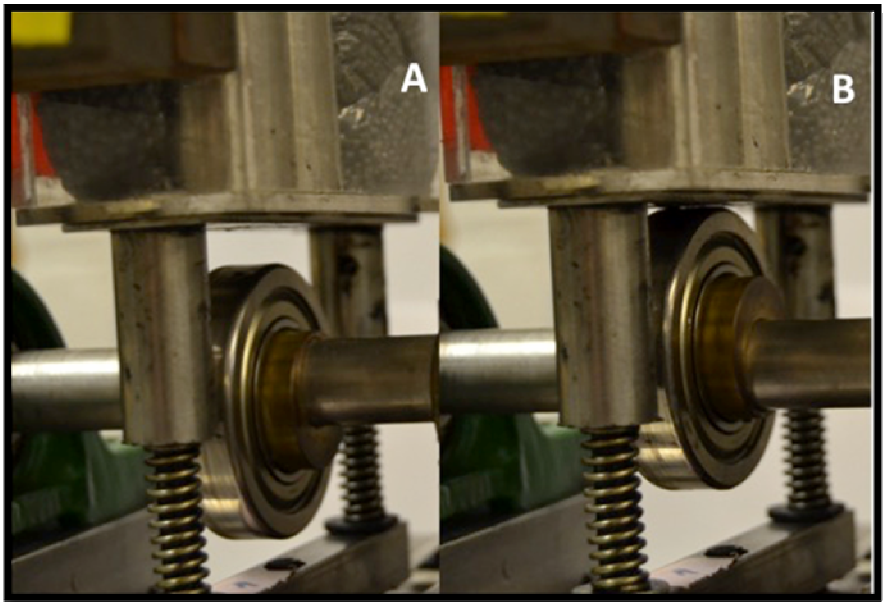

Figura 7. Pesos suportados pelo conjunto de molas. (A) Motor rotativo com movimento em posição centralizada. (B) Motor rotativo em movimento em contato com o nicho onde estão alojados os pesos.

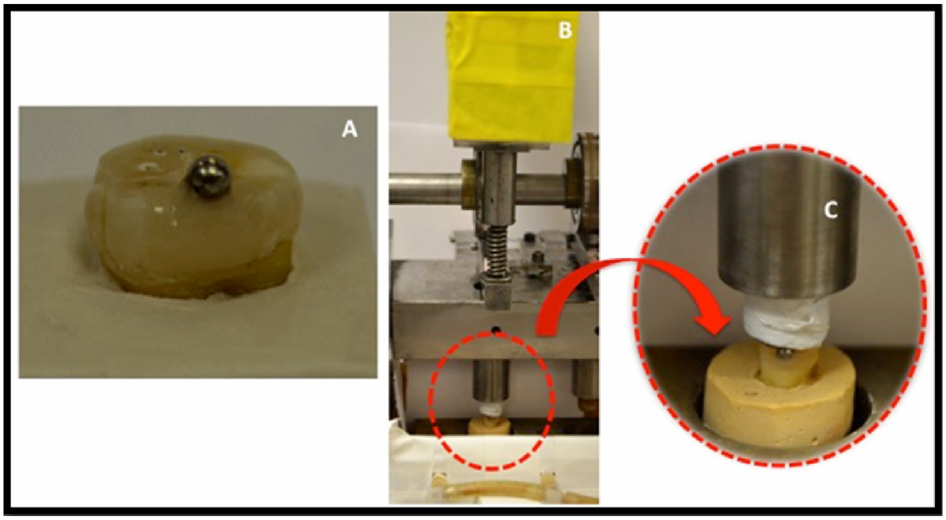

Figura 8. (A) Superfície oclusal da restauração com a esfera de aço anexada. (B) Amostra inserida na máquina de ensaio para ciclagem mecânica. (C) Parafuso de aço em contato com a superfície da esfera.

Preparo dos palitos para o Ensaio de Resistencia de União à Microtração

Antes das amostras serem cortadas, um prolongamento com resina composta foi adicionado (Figura 9) a fim de aumentar o comprimento dos palitos e, dessa forma, facilitar a fixação no dispositivo para o ensaio de resistência de união à microtração. Após essa etapa, as amostras foram cortadas perpendicularmente ao longo eixo do dente e, então, cada fatia foi identificada com cores diferentes, e o processo de corte era reiniciado para obter os palitos com cerca de 1 mm^2 de área de secção transversal.

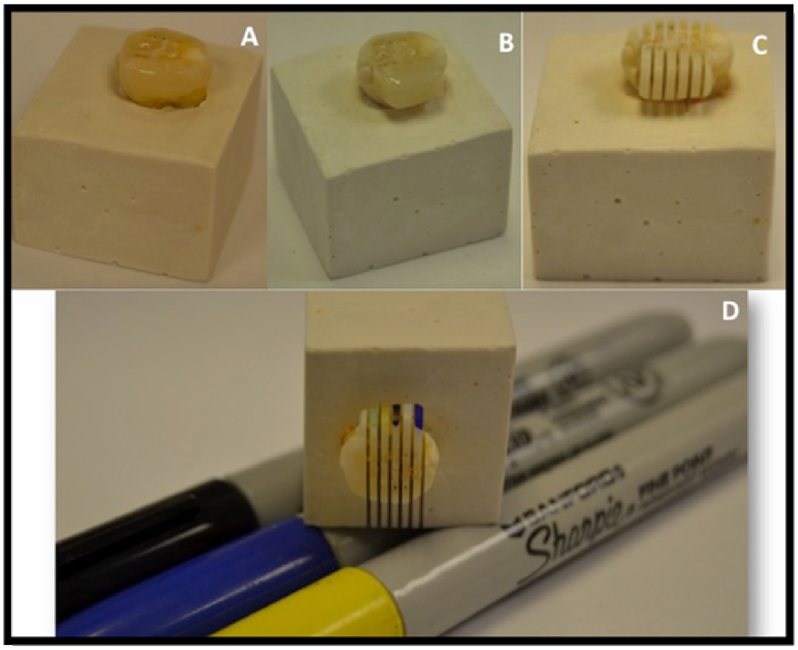

Figura 9. (A) Restauração após acabamento e polimento (B) Prolongamento de resina composta. (C) Primeira sequência de cortes realizada perpendicularmente ao longo eixo do dente. (D) Fatias identificadas com cores diferentes.

ANEXO

Comitê de Ética em Pesquisa - Certificado 30/11/14 02:14

COMITÊ DE ÉTICA EM PESQUISA
FACULDADE DE ODONTOLOGIA DE PIRACICABA
UNIVERSIDADE ESTADUAL DE CAMPINAS

CERTIFICADO

O Comitê de Ética em Pesquisa da FOP-UNICAMP certifica que o projeto de pesquisa **"Estudo da adaptação e das propriedades físicas e mecânicas de compósitos restauradores associados aos sistemas adesivos convencional e à base de silorano"**, protocolo nº 029/2012, dos pesquisadores Ana Paula Piovezan Fugolin e Simonides Consani, satisfaz as exigências do Conselho Nacional de Saúde - Ministério da Saúde para as pesquisas em seres humanos e foi aprovado por este comitê em 27/06/2012**.**

The Ethics Committee in Research of the Piracicaba Dental School - University of Campinas, certify that the project **"Study of adaptation and physical and mechanical properties of resin composites associated with conventional and silorane based adhesive systems"**, register number 029/2012, of Ana Paula Piovezan Fugolin and Simonides Consani, comply with the recommendations of the National Health Council - Ministry of Health of Brazil for research in human subjects and therefore was approved by this committee on Jun 27, 2012.

Lívia M. A. Tenuta
Profa. Dra. Lívia Maria Andaló Tenuta
Secretária
CEP/FOP/UNICAMP

Prof. Dr. Jacks Jorge Junior
Coordenador
CEP/FOP/UNICAMP

Nota: O título do protocolo aparece como fornecido pelos pesquisadores, sem qualquer edição.
Notice: The title of the project appears as provided by the authors, without editing.

http://www.fop.unicamp.br/cep/sistema/certificado.php?Protocolo=029/2012&Id=1805&Passo=2&DataPar=2012-06-27 Página 1 de 1