AMÉRICO BORTOLAZZO CORRER

INFLUÊNCIA DO TAMANHO DA CAVIDADE, TÉCNICA DE FOTOATIVAÇÃO E SISTEMA RESTAURADOR SOBRE A ADAPTAÇÃO MARGINAL, DUREZA KNOOP E RESISTÊNCIA DE UNIÃO À DENTINA RADICULAR

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para a obtenção do Título de Doutor em Materiais Dentários.

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"Bom mesmo é ir à luta com determinação, abraçar a vida e viver com paixão, perder com classe e vencer com ousadia, pois o triunfo pertence a quem se atreve... E a vida é muito para ser insignificante."

(Charles Chaplin)

RESUMO

O objetivo deste estudo foi verificar a influência do volume de compósito, técnica de fotoativação e sistema restaurador sobre a adaptação marginal, dureza Knoop e resistência de união de compósitos à dentina humana radicular. Este estudo foi dividido em 2 Capítulos. O Capítulo I verificou o efeito do volume de compósito (mantendo-se o mesmo fator C) e sistema restaurador sobre a adaptação marginal, dureza Knoop e resistência de união “push-out” a dentina radicular. Foram utilizados 90 pré-molares hígidos divididos em 9 grupos (n=10), de acordo com o volume de compósito (pequena, média e grande) e sistema restaurador (Filtek Z350, Filtek Z350 Flow e Filtek LS). Os compósitos foram fotoativados por LED Ultralume 5 (Ultradent) por 20 s para Filtek Z350, Filtek Z350 Flow e 40 s para Filtek LS. A adaptação marginal foi avaliada após 24h utilizando o método do corante superficial, medindo-se a porcentagem corada em relação ao perímetro da cavidade (Caries Detector). Após a análise da adaptação marginal, as amostras foram submetidas ao ensaio de resistência de união “push-out” e de dureza Knoop na superfície do compósito. Os dados foram submetidos à análise de variância dois fatores e teste de Tukey ($p \leq 0,05$). O volume de compósito não influenciou a adaptação marginal dos compósitos. Filtek LS apresentou os melhores resultados de adaptação marginal e resistência de união. Os menores valores de resistência foram apresentados pelo Filtek Z350. O volume de compósito influenciou nos valores de resistência de união e de dureza Knoop, dependendo do compósito utilizado. Os maiores valores de dureza foram apresentados pelos volumes de compósito médios. A maior dureza foi do compósito Filtek Z350, seguido por Filtek LS e Filtek Z350 flow. No Capítulo II o objetivo foi verificar a influência da modulação da intensidade da luz durante a fotoativação e volume de compósito sobre a dureza Knoop e resistência de união do compósito Filtek Z350 à dentina radicular. Foram utilizados 90 pré-molares hígidos divididos em 9 grupos (n=10), segundo o volume de compósito (pequena, média e grande) e método de fototivação (luz contínua, “pulse delay” e “soft-start”). O compósito foi fotoativado

por LED Ultralume 5 (Ultradent) pelos métodos citados anteriormente. Após 24h foi realizado o ensaio de resistência de união “push-out” e de dureza Knoop na superfície do compósito. Os dados foram submetidos à análise de variância dois fatores e teste de Tukey ($p \leq 0,05$). Os resultados de resistência de união mostraram que os métodos de modulação da intensidade da luz foram superiores ao contínuo, sem diferença entre eles. Cavidade média foi significativamente superior que cavidade pequena e grande para o método contínuo. O ensaio de dureza Knoop mostrou que o melhor comportamento foi apresentado pelo método *pulse delay* e para cavidades médias. O volume de compósito influenciou os valores de resistência de união, dureza Knoop e adaptação marginal a dentina radicular. Os maiores valores de resistência de união e adaptação marginal foram apresentados pelo compósito Filtek LS e os maiores valores de dureza pelo compósito Filtek Z350. Os métodos de modulação da intensidade de luz mostraram os melhores resultados de resistência de união, sem detrimento dos valores de dureza Knoop.

Palavras-chave: fotoativação, resina composta, resistência de união, adaptação marginal, dureza Knoop, fator C.

ABSTRACT

The polymerization shrinkage is one of the major drawbacks of the resin composites. The polymerization stress depends on the viscosity of the composite, rate of reaction, geometric configuration of the cavity and irradiance used during photoactivation. However, the volume of composite and low shrink monomers has been investigated. The aim of this study was to evaluate the influence of the cavity size, photoactivation technique and composite composition on marginal adaptation, Knoop hardness and push-out bond strength of composites to root dentin. This study was divided into 2 Chapters. In the Chapter 1 was to evaluate the effect of the cavity size (with similar C-factor) and restorative system on marginal adaptation, Knoop hardness and push-out bond strength to root dentin. Ninety premolars were divided into 9 groups, according to cavity size (small, middle and large) and resin composite (Filtek Z350, Filtek Z350 Flow e Filtek P90). The resin composites Filtek Z350, Filtek Z350 Flow were photoactivated with LED Ultralume 5 (Ultradent) for 20 s and Filtek P90 for 40 s. The marginal adaptation was analyzed after 24 h using Caries Detector. After that, the specimens were submitted to push-out Bond strength. The Knoop hardness examination was performed at the top surface. The data were submitted to ANOVA two-way and post hoc Tukey-s test at 95% significance level. The cavity size had no influence on marginal adaptation of the resin composites. Filtek P90 presented the best marginal adaptation, significantly better than other composites for large cavity. Filtek P90 showed the highest bond strength, significantly higher than other composites. Filtek Z350 showed the lowest bond strength results. Middle cavity presented Bond strength significantly higher than large cavity with the composite Filtek Z350. The highest Knoop hardness was showed by Filtek Z350, followed by Filtek P90 and Filtek Z350 flow. The aim of the Chapter 2 was to evaluate the influence of modulated photoacivation methods and cavity size on Knoop hardness and push-out bond strength of the composite Filtek Z350 to root dentin. Ninety premolars were divided into 9 groups, according to cavity size (small, middle and

large) and photoactivation method (continuous light, pulse delay and soft-start). The composite was inserted and photoactivated with LED Ultralume 5 (Ultradent) by the methods aforementioned. The specimens were submitted to push-out Bond strength and Knoop hardness test. The data were submitted to ANOVA two-way and post hoc Tukey-s test at 95% significance level. Pulse delay showed the highest Bond strength results, significantly higher continuous light for small and large cavities. Middle cavity showed Bond strength significantly higher than small and large cavities for continuous light. Continuous light presented Knoop hardness significantly higher than soft-start for small cavity. For middle cavity, pulse delay showed Knoop hardness significantly higher than continuous light and soft-start. Pulse delay and soft-start showed Knoop hardness significantly higher than continuous light. The cavity size had influence on marginal adaptation, Knoop hardness and bond strength to root dentin. Filtek LS showed the highest bond strength and marginal adaptation results and Filtek Z350 presented the highest Knoop hardness results. The light intensity modulation methods showed the best bond strength results without decrease the Knoop hardness values.

Key-words: photoactivation, resin composite, bond strength, marginal adaptation, Knoop hardness, C-factor.

SUMÁRIO

INTRODUÇÃO GERAL

Os compósitos fotoativados são os materiais restauradores estéticos mais utilizados para restaurações diretas em odontologia. Modificações têm sido feitas desde o seu desenvolvimento, com o objetivo de melhorar suas propriedades estéticas e mecânicas. Dentre as melhorias nos compósitos, os avanços ocorreram nas partículas de carga, tanto no tamanho quanto tipo de partícula. As melhorias nas partículas de carga possibilitaram a utilização dos compósitos em restaurações posteriores, pela maior inclusão de partículas nos compósitos, aumentando sua resistência mecânica. A diminuição no tamanho das partículas possibilitou o menor desgaste dos compósitos e melhora das propriedades estéticas, pela possibilidade de maior polimento e manutenção do brilho, aumentando sua longevidade.

Embora o desenvolvimento tecnológico tenha possibilitado a evolução dos compósitos, eles possuem uma desvantagem que é inerente à sua reação de cura, que é a contração. Durante a reação de polimerização, os monômeros se aproximam (na cadeia e entre as cadeias), levando a diminuição volumétrica do compósito. A contração de polimerização é determinada por diversos fatores como grau de conversão (Silikas *et al.*, 2000), tipo e peso molecular dos monômeros e quantidade de partículas de carga. O grau de conversão está relacionado com as propriedades mecânicas e estéticas dos compósitos (Asmussen, 1982). Portanto, a diminuição do grau de conversão pode afetar negativamente as propriedades mecânicas dos compósitos e sua biocompatibilidade (Caughman *et al.*, 1991).

Compósitos atuais utilizam em suas formulações monômeros com alto peso molecular, resultando em menor contração de polimerização (Ferracane, 2005). O Bisfenol-A glicidil dimetacrilato (Bis-GMA) é o monômero mais utilizado nos compósitos odontológicos. Ele apresenta alto peso molecular e relativamente baixa contração de polimerização (Anseth *et al.*, 1996). Devido à alta viscosidade do Bis-GMA, são introduzidos monômeros diluentes com o objetivo de melhorar a manipulação dos compósitos e possibilitar a maior incorporação de partículas de

carga. Dentre os monômeros diluentes, um dos mais utilizados é o trietileno glicol dimetacrilato (TEGDMA), que é mais flexível, tem menor peso molecular e possui viscosidade muito menor que o Bis-GMA (Anseth *et al.*, 1996; Dulik *et al.*, 1981). Entretanto, a introdução do TEGDMA leva ao aumento na sorção de água e na contração de polimerização (Dulik *et al.*, 1981). Mais recentemente foi introduzido nos compósitos o bisfenol-A dimetacrilato etoxilado (Bis-EMA). Este monômero apresenta menor viscosidade comparado ao BisGMA e, portanto, pode minimizar a quantidade de TEGDMA dentro do compósito. O BisEMA é estruturalmente semelhante ao Bis-GMA, com um anel fenílico central rígido. Entretanto, o BisEMA não possui os grupos hidroxilas pendentes, que são responsáveis pela maior hidrofilia e viscosidade do Bis-GMA, devido às pontes de hidrogênio com os grupos carbonílicos (Kalachandra *et al.*, 1997; Lemon *et al.*, 2007). Recentemente foi introduzido no mercado o compósito restaurador Filtek LS (3M/ESPE), que apresenta em sua formulação outro tipo de monômero, o silorano. Estes monômeros apresentam durante a reação de polimerização a abertura de anéis, possibilitando redução na contração de polimerização.

Outra forma de diminuir a contração de polimerização é acrescentar maior quantidade de partículas de carga. Assim há redução na quantidade de matriz orgânica, que é o fator responsável pela contração desses materiais restauradores. Durante a contração de polimerização, tensões são geradas no material restaurador. Dependendo do módulo de elasticidade do material, essa tensão pode ser dissipada dentro do próprio material ou pode ser transmitida para a interface de união e afetar negativamente a união entre o material restaurador e a parede cavitária (Koran & Kurschner, 1998). Se a intensidade da tensão transmitida para a interface de união for muito alta, pode ocorrer a formação de fendas entre o material restaurador e o dente (Unterbrink & Muessner, 1995), ou ainda fraturas do esmalte ou do material restaurador, diminuindo a vida útil das restaurações (Davidson *et al.*, 1984). A presença de fendas entre o material restaurador e o dente pode ser verificada através da análise em microscópio óptico, microscópio eletrônico de varredura ou pela utilização de corantes que

penetram nas fendas presentes entre o material restaurador e o dente (Alonso *et al*., 2006).

A tensão de contração é influenciada pela composição do material, configuração da cavidade e pela técnica de fotoativação. Outro modo de reduzir a tensão gerada durante a contração de polimerização é inserir o compósito em incrementos, diminuindo o fator C. Segundo Feilzer *et al*., 1987, quanto maior a área aderida, ou seja, quanto maior o fator C, menor a capacidade de escoamento do compósito durante a polimerização, de modo que as forças de adesão tornam-se insuficientes para preservar a união na interface dente-material restaurador. A inserção do compósito em incrementos oblíquos reduziria o fator C, diminuindo as tensões de contração e melhoraria a adaptação marginal das restaurações (He *et al*., 2007).

A formulação dos compósitos tem influência significativa na tensão de contração. A tensão de contração pode ser influenciada pelo tipo de monômero, pois monômeros com alto peso molecular contraem menos que monômeros de baixo peso molecular; pela quantidade de partículas de carga; pois quanto maior a quantidade de partículas de carga; menor a contração; pela quantidade de fotoiniciadores, pois quanto maior a quantidade de fotoiniciadores, maior a quantidade de radicais livres gerados durante o início da fotoativação e maior a tensão gerada no material restaurador (Ferracane, 2005).

A tensão de contração está intimamente relacionada com a velocidade da reação de polimerização, e pode ser controlada pela diminuição ou aumento da intensidade de luz (Irie *et al*., 2002). Muitas técnicas têm sido propostas com o objetivo de controlar a velocidade durante o processo de polimerização (Uno & Asmussen, 1991). As técnicas se baseiam na polimerização inicial com intensidade de luz reduzida, seguido de polimerização com elevada intensidade, ou, utilizando ciclos com presença e ausência de luz (Feilzer *et al*., 1995; Koran & Kurschner, 1998; Pires *et al*., 1993; Unterbrink & Muessner, 1995). Assim, a menor taxa de conversão monomérica inicial permite o escoamento do material, gerando baixa tensão que pode ser dissipada internamente no material restaurador, e

posteriormente, a alta intensidade na polimerização promove adequado grau de conversão para obtenção de propriedades físicas e biológicas satisfatórias (Davidson *et al.*, 1984; Feilzer *et al.*, 1995; Watts & al Hindi, 1999).

A redução das tensões geradas durante a reação de polimerização é de fundamental importância para o sucesso das restaurações odontológicas. Entretanto, a influência do volume de compósito utilizando diferentes modos de fotoativação e materiais restauradores com diferentes módulos de elasticidade sobre a contração de polimerização e resistência de união ainda não foi esclarecido, sendo a fonte de investigação deste estudo.

Em vista do questionamento a respeito da influência da contração de polimerização sobre a união compósito/substrato dental, o propósito deste estudo foi verificar a influência do volume de compósito (mantendo o mesmo fator C), técnica de fotoativação (Luz contínua, *pulse-delay* e *soft*-start) e sistema restaurador (Filtek Z350, Filtek Z350 Flowable e Filtek LS) sobre a adaptação marginal, dureza Knoop e resistência de união de compósitos à dentina radicular.[1]

[1] Este trabalho foi realizado no formato alternativo com base na Informação da Comissão Central de Pós-Graduação (CCPG) da Universidade Estadual de Campinas (UNICAMP) nº002/06.

CAPÍTULO 1

Effect of volume of composite and low-shrinkage dental composite on marginal adaptation, Knoop hardness and push-out bond strength to root dentin

ABSTRACT

The aim of this study was to evaluate the effect of the volume of composite (with similar C-factor) and restorative system on marginal adaptation, Knoop hardness and push-out bond strength to root dentin. Truncated cone preparations were made in ninety premolars, divided into 9 groups, according to volume of composite (small, medium and large) and resin composite (Filtek Z350, Filtek Z350 Flow and Filtek LS, shade A3) (n=10). The resin composites Filtek Z350, Filtek Z350 Flow were photoactivated with LED Ultralume 5 (Ultradent) for 20 s and Filtek LS for 40 s. The marginal adaptation was evaluated after 24 h using Caries Detector. After this, the specimens were submitted to the push-out bond strength test. The Knoop hardness examination was performed on the top surface, 7 days after the restorative procedure. The data were submitted to ANOVA two-way and post hoc Tukey's test at a 95% level of significance. The volume of composite had no influence on marginal adaptation of the resin composites. Filtek LS presented the best marginal adaptation, significantly better than other composites for the large volume. Filtek LS showed the highest bond strength, significantly higher than that of the other composites. Filtek Z350 showed the lowest bond strength results. The medium-sized volume presented significantly higher bond strength than the large volume with the composite Filtek Z350. The highest Knoop hardness was shown by Filtek Z350, followed by Filtek LS and Filtek Z350 flow. The low-shrinkage composite increased the marginal adaptation and the bond strength to dentin and

showed Knoop hardness lower than that of the conventional composite. The volume of composite did not affect the marginal adaptation, but showed an influence on bond strength and Knoop hardness values.

Key-words: resin composite, bond strength, marginal adaptation, Knoop hardness, C-factor

INTRODUCTION

Polymerization shrinkage of resin composites remains a major obstacle to their clinical success as dental restorative materials. Polymerization shrinkage is clinically undesirable because it stresses tooth-composite adhesive interfaces and deforms the tooth itself[1]. Photoinitiated polymerization, which occurs more rapidly than chemically initiated reactions, may produce more shrinkage stress[2]. These stresses may cause microfractures in the tooth enamel, marginal gap formation and subsequent microleakage, or pain[3,4]. Reduction in polymerization shrinkage stress can be obtained in several ways. Attempts have been made to achieve this by using incremental layering of the composites during insertion[5,6] and by using low viscosity, low-e-modulus resin between the bonding agent and restorative resin to act as an “elastic buffer” or “stress breaker” capable of relieving contraction stresses and improving marginal integrity[7-9]. A second alternative is the so called slow-polymerization technique[7,10].

In addition to polymerization shrinkage, several other factors may influence shrinkage stress and gap formation at the tooth-resin composite interface. Feilzer et al.[11] showed that shrinkage stress is related to the cavity configuration, the C-factor, defined as the ratio of bonded to unbonded surfaces of the restoration[11]. Restoring a box-shaped preparation with incremental placement of resin composite has been suggested, based on the concept of reducing the volume of resin to be photopolymerized[4,11-15].

According to the aforementioned authors, in cavities with a C-factor of less than 1, shrinkage stress develops slowly and the resin composite remains bonded to the cavity walls. Braga et al[16], using a photoelastic analysis, showed that cylindrical cavities with the same volume of resin composite developed numerically higher fringe orders at internal angles when the C-factor was higher. Watts & Satterthwaite[17] found that the axial shrinkage-stress depends upon both C-factor and composite mass. The extent of shrinkage stress is also dependent on the viscoelastic properties of the resin composite[18,19]. At a given polymerization shrinkage, the most rigid resin composite will produce the highest shrinkage stress, and consequently, increase gap formation at the tooth-resin composite interface[19,20].

The shrinkage intrinsic to methacrylate resin has remained a major challenge. Therefore, exchanging the resin seems to be the most promising pathway to solving the shrinkage problem. Recently a new commercial resin composite was introduced; the so called low-shrinkage restorative material Filtek LS. The polymerization process of Filtek LS restorative occurs via a cationic ring-opening reaction, which results in lower polymerization shrinkage, compared with the methacrylate-based resins, which polymerize via a radical addition reaction of their double bonds. The low-shrinkage Filtek LS restorative is based on the new ring-opening silorane chemistry. Siloranes are a totally new class of compounds for use in dentistry. The name silorane derives from its chemical building blocks siloxanes and oxiranes. In contrast to the linear-reactive groups of methacrylates, the ring-opening chemistry of the siloranes starts with the cleavage and opening of the ring systems. This process gains space and counteracts the loss of volume that occurs in the subsequent step, when the chemical bonds are formed. In total, the ring-opening polymerization process yields a volumetric shrinkage reduced to less than 1%[21].

Since shrinkage is caused by the resin matrix, the lower the proportion of resin in a composite, the lower the shrinkage will be. However, the question of whether resin–dentin bond strength can be affected by volume of composite and

resin composite with different characteristics still remains. The aim of this study was to evaluate the effect of the volume of composite and restorative system on the push-out bond strength, marginal adaptation and Knoop hardness of composite restorations. The tested hypothesis is that the low-shrinkage composites increase the bond strength and marginal adaptation of the composite restorations. The second hypothesis is that the small volumes present higher bond strength and marginal adaptation than large volumes, irrespective of the resin composite. The third hypothesis is that the Knoop hardness of the three composites and volume of composites are similar.

MATERIALS AND METHODS

Selection and teeth preparation

Ninety premolars were stored in 0.1% aqueous solution of thymol at 4°C. The crowns were cut off at the cement-enamel junction using a double-faced diamond disk under water cooling (KG Sorensen). Slices 3 mm thick were obtained from the cervical portion of root and ground with 400-grit SiC paper to thicknesses of 1.5 mm, 2.1 mm, and 2.5 mm. Truncated cone preparations of 3 different sizes (large – 2.3 mm top diameter x 1.6 mm bottom diameter x 2.5 mm deep; medium - 1.9 mm top diameter x 1.4 mm bottom diameter x 2.1 mm deep; small – 1.4 mm top diameter x 1.0 mm bottom diameter x 1.5 mm deep) were prepared in the root canal of each root slice, using a truncated cone diamond tipped bur with the following dimensions (top diameter of 2.4 mm x bottom diameter of 0.8 mm, and 5.0 mm in height) mounted in a high-speed handpiece (KaVo), under constant air-water cooling in a standard cavity preparation device (Figure 1).

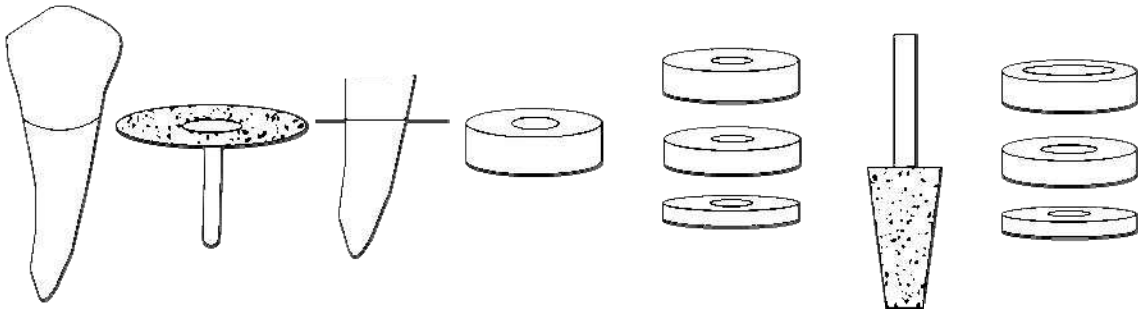

Figure 1. Sequence of specimen preparations for obtaining different cavity sizes.

The diamond burs were replaced after every five preparation. The volumes of the composite were 7.54, 4.81, and 1.71 mm^3 for large, medium and small volumes, respectively, and were calculated according to the following equation:

$$V = 1/3\pi h(R^2 + Rr + r^2)$$

V = volume of composite
h = height
R = higher radius
r = lower radius

The C-factor of the preparations (2.4) was calculated according to the following equation:

$$\text{C-factor} = \frac{\pi(R+r)Vh^2 + (R-r)^2}{(\pi R^2) + (\pi r^2)}$$

h = height
R = higher radius
r = lower radius

Restorative procedures

The specimens were randomly assigned into 9 groups (n=10), according to the restorative system and volume of composite. The restorative materials used in this study are listed on Table 1.

Table 1. Composition of the material used in this study.

Material	Composition	Shade	Batch n°
Scotchbond Etchant	Phosphoric acid 35%, silica gel, water		7KK
Adper Single Bond 2	Ethanol, BisGMA, Silica, HEMA, Dimethacrylate, Copolymer Of Acrylic And Itaconic Acids, Water		8PR
LS System Adhesive Self-Etch Primer and Bond	**Primer**: Phosphorylated methacrylates, Vitrebond™ copolymer, BisGMA, HEMA, Water, Ethanol, silica, Initiators, Stabilizers		7AL
	Adhesive: Hydrophobic dimethacrylate, Phosphorylated methacrylates, TEGDMA, Silane-treated silica filler, Initiators, Stabilizers		7AF
Filtek Z350	Silane treated ceramic/silica (59.5%v), BisEMA, DUDMA, BisGMA, BisPMA, TEGDMA, Water	A3	6GX
Filtek Z-350 Flow	Silane treated ceramic/silica (55%v), BisEMA, DUDMA, BisGMA, TEGDMA, Water, Dimethacrylate Polymer	A3	8GC
Filtek LS	Silorane resin, Initiating system: camphorquinone, iodonium salt, electron donor, Quartz filler, Yttrium fluoride, Stabilizers, Pigments	A3	8CF

Bis-GMA = bisphenol-A-glycidyl methacrylate; Bis-EMA = bisphenol-A-ethoxylate glycidyl methacrylate; DUDMA = diurethane dimethacrylate; TEGDMA = triethylene glycol dimethacrylate; Bis-PMA = bisphenol-A-polyethylene glycol diether dimethacrylate

Preparations filled with Filtek Z350 and Filtek Z350 flowable were etched using 35% phosphoric acid for 15 seconds on dentin and rinsed for 15 seconds. Adper Single Bond 2 adhesive system was applied according to the manufacturer's instructions and photoactivated for 10 seconds at 800 mW/cm^2 (Ultralume LED5, Ultradent). In the cavities filled with Filtek LS, the LS System Adhesive Self-Etch Primer was applied for 15 seconds with a black microbrush, followed by gentle air dispersion and 10 seconds of light polymerization. After this, the LS System Adhesive Bond was applied with green microbrush, followed by gentle air dispersion and 10 seconds of light polymerization. After application of the adhesive systems, the resin composites were placed in bulk. A Mylar strip was placed over the cavity and used to force the composite to adapt to the preparation walls and to extrude the excess material. Filtek Z350 and Filtek Z350 flowable and Filtek LS were photoactivated for 20, 20 and 40 seconds, respectively, with 800 mW/cm^2 (Ultralume LED5, Ultradent) at the higher diameter surface. The irradiance was frequently checked by a radiometer (Demetron Research). After concluding the photoactivation procedures, the samples were stored in distilled water at 37˚C for 24 hours and wet-polished with 1200-grit SiC paper for evaluating marginal adaptation.

Marginal Adaptation Evaluation

To determine the marginal adaptation at the surface, a 1.0% acid red propylene glycol solution (Caries Detector, Kuraray, Osaka, Japan) was applied at the restoration margins for 10 s [12]. After dye staining, the specimens were rinsed in tap water for 10 seconds and gently blown dry. A digital image of each specimen was obtained at this stage. The length of staining along the margins was measured using Image Tool 2.0 software (UTHSC, San Antonio, TX, USA). Marginal adaptation (%) was calculated as the ratio of the stained margin by the total length of the margin. Data were transformed (arc-sen x/100) and submitted to two-way ANOVA and Tukey's post-hoc test at a predetermined significance level of .05

Bond Strength Test

The bond strength test was conducted using a push-out test [19]. The sample was placed on top of a metal device with an aperture that allowed the smaller diameter of the restoration to be in contact with an aspheric device, connected to the load cell of a universal testing machine (Instron, model 4411). This aspheric device applied a compressive force on the smaller diameter surface of the restoration until the tooth-composite bond ruptured. The push-out test was performed at a crosshead speed of 0.5 mm/min. Maximum load was divided by bonded area, and the bond strength results (MPa) data were transformed (root X+0) and submitted to two-way ANOVA and Tukey's post-hoc test at a predetermined significance level of .05.

Knoop hardness test

After the push-out bond strength test, the composites were embedded in acrylic resin and ground and polished using 600, 1200, and 2000 SiC papers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE 53600-000, Brazil) on an automated polisher under water cooling. The specimens were dried and submitted to the Knoop hardness measurements in a microhardness tester (HMV-2000, Shimadzu, Tokyo 101, Japan), with load of 50 g and time of 10 s. Three readings were performed at the top surface, and an mean value was calculated. The data were submitted to two-way ANOVA and Tukey post-hoc test, at a predetermined significance level of .05.

RESULTS

ANOVA detected a significant difference for resin composite (p=.00025) in the marginal adaptation evaluation. The factor volume of composite individually and the interaction between volume of composite and restorative system were not

significant (p=0.07250 and p=0.66167, respectively). The Filtek LS inserted in large cavities showed significantly lower gap formation than the other resin composites. There was no significant difference among small, medium, and large volume groups (Table 2).

Table 2. Means (standard deviation) of marginal adaptation (%) of resin composites inserted in cavities with different volumes.

Resin Composite	Gap (%)	
Filtek z350	19.70 (17.63)	A
Filtek flow Z350	17.73 (12.82)	A
Filtek LS	4.07 (7.63)	B

Means followed by different letter represent statistical difference (p<.05).

With regard to the push-out bond strength, ANOVA detected a statistically significant difference for resin composite (p=.00001) and for the interaction between restorative system and volume of composite (p=.018, which means that depending on the restorative system, the volume of composite had a different effect on bond strength. The factor volume of composite individually was not significant (p=.99). The results, according to the Tukey's test, are listed in Table 3. Filtek LS resin composite showed the highest bond strength results, statistically higher than Filtek Z350 and Filtek Z350 flowable for all volume of composites. Filtek Z350 flowable showed statistically higher bond strength results than Filtek Z350 for small and large volumes. The large volume showed significantly lower bond strength than medium volumes, only when the resin composite Filtek Z350 was used.

Table 3. Means (standard deviation) of push-out bond strength (MPa) of resin composites inserted in cavities with different volumes.

Material	Volume of composite		
	Small	**Middle**	**Large**
Filtek z350	4.13 (2.25) c, AB	5.70 (1.40) b, A	3.54 (1.63) c, B
Filtek flow Z350	6.25 (2.14) b, A	4.73 (2.17) b, A	6.59 (2.70) b, A
Filtek LS	9.79 (2.96) a, A	9.34 (2.04) a, A	9.99 (1.22) a, A

Means followed by different small letter in the column and capital letter in the row represent statistical difference ($p<0.05$).

For Knoop hardness, ANOVA detected a statistically significant difference for resin composite (p=.00001) and for the interaction between volume of composite and restorative system (p=.00001). The factor volume of composite individually was not significant (p=.07503). The Tukey test results are presented in Table 4. Medium and small volumes presented statistically higher Knoop hardness than the large volume with Filtek Z350 and for Filtek LS, the medium volume value was statistically higher than that of small and large volumes. For Filtek Z350 flowable, the large volume showed statistically higher Knoop hardness than small and medium volumes. Filtek Z350 showed the highest Knoop hardness, significantly different from Filtek LS and Filtek Z350 for all volume of composites. Filtek LS was significantly harder than Filtek Z350 flowable for small and medium volumes.

Table 4. Means (standard deviation) of Knoop hardness of resin composites inserted in cavities with different volumes.

Material	Volume of composite					
	Small		Middle		Large	
Filtek z350	61.97 (1.54)	a, A	62.12 (1.67)	a, A	58.80 (1.80)	a, B
Filtek flow Z350	35.16 (1.33)	c, B	34.03 (1.06)	c, B	37.50 (1.62)	b, A
Filtek LS	38.21 (1.77)	b, B	40.57 (1.07)	b, A	37.15 (1.38)	b, B

Means followed by different small letter in the column and capital letter in the row represent statistical difference ($p<0.05$).

Failure mode classification is shown in Figure 1. Small and Medium volumes showed adhesive failure as the most frequent failure mode. Medium volumes showed equal percentages of adhesive and mixed failures. Large volumes showed 100% of adhesive failure for Filtek Z350 and predominance of mixed failure for Filtek LS. Cohesive failure in resin was found in Filtek LS groups inserted in medium and large volumes.

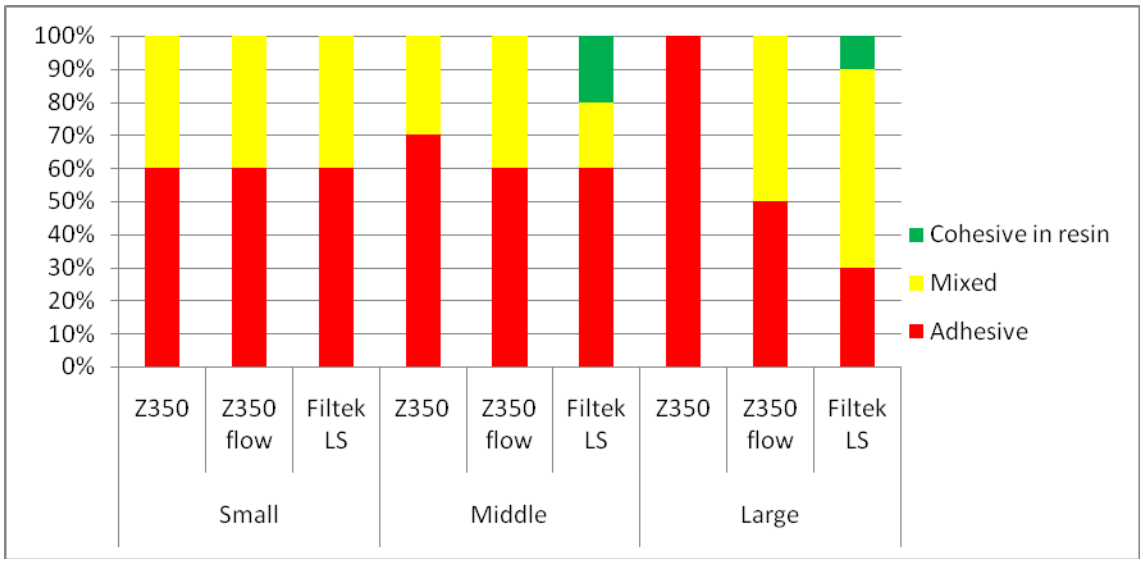

Figure 2. Failure mode (%) after shear bond strength test for different resin composites.

DISCUSSION

The first hypothesis was accepted because the low-shrinkage resin composite Filtek LS increased the bond strength and the marginal adaptation in comparison with methacrylate-based composites. The second hypothesis was rejected, because the volume of composite had no significant influence on the bond strength and the marginal adaptation of the resin composites. The third hypothesis was rejected, because the Filtek Z350 presented significantly higher Knoop hardness than the other composites. The volume of composite influenced the Knoop hardness results, with the small and medium volumes showing the highest results for Filtek Z350, medium volume showing the highest results for Filtek LS, and the large volume showing the highest values for Filtek Z350 flowable.

When monomers in proximity react to establish a covalent bond, the distance among them is reduced from 4°A to 1.5°A, and a reduction in free volume occurs, leading to volumetric shrinkage. The magnitude of volumetric shrinkage is determined by their filler volume fraction, and the composition of the resin matrix[22] and has been shown to be proportional to its degree of conversion[23].

The marginal adaptation results showed that there was no significant difference among small, medium, and large volume groups. When composite shrinkage is restricted by adhesion to the cavity walls, two variables must be considered. First, the level of confinement imposed on the material, which is estimated as the percentage of composite surface that is bonded to the substrate in relation to the total surface area, and secondly, the compliance of the bonding substrate[22]. Substrate compliance is characterized by the stiffness and mobility of its walls. The effect of confinement and compliance of the bonding substrate on stress values and on the integrity of the bonded interface has been the object of intense debate in the literature. The C-factor of the preparations was standardized (2.4) and the bonding substrate was the dentin root. There was a great difference in the volume of composite (7.54, 4.81, and 1.71 mm^3 for large, medium and small

cavities, respectively). However, even for the large cavities, it is possible that the volume of composite was very small for great differences to be observed in marginal adaptation and bond strength of the resin composites tested. A higher volume of composites was not used due to limitation of the restorative technique and the dentin substrate selected for this study. If a higher volume of composite had been used, the composite should have been inserted in incremental layers, due to the limited depth of polymerization of the resin composites at depths exceeding 2.0 mm[24]. Moreover, if the diameter of the cavities had been increased, the side walls of the preparations would have been too thin and could have fractured during the push-out bond strength test.

Filtek LS showed significantly less gap formation than the other composites. Ernst et al.[25] related that there was no significant difference in marginal adaptation between silorane-based all-in-one self-etching adhesive and self-etching adhesives at the cementum margins. However, in this study an improved two-step self-etching adhesive was used instead of the all-in-one mixed product that was used in the above-mentioned investigation. Several reports indicate that BisGMA in Single Bond 2 does not infiltrate into acid-etched dentine as well as HEMA, creating a HEMA-rich, BisGMA-poor lower half of such hybrid layers[26]. Some believe that HEMA mixing with water in the bottom half of the hybrid layer, may produce hydrogels and that silver uptake in these sites reflects the presence of such water. Filtek LS (3M ESPE) is a two-step adhesive system. The first step involves placing a self-etching primer on the smear layer covered dentine. It also demineralizes the intertubular dentin to a depth of 1–1.5 µm. After evaporating the solvent, the hydrophilic primer is light polymerized. The primer converts a wet hydrophilic, collagenous surface, into a dry hydrophobic surface that can couple with the silorane adhesive. After that, it is covered with a thick layer of a very hydrophobic adhesive. The hydrophobic nature of the Silorane adhesive is manifested as a lack of water diffusion through this layer. The use of a very hydrophobic sealer is a logical expansion of the use of the non-solvented BisGMA-rich adhesive that is used in Optibond FL and Scotchbond Multi-Purpose

adhesives[27,28]. The thicker layer of the hydrophobic adhesive may act as a stress relief material, leading to stronger bond strength and interface preservation[29].

The results of push-out bond strength test showed that the bond strengths of medium volumes were significantly higher than those of large volumes for Filtek Z350. He et al.[30] compared the effect of volume of composite on microtensile bond strength of resin composite to bovine dentin, reporting that bond strength to the cavity floor was greatly affected by the volume of polymerizing resin composite. However, they prepared cavities 3 and 5 mm deep, and the composite could be not efficiently polymerized in large volumes, leading to reduced bond strength for those groups. Moreover, the bond strength was checked at the cavity floor, which has been reported to be more susceptible to failure. In our study, the bond strength was measured at the side walls of the preparations, because the cavity floor was removed during the cavity preparation.

The push-out bond strength test results showed significantly higher bond strength for Filtek LS in comparison with other resin composites. The different organic matrix formulations significantly affected the polymerization shrinkage and rheological properties of the resin composites[31]. The extent of polymerization shrinkage depends on the relative mobility, molecular weight, and functionality of the monomers. Although siloranes exhibit low polymerization shrinkage, they also exhibited an atypical time-cuspal displacement curve, with a 30 s period of no dimensional change[32]. The Filtek LS used a three component initiating system comprising camphoroquinone, an iodonium salt and an electron donor. This complex initiation system may lead to a slower polymerization, allowing time for the material flow and stress relaxation, resulting in a lower final degree of polymerization stress at the bonding interface. This tendency was confirmed by analysis of the failure mode of the specimens. For Filtek LS inserted in large volumes, a reduction in the frequency of adhesive failure was observed, which could be associated with partial preservation of the bond interface. Recent studies with siloranes have demonstrated a polymerization reaction with a slow onset

because of the time needed for cation formation[33,34]. In the current studies, other materials were polymerized via free radical mechanisms, which are inherently faster[34]. Moreover, the thicker layers of an adhesive providing a low modulus of elasticity, as observed for Filtek LS, are capable of reducing the polymerization stress acting at the interface of resin composites[35]. The hybrid layer has a stress absorbing property, creating an area of low elastic modulus between the restoration and dentin[36].

The Filtek Z350 flowable showed significantly higher bond strength than Filtek Z350 for small and large volumes. The viscosity of composites is greatly affected by the resin matrix formulation, the interlocking between the filler particles, and the interfacial interaction between filler particles and the resin matrix[37,38]. In spite of the resin matrix of Filtek Z350 and Filtek Z350 flowable being very similar, these composites have different filler contents. Using various types and ratios of inorganic filler for an identical resin matrix, the composite viscosity exponentially increased as the filler volume is increased; and in the case of identical filler volumes, as the filler size decreased, viscosity increased[38]. The more flowable the composite, the less axial shrinkage is measured by the compensational radial flow. Therefore, the effect of consistency should always be considered when measuring and comparing the amount of polymerization shrinkage of composites. Condon et al.[39] showed a strong correlation between filler volume of commercial resin composites and shrinkage stress. Increasing the filler load resulted in higher stresses. The combination of this result with the increasing filler load (e.g. flowable resin composites/non-flowable resin composites) results in an increase of the tensile modulus, showing a positive correlation between tensile modulus and shrinkage stress. In short, increasing the filler load results in less shrinkage, more stress and a higher tensile modulus[40]. However, as found in this study, a flowable composite does not produce gap-free resin margins and the use of a flowable composite does not guarantee gap-free restorations, but improved bond strength of resin to dentin in bulk-filled restorations. Clinically, the magnitude of stress can be reduced by between 22 and 53% by applying a low stiffness material

between the composite restorative and the cavity walls to increase the compliance of the bonding substrate[41]. Another benefit of this procedure is that stress distribution is more uniform along the low elastic modulus layer[42].

Microhardness measurement is used as an indirect method to assess the extent of polymerization in the resin material inside the cavity[43]. The hardness of the composites is influenced by several factors, such as composition of the organic matrix[44], type and amount of filler particles[45] as well as the degree of conversion[46]. In this study, the Knoop hardness values of the composite Filtek Z350 were significantly higher than those of the other composites. The organic matrix of the composite Filtek Z350 is similar to the Filtek Z350 flowable. However, the higher Knoop hardness values for Filtek Z350 when compared with Filtek Z350 flowable may be explained by differences in filler content (60%v and 55%v for Z350 and Z350 flowable, respectively). On the other hand, Filtek LS is filled with a combination of fine quartz particles and radiopaque yttrium fluoride. The organic matrix is composed of the new generation of dental resin, silorane. The differences between Filtek Z350 and Filtek LS may be explained by differences in polymer structure and filler composition. Composites with harder filler particles exhibit higher surface hardness. However, the bonding of the filler particles to the polymeric matrix also affects their hardness values. The differences among the different volume of composites are unclear. One of the possible explanations is that the shear bond strength test performed before the hardness evaluation may have modified the surface of the composite, leading to differences observed among the samples. Moreover, differences among the three volumes of composites during the polymerization shrinkage may produce sites of stress concentration that can influence the hardness results.

Further studies are suggested in order to investigate the influence of volume of composite with different cavity geometry on stress generation at the bonding interface. The silorane-based resin composites appear to be a promising restorative dental material. However, further investigations must be conducted with

regard to the polymer network, longevity of the bond interface, and stress generation of the silorane materials.

CONCLUSION

The low-shrinkage resin composite Filtek LS showed the highest bond strength and the best marginal adaptation when compared with the methacrylate-based composites. The volume of composite influenced the bond strength and the marginal adaptation of the resin composites, with medium volumes showing the best results. Filtek Z350 presented the highest Knoop hardness values. The volume of composite influenced the Knoop hardness results, with the medium volume showing the highest values for Filtek Z350 and Filtek LS, and large volume showing the highest values for Filtek Z350 flowable. The analysis of the failure pattern showed predominance of adhesive failure.

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CAPÍTULO 2

Influence of volume of composite and photoactivation method on push-out bond strength and Knoop hardness of a resin composite

ABSTRACT

The aim of this study was to evaluate the influence of modulated photoactivation methods and volume of composite on Knoop hardness and push-out bond strength of the composite Filtek Z350 to root dentin. Three volume of composites (small, middle and large) were prepared in 90 premolars, restored with Filtek Z350 and photoactivated with continuous light, pulse delay or soft-start. The specimens were submitted to push-out bond strength and Knoop hardness tests. The data were submitted to ANOVA two-way and post hoc Tukey's test at 95% level of significance. The modulated photoactivation methods increased the bond strength to dentin and influenced the Knoop hardness results. The volume of composite affected the bond strength and the Knoop hardness values, with medium volumes showing the best results.

Key-words: photoactivation, resin composite, bond strength, marginal adaptation, Knoop hardness.

INTRODUCTION

Notwithstanding the advancement in resin composite formulation, the major problem of these restorative materials continues to be polymerization shrinkage. The rapid conversion rate in light-cured composites quickly induces an increase in composite stiffness, causing high shrinkage stresses at the bonded interface[1]. If shrinkage forces exceed the bond strength at the interface, the resulting interfacial

gap can lead to staining, marginal leakage[2], post-operative sensitivity[3] and recurrent caries[4]. If the interface is preserved, the shrinkage forces can be transferred to neighboring dental structures causing cuspal deflection[5, 6] or fractures in the enamel[7].

Alternative photoactivation methods are recommended to modify the polymerization rate and therefore minimize the harmful effects of stress developing at the adhesive interface. The soft-start method uses a low initial irradiation, followed by a second high irradiance to maximize the mechanical and biological properties of composites. The pulse delay photoactivation method is similar to soft-start. The major difference is the longer lag period in pulse-delay technique that permits more time for stress relief and thus decreases the additional stress located at the bond interface during the continuing polymerization[8]. The dark period for the pulse-delay method has been shown to be important to stress relief, permitting a molecular rearrangement and consequently stress relief. Moreover, in this dark period, monomer conversion occurs as well when samples are exposed to low irradiance. This is due to the free radicals that persist in the network after irradiation has ceased. The lower initial conversion produces greater mobility and allows more dark-polymerization and stress relief[9]. Cunha et al.[10] found that the maximum stress rate reached by soft-start (0.21 MPa/s) and pulse-delay (0.15 MPa/s) methods was 34 and 53% respectively, lower than that presented by continuous light (0.32 MPa/s). For soft-start and pulse-delay, the initial low irradiance led to a decreased initial polymerization, reflected as a reduction in the stress rate, thereby modifying the generation and distribution of stress. The reduction in the stress rate observed for soft-start and pulse-delay could be related to improved bond preservation.

It is well known that the stress resulting from polymerization shrinkage is influenced by restorative techniques, modulus of resin elasticity, polymerization rate[11, 12], photoactivation method and cavity configuration or "*C*-factor" which is defined as the quotient between bonded and unbonded resin composite surface area[13]. Yoshikawa et al.[14] found decreased bond strength under high *C*-factors.

In most of the studies, the *C*-factor has been manipulated with a fixed volume of composite to evaluate the bond strength to dentin[15]. However, in the literature there are few data regarding the dentin bond strength to cavities of different sizes with fixed *C*-factor. Composite shrinkage during polymerization is well known. Therefore, it would be interesting to evaluate the effect of the increase in the volume of composite on bond strength, when a similar C-factor is maintained, in order to understand the factors acting on stress shrinkage of the composites. Pfeifer et al.[16] found a strong correlation between the cavity size and microleakage in cavities with similar C-factor. However, there are few studies investigating the question of the effect of volume of composite and photoactivation methods on the resin–dentin bond strength.

The aim of this study was to evaluate the effect of the photoactivation methods and volume of composite on the push-out bond strength and Knoop hardness of composite restorations. The tested hypothesis is that the modulated photoactivation methods increase the bond strength and maintain the Knoop hardness in composite restorations. The second hypothesis is that the small volumes present higher bond strength than large volumes with similar Knoop hardness.

MATERIALS AND METHODS

Selection and teeth preparation

Ninety premolars were stored in 0.1% aqueous solution of thymol at 4°C for no longer than 1 month before being used. The crowns were cut off at the cement-enamel junction using a double-faced diamond disk under water cooling (KG Sorensen). Slices 3 mm thick were obtained from the root and ground with 400-grit SiC paper to thicknesses of 1.5 mm, 2.1 mm, and 2.5 mm. Truncated cone preparations of 3 different sizes (large – 2.3 mm top diameter x 1.6 mm bottom diameter x 2.5 mm deep; medium - 1.9 mm top diameter x 1.4 mm bottom diameter x 2.1 mm deep; small – 1.4 mm top diameter x 1.0 mm bottom diameter x

1.5 mm deep) were prepared in the root canal of each root slice using a truncated cone diamond tipped bur with the following dimensions (top diameter of 2.4 mm x bottom diameter of 0.8 mm, and 5.0 mm in height) mounted in a high-speed handpiece (Kavo), under constant air-water cooling in a standard cavity preparation appliance. The diamond burs were replaced after every fifth preparation. The C-factor of the preparation was calculated to be 2.4.

Restorative procedures

Preparations were etched using 35% phosphoric acid (Scotchbond Etchant, batch No. 7KK, 3M ESPE) for 15 seconds on dentin and rinsed for 15 seconds. Adper Single Bond 2 adhesive system (batch No. 8PR, 3M ESPE) was applied according to the manufacturer's instructions and photoactivated for 10 seconds at 800 mW/cm^2 (Ultralume 5, Ultradent). The resin composite (Filtek Z350, shade A3, batch No. 6GX, 3M/ESPE) was placed in bulk. A Mylar strip was placed over the cavity and used to force the composite to adapt to the preparation walls and to extrude the excess material. The specimens were randomly assigned into 9 groups (n=10) according to the photoactivation method (Table 1) and volume of composite. The irradiance was frequently checked by a radiometer (Demetron Research). To reduce the irradiance, the tip of the curing units was moved away from composite surface. In order to standardize the photoactivation distance, acrylic resin spacers (1.6 mm) were interposed between the surface of the composite and the tip of the light curing unit. After the photoactivation procedures were concluded, the specimens were stored in distilled water at 37˚C for 24 hours and wet-polished with 1200-grit SiC paper for push-out and Knoop hardness evaluation.

Table 1. Photoactivation methods evaluated with their outputs and the respective radiant exposure.

Photoactivation method	Irradiance and time exposure	Energy density
Continuous light	800 mW/cm^2 for 20s	16 J/cm^2
Soft-start	3 s at 150 mW/cm^2 + 20 s at 800 mW/cm^2	16.45 J/cm^2
Pulse-delay	3 s at 150 mW/cm^2 + 1 min + 20 s at 800 mW/cm^2	16.45 J/cm^2

The reduction of the power density in these groups was obtained using a standard separator.

Bond strength test

The bond strength test was conducted using a push-out test. The sample was placed on top of a metal device with an aperture that allowed the smaller diameter of the restoration to be in contact with an aspheric device, connected to the load cell of a universal testing machine (Instron, model 4411). This aspheric device applied a compressive force on the smaller diameter surface of the restoration until the tooth-composite bond ruptured. The push-out test was performed at a crosshead speed of 0.5 mm/min. Maximum load was divided by bonded area, and the bond strength results submitted to two-way ANOVA and Tukey post hoc test at a predetermined significance level of .05.

Knoop hardness test

After the push-out bond strength test, the composites were embedded in acrylic resin and ground and polished using 600, 1200, and 2000 SiC papers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE 53600-000, Brazil) on an automated polisher under water cooling. The specimens

were dried and submitted to the Knoop hardness measurements in a microhardness tester (HMV-2000, Shimadzu, Tokyo, Japan) with 50 g load and time of 10 s. Three readings were performed at the top surface, and a mean value was calculated. The data were submitted to two-way ANOVA and Tukey post hoc tests at a predetermined level of significance of .05.

RESULTS

With regard to push-out bond strength, ANOVA detected a statistically significant difference for photoactivation method (p=.00013) and for the interaction between composite volume and photoactivation methods (p=.0083). The results, according to the Tukey test, are listed in Table 2. Continuous light caused the lowest bond strength results. Modulated photoactivation methods, especially pulse-delay, showed a significant increase in bond strength when compared with the continuous photoactivation method.

Only for the continuous photoactivation method did the composite volume have significant influence on the push-out bond strength, in which medium volume presented significantly higher bond strength than small and large volumes.

Table 2. Means (standard deviation) of push-out bond strength of resin composite inserted in cavities with different volumes photoactivated with different methods.

Photoactivation method	Cavity size					
	Small		Middle		Large	
Continuous	4.13 (2.25)	**b, B**	5.70 (1.40)	**a, A**	3.54 (1.63)	**b, B**
Soft-start	5.47 (1.76)	**ab, A**	6.33 (1.31)	**a, A**	6.39 (1.04)	**a, A**
Pulse-delay	7.14 (2.00)	**a, A**	5.62 (2.37)	**a, A**	6.10 (1.12)	**a, A**

Means followed by different small letter in the column and capital letter in the row represent statistical difference ($p<0.05$).

For Knoop hardness, ANOVA detected a statistically significant difference for photoactivation method (p=0.00001), composite volume (p=0.00001), and for the interaction between composite volume and photoactivation methods (p=0.00002). The results of Knoop hardness according to the Tukey test are listed in Table 3. Medium composite volumes presented the highest Knoop hardness values, statistically higher than large volume with continuous and pulse-delay photoactivation methods, and statistically higher than small volume with soft-start and pulse-delay photoactivation methods. For medium and large composite volumes, the continuous method presented significantly lower Knoop hardness than pulse-delay. The soft-start method presented significantly lower Knoop hardness than the continuous method in small volume and lower than pulse-delay in medium volume.

Table 3. Means (standard deviation) of Knoop hardness of resin composite inserted in cavities with different volumes photoactivated with different methods.

Photoactivation method	Cavity size		
	Small	Middle	Large
Continuous	61.97 (1.54) **a, A**	62.12 (1.67) **b, A**	58.80 (1.80) **b, B**
Soft-start	60.34 (1.45) **b, B**	62.72 (1.48) **b, A**	61.72 (1.22) **a, AB**
Pulse-delay	61.80 (1.06) **ab, B**	65.88 (1.30) **a, A**	61.67 (1.25) **a, B**

Means followed by different small letter in the column and capital letter in the row represent statistical difference (p<0.05).

Failure mode classification is shown in Figure 1. Adhesive failure was the most frequent failure mode. Large volumes showed 100% of adhesive failure when photoactivated by the continuous method. The pulse-delay method in large volumes showed predominance of mixed failure. No cohesive failure in resin was found in this study.

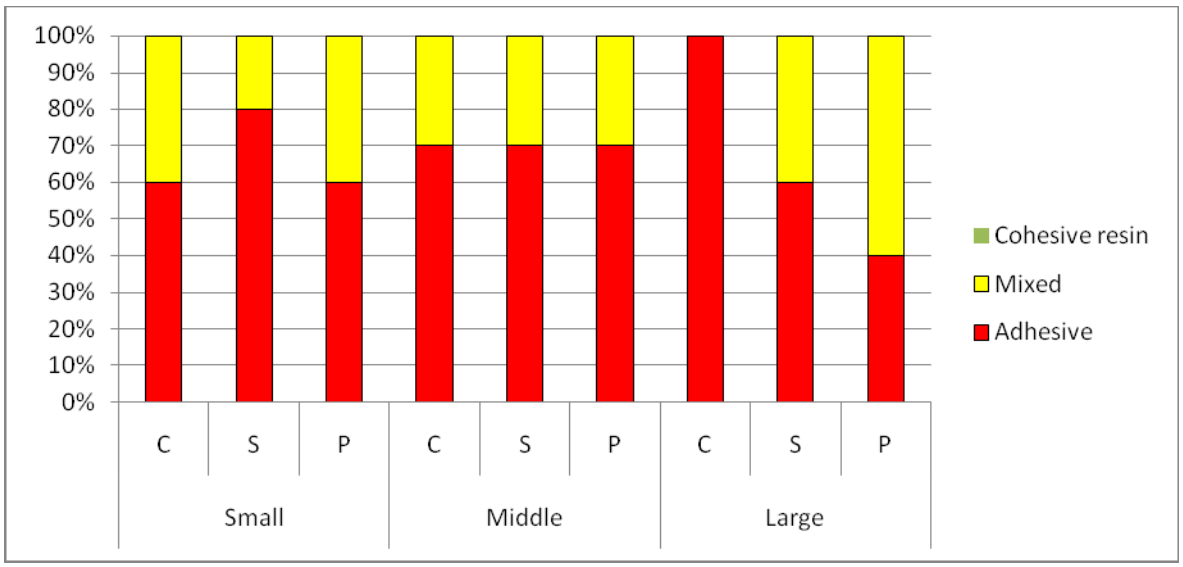

Figure 1. Failure mode (%) after shear bond strength test for different photoactivation methods (C – continuous; S – Soft-start; P – Pulse-delay).

DISCUSSION

In this study, because of the difficulty of finding 2.5 mm thick dentin substrate for preparing a large sized cavity, the roots were used for making standard cavities. The test design for this study was a push-out model. Previous studies[17] making use of a conical version of the push-out design demonstrated the different bonding properties of direct restorations with highly reproducible measurements. Moreover, the cavity-like configuration of the bonding area allows the composite bond strength, marginal adaptation, and Knoop hardness to be determined in the same sample[17].

When composite polymerization shrinkage is restricted to the cavity walls by adhesion, stresses build up at the bonded interface. Most authors agree that in situations where the resin composite is highly confined (high C-factor), high stresses are expected[18]. If the shrinkage stress overcomes the adhesive strength, a gap may be formed and will decrease the bond strength of the resin composite to dental substrate. Modulated photoactivation methods showed a significant increase

in bond strength when compared with continuous light. This result is in agreement with previous studies[10, 19] that found the highest mean values for modulated photoactivation methods to be significantly higher than those of continuous methods. The polymerization shrinkage stress caused bond disruption, decreasing the bond strength values and increasing the internal gap formation, especially in the continuous method, in which the shrinkage stress is higher[20]. It was shown that stress development seems to be directly proportional to the increase in irradiance. The reduction in the stress rate observed for soft-start and pulse-delay could be related to improved bond preservation[21]. The Soft-start technique also improved the internal adaptation of composite restorations[21]. Such results could be related to the increased ability of the composite to flow. This phenomenon is caused by the slower formation of polymer network and crosslinking, which supply favorable conditions for the adaptation of molecules within the polymeric chain that has been developed[22]. The introduction of delay in the early portion of the light-polymerization routine may prolong the low modulus phase, allowing the relief of stress development by polymer flow and deformation[23], while maximizing both the degree of conversion and shrinkage that occurred before the composite became predominantly rigid.

The initial low irradiance only retards the polymerization rate in the early periods but brings about the same final shrinkage as does a higher light intensity[24]. Therefore, differences in shrinkage stress cannot be accounted for by differences in the extent of polymerization or volumetric shrinkage. This lends support to the hypothesis that a slower polymerization reaction accompanied by a slow development of elastic modulus are responsible for reduced stress with step-cure polymerization[25]. The step-cure methods probably produced similar overall conversion to that of the continuous exposure polymerization method, whereas, the energy density was similar for all irradiation conditions[26].

The volume of composite did not have any influence on the push-out bond strength. Only for continuous photoactivation method did the volume of composite have significant influence on push-out bond strength, in which medium volume

presented significantly higher bond strength than small and large volumes. As the composite shrinkage is dependent on the amount of organic matrix, the highest bond strength was expected for small volumes, due to lower composite volume. However, the highest bond strength was found for medium volumes.

The large volume group exhibited significantly lower bond strength than did the medium volume group. This result could indicate that the effect of *C*-factor on bonding depends on the volume of composite. This can be explained by the high polymerization shrinkage stresses created during the photoactivation of a higher volume of composite resin in large volumes under a high *C*-factor. A direct relationship between stress and confinement seems to hold true in cases in which stress values are measured in a rigid testing apparatus[18]. The confinement conditions imposed on the material affect the composite flow ability in the earlier stages of polymerization. It is easier for the composite to yield to shrinkage forces by deforming its free surface, referred to as "macroscopic" flow, in comparison with "microscopic" (internal) flow (molecular rearrangement)[27].

Irrespective of the light modulation method, it has been recommended that similar energy densities be used[28]. The degree of conversion depends more on the energy density that is supplied to the composite than on the photoactivation method[28]. Hardness is a mechanical property indirectly related to the degree of conversion of composites. Higher hardness means can be obtained by increase in the degree of conversion, depending on the light polymerization method[29]. Recommendations on adequate radiant exposure values are based on different properties and materials, and therefore, vary greatly. Some studies have shown that uniform degree of conversion and microhardness were achieved through a 2mm thickness of composite with 21–24 J/cm^2[30, 31]. Other authors recommend lower radiant exposure values (12–15 J/cm^2) based on mechanical properties, such as microhardness[32], compressive and diametral tensile strengths[33]. The energy density used in this study was 16 J/cm^2 for continuous light and 16.45 J/cm^2 for modulated photoactivation methods.

The highest Knoop hardness results were shown by pulse-delay in the medium volume (65.88) and the lowest results for continuous light in large the volume (58.80). This is explained by the fact that polymerization of dimethacrylate monomers is limited by diffusion. The diffusion limitations occur in two stages with different effects. First, at very low conversion (in relatively high viscosity dental resins and composites), termination reactions involving the free radicals become mobility limited, which results in autoacceleration. At moderate conversion, the propagation reactions reliant on monomer diffusion also become limited leading to autodeceleration. As the reaction progresses further, the onset of vitrification occurs and the rate drops rapidly[34]. This non-linear relationship between radiant exposure and degree of conversion is clinically relevant because, as degree of conversion levels off, physical properties may also not improve significantly. In fact, non-linear relationships between radiant exposure and elastic modulus and microhardness have been demonstrated[35, 36]. The modulated photoactivation methods may prolong the stage of polymeric chain diffusion, leading to stress relaxation in material.

However, the modulated photoactivation methods may be related to higher softening than the standard mode. Although the radiant exposure was kept similar for photoactivation methods, a polymer with different crosslink density for the distinct photoactivation methods can exist. An initially slow cure may be associated with relatively few centers of polymer growth that favor the formation of a relatively linear polymer structure[37, 38, 39] and consequently, with lower Knoop hardness.

The highest Knoop hardness for medium volume of composite may be explained by differences on reflection light among the cavity sizes. When the light pass through composite and reaches the dentin it can be scattering, absorbed or reflected. The Knoop hardness measurements were performed at the center and margins of the resin composite restorations. The reflected light can collaborate with the increase of the Knoop hardness by increasing the degree of conversion on adjacent areas of the dentin. Thus, the increase on Knoop hardness of the small

and medium volume of composites may be occurred by increase on polymerization at the margin of restorations.

CONCLUSION

The modulated photoactivation methods increased the bond strength and maintained the Knoop hardness at a level similar to or even higher than that obtained by the continuous method. The medium volume showed higher bond strength than the large volume only for the continuous method. The Knoop hardness test showed that the volume size had an influence on Knoop hardness, with the medium volume showing the highest results. The analysis of the failure pattern showed predominance of adhesive failure.

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CONSIDERAÇÕES GERAIS

A contração de polimerização é um fator inerente à reação de cura dos compósitos. Durante a reação de polimerização, tensões são geradas devido à contração do compósito, podendo levar ao aparecimento de fendas e reduzir a resistência de união. Apesar do cirurgião-dentista não poder controlar a contração dos compósitos, alguns métodos foram propostos com o objetivo de reduzir a tensão gerada durante a reação de polimerização. Portanto, é importante que o cirurgião-dentista tenha conhecimento destes métodos para que as restaurações em compósito tenham sucesso.

Assim, este estudo procurou verificar se o volume de compósito, inserido em cavidades com diferentes dimensões, mas mantendo o mesmo fator C (2,4) seria um importante fator na contração dos compósitos. Para verificar esta influência, foi utilizada a dentina do conduto radicular. Inicialmente, o objetivo era utilizar a dentina coronária bovina ou humana. Entretanto, devido à dificuldade na obtenção de dentina com espessuras de 2,5 mm, foi selecionada a dentina do conduto radicular, que satisfazia os requisitos relacionados às dimensões do preparo cavitário e ao tipo de teste mecânico utilizado (*push-out*).

Durante a reação de polimerização as tensões geradas pela contração de polimerização são dissipadas no material, através do deslizamento das cadeias poliméricas; na superfície não aderida através de alterações de forma do material; ou são direcionadas para a interface de união. As tensões transmitidas para a interface dente/restauração podem causar ruptura do selamento marginal, possibilitando infiltração de fluidos, microrganismos e cáries recorrentes. Os resultados deste estudo mostraram que o volume de compósito influenciou os valores de adaptação marginal e resistência de união. Quanto maior for o volume de compósito, maior será a quantidade de duplas ligações convertidas e, portanto, maior a tensão no material. Em cavidades pequenas, a quantidade de superfície não aderida foi muito pequena, dificultando a liberação das tensões. Entretanto,

nas cavidades médias o compósito teve possibilidade de escoar e liberar as tensões na superfície não-aderida, proporcionando a este grupo os melhores resultados.

Os resultados deste estudo mostraram que a resistência de união pode ser melhorada pela modulação da luz durante a fotoativação, pela utilização de compósitos compostos por monômeros de baixa contração e por compósitos de alto escoamento (com baixo módulo de elasticidade). Com o surgimento dos compósitos fotoativados, o método de fotoativação passou a ser uma das principais etapas para que o procedimento restaurador obtivesse sucesso. Após a fotoativação, o compósito deve apresentar alto grau de conversão e propriedades físicas e biológicas adequadas. Como foi mostrado neste estudo, o método de fotoativação tem influência direta nas propriedades finais dos compósitos. Os métodos de modulação da luz durante a fotoativação se apresentaram eficientes para aumentar a resistência de união do compósito à dentina radicular. Esses métodos consistem na fotoativação inicial com baixa irradiância, seguido pelo complemento da fotoativação com a irradiância máxima da fonte de luz. Neste método de fotoativação, devido à baixa irradiância durante os períodos iniciais e ao tempo de espera, as tensões de contração poderiam ser liberadas internamente no material, havendo menor concentração na interface de união. Portanto, menos fendas são geradas e o material apresenta maior resistência de união. A diferença entre o pulso interrompido e o método soft-start é o tempo de espera após a fotoativação inicial.

Outro fator que influenciou significativamente a resistência de união foi o compósito resinoso utilizado. O compósito que utiliza monômeros com baixa contração de polimerização apresentou os maiores valores de resistência de união e a menor formação de fenda. Esse compósito utiliza monômeros a base de silorano, que durante a reação de polimerização apresentam a abertura de seus anéis, proporcionando redução na contração de polimerização. Além disso, o sistema fotoiniciador destes compósitos é diferente dos compósitos a base de metacrilato. O compósito Filtek LS apresenta um sistema iniciador de 3

componentes, através de reação via catiônica. Este sistema faz com que o início da reação seja mais lento, havendo menor velocidade inicial de reação, gerando menor tensão de contração, além de permitir maior tempo de trabalho para o cirurgião-dentista. Além da menor contração de polimerização do Filtek LS, as diferenças entre os sistemas adesivos utilizados também podem ter influenciado nos valores de resistência de união. Para os compósitos Filtek Z350 e Filtek Z350 flow foi utilizado o sistema adesivo Single Bond 2, que utiliza a técnica úmida com condicionamento com ácido fosfórico. Já o compósito Filtek LS utiliza um sistema adesivo autocondicionante de dois passos. Primeiro aplica-se o primer, que é fotoativado, e em seguida o adesivo com características hidrófobas. Portanto, as diferentes características entre os dois sistemas adesivos podem ter ocasionado diferentes interações com a dentina, levando às diferenças nos valores de resistência de união.

O compósito Filtek LS apresentou-se efetivo na redução da contração de polimerização. Entretanto, esse compósito é indicado para uso em restaurações posteriores. Assim, mais pesquisas são necessárias para compreender o fator responsável pelo aumento da resistência de união com o compósito Filtek LS, se é pelo processo de abertura dos anéis ou pela menor velocidade inicial da reação devido ao sistema iniciador, ou ainda, pelo seu sistema de união. Além disso, o grau de conversão destes materiais precisa ser investigado, pois influencia as propriedades mecânicas e biológicas dos compósitos.

CONCLUSÕES GERAIS

Com base nos resultados obtidos nos dois estudos, pôde-se concluir que:

1. O tamanho da cavidade influenciou os valores de resistência de união, dureza Knoop e adaptação marginal a dentina radicular;
2. O compósito resinoso empregado influenciou os valores de resistência de união, dureza Knoop e adaptação marginal das restaurações, sendo que os maiores valores de resistência de união e adaptação marginal foram apresentados pelo compósito Filtek LS e os maiores valores de dureza pelo compósito Filtek Z350.
3. A técnica de fotoativação influenciou os valores de resistência de união e dureza Knoop, com os métodos de modulação da intensidade de luz apresentando os melhores resultados.
4. Houve predominância de falhas adesivas, com exceção do compósito Filtek Z350 inserido em grande volume e fotoativado pelo método *pulse*-delay e para o compósito Filtek LS inserido em grande volume, em que houve predominância de falhas mistas.

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[*] De acordo com a norma da UNICAMP/FOP, baseadas na norma do International Committee of Medical Journal Editors – Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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APÊNDICE

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DECLARAÇÃO

As cópias de artigos de minha autoria ou de minha co-autoria, já publicados ou submetidos para publicação em revistas científicas ou anais de congressos sujeitos a arbitragem, que constam da minha Dissertação/Tese de Doutorado, intitulada "Influência do tamanho da cavidade, técnica de fotoativação e sistema restaurador sobre a adaptação marginal, dureza e resistência de união à dentina", não infringem os dispositivos da Lei nº 9.610/98, nem o direito autoral de qualquer editora.

Piracicaba, 08 de Março de 2009.

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RG Nº 29715856-9
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RG Nº
ORIENTADOR

ANEXO I

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

CERTIFICADO

O Comitê de Ética em Pesquisa da FOP-UNICAMP certifica que o projeto de pesquisa **"Influência do volume de compósito, fator C e técnica de fotoativação sobre a adaptação marginal, dureza Knoop e resistência de união de compósitos à dentina"**, protocolo nº 124/2008, dos pesquisadores Américo Bortolazzo Correr e Mário Alexandre Coelho Sinhoreti, satisfaz as exigências do Conselho Nacional de Saúde - Ministério da Saúde para as pesquisas em seres humanos e foi aprovado por este comitê em 15/10/2008.

The Ethics Committee in Research of the School of Dentistry of Piracicaba - State University of Campinas, certify that the project **"Effect of resin composite volume, C-factor and photoactivation technique on marginal adaptation, Knoop hardness and push-out bond strength to dentin"**, register number 124/2008, of and Mário Alexandre Coelho Sinhoreti, comply with the recommendations of the National Health Council - Ministry of Health of Brazil for research in human subjects and therefore was approved by this committee at .

Prof. Dr. Pablo Agustin Vargas
Secretário
CEP/FOP/UNICAMP

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

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

ANEXO II

Prof. Mario Sinhoreti

De: "Journal of Dentistry" <JoD@elsevier.com>
Para: <sinhoret@fop.unicamp.br>
Enviada em: quarta-feira, 18 de março de 2009 10:57
Assunto: Submission Confirmation for Journal of Dentistry

Dear Dr. Sinhoreti,

Your submission entitled "Influence of volume of composite and photoactivation method on push-out bond strength and Knoop hardness of a resin composite" has been received by the Journal of Dentistry.

You will be able to check on the progress of your paper by logging on to Elsevier Editorial System as an author. The URL is http://ees.elsevier.com/jjod/.

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Thank you for submitting your work to this journal.

Kind regards,

Marie Dymond
Journal Manager
Journal of Dentistry