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CAIO JUNJI TANAKA

AVALIAÇÃO DA EFETIVIDADE DE RESINAS COMPOSTAS BULK-FILL

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"O que você fizer, será mais importante do que aquilo que conseguir.
E como você se sentir por isso, será ainda mais importante do que o que tiver feito."
(Jerry Gilles)

RESUMO

A presente dissertação teve o objetivo de avaliar a efetividade de resinas compostas Bulk-Fill (RCBF) através de dois estudos, testando a resistência à degradação, e os efeitos de diferentes fontes de luz na formação de fendas, polimerização e microdureza Knoop (KHN). Quando avaliada a resistência à degradação, um total de 60 espécimes de resina composta (n=10) foram preparados utilizando uma matriz, recebendo um único incremento de RCBF e dois incrementos de resina convencional (RCC), sendo ativados por luz de acordo com a recomendação do fabricante. Os fatores testados foram: resina composta em 3 níveis (RCC como controle, RCBF fluida e RCBF regular), meio de degradação in vitro em 2 níveis (imersão em 100% de etanol ou água), tempo em 4 níveis (linha de base de controle; e imersão por 24 horas, 7 ou 30 dias) e profundidade com medidas de KHN em 7 níveis (0,5; 1,0; 1,5; 2,0; 2,5; 3,0 e 3,5 mm da superfície irradiada). Em cada amostra, foram realizados 3 indentações Knoop em cada profundidade, com 25g de carga por 20s. Ambas as imersões em água destilada e etanol são capazes de degradar RCC e RCBF. Degradação severa foi observada após a imersão em etanol. Na avaliação dos efeitos de diferentes fontes de luz na formação de fendas, fotopolimerização e KHN, foram avaliados dois fatores: RCBF (Filtek Bulk-Fill Flow [FBF]; e Palfique Bulk-Fill Flow [PBF] - e fonte de luz (Halógena Optlux [QLF]; LED monowave Elipar [MW] e LED polywave Valo [PW]) compondo 6 grupos experimentais. As matrizes foram fabricadas com resina composta fluida, tratadas com jato de oxido de alumínio, condicionadas com ácido fosfórico 35% (K-Etchant gel), seguido de um primer cerâmico (Clearfil Ceramic Primer) e adesivo Clearfil SE BOND 2. As RCBF foram inseridas em incremento único e fotoativadas. Os espécimes foram observados por tomografia de coerência óptica e mensurada a área média das fendas no assoalho da cavidade. Em seguida foram realizadas imagens por Microscopia Confocal Laser e Eletrônica de Varredura, confirmando a presença das fendas. Dependendo das diferentes fontes de luz e RCBF, vários graus de desenvolvimento de fendas foram observados. O compósito FBF apresentou a menor formação de fenda quando ativadfo pela unidade de MW.

Palavras-chave: *resina composta, degradação, fontes de luzes, fendas*

ABSTRACT

The aim of this study was to evaluate the effectiveness of Bulk-Fill resin composites (BFRC) by two studies, testing the resistance to degradation, and the effects of different light curing units on gap formation, polymerization and Knoop microhardness (KHN). When evaluated for resistance to degradation, a total of 60 composite resin specimens (n=10) were prepared using a matrix, receiving a single increment of BFRC and two increments of conventional resin composites (CRC). They were activated according to the manufacturer's recommendation. The factors tested were: composite resin in 3 levels (CRC as control, BFRC fluid and regular BFRC), degradation in vitro in 2 levels (immersion in 100% ethanol or water), time in 4 levels (baseline of and depth with KHN measures in 7 levels (0.5, 1.0, 1.5, 2.0, 2.5, 3.0 and 3.5 mm radiated surface). In each sample, three Knoop indentations were made at each depth, with 25g of load for 20s. Both immersions in distilled water and ethanol are able to degrade CRC and BFRC. Severe degradation was observed after immersion in ethanol. In the evaluation of the effects of different light curing units on gap formation, polymerization and KHN, two factors were evaluated: Filtek Bulk-Fill Flow (FBF) and Palfique Bulk-Fill Flow (PBF) Optlux [QTH], LED monowave Elipar [MW] and LED polywave Valo [PW]) in 6 experimental groups. The matrices were made with flowable composite resin, treated with aluminum oxide spray, conditioned with phosphoric acid 35% Etchant gel), followed by a ceramic primer (Clearfil Ceramic Primer) and Clearfil SE BOND 2 adhesive. RCBF were inserted in a single increment and photoactivated. The specimens were observed by optical coherence tomography and the mean area of the The images were then analyzed by Confocal Laser and Scanning Electron Microscopy, confirming the presence of the gaps. Depending on the different sources of light and BFRC. The composite FBF showed the smallest slit formation activated by MW.

Keywords: *composite resin, degradation, light sources, cracks*

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1- INTRODUÇÃO

As resinas compostas convencionais devem ser inseridas nas cavidades em incrementos de no máximo 2mm com o objetivo de reduzir os efeitos da contração de polimerização (Lazarchik et al. 2007; Van Ende et al. 2013). A contração de polimerização resulta da conversão dos monômeros em uma rede de polímeros após a fotoativação (Van Ende et al. 2013). Essa contração pode induzir estresse e tensão nas paredes da cavidade do preparo e no corpo da restauração podendo resultar ainda em deflexão de cúspides, fendas na linha de união e fraturas das bordas do esmalte dental no angulo cavo-superficial que podem resultar em sensibilidade pós operatória, microinfiltração e subsequente desenvolvimento de cárie secundária em função do risco de cárie do paciente (Park et al. 2008; Van Ende et al. 2012).

Diversas técnicas clínicas tem sido sugeridas para redução do estresse da contração de polimerização, como o controle da intensidade da luz, o uso da "técnica sanduiche" com ionômeros de vidro, o uso de resinas compostas fluidas como base; o uso de resina composta com pouco monômero diluente, a redução das partículas de carga por meio da nanotecnologia e o uso de resinas sem metacrilato (Ferracane, 2005; Ilie, Hickel 2011). Entretanto, nenhum desses métodos demonstrou resultados tão efetivos quanto os apresentados pela técnica incremental na redução do estresse da contração de polimerização (Park et al 2008).

A fim de superar clinicamente, ou reduzir a contração de polimerização, a técnica de preenchimento incremental é obrigatória para restaurações de resinas compostas (Lutz et al., 1986). Foi sugerido que se o volume de resina fosse reduzido em cada incremento, usando incrementos oblíquos ou horizontais de 2 mm de espessura, poderia diminuir as tensões de contração na interface dente-restauração.

Como a magnitude do estresse da contração de polimerização é afetado pela configuração cavitária, a restauração com a técnica incremental permite que a inserção contínua de incrementos, polimerizados a cada inserção, até que a cavidade seja preenchida, diminua a tensão final de polimerização a cada camada inserida (Lazarchik et al., 2007; Nayif et al., 2008; El-Safty et al., 2012; Flury et al., 2012; Van Ende et al., 2012; Tiba et al., 2013). Dessa forma é reduzido o estresse de contração de polimerização visto que o mesmo é o resultado da razão da área de

adesão do incremento em relação a área não aderida obtendo-se um baixo fator C (Lazarchik et al., 2007).

Além disso, o uso de incrementos com menos de 2 mm de espessura permite uma penetração de luz adequada para a subsequente ativação da resina composta, resultando em melhores propriedades físicas, melhor adaptação marginal e redução da citotoxicidade da resina composta.

Entretanto, a técnica incremental apresenta algumas limitações como: (i) a possibilidade de incorporação de bolhas ou detritos entre as camadas, (ii) há um aumento da probabilidade de falhas adesivas entre os incrementos, (iii) há uma maior dificuldade de inserção em preparos extremamente conservadores devido ao acesso e (iv) aumenta o tempo clínico em virtude da necessidade de polimerização de cada camada (Abbas et al., 2003; Sarrett 2005; Lazarchik et al., 2007; El-Safty et al., 2012).

Em função disso, novas resinas compostas com contração de polimerização reduzida têm sido desenvolvidas e inseridas no mercado odontológico com a indicação para restauração pela técnica *Bulk-Fill*, de incremento único. Esses materiais apresentam indicação para inserção em camadas com espessura de 4 a 5 mm, e em seguida são polimerizados em uma única etapa (Czasch e Ilie, 2013; Tiba et al., 2013; Ilie et al., 2013). Como vantagens, eles se apresentam como uma opção para realização rápida de restaurações diminuindo expressivamente o tempo clínico e estão sendo amplamente recomendados para restaurações diretas e função dessa capacidade de compensar o alto fator C de cavidades em dentes posteriores, permitindo ainda em alguns casos, que seja possível sua inserção em uma consistência fluida que se adapta melhor as paredes do preparo (Park et al., 2008).

Estudos demonstram que a contração de polimerização das resinas *Bulk-Fill* é estatisticamente menor que das resinas compostas convencionais nano e microhíbridas e das resinas a base de silorano (Burgess et al., 2010; Ilie, Hickel, 2011, Tiba et al., 2013). A tecnologia empregada para a redução da contração de polimerização foi obtida de diferentes formas pelos fabricantes, envolvendo aumento das características de translucidez, redução do módulo de elasticidade, aumento da concentração de partículas de carga, uso de monômeros que controlam a cinética de polimerização, uso de novos fotoiniciadores mais efetivos e até associação com métodos mecânicos para alterar a consistência do compósito (Ilie, Hickel 2011; Czasch and Ilie, 2013; Van Ende et al. 2013).

De acordo com Tiba et al. (2013), algumas questões não estão claras em relação as resinas *Bulk-Fill*, dentre elas, não se sabe ao certo se a luz fotoativadora é capaz de penetrar toda a espessura total de material inserido por incremento único e se a mesma será capaz de polimerizar adequadamente na parte mais profunda da cavidade (Nayif et al. 2008; Tiba et al. 2013).

Observa-se na literatura que uma resina composta convencional inserida em uma cavidade proximal classe II pela técnica incremental ou de incremento único e exposta a luz direta pela face oclusal apresenta valores de dureza que decrescem da oclusal para a parede cervical (Poskus et al., 2004). Flury et al. (2012), observou *in vitro* que o grau de conversão das resinas *Bulk-Fill* também diminui quando se distância da fonte polimerizadora (Flury et al., 2012).

Assim, se a região de base da resina não recebe luz o suficiente para completa polimerização, as propriedades necessárias para resistir ao meio bucal serão prejudicadas. Apesar de que Roggendorf et al. (2011), não tenham observado diferenças estatísticas no desenvolvimento de fendas e na integridade marginal no esmalte dental e dentina de restaurações com resinas *Bulk-Fill* (4 mm), antes e após um desafio térmico-mecânico, sabe-se que de modo geral as RBCs sofrem degradação hidrolítica pela absorção de água, o que pode torná-las mais propensas ao desgate, por perder propriedades mecânicas incluindo a microdureza (Mayworm et al., 2008; Delaviz et al., 2014; Münchow et al., 2014). Assim, substâncias ingeridas durante a alimentação como sucos e bebidas esportivas (Erdemir et al., 2013) podem provocar a perda de dureza de resinas compostas resultando em sua degradação.

Simulando modelos *in vitro* de degradação, estudos observam que as resinas compostas sofrem degradação quando imersas em soluções com pH ácido, como ácido lático, propiônico, acético em diferentes concentrações (Münchow et al., 2014), assim como em álcool absoluto por 24h ou 7 dias resultando em redução de microdureza (Cavalcante et al., 2011; Fonseca et al., 2013).

Além disso, a falta de integridade e o desenvolvimento de fendas na interface adesiva dente-restauração prejudica o sucesso de uma restauração com resina composta (Bakhsh et al., 2011; Opdam et al., 2014). A restauração com resina composta é desafiada desde sua ativação devido à tensão intrínseca de contração de polimerização, pois a distância intermolecular diminui à medida que ocorre a contração de polimerização, causando uma redução volumétrica variando de 1 a 6%

(Labella et al., 1999; Ferracane, Hilton 2016). Assim, se a tensão de contração exceder a força de união na interface dente-restauração, uma fenda será desenvolvida (Sampaio et al., 2015). Além disso, essas tensões podem aumentar com o tempo, causando problemas nas margens da cavidade (Yamamoto et al., 2015). Fendas e margens abertas podem acelerar a deterioração do material restaurador e levar a efeitos deletérios, como infiltração marginal, sensibilidade pós-operatória e contribuir para o desenvolvimento da cárie secundária (Benetti et al., 2015) (Almeida Junior et al., 2017).

Há uma preocupação com esse estresse de contração e a maneira pela qual a sobrevivência a longo prazo da restauração, desde a adaptação marginal da restauração dentária, é considerada uma exigência clínica para que o sucesso do tratamento ocorra inevitavelmente em materiais resinosos (Jang et al. 2015; Bakhsh et al., 2011). Vários fatores interferem na contração de polimerização e estão relacionados a características moleculares dos compósitos como monômeros, sistemas de iniciação, teor de cargas inorgânicas, módulo de elasticidade ou ao fator C da cavidade, substrato a ser aderido, irradiância à luz por unidade de cura e grau de conversão (Labella et al., 1999; Lim et al, 2002; Ferracane, 2005; Feilzer et al., 2005; Venhoven et al., 2003; Sampaio et al., 2015; Papadogiannis et al., 2015; Braga et al., 2005; Versluis et al., 1996; Atria et al., 2017).

Considerando que a grande vantagem das resinas compostas Bulk-Fill é a maior profundidade de cura, até 4 mm, menor desenvolvimento de contração volumétrica do que as resinas compostas convencionais (Benetti et al., 2015) obseva-se que um estudo mostrou o desenvolvimento de fendas no assoalho cavitário de restaurações de classe I (Hayashi et al., 2017). A formação de falhas está relacionada ao grau de polimerização, que também é influenciado pela fonte de ativação da luz. Muitas unidades de foto ativação foram desenvolvidas, cada uma delas com diferentes comprimentos de onda e intensidades de potência (Benetti et al., 2015). A eficiência da luz geralmente é avaliada por testes de profundidade de cura e microdureza como uma fonte indireta do grau de conversão (Ilie e Stark, 2015). Atingir um grau adequado e uniforme de conversão de monômeros é tão essencial para o sucesso e a longevidade das restaurações compostas quanto evitar o desenvolvimento de fendas (Al-Ahdal et al., 2015). O grau de cura das resinas compostas pode ser relacionado à estabilidade dimensional, estabilidade de cor e biocompatibilidade (Maghaire et al. 2018; S et al. 2006).

Em relação a avaliação do selamento marginal de cavidades, Makishi et al. em 2011 demonstrou a eficácia da tomografia de coerência óptica (SS-OCT) através de imagens 3D para detectar fendas em torno das restaurações de resina composta nos dentes extraídos. A Tomografia de coerência óptica (OCT) desenvolvida por Fujimoto et al. em 1991 pode construir imagens através de interferência de onda que ocorre quando a luz de duas fontes são utilizadas. Isto pode detectar seletivamente a luz refletida e não é afetada pelo espalhamento, permitindo assim a recuperação de imagens detalhadas do tecido biológico *in vivo*, que não requer o corte e processamento dos espécimes e permite a visualização de microestruturas de tecidos e biomateriais em tempo real como o desenvolvimento da fenda.

Sabemos que não há relatos clínicos sobre a influência de diferentes dispositivos de fonte de luz de fotoativação no desenvolvimento de fendas marginais com resinas compostas Bulk-Fill. Da mesma forma, existem estudos poucos estudos *in vitro* demonstrando a efetividade de resinas Bulk-Fill em resistir ao meio bucal, restando apenas na literatura poucos estudos clínicos.

Os achados clínicos de restaurações realizadas com resinas *Bulk-Fill*, de van Dijken e Pallesen (2014, 2016), demonstram que em até 5 anos restaurações de resina *Bulk-Fill* de baixa viscosidade apresentam uma taxa de sobrevida similar a resinas convencionais. Cantekin, Gumus (2014), observaram que restaurações realizadas com resinas *Bulk-Fill* em dentes decíduos que sofreram pulpotomia apresentam performance clínica similar a resinas convencionais por 12 meses. Bayraktar et al. (2016) demonstraram que restaurações realizadas com resinas *Bulk-Fill* de baixa e alta viscosidade apresentam longevidade clínica similar a convencional por 12 meses.

Dessa forma, apesar de promissores os resultados iniciais, não existe evidência científica de resultados clínicos e laboratoriais que demonstrem se as resinas *Bulk-Fill* apresentam vantagens significativas em sua indicação, vantagens essas que encorajem os clínicos em implementar esse novo tipo de resina composta (El-Safty et al., 2012).

2- OBJETIVO

O objetivo deste estudo foi avaliar a efetividade de resinas compostas Bulk-Fill através de dois estudos, testando a resistência à degradação, efeitos de diferentes fontes de luz na formação de fendas, fotopolimerização e microdureza Knoop.

3 - DESENVOLVIMENTO

3.1 RESISTANCE TO DEGRADATION OF BULK-FILL COMPOSITES

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Keywords: Composite resin, Bulk-Fill, polymerization, degradation, depth of cure, microhardness

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Abstract

The aim of this study was to assess the resistance to degradation by microhardness test of Bulk-Fill composite resins with regular or low viscosity challenged in water or ethanol. A total of 60 specimens of resin composite (n= 10) were prepared using a 2x2x4mm matrix. Bulk-Fill composites were inserted in a single increment and conventional composite in two increments, and light activated according to manufacturer. They were tested in a factorial design considering composite resin in 3 levels (conventional resin as control, bulk-fill composite with low or regular viscosity), *in vitro* degradation medium in 2 levels (immersion in 100% ethanol or water), time in 4 levels (control baseline; and immersion for 24 hours, 7 or 30 days), and depth with measures of microhardness in 7 levels (at 0.5; 1.0; 1.5; 2.0; 2.5, 3.0 and 3.5mm from the irradiated surface). At each specimen 3 Knoop indentations were made on each depth applying 25g load for 20s.

Both immersion in distilled water and ethanol are able to degrade composite resins, conventional and Bulk-Fill. Severe degradation are expected after immersion in ethanol.

Key Words: Composite Resin, bulk-fill, polymerization, degradation, depth of cure, microhardness.

Introduction

The traditional composite resins (RBCs) must be inserted into the walls in increments of at least 2mm in order to reduce the effects of polymerization shrinkage (Vanarch et al., 2007). Polymerization contraction (CP) results from the conversion of the monomers into a polymer network after photoactivation (Van Ende et al. 2013). This shrinkage may induce stress and tension in the walls of the cavity prepare and in the body of the RBC restoration, which may result in cusp deflection, cracks in the union line and fractures of the edges of the dental enamel at the cavo-superficial angle (Van Ende et al., 2012).

Several clinical techniques have been suggested to reduce CP stress, such as the control of light intensity, the use of the sandwich technique with glass ionomers, the use of RBC as a base, the use of RBCs with low monomer diluents, the reduction of particles size by nanotechnology and the use of non-methacrylate resins (Ferracane, 2005; Ilie, Hickel 2011). However, none of these methods showed as effective as the incremental technique in CP stress reduction (Park et al 2008).

The stress magnitude of CP is affected by the cavitary configuration, the filling by incremental technique allows the continuous insertion of increments and activation after at each insertion, until the cavity is fullfilled. Then final CP is decreased at each inserted layer (Lazarchik et al., 1998) and Van Nielsen), since it is the result of the small adhesion area ratio of incremen in relation to the non adhered 21rea, obtaining a relative low C factor (Lazarchik et al., 2007).

In addition, the use of increments lesser than 2 mm in thickness allows adequate light penetration for subsequent polymerization of RBC, resulting in better physical properties, better marginal adaptation and reduction of RBC cytotoxicity.
However, the incremental technique presents some limitations such as: (i) the possibility of incorporation of bubbles or debris between layers, (ii) there is an increase in the probability of adhesive failures between increments, (iii) there is a greater difficulty of insertion in minimally invasive cavities and (iv) increases the clinical time due to the need for polymerization of each layer (Abbas et al., 2003; Sarrett 2005; Lazarchik et al., 2007; El-Safty et al., 2012).

As a result, new RBCs with reduced CP have been developed and inserted in the dental market with the indication for restoration by the Bulk-Fill technique. These materials are indicated for insertion in layers with a thickness of 4 or 5 mm, and then they are polymerized in a single step (Czasch and Ilie, 2013, Tiba et al., 2013, Ilie et

al., 2013). C-factor of posterior tooth cavities, still allowing in some cases, it is possible to insert it into a fluid consistency that better adapts the preparation walls (Park et al., 2008).

In this paper, we have shown that CP of Bulk-Fill resins is statistically lower than that of conventional nano and microhybrid RBCs and silorane-based resins (Burgess et al., 2010, Ilie, Hickel, 2011, Tiba et al., 2013). The technology used to reduce CP was obtained in different ways by the manufacturers, involving increase of translucency characteristics, reduction of modulus of elasticity, increase in the concentration of filling particles, use of monomers that control polymerization kinetics, use of new photoinitiators and even association with mechanical methods to change the composite's consistency (Ilie, Hickel 2011; Czasch and Ilie, 2013; Van Ende et al., 2013).

According to Tiba et al. (2013), some questions are not clear with regard to Bulk-Fill resins, among them, it is not known if the light is able to penetrate the entire thickness of the single increment inserted material and if even it is able to polymerize in the deepest part of the cavity (Nayif et al., 2008, Tiba et al., 2013).

It is observed in the literature that a RBC inserted into a class II proximal cavity by the incremental or single increment technique and exposed to direct light by the occlusal face presents values of hardness that decrease from the occlusal to the cervical wall (Poskus et al., 2004). Flury et al. (2012), observed in vitro that the degree of conversion of the Bulk-Fill resins also decreases as the distance from the light source increases.

The primary objective of this study was to assess the resistance to degradation by microhardness test of Bulk-Fill composite resins with regular or low viscosity. Secondary objectives were to observe the influence of depth of cure, degradation medium and time of immersion of degradation of these Bulk-Fill composite resins.

Material and Methods

Experimental Design

The study factorial design considered to observe the resistance to degradation comprised the factors “Composite Resin” in 3 levels: conventional resin as control, bulk-fill composite with low or regular viscosity; *in vitro* degradation “Medium” in 2 levels: immersion in 100% ethanol or water (table 1), with repeated measurements of

"Time" in 4 levels: control baseline; and immersion for 24 hours, 7 or 30 days; and "Depth" with measures of microhardness in 7 levels: at 0.5; 1.0; 1.5; 2.0; 2.5, 3.0 and 3.5mm from the top irradiated surface, in a total of 18 groups with 10 samples each. The response variable was Knoop microhardness (KHN).

The Bulk-Fill resin composites investigated were Tetric N-Ceram Bulk-fill (TEC), a regular-viscosity bulk-fill composite resin; Tetric N-Flow Bulk Fill (TEF), a low-viscosity bulk-fill composite resin; and Tetric N-Ceram (TET), a regular-viscosity conventional composite resin (Ivoclar Vivadent, Schaan, Liechtenstein).

Table 1- Factorial distribution of studied groups

Composite resin	Composition	Degradation midium
Tetric N-Ceram (TEBR) Ivoclar Vivadent Convencional regular-viscosity Batch: V39396 Shader: A2	Urethane dimetacrylate, Bis-GMA 15.0%, Ethoxylated Bis-EMA 3.8%, Triethylenoglycol dimethacrylate, Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide 63.5%, Prepolymers 17.0%, Additives, stabilizers, catalysts, pigments 0.7%	100% ethanol (n=10)
		Water (n=10)
Tetric N-Flow Bulk Fill (TEBF) Ivoclar Vivadent regular-viscosity bulk-fill Batch: W15463 Shade: IVA	Urethane dimetacrylate, Bis-GMA 27.8%, Ethoxylated Bis-EMA, Triethylenoglycol dimethacrylate 7.3%, Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide 63.8%, Prepolymers, Additives, stabilizers, catalysts, pigments 1.1%	100% ethanol (n=10)
		Water (n=10)
Tetric N-Ceram Bulk Fill (TEC) Ivoclar Vivadent Bulk-Fill/regular viscosity Batch: W27100 Shade: IVA	Urethane dimetacrylate, Bis-GMA 15.0%, Ethoxylated Bis-EMA 3.8%, Triethylenoglycol dimethacrylate, Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide 63.5%, Prepolymers 17.0%, Additives, stabilizers, catalysts, pigments 0.7%	100% ethanol (n=10)
		Water (n=10)

Sample preparation

A 2x2mm matrix with 4mm depth was used to prepare 20 samples of each composite resin. Bulk-Fill composite resins were inserted into the matrix in a single increment followed by light activation. The conventional composite resin was inserted

in two increments. To the light activation the mylar strip was positioned on the filled matrix, followed by the light device tip at the top surface of the sample, in order to minimize the effects of oxygen polymerization inhibition, and composite resins were activated according manufacturer for 20s (Valo, Ultradent). After activation the specimens were stored in a dark environment with 100% humidity for 24 hours.

Randomly one lateral surface of the specimen was selected to be polished using a 600, 1200 and 2600 grit Al_2O_3 abrasive papers (Carborundum/3M do Brasil Ltda, Sumare- Brazil) to obtain flat a surface to proceed microhardness test.

Microhardness test

Microhardness measurements were performed in each sample before immersion in the degradation medium (baseline) and after immersion for 24 hours, 7 days and 30 days. Knoop microhardness was measured keeping the long axis of the diamond parallel to the polished lateral surface using a microhardness tester (Microdurometro Digital 10A 1000 HVS-1000A, Panambra Ind. e Tec. S.A., Sao Paulo, SP, Brazil). For each specimen, 3 indentations were made on each depth, at 0.5; 1.0; 1.5; 2.0; 2.5, 3.0 and 3.5mm from the top irradiated surface applying 10g load for 20s.

In vitro degradation challenge

After microhardness baseline measurements, samples were randomized to degradation medium immersion, distilled Water or Ethanol (Ethyl Alcohol 100%). After immersion the samples were located into a dark environment and immersion mediums were changed every 24 hours.

Statistical analysis

The mean of the three measurements of microhardness at each depth for each sample was calculated and used to statistical analysis. ANOVA with repeated measurements was used considering the factors “Composite Resin” “Medium” “Time” and “Depth”, followed by Bonferroni Test.

Results

Four-way ANOVA revealed no significant difference for the interaction of main factors ($p>0.98$), the interaction of the three factors “Composite Resin” “Degradation” “Time” and “Composite Resin” “Medium” “Depth” showed statistically significant difference ($p<0.05$). Mean microhardness values for these triple interactions are presented in Tables 2 and 3.

The triple interaction among “Composite Resin” “Degradation” “Time” showed that composite resins have statistical different microhardness, since baseline measurements. It can be observed that water and ethanol reduced composite resin microhardness, indirectly related to degradation. The microhardness of specimens immersed in ethanol were statistically lower than specimens immersed in water at 24 hours, 7 and 30 days.

Table 2 – Means, standard deviation and statistical differences of composite resins microhardness in function of water and ethanol medium and immersion time.

	Ethanol			Water		
	TEBF	TEBR	TEC	TEBF	TEBR	TEC
Baseline	32.5 (0.49)Ba*	41.7 (2.12)Aa#	31.8 (0.31)Ca*	32.4 (0.98)Ba*	42.9 (0.24)Aa*	31.7 (0.28)Ca*
24hs	24.5 (0.78)Bb#	37.7 (1.43)Ab#	29.6 (0.42)Cc#	31.4 (0.94)Bb*	41.4 (0.36)Ac*	31.6 (0.31)Bab*
7 days	19.0 (0.38)Bc#	31.0 (1.29)Ac#	31.3 (1.01)Ab#	31.0 (0.72)Bc*	41.0 (0.63)Ad*	31.4 (0.55)Bb*
30 days	18.7 (0.38)Cd#	30.1 (0.53)Ad#	24.9 (0.70)Bd#	31.4 (0.97)Bb*	42.2 (0.71)Ab*	30.2 (0.44)Cc*

Statistically significant differences are showed to factor “Composite Resin” by different capital letters, to factor “Time” by lowercase letters, and to factor “medium” by different symbols.

It is observed in the Table that the resins stored in water did not present a decrease in microhardness as a function of depth, and this drop can be observed in the regular Bulk-Fill resin stored in Ethanol. it is also observed that the microhardness of the resins stored in Ethanol is lower than the microhardness of the resins stored in water.

Table - Means and standard deviation of the microhardness of the composite resins under study as a function of depth and degradation medium.

Microhardness mean (SD) and statististical significant differences for the comparison of Composite, Degradation and Depth

	Ethanol			Water		
	TEBF	TEBR	TEC	TEBF	TEBR	TEC
0.1mm	23.7 (5.73)Ba#	35.4 (5.19)Aa#	29.5 (2.73)Ca#	31.8 (0.90)Ba*	41.9 (0.96)Aa*	31.3 (0.75)Ca*
1.0mm	23.8 (5.80)Ba#	35.4 (5.19)Aa#	29.4 (2.90)Ca#	31.7 (0.89)Ba*	41.9 (0.89)Aa*	31.4 (0.74)Ba*
1.5mm	23.7 (5.65)Ba#	35.2 (5.14)Aab#	29.3 (2.74)Ca#	31.8 (0.88)Ba*	41.9 (0.86)Aa*	31.2 (0.77)Ca*
2.0mm	23.7 (5.52)Ba#	35.3 (5.09)Aab#	29.3 (2.87)Ca#	31.7 (0.87)Ba*	41.9 (1.01)Aa*	31.1 (0.70)Ca*
2.5mm	23.8 (5.72)Ba#	34.7 (5.00)Ac#	29.4 (2.74)Ca#	31.7 (0.84)Ba*	41.8 (0.93)Aa*	31.2 (0.67)Ca*
3.0mm	23.6 (5.87)Ba#	34.9 (4.97)Acb#	29.3 (2.89)Ca#	31.7 (0.86)Ba*	41.9 (0.86)Aa*	31.4 (0.75)Ba*
3.5mm	23.6 (5.59)Ba#	34.8 (4.88)Ac#	29.3 (2.69)Ca#	30.9 (1.54)Bb*	42.0 (0.92)Aa*	31.2 (0.81)Ba*

Statistically significant differences are showed to factor “Composite Resin” by different capital letters, to factor “Depth” by lowercase letters, and to factor “Degradation” by different symbols.

Discussion

The results of this study indicate that both distilled water and alcohol promoted the degradation of composite resins. Distilled water is indicated as solvent for resin restorative materials aging (Mortier E et al., 2005), because it simulates the intraoral wet environment resulting from the presence of saliva and water. When the resin composite is immersed in an aqueous solution, the sorption of water occurs between polymers, and although silanes are used to improve its mechanical properties, degradation and damage to the adhesive resistance between the resin matrix and the inorganic filler occurs (Cattani-Lorente MA 1999).

Ethanol has been choosen to simulate accelerated aging of resin composite restorations, since it has a solubility parameter, which coincides with that of BisGMA (Ito et al., 2005). When the ethanol enters the polymer network it causes an expansion of the polymer structure, allowing the release of residual monomers and causing the dissolution of the linear chain of the polymer (Ferracane et al., 1994). The chemical degradation of the resin composites can occur as a consequence of the diffusion of molecules and ions of the residual monomers. This way they can be used in studies as an aging model to predic clinical outcome. The immersion in alcohol resulted in a higher degradation pattern 24 hours after immersion. This is an relevant fact and should be considered to accelerate degradadation on the studies of resin composites aging.

These results are in agreement with those presented by other studies, which reported the reduction of hardness after immersion in alcohol (Tanthanuch et al., 2014). For 30 days of immersion in alcohol, TEBF showed significant lower hardness than distilled water. But this situation did not occur with composite resins TEC and TEBF. These results are in agreement with other studies (Sarret et al., 2000) has showed no differences between alcohol and water after 14 days of immersion.

The regular Bulk-Fill resin composite showed highest degradation as a function of depth. This can probably be justified by the presence of filler particles that impairs light diffusion in depth areas.

The degradation of the resin interface and the inorganic filler may also be useful in reducing the surface hardness (El-Sharkawy et al., 2012). The difference of hardness in the composite resins immersed in alcohol is also associated to the chemical composition (Abu-Bakr et al., 2000).

The Tetric resin composites have in organic matrix BisEMA monomer with high molecular weight and it is more resistant to degradation due to removal of terminals OH-groups, which are susceptible to absorption and solubility (Ferracane et al., 2006). The composite resin TEBR has the highest percentage of organic matrix, based on UDMA, and a lower percentage of inorganic filler, which may explain the lowest values of surface hardness. Moreover, UDMA, TEGDMA, and Bis-GMA are monomers highly susceptible to absorption and solubility in contact with alcohol, causing softening and degradation of the organic matrix (Yap et al., 2003; Sarret et al., 2000; Villalta et al., 2006).

The loss of hardness may contribute to deterioration of the composite resin restorations, including loss of anatomical form and discoloration (Garcia-Godoy et al., 2006). Furthermore, chemical softening may have a negative effect on wear and abrasion decreasing the long term-sucess of these restorations (Yap Au et al., 2000).

According to the presented results, both TEBR and TEC composites had higher initial hardness values than TEBF. The difference in organic matrix contents and higher filling load may explain the behavior of these materials compared to TEBF. After immersion, all materials showed a significant reduction of hardness, and TEBF showed the highest loss of hardness than the other two composites, with distilled water being the most lossy medium.

Lutz, Krejci & Barbakow (1992) observed that resin composites were chemically degraded by immersion in ethanol. The decrease in hardness observed for all storage solutions was probably originated from the organic molecules hydrolysis (Prakki et al., 2005). The absorption of liquids can release unreacted components from the resin matrix, causing a reduction of the mechanical properties. Also, the diffusion of the solvents as water and alchool inside the resin composite matrix interferes with the bonding, separates the polymer chains and interrupts the arrangement of the polymer chain in the composite, causing significant reduction of the mechanical properties of the material (Sideridou et al., 2007).

Hardness parameters depend on several factors, such as fillers particle size, percentage of surface area, degree of polymer matrix conversion and particle-matrix interaction (Marghalani, 2010). In the present study, the highest average knoop microhardness corresponds to the resin that presents the highest fillers particle size TEBF. The influence of chemical factors are related to the clinical success and long-term performance of dental composite restorations. The chemical degradation of the composite resins can occur as a consequence of the diffusion of molecules and ions of the residual monomers. When the resin is immersed in an aqueous solution, the sorption of water occurs between polymers, and although silanes are used to improve their mechanical properties, degradation and damage to the adhesive resistance between the resin matrix and the inorganic filler occurs (Catanni-Lorente et al. al., 1999). McKinney et al., 1985 observed that resin composites showed an increase in sorption on water immersion medium, which can lead to softening of restorative materials. Alcohol solutions simulate alcoholic liquids and is the medium of choice for the accelerated aging of composite resin restorations, as their

parameter is only comparable to that of Bis-GMA, which is the resinous monomer most commonly used in composite resins (McKinney et al., 1985).

Bulk-Fill composites showed similar behavior to conventional in vitro degradation and may be indicated clinically, especially in deep walls to reduce the work time.

Conclusion

Both immersion in distilled water and ethanol are able to degrade composite resins, conventional and Bulk-Fill. Severe degradation are expected after immersion in ethanol.

References

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;31:437–44.

Abu-Bakr N, Han L, Okamoto A, Iwaku M. Changes in the mechanical properties and surface texture of compomer immersed in various media. J Prosthet Dent 2000; 84: 444-452

Bayraktar Y, Ercan E, Hamidi MM, Çolak H. One-year clinical evaluation of different types of bulk-fill composites. J Investig Clin Dent. 2016 Jan 22. doi: 10.1111/jicd.12210.

Burgess J, Cakir D. Comparative properties of low-shrinkage composite resins. Compendium of Continuing Education in Dentistry 2010;31:10–5. (Spec Iss 2).

Cantekin K, Gumus H. In vitro and clinical outcome of sandwich restorations with a bulk-fill flowable composite liner for pulpotomized primary teeth. J Clin Pediatr Dent. 2014 Summer;38(4):349-54.

CATTANI-LORENTE, M. A.; DUPUIS, V.; PAYAN, J.; MOYA, F.; MEYER, J. M. Effect of water on the physical properties of resin-modified glass ionomer cements. Dent. Mater., v. 15, p. 71-78, 1999.

Cavalcante LM, Schneider LF, Silikas N, Watts DC. Surface integrity of solvent-challenged ormocer-matrix composite. Dent Mater. 2011 Feb;27(2):173-9.

Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Investig. 2013 Jan;17(1):227-35.

El-Safty S, Silikas N, Watts DC. Creep deformation of restorative resin-composites intended for bulk-fill placement. Dent Mater. 2012 Aug;28(8):928-35.

El-Sharkawy FM, Zaghloul NM, Ell-Kappaney AM. Effect of water absorption on color stability of different resin based restorative materials *in vitro* study. Int J Compos Mater 2012; 2: 7-10.

Erdemir U, Yildiz E, Eren MM, Ozel S. Surface hardness evaluation of different composite resin materials: influence of sports and energy drinks immersion after a short-term period. J Appl Oral Sci. 2013 Mar-Apr;21(2):124-31.

Ferracane JL. Evolution of leachable components from composites. J Oral Rehabil. 1994; 21(4):441-52.

Ferracane JL. Developing a more complete understanding of stresses produced in dental composites during polymerization. Dental Materials 2005;21:36–42.

Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? Dent Mater. 2012 May;28(5):521-8. doi: 10.1016/j.dental.2012.02.002.

Fonseca AS, Gerhardt KM, Pereira GD, Sinhoreti MA, Schneider LF. Do new matrix formulations improve resin composite resistance to degradation processes? Braz Oral Res. 2013 Sep-Oct;27(5):410-6.

García-Godoy F, García-Godoy A, García-Godoy F. Effect of APF Minute-Foam on the surface roughness, hardness, and micromorphology of high-viscosity glass ionomers. J Dent Child (Chic) 2003; 70: 19-23.

Ilie N, Hickel R. Investigation on a methacrylate-based flowable composite based on the SDRTM technology. Dental Materials 2011;27:348–55.

Ilie N, Keßler A, Durner J. Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites. J Dent. 2013 Aug;41(8):695-702.

Ito S, Hashimoto M, Wadgaonkar B, Svizero N, Carvalho RM, Yiu C, et al. Effect of resin hydrophilicity on water sorp- tion and changes in modulus of elasticity. Biomaterials. 2005; 26(33):6449-59.

Lazarchik DA, Hammond BD, Sikes CL, Looney SW, Rueggeberg FA. Hardness

comparison of bulk-filled/transtooth and incremental-filled/occlusally irradiated composite resins. J Prosthet Dent. 2007 Aug;98(2):129-40.

LUTZ, F., KREJCI, I. & BARBOKOW, F. (1992) Chewing pressure vs. wear of composites and opposing enamel cusps. Journal of Dental Research. 71, 152

MARGHALANI, H. Y. Effect of filler particles on surface roughness of experimental composite series. J. Appl. Oral. Sci., v. 18, p. 59-67, 2010.

Mayworm CD, Camargo JRSS, Bastian FL. Influence of artificial saliva on abrasive wear and microhardness of dental composites filled with nanoparticles. J Dent 2008;36(9):703–10.

McKinney, J. E.; Wu, W. Chemical softening and wear of dental composites. J. Dent. Res., v. 64, p. 1326-1331, 1985.

Mortier E, Gerdolle DA, Dahoun A, Panighi MM. Influen- ce of initial water content on the subsequent water sorp- tion and solubility behavior in restorative polymers. Am J Dent. 2005; 18 (3): 177-81.

Münchow EA, Ferreira AC, Machado RM, Ramos TS, Rodrigues-Junior SA, Zanchi CH. Effect of acidic solutions on the surface degradation of a micro-hybrid composite resin. Braz Dent J. 2014;25(4):321-6.

Nayif MM, Nakajima M, Foxton RM, Tagami J. Bond strength and ultimate tensile strength of resin composite filled into dentine cavity; effect of bulk and incremental filling technique. J Dent. 2008 Mar;36(3):228-34.

Park J, Chang J, Ferracane J, Lee IB. How should composite be layered to reduce shrinkage stress: incremental or bulk filling? Dent Mater. 2008 Nov;24(11):1501-5.

Poskus LT, Placido E, Cardoso PE. Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations. Dent Mater 2004;20:726-32.

Prakki, A.; Cilli, R.; Mondellil, R. F.; Kalachandra, S.; Pereira, J. C. Influence of pH environment on polymer based dental material properties. J. Dent., v. 33, p. 91-98, 2005.

Roggendorf MJ, Krämer N, Appelt A, Naumann M, Frankenberger R. Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. J Dent. 2011 Oct;39(10):643-7.

Sarrett DC. Clinical challenges and the relevance of materials testing for posterior composite restorations. Dental Materials 2005;21:9–20.

Sideridou, I. D.; Karabela, M. M.; Bikiaris, D. N. Aging studies of light cured dimethacrylate-based dental resins and a resin composite in water or ethanol/water. Dent. Mater., v. 23, p. 1142-1149, 2007.

Tanthanuch S, KukiattrakoonB, Siriporananon C,Ornprasert N, Mettasitthikorn W, Likhitpreeda S, Waewsanga S. The effect of different beverages on surface hardness of nanohybrid composite resin and giomer. J Conserv Dent 2014; 17: 261- 265.

Tiba A, Zeller GG, Estrich CG, Hong A. A laboratory evaluation of bulk-fill versus traditional multi-increment-fill resin-based. JADA 2013;144(10):1182-3.

van Dijken JW. A clinical evaluation of anterior conventional, microfiller, and hybrid composite resin fillings. A 6-year follow-up study. Acta Odontol Scand. 1986 Dec;44(6):357-67.

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 Sep;30(9):e245-51.

van Dijken JW, Pallesen U. Posterior bulk-filled resin composite restorations: A 5-year randomized controlled clinical study. J Dent. 2016 Aug;51:29-35.

Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, Van Meerbeek B. Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity- bottom dentin. Dent Mater. 2013 Mar;29(3):269-77.

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 Apr;40(4):295-303.

Villalta P, Lu H, Okte Z, Garcia-Godoy F, Powers JM. Effects of staining and bleaching on color change of dental composite resins. J Prosthet Dent 2006; 95: 137-142.

Yap AU, Wattanapayungkul P, Chung SM. Influence of the polymerization process on composite resistance to chemical degradation by food-simulating liquids. Oper Dent 2003; 28: 723-727.

3.2 EFFECTS OF LIGHT CURING UNITS ON BULK-FILL FLOW COMPOSITES: POLYMERIZATION, GAP DEVELOPMENT AND KNOOP MICROHARDNESS TEST

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Key words: Light curing units, composite resins, dental restoration failure, optical coherence tomography

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ABSTRACT

The objective of this study was to evaluate the gap development in Bulk-Fill resins activated with different light sources [FL]. Two factors were evaluated Composite resin: Filtek Bulk Fill Flowable (FBF - 3M) and PALFIQUE Estelite Low Flow (PBF - Tokuyama) and light source: Halogen (QLF – Optlux 501, Kerr)]; LED monowave (MW - Elipar 3M) and LED polywave (PW - Valo Ultradent), in 6 experimental groups. A matrix was fabricated with Estelite Flow resin with a circular cavity with 3 mm of diameter and 4 mm of depth. The cavity was conditioned (K-Etchant gel) and treated with a ceramic primer (Clearfil Ceramic Primer Plus) followed by the Clearfil SE BOND 2 (Kuraray). The composite resins were inserted in a single increment and photo activated. Swept-Source Optical Coherence Tomography (ss-OCT) images were imported into the ImageJ software and the mean lenght of gap on the the 6-section slices (0^o, 30^o, 60^o, 90^o, 120^o e 150^o) on the cavity floor was measured. Then images by Microscopy Confocal Laser (CLSM) and Scanning Electronics (SEM) were obtained to confirm the presence of gaps. Three indentations, 1,3 mm from the specimen lateral margins and 200μm from each other, at 1.0; 1.5; 2.0; 2.5; 3.0; 3.5; and 4.0mm from the irradiated surface were made under a load of 49g for 20 sec. The average yielded a single microhardness number (KHN) for each depth evaluated. The 2-way ANOVA showed statistical difference for RC, FL and to the interaction RC * FL: FBFQTH = 94.54 ± 0.7; FBFMW = 35.90 ± 6.10; FBFPW = 73.38 ± 3.68; PFBQTH = 64.04 ± 8.04; PFBMW = 52.62 ± 4.60; PFBPW = 67.84 ± 4.83. CLSM and MEV confirmed the presence of gaps. Significant statistical difference was observed for the interaction between the three factors (p= 0.04), for the interactions between the "LIGHT" and "DEPTH" factors (p <0.01), "COMPOSITE RESIN" and "DEPTH" (p <0.01), "LIGHT" (p <0.01), "COMPOSITE RESIN" and "LIGHT" (p <0.01), and "DEPTH" (p = 0.16), Composite hardness and gap formation are affected by light curing unit. Filtek Bulk-Fill Flow (3M) composite showed the smallest gap formation polymerized by LED Monowave light cure unit.

Key Words: Composite Resin, bulk-fill, polymerization, SS-OCT, gap formation, microhardness.

INTRODUCTION

The lack of integrity and gap development at the tooth-restoration adhesive interface impairs the success of a resin composite restoration (Bakhsh et al., 2011; Opdam et al., 2014). The resin composite restoration is challenged since its activation in the reason of the intrinsic polymerization shrinkage stress. Since intermolecular distance decreases as polymerization occurs, there is a volumetric reduction ranging from 1 to 6% (Labella et al., 1999; Ferracane, Hilton 2016) and the stress is transferred to cavity walls. Then, if shrinkage stress exceeds the bond strength at the resin-tooth interface a gap will be developed (Sampaio et al., 2015). Furthermore, these stresses may increase with time, causing delayed damage to cavity margins (Yamamoto et al., 2015). Gaps and unbonded margins spaces may accelerate material deterioration, and lead to deleterious effects such as marginal infiltration, postoperative sensitivity and contribute to secondary caries development (Benetti et al., 2015; Almeida Junior 2017).

There is concern over this shrinkage stress and the manner in which it influences the long-term survival of the restorarion. Marginal adaptation of tooth restoration is considered a clinical requirement for the success of treatment, but shrinkage occurs inevitably in resin-based materials (Jang et al. 2015; Bakhsh et al., 2011). Varoius factors interfere on resin composite polymerization shrinkage as monomers molecular characteristics, initiation systems, inorganic fillers content, elastic modulus, the cavity C-factor, the substrate to be bonded, light irradiance delivered by curing unit as the degree of conversion (Labella et al., 1999; Lim et al, 2002; Ferracane, 2005; Feilzer et al., 2005; Venhoven et al., 2003; Sampaio et al., 2015; Papadogiannis et al., 2015; Braga et al., 2005; Versluis et al., 1996; Atria et al 2017).

In order to clinically overcame or reduce polymerization shrinkage the incremental filling technique is mandatory to conventional composite resin restorations (Lutz et al., 1986). It has been suggested that the reduced volume of the resin at each increment, using either 2-mm-thick oblique or horizontal increments, may decrease the shrinkage stresses at the tooth–restoration interface. However incremental technique is time consuming, and bubles could be incorporated reducing the composite mechanical properties. In the last decade, several approaches have been taken by the manufactures to minimize polymerization shrinkage by

incorporating more fillers and introducing new chemical resin formulations resulting on the Bulk-Fill composite resins development.

The bulk-fill composite resins major advantages are the larger curing depth, up to 4 mm, lower volumetric shrinkage development than conventional composite resins (Benetti et al., 2015). As consequence, restorative technique is simplified, chair time is substantially reduced and fewer clinical steps are required. These improvements were accomplished by optimization of matrix and initiator chemistry, as well as filler technology and increased translucency of the bulk-fill composites, which allows greater light transmission (Bucuta, Ilie et al 2000; Benetti et al., 2015, Hirata et al., 2015).

Despite several in vitro studies demonstrate favorable outcomes for bulk-fill composites, there is few short-term clinical survivals, and a previous study of bulk-fill composites showed the gap development in the cavity floor of class I restorations (Hayashi et al., 2017). Gap formation are related to the degree of polymerization, which is also influenced by light activation source, recently many light curing units have been introduced, each of them with different wavelengths and power intensities (Benetti *et al.*, 2015).

The efficiency of light unites is usually evaluated by curing depth and microhardness tests as an indirect source of conversion degree (Ilie and Stark, 2015). Achieve an equal and adequate degree of monomers conversion is as essential for the success and longevity of the composite restorations as avoid gap development (Al-Ahdal *et al.*, 2015). The curing degree of the composite resins can be related to dimensional stability, color stability and biocompatibility (Maghaire et al. 2018; S et al. 2006). Makishi et al. in 2011 demonstrated the effectiveness of SS-OCT through 3D imaging for detecting gaps around the composite resin restorations in extracted teeth. Optical coherence tomography (OCT) developed by Fujimoto et al. in 1991 can construct images through wave interference that occurs when light from two sources is used. This can selectively detect reflected light and is unaffected by scattering, thereby allowing retrieval of detailed images from in vivo biological tissue, which does not require cutting and processing of the specimens and allows the visualization of microstructures of tissue and biomaterials in the real time as gap development.

The best we know, there is no report regarding the influence of light curing units on interfacial gap developmet in clinically relevant setup with flowable bulk-fill composite resins activated with different light source devices.

OBJECTIVE

The aim of this study was to compare polymerization and the gap development of bulk-fill composites restoration activated by light curing units with different irradiant wavelengths.

MATERIALS AND METHODS

Experimental design

In this study, the influence of two factors were assessed: “COMPOSITE RESIN” in two levels (Table 1): FBF- 3M Filtek Bulk-Fill Flow (3M ESPE); PBF- Palfique Bulk-Fill Flow (Tokuyama Dental Corporation, Tokyo, Japan); “LIGHT” in three levels (QTH- Halogen Optilux 501, MW- LED Monowave Elipar Deepcure-L 3M, and PW- LED Polywave Valo Ultradent); with an additional factor for microhardness test “DEPTH” in 7 levels (1.0mm; 1.5mm; 2.0mm; 2.5mm; 3.0mm; 3.5mm; 4.0mm from composite surface). In a factorial design six groups, with 8 samples (n=8), were prepared according to COMPOSITE RESIN and LIGHT source.

The response variables were gap development, quantitatively evaluated in μm by swept-source optical coherence tomography (SS-OCT) images and composite resin depth of cure evaluated indirectly by Knoop Hardness in KHN.

Table 1- Materials used, their composition, and manufacturer.

Material	Composition	Manufacturer (Batch number)	Time Irradiance
FBF: Filtek Bulk Fill Flowable Shade: A2	UDMA, BISEMA-6, Bis-GMA, benzotriazol, TEGDMA, ethyl 4-dimethyl aminobenzoate. Silane-treated ceramics, YbF3, filler loading: 64.5 wt%	3M ESPE, St Paul, MN, USA (00133A)	LED/QTH 550-1000mW/cm^2 U:20s A1, A2, A3: 40s LED 1000-2000mW/cm^2 U:10s A1, A2, A3: 20s
PBF: PALFIQUE Estelite Low Flow Shade: A1	Bis-GMA, TEGDMA, Bis-MPEPP, photoinitiator. Silica–zircônia filler, 71 wt.%.	Tokuyama Dental Corporation, Tokyo, Japan LOT: 670	QTH: 5-10 s LED : 5**-10 s (**5 sec with 900mW/cm^2)

UDMA: diurethane dimethacrylate, BISEMA-6: dimethacrylate, bisphenol Apolyethylene glycol dietherdimethacrylate, Bis-GMA: bisphenol-A glycidyl dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, Bis-MPEPP: Bisphenol A polyethoxy methacrylate, YbF3: ytterbium fluoride, wt%: weight percentage.

Sample preparation

The resin composite Estelite Flow Quick (Tokuyama Dental Corporation, Tokyo, Japan) was used to prepare standardized matrices with a central circular cavity, with Ø= 3mm and 4mm of depth and C-factor= 6.33 (Czasch and Ilie, 2013).

After 24 hours, the composite matrices were sand-blasted and all composite cavities were cleaned with 40% phosphoric acid (K-Etchant gel, Kuraray Medical, Tokyo, Japan). The Clearfil ceramic primer plus (Kuraray Medical, Tokyo, Japan) was used to perform the silanization and the Clearfil SE BOND 2 bonding agent (Kuraray Medical, Tokyo, Japan) was applied as adhesive agent and activated with a PW (Valo Ultradent, Utah, USA).

According to each experimental group, Bulk-Fill flowable composites (Table 1) were inserted in the matrices in a bulk increment, followed by the respective activation light (QTH: Optlux 501 Kerr, Ca, USA 600mW/cm^2 for 40s; MW: 3M ESPE Elipar DeepCure – L, St. Paul, Mn, USA; 1.000mW/cm^2 for 20s; PW: Valo Ultradent, Utah, USA, 1.200mW/cm^2 for 20s) in contact with composite.

Swept Source - Optical Coherence Tomography Imaging

After matrices filling, the restoration floor on specimens were observed with a nondestructively swept-source OCT system (SS-OCT, Santec OCT-2000, Komaki, Japan). In this system, the center wavelength is 1.330 nm (bandwidth 110 nm) with a 30-kHz sweep rate. The optical resolution is 20 μm transversally and 12 μm axially in air. The laser beam scans the object in X and Z dimensions. Collected backscattered light returned to the system, which is digitized in time scale, and analyzed in the Fourier domain to form a depth-resolving scan (A-scan) at each point. A serial set of A-scans along a certain section creates a cross-sectional B-scan or 2-dimensional (2D) image. Serial B-scans over a region of interest can create a 3D data set (Turkistani et al. 2015).

The handheld scanning probe connected to the OCT was set at a fixed distance (5 cm) over the specimen, with the scanning beam oriented 90° with respect to the restoration surface. Cross-sectional images were recorded from the OCT system at a resolution of 800 × 600 pixels and 20 frames/s at a central mesiodistal plane through the restoration. The gap formation in each specimen was observed and quantitatively evaluated.

Six cross-sectional SS-OCT at 0˚, 30˚, 60˚, 90˚, 120˚ and 150˚ were selected and imported to ImageJ software (version 1.48; National Institutes of Health, USA). It is known that when there is a microgap between two media of different refractive indices, the reflections of light at the interface will be dissimilar (Sadr et al., 2011). The presence of air, which means a microgap between the matrice and restorative material, is visualized as a bright spot on the SS-OCT image. To measure the gap development, the length of the bright spots on the cavity floor was measured. Gap development at the cavity floor was measured in μm. The percentage of gap development (%GAP) was defined as the percentage from dividing the sum of portions where signal intensities were above the threshold by the entire length of a floor (Han et al., 2016).

Confocal Laser Scanning Microscopy

Representative specimens were observed at 1.250x magnification in a 3D Confocal Laser Scanning Microscope (VK-X150 KEYENCE, Osaka, Japan) using a software interfaced to CLSM (Multi-file Analysis Application VK-H1XM; KEYENCE, Osaka, Japan).

Microhardness test

The microhardness was measured using a Knoop Micro-hardness Tester (HVS-1000, Pantec, Sao Bernardo do Campo, SP, Brazil). The measurements were conducted at room temperature (23˚C) under a load of 49g with a dwell time of 20 seconds. Three indentations, 1,3 mm apart from the specimen lateral margins and 200μm from each other, at 1.0; 1.5; 2.0; 2.5; 3.0; 3.5; and 4.0mm from the irradiated surface were made and averaged to yield a single micro-hardness number (KHN) for each depth evaluated.

STATISTICAL ANALYSIS

The data of gap length percentage from OCT images were imported to SPSS software for statistical analysis. The average percentage of 6 cross sections form gap length was submitted to analysis of variance 2-WAY ANOVA and Tukey's post hoc test (α= 0.05), considering the factors "COMPOSITE RESIN" and "LIGHT". Confocal Laser Scanning (CSLM) was used to describe the pattern of gap development on experimental groups.

The average of hardness values obtained from the three indentations at each depth was calculated. These data were submitted to analysis of variance 3-WAY ANOVA, followed by the Bonferroni test (α= 0.05), considering the factors "COMPOSITE RESIN", "LIGHT" and" DEPTH ".

RESULTS

SS-OCT

Significant statistical difference was observed for the interaction between the two factors ($p < 0.04$), and main factors "COMPOSITE RESIN" and "LIGHT" ($p < 0.01$), the values can be observed in Table 2. From the results of multiple comparisons by Dunnett's T3 test, statistically significant differences were observed

among 3 different light curing units on FB group, QTH showed the highest gap development, and MW lowest values. On the other hand, in PB group there was no significant difference between LED PW and QTH, and LED MW statistically differed from other groups with low gap development. Comparing composite resins activated with same light source, statistically significant difference was observed only for MW, with higher gap development to PB than FB.

Table 2– Means, standard deviation and results of the Tukey's test of gap lenght percentage (%) between composite resins studied as a function of the light source.

	MW	PW	QTH
FB	35.90 (6.10) Ca	73.38 (3.68) Ba	94.54 (0.72) Aa
PB	52.62 (4.60) Bb	67.84 (4.83) Aa	64.04 (8.04) Aa

Upper case letters indicate significant statistical difference among light sources, and lower case between composite resins.

Representative optical coherence tomography (OCT) cross-sectional images from each group are presented in figure 1. High signal intensity could be observed at the cavity floors, which indicate gap development in all groups. PBF specimens showed high signal intensities indicating composite crack associated with gap development.

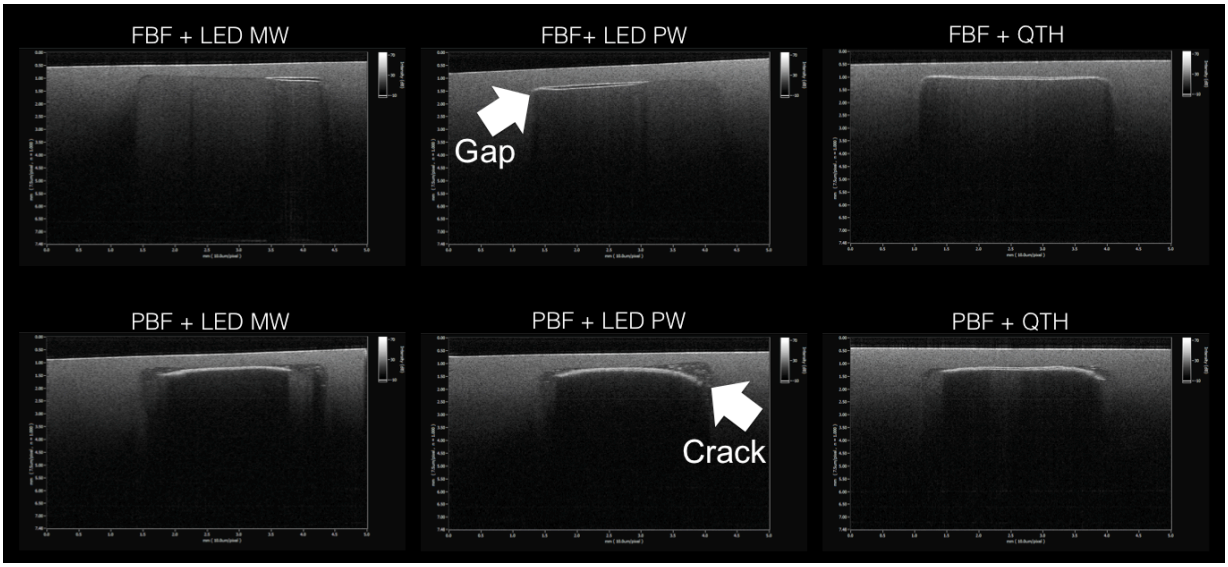

Figure 1. Representative swept source optical coherence tomography (ss-OCT) cross-sectional images from each group. High signal intensities at the cavity floors indicate gap development. PBF specimens showed higher signal intensities, which indicate crack development.

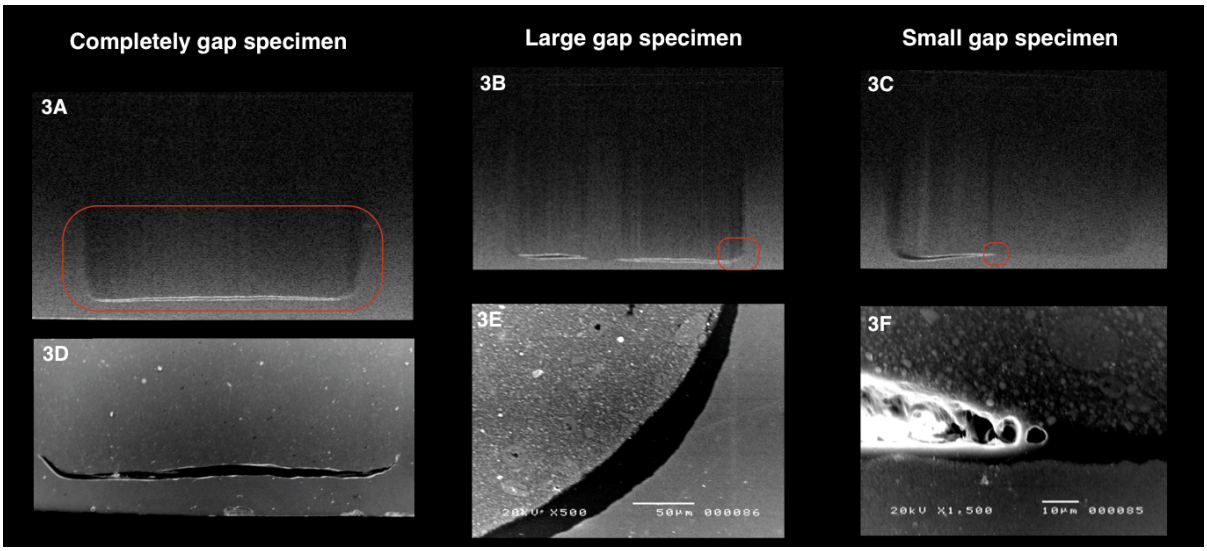

Figure 2. Representative images of three patterns of gap development along cavity floor wall with gap development, large gap and small gap. The top images were collected with OCT and the bottom image with SEM.

Figure 2 shows representative images of three patterns of gap development along cavity floor wall. In the figure 3A, 3D gap developed all over the cavity floor, in the figure 3B, 3E it can observe a large gap formation and, in the figure 3F, a small

gap formation. In the Figure 3F it can observe at high magnification the end point of the gap progress nearby the center of the cavity floor.

MICROHARDNESS TEST

Significant statistical difference was observed for the interaction between the three factors (p= 0.04), for the interactions between the "LIGHT" and "DEPTH" factors (p <0.01), "COMPOSITE RESIN" and "DEPTH" (p <0.01), "LIGHT" (p <0.01), "COMPOSITE RESIN" and "LIGHT" (p <0.01), and "DEPTH" (p = 0.16), the values can be observed in table 3.

It has been observed that the PB microhardness was statistically higher than FB. No differences were observed in the microhardness of the FB composite resin in depth function. For PB composite resin, no differences were observed in the microhardness in depth function activated with MW and QTH. The composite resin PB showed lower microhardness at depths below 2.5mm (Table 3) activated with PW.

Comparing light sources, the composite resin FB showed significantly higher hardness at all depths when activated with the MW light source, followed by the PW with intermediate values, and the QTH with the lowest values. Also PB resin, showed at all depths, the highest values of hardness when activated with the MW light source, however the intermediate values were observed in the activation with the QTH light source and the lowest values were observed with the PW LED (Table 3).

Table 3- Means, standard deviation and results of the Bonferroni test between the microhardness of the resin composites studied at different depths as a function of the light source, and boton/top rate.

	FB*			PB#		
	MW	PW	QTH	MW	PW	QTH
1.0mm	44.9 (0.97) Aa	37.2 (0.65) Ab	32.9 (0.40) Ac	56.5 (0.77) Aa	51.9 (0.54) ABc	54.5 (0.36) Ab
1.5mm	45.1 (0.57) Aa	37.0 (0.53) Ab	32.7 (0.31) Ac	57.1 (0.94) Aa	52.1 (0.18) Ac	54.3 (0.21) Ab

2.0mm	44.8 (0.74) Aa	37.5 (0.34) Ab	32.7 (0.37) Ac	57.5 (0.62) Aa	51.5 (0.55) ABc	54.5 (0.28) Ab
2.5mm	45.4 (0.89) Aa	36.4 (0.36) Ab	32.8 (0.24) Ac	57.3 (0.78) Aa	50.9 (0.46) BCc	54.8 (0.19) Ab
3.0mm	45.4 (0.47) Aa	36.4 (1.07) Ab	32.9 (0.33) Ac	56.5 (0.64) Aa	50.5 (0.18) Cc	54.7 (0.81) Ab
3.5mm	45.3 (0.88) Aa	36.6 (0.54) Ab	32.9 (0.38) Ac	56.9 (0.85) Aa	50.3 (0.21) Cc	54.3 (1.03) Ab
4.0mm	45.9 (0.65) Aa	36.4 (0.79) Ab	33.2 (0.68) Ac	57.3 (0.60) Aa	50.3 (0.43) Cc	53.5 (0.97) Ab
Boton/top ratio	1.02	0.94	1.01	1.01	0.97	0.98

Upper case letters indicate significant statistical difference between depths; Lower case letters indicate significant statistical difference between light sources; Symbols indicate statistically significant differences between composite resins.

DISCUSSION

Composite resin polymerization is a dynamic process, as soon as activation light is applied and reaction reachs the gel-phase begins the polymerization contraction strain and shrinkage stress, then gaps may be developed during the course of polymerization. Although the volumetric polymerization shrinkage varied among the different types of flowable resin composite restorative materials (Sampaio et al., 2017), in general on this study where two flowable Bulk-Fill composite resins were activated with three different light sources, excepted for FBF activated with MW light source which resulted in less gap development the gap formation behavior to QTH and PW was similar for both composites, whereas PBF microhardness was higher than FBF. Also, an absence of marginal gaps was observed in all groups, in agreement with Almeida Junior et al., also observed that all class I restorations filled with bulk-fill composite resins presented a gap only at the interface between the composite resin and the pulp wall (Almeida Junior et al., 2017).

The abcence of gap development in lateral walls could be explained by the influence of light emitted on the top surface and adhesive model that restricts the

direction of the shrinkage vectors downwards (Sampaio et al., 2017B; Kaisarly et al., 2018). Because the intensity of the light is highest at the top surface and decreases as it penetrates into the composite resin body, superficial layers shrink first and faster than do deeper layers, and it might be assumed that deeper cavities have higher shrinkage stress and, consequently, more gap development (Versluis et al., 1998). However, in a clinical situation the shrinkage on a cavity preparation may be influenced by the adhesion inside on enamel and dentin substrates, shrinkage vectors might be oriented toward the strongest bonding sites due to detachment from the weaker areas, or even directed toward irregularities from the preparation in an un bounded situation (Versluis et al., 1998; Chiang et al., 2010).

In contrast, bonding failures in the cavity floor might occur as a result of lower copolymerization of monomers, in the reason of insufficient delivery of light energy at this location by composite absorption and scattering (Cho te al., 2011; Leprince et al., 2013; Bucuta, Ilie, 2014). However, the radiance emitted by the light devices used in this study likely favored satisfactory excitation of photoinitiators from the tops to the bottoms of the cavities, resulting in bottom-to-top hardness ratios 0.9, since 0.8 ratio has been considered satisfactory (Alshaa *et al.*, 2016). In addition, Kumagai et al (2015), using MOD cavity preparations on ex-vivo teeth demonstrated that bond strength values in gingival walls were significantly higher when a flowable bulk-fill composite (Surefil SDR flow, Dentsply Caulk) was used in comparison with a regular nanofilled composite (Filtek Z350, 3M Oral Care), either placed incrementally or in bulk.

The gap development on the cavity floor depended on multiple factors as the polymerization of composites as the composite resin filler contents (Kishikawa et al., 2005). Both composite resins presented high filler contains (FBF: 64.5% and PBF: 71% by weight) which are probalbly the factor responsible to reduce volumetric shrinkage, literature has pointed out that composite resins with a lower percentage of fillers may have a higher shrinkage than those with higher percentages (Braga et al., 2005). Although the high filler content results in high elastic modulus due to produce more rigid restorations, which increase the effect of polymerization shrinkage stresses, since shrinkage occurs in the course of monomer conversion to polymer, the higher the filler content, the lower will the resultant shrinkage (Versluis et al 2004; Gonçalves et al., 2010).

Other factors such as variations in restoration volume, C-Factor also affected free surface displacement and shrinkage stresses, and an increase in value for any of those factors would be linked to increases in shrinkage stress (Boaro et al., 2014). However, these factors were isolated in the present study by opting for standard preparations in a composite resin substrate to establish a more uniform bonding condition. As bonding to enamel is far better established than bonding to dentin (Van Meerbeek et al., 2010; Van Ende et al., 2015), the dentin bond is more likely to fail due to the stress development and the shrinkage may thus be directed by the stronger enamel bond and the effect of different light sources on gap development could be misinterpreted.

The bulk-fill composite FBF, in turn, contains procrylat resins and Bis-EMA, which have high molecular weights and low viscosities, compared to Bis-GMA, as a result of the absence of hydroxyl groups in their structures (Stansbury, 2012). Dental polymers based on Bis-EMA and containing lower viscosity urethane monomers tend to exhibit higher DC values than typical Bis-GMA/TEGDMA resins (Tarle et al. 2015).

The hardenss difference of composite resins could be related to their composition (Table 1), PBF have higher filler content (71%) than FPF (64.5%) which might result on higher hardness. In addition, PBF has a catalyst technology that reduces activation time.

In agreement with present results, Alshaafi et al found no significant differences in the Knoop microhardness measurements made at the top of the Bulk-Fill RBCs, however they found significant differences in the KHN at the bottom between the different thicknesses of the RBCs. Such difference was observed on PBF PW.

Our results demonstrated that LED MW showed the lowest gap length percentage. followed by LED PW and halogen. On the other hand. tested LED MW features the highest power intensity. followed by LED PW and halogen. It can see bigger decline of light intensity of tested LED polywave through 7mm from the light source compared to LED monowave.

High power light can reach more energy to the bottom of restoration. It can achieve maturing of adhesion at the cavity bottom at an early stage. Among LED, tested monowave shows smaller percentage compared to the polywave.

Ununiformed light produces an ununiformed degree of cure at the cavity floor, thus the partial weak adhesion area would become a starting point of gap progress.

Regarding the different tendency between two tested composites, the restorations with the FBF composite presented significant differences between the light curing units. In the other hand, PBF composite did not present a statistically significant difference among light curing units.

Moreover, PBF composite showed crack formation during light curing. From the information provided by manufacturer. Palfique Bulk-Fill Flow (Tokuyama) composite would reach to the gel phase at an early stage because of RAP technology. Some previous important literature suggests that shorten of the pre-gel phase would cause loss of the chance to relaxation.

CONCLUSION

Composite hardness and gap formation are affected by light curing unit. Filtek Bulk-Fill Flow (3M) composite showed the smallest gap formation polymerized by LED Monowave light cure unit.

REFERENCES

Algamaiah H, Sampaio CS, Rigo LC, Janal MN, Giannini M, Bonfante EA, Coelho PG, Reis AF, Hirata R. Microcomputed Tomography Evaluation of Volumetric Shrinkage of Bulk-Fill Composites in Class II Cavities. J Esthet Restor Dent. 2017 Apr;29(2):118-127. doi: 10.1111/jerd.12275.

Al-Ahdal, K., Ilie, N., Silikas, N. and Watts, D. C. (2015) 'Polymerization kinetics and impact of post polymerization on the Degree of Conversion of bulk-fill resin-composite at clinically relevant depth.', *Dental materials : official publication of the Academy of Dental Materials*, 31(10), pp. 1207–13. doi: 10.1016/j.dental.2015.07.004.

Almeida Junior LJDS, Penha KJS, Souza AF, Lula ECO, Magalhães FC, Lima DM, Firoozmand LM. Is there correlation between polymerization shrinkage, gap formation, and void in bulk fill composites? A μCT study. Braz Oral Res. 2017 Dec 18;31:e100. doi: 10.1590/1807-3107bor-2017.vol31.0100.

Alshaa, M. M., Haenel, T., Sullivan, B., Labrie, D., Alqahtani, M. Q. and Price, R. B. (2016) 'Effect of a broad-spectrum LED curing light on the Knoop microhardness of four posterior resin based composites at 2 , 4 and 6-mm depths', 45, pp. 14–18.

Alshali, R. Z., Silikas, N. and Satterthwaite, J. D. (2013) 'Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals.', *Dental materials : official publication of the Academy of Dental Materials*, 29(9), pp. e213-7. doi: 10.1016/j.dental.2013.05.011.

Atria PJ, Sampaio CS, Cáceres E, Fernández J, Reis AF, Giannini M, Coelho PG, Hirata R. Micro-computed tomography evaluation of volumetric polymerization shrinkage and degree of conversion of composites cured by various light power outputs. Dent Mater J. 2018 Jan 30;37(1):33-39. doi: 10.4012/dmj.2016-430.

Bakhsh TA, Sadr A, Shimada Y, Mandurah MM, Hariri I, Alsayed EZ, Tagami J, Sumi Y. Concurrent evaluation of composite internal adaptation and bond strength in a class-I cavity. J Dent. 2013 Jan;41(1):60-70. doi: 10.1016/j.jdent.2012.10.003.

Bakhsh TA, Sadr, Shimada, Tagami J, Sumi Y. Non-invasive quantification of resin-dentin interfacial gaps using optical coherence tomography: validation against confocal microscopy. Dent Mater. 2011 Sep;27(9):915-25. doi: 10.1016/j.dental.2011.05.003.

Benetti AR, Havndrup-Pedersen C, Honoré D, Pedersen MK, Pallesen U. Bulk-fill resin composites: polymerization contraction, depth of cure, and gap formation. Oper Dent. 2015 Mar-Apr;40(2):190-200. doi: 10.2341/13-324-L.

Benetti AR, Peutzfeldt A, Lussi A, Flury S. Resin composites: Modulus of elasticity and marginal quality. Dent. 2014 Sep;42(9):1185-92. doi: 10.1016/j.jdent.2014.07.004.

Boaro LC, Brandt WC, Meira JB, Rodrigues FP, Palin WM, Braga RR. Experimental and FE displacement and polymerization stress of bonded restorations as a function of the C-Factor, volume and substrate stiffness. J Dent. 2014 Feb;42(2):140-8. doi: 10.1016/j.jdent.2013.11.016.

Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resincomposites: A systematic review. Dent Mater 2005; 21: 962- 970.

Bucuta S, Ilie N. Light transmittance and micro-mechanicalproperties of bulk fill vs. conventional resin basedcomposites. Clin Oral Investig 2014;18(8):1991–2000.

Chiang YC, Rösch P, Dabanoglu A, Lin CP, Hickel R, Kunzelmann KH. Polymerization composite shrinkage evaluation with 3D deformation analysis from microCT images. Dent Mater. 2010 Mar;26(3):223-31. doi: 10.1016/j.dental.2009.09.013.

Cho E, Sadr A, Inai N, Tagami J. Evaluation of resin composite polymerization by three dimensional micro-CT imaging and nanoindentation. Dent Mater. 2011 Nov;27(11):1070-8. doi: 10.1016/j.dental.2011.07.008.

Czasch, P. and Ilie, N. (2013) 'In vitro comparison of mechanical properties and degree of cure of bulk fill composites', pp. 227–235. doi: 10.1007/s00784-012-0702-8.

D'Alpino PH, Bechtold J, dos Santos PJ, Alonso RC, Di Hipólito V, Silikas N, Rodrigues FP. Methacrylate- and silorane-based composite restorations: hardness, depth of cure and interfacial gap formation as a function of the energy dose. Dent Mater. 2011 Nov;27(11):1162-9. doi: 10.1016/j.dental.2011.08.397.

Feilzer A, Dooren L, Gee Ad, Davidson C. Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface. Eur J Oral Sci 2005; 103: 322- 326.

Ferracane JL, Hilton TJ. Polymerization stress--is it clinically meaningful? Dent Mater. 2016 Jan;32(1):1-10. doi: 10.1016/j.dental.2015.06.020.

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

Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LE, Martin AA, Ambrosano G, Giannini M. Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites. Dent Mater. 2015 Dec;31(12):1542-51. doi: 10.1016/j.dental.2015.10.001.

Gonçalves F, Kawano Y, Braga RR. Contraction stress related to composite inorganic content. Dent Mater 2010; 26:704–9.

Han SH, Sadr A, Tagami J, Park SH. Internal adaptation of resin composites at two configurations: Influence of polymerization shrinkage and stress. Dent Mater. 2016 Sep;32(9):1085-94. doi: 10.1016/j.dental.2016.06.005.

Hayashi, J., Shimada, Y., Tagami, J., Sumi, Y. and Sadr, A. (2017) 'Real-Time Imaging of Gap Progress during and after Composite Polymerization'. doi: 10.1177/0022034517709005.

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. 2015 May;103(4):798-806.

Ilie, N. and Stark, K. (2015) 'Effect of different curing protocols on the mechanical properties of low-viscosity bulk-fill composites', pp. 271–279. doi: 10.1007/s00784-014-1262-x.

Ishibashi K, Ozawa N, Tagami J, Sumi Y. Swept-source optical coherence tomography as a new tool to evaluate defects of resin-based composite restorations. J Dent. 2011 Aug;39(8):543-8. doi: 10.1016/j.jdent.2011.05.005.

Jang, J.-H., Park, S.-H. and Hwang, I.-N. (2015) 'Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin.', *Operative dentistry*, 40(2), pp. 172–80. doi: 10.2341/13-307-L.

Kaisarly D, El Gezawi M, Xu X, Rösch P, Kunzelmann KH. Shrinkage vectors of a flowable composite in artificial cavity models with different boundary conditions: Ceramic and Teflon. J Mech Behav Biomed Mater. 2018 Jan;77:414-421. doi: 10.1016/j.jmbbm.2017.10.004.

Kishikawa R, Koiwa A, Chikawa H, Cho E, Inai N, Tagami J. Effect of cavity form on adhesion to cavity floor. Am J Dent 2005;18:311–4.

Labella R, Lambrechts P, Van Meerbeek B, Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. Dent Mater 1999; 15: 128-137.

Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. Dent Mater 2013;29(2):139–56.
Lim BS, Ferracane J, Sakaguchi R, Condon J. Reduction of polymerization contraction stress for dental composites by two-step light-activation. Dent Mater 2002; 18: 436-444.

Lutz E, Krejci I, Oldenburg TR. Elimination of polymerization stresses at the margins of posterior composite resin restorations: a new restorative technique. Quintessence Int 1986;17(12):777–84.

Makishi, P., Shimada, Y., Sadr, A., Tagami, J. and Sumi, Y. (2011) 'Non-destructive 3D imaging of composite restorations using optical coherence tomography : Marginal adaptation of self-etch adhesives', *Journal of Dentistry*. Elsevier Ltd, 39(4), pp. 316–325. doi: 10.1016/j.jdent.2011.01.011.

Opdam, N. J. M., van de Sande, F. H., Bronkhorst, E., Cenci, M. S., Bottenberg, P., Pallesen, U., Gaengler, P., Lindberg, A., Huysmans, M. C. D. N. J. M. and van Dijken, J. W. (2014) 'Longevity of posterior composite restorations: a systematic review and meta-analysis.', *Journal of dental research*, 93(10), pp. 943–949. doi: 10.1177/0022034514544217.

Papadogiannis D, Tolidis K, Gerasimou P, Lakes R, Papadogiannis Y. Viscoelastic properties, creep behavior and degree of conversion of bulk fill composite resins. Dent Mater 2015; 31: 1533-1541.

Pedalino, I., Hartup, G. R. and Vandewalle, K. S. (no date) 'Depth of cure of bulk-fill flowable composite resins.', *General dentistry*, 63(5), pp. e28-34. Available at: http://www.ncbi.nlm.nih.gov/pubmed/26325654 (Accessed: 2 May 2017).

S, A. A. A.-S. B. D., Sc, M. and Ph, D. (2006) 'Effectiveness of Four Different Light-activated Composites Cure with Different Light Energy Densities', (3), pp. 1–6.

Sadr A, Shimada Y, Mayoral JR, Hariri I, Bakhsh TA, Sumi Y. Swept source optical coherence tomography for quantitativeand qualitative assessment of dental composite restorations. Proc SPIE 2011;7884:78840C.

Sampaio CS, Rodrigues RV, Souza-Junior EJ, Freitas AZ, Ambrosano G, Pascon FM, Rontani RP. Effect of restorative system and thermal cycling on the tooth-restoration interface —OCT Evaluation. Oper Dent 2015; 41: 162-170.

Sampaio CS, Chiu KJ, Farrokhmanesh E, Janal M, Puppin-Rontani RM, Giannini M, Bonfante EA, Coelho PG, Hirata R. Microcomputed Tomography Evaluation of Polymerization Shrinkage of Class I Flowable Resin Composite Restorations. Oper Dent. 2017A Jan/Feb;42(1):E16-E23. doi: 10.2341/15-296-L.

Sampaio CS, Atria PJ, Rueggeberg FA, Yamaguchi S, Giannini M, Coelho PG, Hirata R, Puppin-Rontani RM. Effect of blue and violet light on polymerization shrinkage vectors of a CQ/TPO-containing composite. Dent Mater. 2017B Jul;33(7):796-804. doi: 10.1016/j.dental.2017.04.010.

Stansbury JW. Dimethacrylate network formation andpolymer property evolution as determined by the selectionof monomers and curing conditions. Dent Mater 2012;28(1):13–22.
Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, TauböckTT. Influence of irradiation time on subsurface degree ofconversion and microhardness of high-viscosity bulk-fillresin composites. Clin Oral Investig 2015;19(4):831–40.

Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van EndeA, Neves A, et al. Relationship between bond-strength testsand clinical outcomes. Dent Mater 2010;26:e100–21,http://dx.doi.org/10.1016/j.dental.2009.11.148.

Venhoven B, De Gee A, Davidson C. Polymerization contraction and conversion of light-curing BisGMA-based methacrylate resins. Biomaterials 1993; 14: 871-875.)

Versluis A, Douglas W, Cross M, Sakaguchi R. Does an incremental filling technique reduce polymerization shrinkage stresses? J Dent Res 1996; 75: 871-878.

Versluis A, Tantbirojn D, Douglas W. Do dental composites always shrink toward the light? J Dent Res 1998;77:1435–45.

Versluis A, Tantbirojn D, Pintado MR, et al. Residual shrinkage stress distributions in molars after composite restoration. Dent Mater 2004;20:554–64.

Van Ende A, Van de Casteele E, Depypere M, De Munck J, Li X, Maes F, Wevers M, Van Meerbeek B. 3D volumetric displacement and strain analysis of composite polymerization. Dent Mater. 2015 Apr;31(4):453-61. doi: 10.1016/j.dental.2015.01.018.

Yamamoto T, Hanabusa M, Momoi Y, Sakaguchi RL. Polymerization stress of dental resin composite continuesto develop 12 hours after irradiation. J Esthet Restor Dent 2015;27:44–54.

4 - CONCLUSÕES

Em função da metodologia empregada neste estudo, pode-se concluir que ambas as imersões em água destilada e etanol são capazes de degradar resina composta convencional e resina composta Bulk-Fill. A degradação grave foi observada após a imersão em etanol. Dependendo das diferentes fontes de luzes e resinas compostas Bulk-Fill, vários graus de desenvolvimento de fendas foram observados. O compósito Filtek Bulk-Fill flow apresentou a menor formação de fenda polimerizada pela unidade de fotopolimerização LED monowave.

5- REFERÊNCIAS

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;31:437–44.

Algamaiah H, Sampaio CS, Rigo LC, Janal MN, Giannini M, Bonfante EA, Coelho PG, Reis AF, Hirata R. Microcomputed Tomography Evaluation of Volumetric Shrinkage of Bulk-Fill Composites in Class II Cavities. J Esthet Restor Dent. 2017 Apr;29(2):118-127. doi: 10.1111/jerd.12275.

Al-Ahdal, K., Ilie, N., Silikas, N. and Watts, D. C. (2015) 'Polymerization kinetics and impact of post polymerization on the Degree of Conversion of bulk-fill resin-composite at clinically relevant depth.', *Dental materials : official publication of the Academy of Dental Materials*, 31(10), pp. 1207–13. doi: 10.1016/j.dental.2015.07.004.

Almeida Junior LJDS, Penha KJS, Souza AF, Lula ECO, Magalhães FC, Lima DM, Firoozmand LM. Is there correlation between polymerization shrinkage, gap formation, and void in bulk fill composites? A μCT study. Braz Oral Res. 2017 Dec 18;31:e100. doi: 10.1590/1807-3107bor-2017.vol31.0100.

Alshaa, M. M., Haenel, T., Sullivan, B., Labrie, D., Alqahtani, M. Q. and Price, R. B. (2016) 'Effect of a broad-spectrum LED curing light on the Knoop microhardness of four posterior resin based composites at 2 , 4 and 6-mm depths', 45, pp. 14–18.

Alshali, R. Z., Silikas, N. and Satterthwaite, J. D. (2013) 'Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals.', *Dental materials : official publication of the Academy of Dental Materials*, 29(9), pp. e213-7. doi: 10.1016/j.dental.2013.05.011.

Atria PJ, Sampaio CS, Cáceres E, Fernández J, Reis AF, Giannini M, Coelho PG, Hirata R. Micro-computed tomography evaluation of volumetric polymerization shrinkage and degree of conversion of composites cured by various light power outputs. Dent Mater J. 2018 Jan 30;37(1):33-39. doi: 10.4012/dmj.2016-430.

Bakhsh TA, Sadr A, Shimada Y, Mandurah MM, Hariri I, Alsayed EZ, Tagami J, Sumi Y. Concurrent evaluation of composite internal adaptation and bond strength in a class-I cavity. J Dent. 2013 Jan;41(1):60-70. doi: 10.1016/j.jdent.2012.10.003.

Bakhsh TA, Sadr, Shimada, Tagami J, Sumi Y. Non-invasive quantification of resin-dentin interfacial gaps using optical coherence tomography: validation against confocal microscopy. Dent Mater. 2011 Sep;27(9):915-25. doi: 10.1016/j.dental.2011.05.003.

Bayraktar Y, Ercan E, Hamidi MM, Çolak H. One-year clinical evaluation of different types of bulk-fill composites. J Investig Clin Dent. 2016 Jan 22. doi: 10.1111/jicd.12210.

Benetti AR, Havndrup-Pedersen C, Honoré D, Pedersen MK, Pallesen U. Bulk-fill resin composites: polymerization contraction, depth of cure, and gap formation. Oper Dent. 2015 Mar-Apr;40(2):190-200. doi: 10.2341/13-324-L.

Burgess J, Cakir D. Comparative properties of low-shrinkage composite resins. Compendium of Continuing Education in Dentistry 2010;31:10–5. (Spec Iss 2).

Benetti AR, Peutzfeldt A, Lussi A, Flury S. Resin composites: Modulus of elasticity and marginal quality. Dent. 2014 Sep;42(9):1185-92. doi: 10.1016/j.jdent.2014.07.004.

Boaro LC, Brandt WC, Meira JB, Rodrigues FP, Palin WM, Braga RR. Experimental and FE displacement and polymerization stress of bonded restorations as a function of the C-Factor, volume and substrate stiffness. J Dent. 2014 Feb;42(2):140-8. doi: 10.1016/j.jdent.2013.11.016.

Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resincomposites: A systematic review. Dent Mater 2005; 21: 962- 970.

Bucuta S, Ilie N. Light transmittance and micro-mechanicalproperties of bulk fill vs. conventional resin basedcomposites. Clin Oral Investig 2014;18(8):1991–2000.

Cantekin K, Gumus H. In vitro and clinical outcome of sandwich restorations with a bulk-fill flowable composite liner for pulpotomized primary teeth. J Clin Pediatr Dent. 2014 Summer;38(4):349-54.

Cavalcante LM, Schneider LF, Silikas N, Watts DC. Surface integrity of solvent-challenged ormocer-matrix composite. Dent Mater. 2011 Feb;27(2):173-9.

Chiang YC, Rösch P, Dabanoglu A, Lin CP, Hickel R, Kunzelmann KH. Polymerization composite shrinkage evaluation with 3D deformation analysis from microCT images. Dent Mater. 2010 Mar;26(3):223-31. doi: 10.1016/j.dental.2009.09.013.

Cho E, Sadr A, Inai N, Tagami J. Evaluation of resin composite polymerization by three dimensional micro-CT imaging and nanoindentation. Dent Mater. 2011 Nov;27(11):1070-8. doi: 10.1016/j.dental.2011.07.008.

Czasch, P. and Ilie, N. (2013) 'In vitro comparison of mechanical properties and degree of cure of bulk fill composites', pp. 227–235. doi: 10.1007/s00784-012-0702-8.

D'Alpino PH, Bechtold J, dos Santos PJ, Alonso RC, Di Hipólito V, Silikas N, Rodrigues FP. Methacrylate- and silorane-based composite restorations: hardness, depth of cure and interfacial gap formation as a function of the energy dose. Dent Mater. 2011 Nov;27(11):1162-9. doi: 10.1016/j.dental.2011.08.397.

El-Safty S, Silikas N, Watts DC. Creep deformation of restorative resin-composites intended for bulk-fill placement. Dent Mater. 2012 Aug;28(8):928-35.

Erdemir U, Yildiz E, Eren MM, Ozel S. Surface hardness evaluation of different composite resin materials: influence of sports and energy drinks immersion after a

short-term period. J Appl Oral Sci. 2013 Mar-Apr;21(2):124-31.

Feilzer A, Dooren L, Gee Ad, Davidson C. Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface. Eur J Oral Sci 2005; 103: 322- 326.

Ferracane JL, Hilton TJ. Polymerization stress--is it clinically meaningful? Dent Mater. 2016 Jan;32(1):1-10. doi: 10.1016/j.dental.2015.06.020.

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

Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? Dent Mater. 2012 May;28(5):521-8. doi: 10.1016/j.dental.2012.02.002.

Fonseca AS, Gerhardt KM, Pereira GD, Sinhoreti MA, Schneider LF. Do new matrix formulations improve resin composite resistance to degradation processes? Braz Oral Res. 2013 Sep-Oct;27(5):410-6.

Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LE, Martin AA, Ambrosano G, Giannini M. Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites. Dent Mater. 2015 Dec;31(12):1542-51. doi: 10.1016/j.dental.2015.10.001.

Gonçalves F, Kawano Y, Braga RR. Contraction stress related to composite inorganic content. Dent Mater 2010; 26:704–9.

Han SH, Sadr A, Tagami J, Park SH. Internal adaptation of resin composites at two configurations: Influence of polymerization shrinkage and stress. Dent Mater. 2016 Sep;32(9):1085-94. doi: 10.1016/j.dental.2016.06.005.

Hayashi, J., Shimada, Y., Tagami, J., Sumi, Y. and Sadr, A. (2017) 'Real-Time Imaging of Gap Progress during and after Composite Polymerization'. doi: 10.1177/0022034517709005.

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. 2015 May;103(4):798-806.

Ilie, N. and Stark, K. (2015) 'Effect of different curing protocols on the mechanical properties of low-viscosity bulk-fill composites', pp. 271–279. doi: 10.1007/s00784-014-1262-x.

Ilie N, Hickel R. Investigation on a methacrylate-based flowable composite based on the SDRTM technology. Dental Materials 2011;27:348–55.

Ilie N, Keßler A, Durner J. Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites. J Dent. 2013 Aug;41(8):695-702.

Ishibashi K, Ozawa N, Tagami J, Sumi Y. Swept-source optical coherence tomography as a new tool to evaluate defects of resin-based composite restorations. J Dent. 2011 Aug;39(8):543-8. doi: 10.1016/j.jdent.2011.05.005.

Jang, J.-H., Park, S.-H. and Hwang, I.-N. (2015) 'Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin.', *Operative dentistry*, 40(2), pp. 172–80. doi: 10.2341/13-307-L.

Kaisarly D, El Gezawi M, Xu X, Rösch P, Kunzelmann KH. Shrinkage vectors of a flowable composite in artificial cavity models with different boundary conditions: Ceramic and Teflon. J Mech Behav Biomed Mater. 2018 Jan;77:414-421. doi: 10.1016/j.jmbbm.2017.10.004.

Kishikawa R, Koiwa A, Chikawa H, Cho E, Inai N, Tagami J. Effect of cavity form on adhesion to cavity floor. Am J Dent 2005;18:311–4.

Labella R, Lambrechts P, Van Meerbeek B, Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. Dent Mater 1999; 15: 128-137.

Lazarchik DA, Hammond BD, Sikes CL, Looney SW, Rueggeberg FA. Hardness comparison of bulk-filled/transtooth and incremental-filled/occlusally irradiated composite resins. J Prosthet Dent. 2007 Aug;98(2):129-40.

Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. Dent Mater 2013;29(2):139–56.

Lim BS, Ferracane J, Sakaguchi R, Condon J. Reduction of polymerization contraction stress for dental composites by two-step light-activation. Dent Mater 2002; 18: 436-444.

Lutz E, Krejci I, Oldenburg TR. Elimination of polymerization stresses at the margins of posterior composite resin restorations: a new restorative technique. Quintessence Int 1986;17(12):777–84.

Makishi, P., Shimada, Y., Sadr, A., Tagami, J. and Sumi, Y. (2011) 'Non-destructive 3D imaging of composite restorations using optical coherence tomography : Marginal adaptation of self-etch adhesives', *Journal of Dentistry*. Elsevier Ltd, 39(4), pp. 316–325. doi: 10.1016/j.jdent.2011.01.011.

Mayworm CD, Camargo JRSS, Bastian FL. Influence of artificial saliva on abrasive wear and microhardness of dental composites filled with nanoparticles. J Dent 2008;36(9):703–10.

Münchow EA, Ferreira AC, Machado RM, Ramos TS, Rodrigues-Junior SA, Zanchi CH. Effect of acidic solutions on the surface degradation of a micro-hybrid composite resin. Braz Dent J. 2014;25(4):321-6.

Nayif MM, Nakajima M, Foxton RM, Tagami J. Bond strength and ultimate tensile strength of resin composite filled into dentine cavity; effect of bulk and incremental filling technique. J Dent. 2008 Mar;36(3):228-34.

Opdam, N. J. M., van de Sande, F. H., Bronkhorst, E., Cenci, M. S., Bottenberg, P., Pallesen, U., Gaengler, P., Lindberg, A., Huysmans, M. C. D. N. J. M. and van Dijken, J. W. (2014) 'Longevity of posterior composite restorations: a systematic review and meta-analysis.', *Journal of dental research*, 93(10), pp. 943–949. doi: 10.1177/0022034514544217.

Papadogiannis D, Tolidis K, Gerasimou P, Lakes R, Papadogiannis Y. Viscoelastic properties, creep behavior and degree of conversion of bulk fill composite resins. Dent Mater 2015; 31: 1533-1541.

Park J, Chang J, Ferracane J, Lee IB. How should composite be layered to reduce shrinkage stress: incremental or bulk filling? Dent Mater. 2008 Nov;24(11):1501-5.

Pedalino, I., Hartup, G. R. and Vandewalle, K. S. (no date) 'Depth of cure of bulk-fill flowable composite resins.', *General dentistry*, 63(5), pp. e28-34. Available at: http://www.ncbi.nlm.nih.gov/pubmed/26325654 (Accessed: 2 May 2017).

Poskus LT, Placido E, Cardoso PE. Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations. Dent Mater 2004;20:726-32.

Roggendorf MJ, Krämer N, Appelt A, Naumann M, Frankenberger R. Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. J Dent. 2011 Oct;39(10):643-7.

Sarrett DC. Clinical challenges and the relevance of materials testing for posterior composite restorations. Dental Materials 2005;21:9–20.

S, A. A. A.-S. B. D., Sc, M. and Ph, D. (2006) 'Effectiveness of Four Different Light-activated Composites Cure with Different Light Energy Densities', (3), pp. 1–6.

Sadr A, Shimada Y, Mayoral JR, Hariri I, Bakhsh TA, Sumi Y. Swept source optical coherence tomography for quantitativeand qualitative assessment of dental compositerestorations. Proc SPIE 2011;7884:78840C.

Sampaio CS, Rodrigues RV, Souza-Junior EJ, Freitas AZ, Ambrosano G, Pascon FM, Rontani RP. Effect of restorative system and thermal cycling on the tooth-restoration interface —OCT Evaluation. Oper Dent 2015; 41: 162-170.

Sampaio CS, Chiu KJ, Farrokhmanesh E, Janal M, Puppin-Rontani RM, Giannini M, Bonfante EA, Coelho PG, Hirata R. Microcomputed Tomography Evaluation of Polymerization Shrinkage of Class I Flowable Resin Composite Restorations. Oper Dent. 2017A Jan/Feb;42(1):E16-E23. doi: 10.2341/15-296-L.

Sampaio CS, Atria PJ, Rueggeberg FA, Yamaguchi S, Giannini M, Coelho PG, Hirata R, Puppin-Rontani RM. Effect of blue and violet light on polymerization shrinkage vectors of a CQ/TPO-containing composite. Dent Mater. 2017B Jul;33(7):796-804. doi: 10.1016/j.dental.2017.04.010.

Stansbury JW. Dimethacrylate network formation andpolymer property evolution as determined by the selectionof monomers and curing conditions. Dent Mater 2012;28(1):13–22.

Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, TauböckTT. Influence of irradiation time on subsurface degree ofconversion and microhardness of high-viscosity bulk-fillresin composites. Clin Oral Investig 2015;19(4):831–40.

Tiba A, Zeller GG, Estrich CG, Hong A. A laboratory evaluation of bulk-fill versus traditional multi-increment-fill resin-based. JADA 2013;144(10):1182-3.

van Dijken JW. A clinical evaluation of anterior conventional, microfiller, and hybrid composite resin fillings. A 6-year follow-up study. Acta Odontol Scand. 1986 Dec;44(6):357-67.

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 Sep;30(9):e245-51.

van Dijken JW, Pallesen U. Posterior bulk-filled resin composite restorations: A 5-year randomized controlled clinical study. J Dent. 2016 Aug;51:29-35.

Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, Van Meerbeek B. Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity- bottom dentin. Dent Mater. 2013 Mar;29(3):269-77.

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 Apr;40(4):295-303.

Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van EndeA, Neves A, et al. Relationship between bond-strength testsand clinical outcomes. Dent Mater 2010;26:e100–21,http://dx.doi.org/10.1016/j.dental.2009.11.148.

Venhoven B, De Gee A, Davidson C. Polymerization contraction and conversion of light-curing BisGMA-based methacrylate resins. Biomaterials 1993; 14: 871-875.)

Versluis A, Douglas W, Cross M, Sakaguchi R. Does an incremental filling technique reduce polymerization shrinkage stresses? J Dent Res 1996; 75: 871-878.
Versluis A, Tantbirojn D, Douglas W. Do dental composites always shrink toward the light? J Dent Res 1998;77:1435–45.

Versluis A, Tantbirojn D, Pintado MR, et al. Residual shrinkage stress distributions in molars after composite restoration. Dent Mater 2004;20:554–64.

Van Ende A, Van de Casteele E, Depypere M, De Munck J, Li X, Maes F, Wevers M, Van Meerbeek B. 3D volumetric displacement and strain analysis of composite polymerization. Dent Mater. 2015 Apr;31(4):453-61. doi: 10.1016/j.dental.2015.01.018.

Yamamoto T, Hanabusa M, Momoi Y, Sakaguchi RL. Polymerization stress of dental resin composite continuesto develop 12 hours after irradiation. J Esthet Restor Dent 2015;27:44–54.