



Interfacial Properties and Bottom/Top Hardness Ratio Produced by Bulk Fill Composites in Dentin Cavities

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The aim of this study was to evaluate the bond strength, bottom/top hardness ratio, marginal adaptation, and interfacial nanoleakage of regular viscosity bulk fill composites (RVBFC) and regular viscosity traditional composites (RVTC). Two RVBFC (Filtek Bulk Fill and Aura Bulk Fill) and two RVTC (Filtek Z250XT and Aura) were assessed. Forty conical cavities (4.8×2.8×4.0) were prepared in bovine dentin and restored with composites (n=10). After 24h in water, marginal adaptation was evaluated by staining with a caries detector. The top and bottom surfaces of the conical restorations were stained for five seconds and the gap percentage in the composite/dentin interface was determined using digital images on a measurement program (ImageTool). The Vickers microhardness was measured and the bottom/top microhardness ratio (B/T) was determined. Push-out bond strength test was performed in a universal testing machine (0.5mm/min) and failure modes were evaluated in a stereomicroscope (20×). Other specimens (n=3) were produced for interfacial nanoleakage evaluation. Data were analyzed using one and two-way ANOVA and Tukey's test ($\alpha=0.05$). The gap percentage was higher in the bottom compared to the top. The B/T ratio of the Aura Bulk Fill was statistically lower than other composites. Push-out bond strength were similar among composites. The RVBFC presented lower nanoleakage than the RVTC in the bottom of the conical restoration and there was no difference among the materials in the top surfaces. In conclusion, Filtek Bulk Fill performed better than Aura Bulk Fill regarding the analyzed properties.

Key Words: composite resins, mechanical stress, light-curing of dental resin.

Introduction

One of the challenges in restorative dentistry is finding the balance between simple clinical protocols for restorations with composite resins and clinical longevity of restorations. (1) The traditional filling technique, consisting of multiple 2-mm composite increments, demands long chair time and increases the possibility of internal voids, which can affect mechanical properties of the composite (1). Differently, bulk-fill composites allow the placement of up to 5-mm increments, thereby saving time (2).

Flowable and regular viscosity bulk fill composites are available in the market. Flowable bulk fill composites are used as cavity bases under a covering composite. On the other hand, regular viscosity bulk fill composites have increased viscosity and most do not require a covering composite; thus, they may be used to fill the entire tooth cavity (3).

Adhesion to dentin, polymerization depth, marginal adaptation, and integrity of the hybrid layer are physical properties that may affect the longevity of tooth restorations using regular viscosity bulk fill composites

(4). A reduced polymerization has been shown to lead to inadequate physical properties that can ultimately lead to clinical failure (5). Indeed, a weak bonding to the tooth structure increases the chance of microleakage, and thus favors adverse consequences such as post-restorative hypersensitivity, recurrent caries, marginal discoloration, and injury to the pulp (6).

Conversely, a good bond strength is synonymous with a good adaptation of the restoration to the hybrid layer. However, in high c-factor cavities, an exacerbated polymerization stress may occur, as curing shrinkage causes stress at the tooth-restoration interface. The detrimental effects of polymerization shrinkage stress include bond failure, cuspal flexure, interfacial gap, and subsequent microleakage (7).

A previous review showed that most studies do not follow the manufacturer's instructions concerning photoactivation, exceeding the recommended time and thus yielding unrealistic results about the materials (8). Concerning dentin substrate, previous investigations (9,10) used a flat dentin surface, which has a low c-factor, to evaluate bond strength of bulk fill composites, not

simulating posterior cavities, which have a high c-factor. A higher c-factor is associated with a lower bond strength, which can affect marginal integrity and lead to the formation of cracks on those restorations (11). Therefore, the push-out test can be used to measure bond strength directly on tooth cavities, in which the composite resin shrinks between internal walls causing interfacial stress during specimen fabrication (12). However, few studies evaluated mechanical properties such as bond strength, bottom/top hardness ratio, marginal adaptation, and interfacial nanoleakage of regular viscosity bulk fill composites in high C-factor dentin cavities.

This study aimed to analyze push-out bond strength, bottom/top hardness ratio, marginal adaptation, and interfacial nanoleakage of regular viscosity bulk fill composites in dentin cavities and compare the results obtained with traditional composite resins. The null hypothesis was that there is no significant difference among the materials for all properties tested.

Material and Methods

Study Design

This in vitro study assessed the following response variables: push-out bond strength (BS), bottom/top hardness ratio (B/T), marginal adaptation (MA), and interfacial nanoleakage (IN). For MA and IN, a 4×2 factorial design study evaluated two factors: material [Filtek Z250XT (3M ESPE, St. Paul, MN, USA), Filtek Bulk Fill (3M ESPE, St. Paul, MN, USA), Aura (SDI, Melbourne, Victoria, Australia), and Aura Bulk Fill (SDI)], and surface

(top and bottom of conical specimens). For BS and B/T, only the material variable was studied. Materials that presented different chemical compositions were selected and their compositions are listed in Table 1.

Specimen Fabrication

For B/T, MA and BS analyses, 40 specimens (n=10) were fabricated. Bovine incisors were evaluated in a dissecting microscope (Stereozoom, Bausch & Lomb, Rochester, NY, USA), being selected only the specimens free of cracks and structural defects. The samples were immersed in 0.01% thymol solution at 4 °C for one week. These specimens were prepared based in the push-out method for testing bond strength described by Sousa-Lima et al. (12), and schematically represented in Figure 1.

The roots were removed with diamond discs (South Bay Technology, San Clement, CA, USA) coupled to a precision cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA) under water refrigeration, cut at the cemento-enamel junction (CEJ). Straight, transverse cuts were made in the crowns, 4 mm from the ACJ, creating a 4-mm thick disc with a central void (pulp cavity). The top and bottom surfaces of the specimens were sanded with P400 and P600 sandpapers (Labopol-21, Struers, Copenhagen, Denmark) to obtain a flat dentin surface. To optimize the space of the pulp cavity, Maxicut burs #PM1251 (Edenta, Hauptstrasse 7, CH 9434 AU/SG, Switzerland) were coupled to a cavity preparation standardizing machine, so that the bur penetrated perpendicularly the center of the specimen (pulp cavity), originating standard conical cavities (4.8 mm top

Table 1. Materials utilized in this study

Material	Manufacturer	Chemical composition (wt%)*	Lot number
Filtek Z250 XT A2 shade	3M ESPE, St. Paul, MN, USA	Silane treated ceramic (75-85%), Bisphenol a polyethylene glycol diether dimethacrylate (5-10%), Diurethane dimethacrylate (5-10%), BISGMA (1-10%), TEGDMA <5 %, Water <2%	808770
Filtek Bulk Fill A2 shade	3M ESPE, St. Paul, MN, USA	Silane treated ceramic (60-70%), Aromatic urethane dimethacrylate (10-20%), YBF3 (1-10%), UDMA (1-10%), Silane treated silica (1-10%), DDDMA <5%, Silane treated zirconia <5%, Water <5%, Pentanedioic acid, 2,2-dimethyl-4-methylene- reaction products with glycidyl methacrylate <1%, EDMAB <0.5%, Benzotriazol <0.5%, Titanium dioxide <0,2%	N874606
Aura E2 shade	SDI, Melbourne, Victoria, Australia	Acrylic monomers as (6-46%), Diurethane dimethacrylate (6-46%), Triethylene glycol dimethacrylate (6-46%), 2,2-bis[4-(2-methacryloxy)ethoxy]phenyl]propane (6-46%)	140149
Aura Bulk Fill Universal shade	SDI, Melbourne, Victoria, Australia	Acrylic monomers as (6-46%), Diurethane dimethacrylate (6-46%), Triethylene glycol dimethacrylate (6-46%), 2,2-bis[4-(2-methacryloxy)ethoxy]phenyl]propane (6-46%)	150710
Single Bond Universal	3M ESPE, St. Paul, MN, USA	2-hydroxyethyl methacrylate (15-25%), BISGMA (15-25%), Decamethylene dimethacrylate (5-15%), Ethanol (10-15%), Silane treated silica (5-15%), Water (10-15%), 1,10-decanediol phosphate methacrylate (1-10%), Copolymer of acrylic and itaconic acid (1-5%), Camphorquinone <2%, N,N-dimethylbenzocaine <2%	582957

*According to the Material Safety Data Sheet. BISGMA: Bisphenol a diglycidyl ether dimethacrylate TEGDMA: Triethylene glycol dimethacrylate YBF3: Ytterbium fluoride UDMA: Diurethane dimethacrylate DDDMA: 1, 12-dodecane dimethacrylate EDMAB: Ethyl 4-dimethyl aminobenzoate

diameter, 2.8 mm bottom diameter, and 4 mm depth). The burs were replaced every 5 preparations. Thereby, a cavity with c-factor of 2.2 was obtained.

The cavities were restored with Single Bond Universal (3M ESPE, St. Paul, MN, USA) in the self-etch mode followed by each composite resin, according to the manufacturer's instructions. The specimens were placed on a glass slab for insertion of the adhesive system, which was photoactivated for 10 s using the Bluephase G2 LED - 1200 mW/cm² (Ivoclar Vivadent, Schaan, Liechtenstein). Then, the composites were inserted in the cavities and photoactivated according to the incremental/bulk insertion technique. A glass slab with 1 mm thickness was positioned on the top surface of the specimen to allow a standardized distance from the tip of the curing device to the composite/adhesive system. The restorations were finished and polished with aluminum oxide-coated abrasive discs Sof-Lex

Pop On (3M ESPE) and diamond paste Diamond R (FGM, Joinville, SC, Brazil) in both surfaces 24 h after polymerization.

Bottom to Top Hardness Ratio

Bottom to top hardness ratio was obtained (12) using a Vickers microhardness tester (HV-100 Digimes, São Paulo, SP, Brazil) with a load of 50 g for 5 s. Three indentations were made in each restoration surface and the average hardness value was calculated based on the measurements of the 3 values.

Marginal Adaptation

Following the method described by Souza-Junior et al (13), a solution of 1% Acid Red dye in propylene glycol (Caries Detector, Kuraray, Osaka, Japan) was applied at the margins of the top and bottom restoration surfaces of all specimens for 10 s. The specimens were washed with

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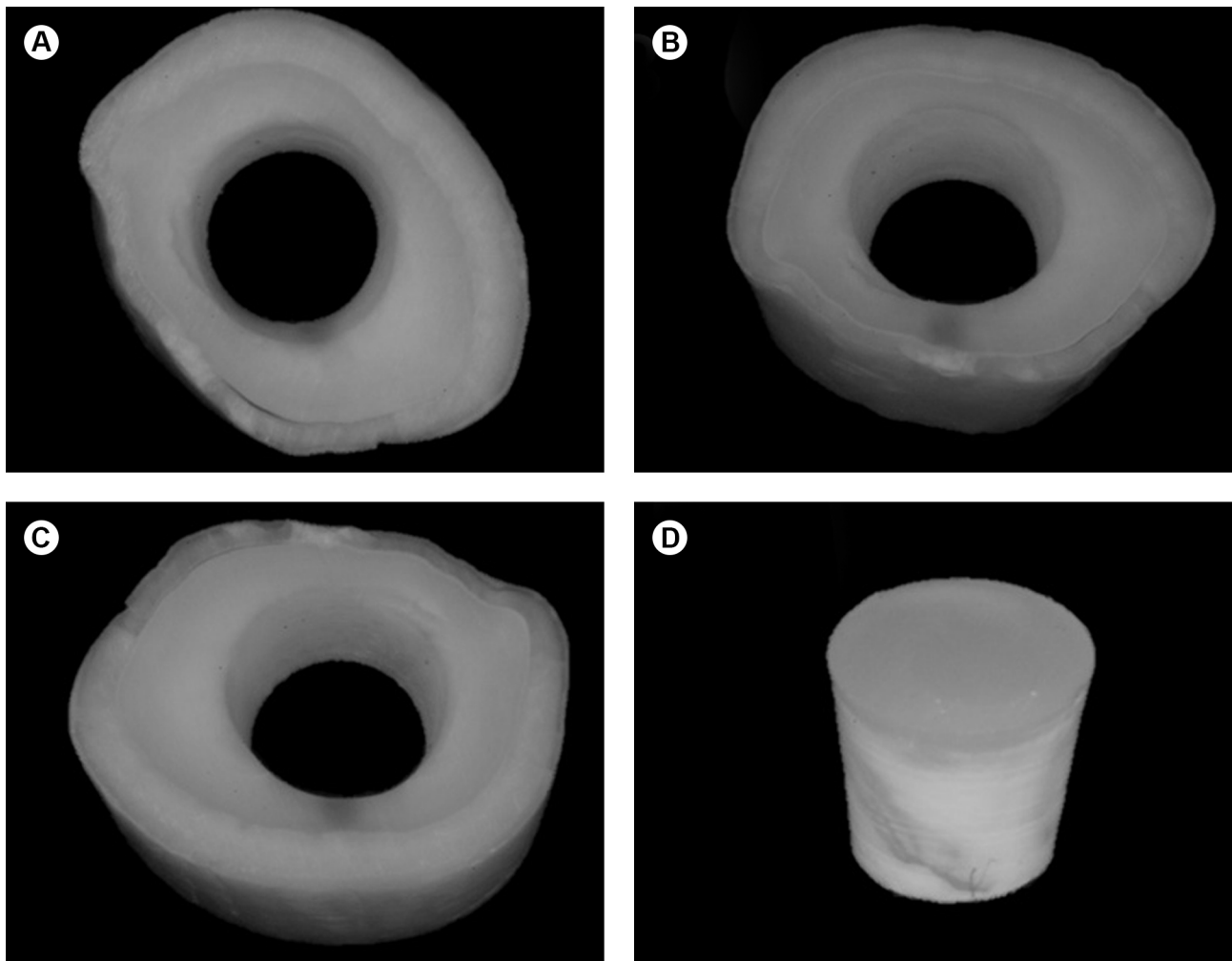


Figure 1. Adhesive failures found in the specimens: Top (A), Buccal (B), and Lingual (C) views of the cavity after debonding. The entire cone-shaped composite specimen was observed (D).

abundant distilled water for 20 s to remove the excess dye. Afterwards, digital images of each specimen were obtained and evaluated by a Leica MZ6 stereomicroscope with 16× magnification.

The length of the marginal gaps was measured in millimeters using the software Image Tool 2.0. The size of the gaps was calculated as a percentage of the total perimeter of the restoration. The specimens were also divided according to the presence or absence of these gaps.

Push-Out Bond Strength

To measure bond strength, a stainless-steel device was fitted to the base of a Universal Testing Machine (Zwicki-Line, Zwick/Roell, Ulm, Germany) with a gap at the center in which the specimens were placed with the smaller base facing upwards. A conic device was fitted on the machine and a compressive force with speed of 0.5 mm/min was applied against the restoration in the center of the smaller base. The failure force was recorded in Newtons (N) and transformed to MPa using the following equation:

$$\text{MPa} = \frac{N}{\pi(R+r)\sqrt{h^2 + (R-r)^2}}$$

where 'R' is the radius of the larger base, 'r' is the radius of smaller base, and 'h' is the thickness of the specimen.

After the test, failure modes were analyzed in a dissecting microscope (Stereozoom, Bausch & Lomb, Rochester, NY, USA), and classified as adhesive failure, cohesive in the composite/dentin interface, or mixed (adhesive and cohesive).

Interfacial Nanoleakage

To evaluate nanoleakage, 12 additional specimens were made (n=3) as described by Sano et al. (14). After being restored, the specimens were immersed in distilled water for 24 h in an oven at 37 °C. Then, they were immersed in silver nitrate solution (25 g of silver

nitrate crystals in 50 mL of distilled water and 50 mL of 28% ammonia hydroxide at pH 11.0). The specimens remained in the dark, stored in sealed black flasks, and wrapped in aluminum paper for 24 h. After, they were washed and immersed in photo reflective solution (Kodak, Rochester, NY, USA) for 8 h under fluorescent light. Once again, the specimens were washed with distilled water and then polished using sandpapers of P600-, 1200-, and 2000-grit size (Labopol-21, Struers, Copenhagen, Denmark) under refrigeration, and 0.3 and 1 μ polishing pastes with felt disc. The specimens were demineralized by immersing in 37% phosphoric acid (Super Etch, SDI, Melbourne, Victoria, Australia) for 5 s, washed with distilled water for 30 s, dried with absorbent paper, and then left for 24 h at room temperature. They were mounted on aluminum supports and examined by scanning electron microscopy (TM 3000, Hitachi, Chiyoda, Tokyo, Japan). Representative images of four quadrants were obtained for each specimen in the top and in the bottom surfaces with 40× magnification. The interfacial penetration of silver nitrate was analyzed descriptively, so that the image that represented the most prevalent pattern in the 4 quadrants was selected.

Statistical Analysis

One-way ANOVA and Tukey's post-hoc tests were used to analyze the bottom-top hardness ratio and the bond strength ($\alpha=0.05$). Marginal adaptation was evaluated using 2-way ANOVA and Tukey's post-hoc tests ($\alpha=0.05$). The software Assistant BETA 7.5 was used for statistical analysis. Interfacial nanoleakage was evaluated descriptively.

Results

Bottom to Top Hardness Ratio

The means of the bottom/top hardness ratio (B/T) according to the type of composite and the means of bond strength according to type of composite are presented in Tables 2 and 3, respectively. Significant differences were

Table 2. Means ± standard deviations of the bottom/top hardness ratio (B/T) according to the type of composite

Composite	B/T
Filtek Z250XT	0.80 ± 0.07 a
Filtek Bulk Fill	0.75 ± 0.08 a
Aura	0.75 ± 0.11 a
Aura Bulk Fill	0.53 ± 0.11 b

Different lowercase letters indicate significant differences between composites ($\alpha=0.05$).

Table 3. Means ± standard deviations of bond strength according to the type of composite

Composite	Bond strength (MPa)
Filtek Z250XT	11.12 ± 2.1 a
Filtek Bulk Fill	10.73 ± 1.9 a
Aura	11.40 ± 2.8 a
Aura Bulk Fill	12.19 ± 2.2 a

Different lowercase letters indicate significant differences between composites ($\alpha=0.05$).

found for bottom/top hardness ratio among materials ($p < 0.01$, power 0.95). Aura Bulk Fill showed significantly lower values than the other materials (Table 2).

Push-out Bond Strength and Failure Modes

No significant difference was found among composites in bond strength ($p = 0.07$, power 0.81, Table 3). Only adhesive failures between adhesive and dentin were found (Fig. 2).

Marginal Adaptation

Significant differences were found for marginal adaptation among composites ($p < 0.01$) and surfaces ($p < 0.01$) (power 0.88). Comparisons among groups are shown in Table 4. Regardless of the composite, the bottom surface showed significantly higher percentage of gaps than the top surface. In both surfaces, Aura Bulk Fill showed the highest percentage of gaps while Filtek Z250 XT showed the lowest percentage.

Interfacial Nanoleakage

Nanoleakage patterns are shown in Figure 2. All composite resins showed greater nanoleakage at the bottom surface than at the top surface. Bulk fill composites showed less nanoleakage at the bottom surface than traditional composites. On the other hand, all composite resins showed similar patterns of low nanoleakage at the top surface. Silver nitrate penetrated in the dentin/adhesive interface more frequently.

Discussion

The null hypothesis tested - that there is no significant difference among the materials for all properties - was rejected. While Aura Bulk Fill showed lower percentage of well-adapted margins and lower bottom/top hardness ratio than the other composites, bond strength was similar among them. This study

evaluated bond strength in dentin cavities, which have larger adhesive area than specimens subjected to microtensile and microshear bond strength tests and might present over/underestimation of bond strength values in comparison with micro tests such as microtensile bond strength. In the push-out test, stress generated by composite polymerization is transferred directly to the adhesive interface as the composite shrinks inside the cavity in bovine dentine, which is

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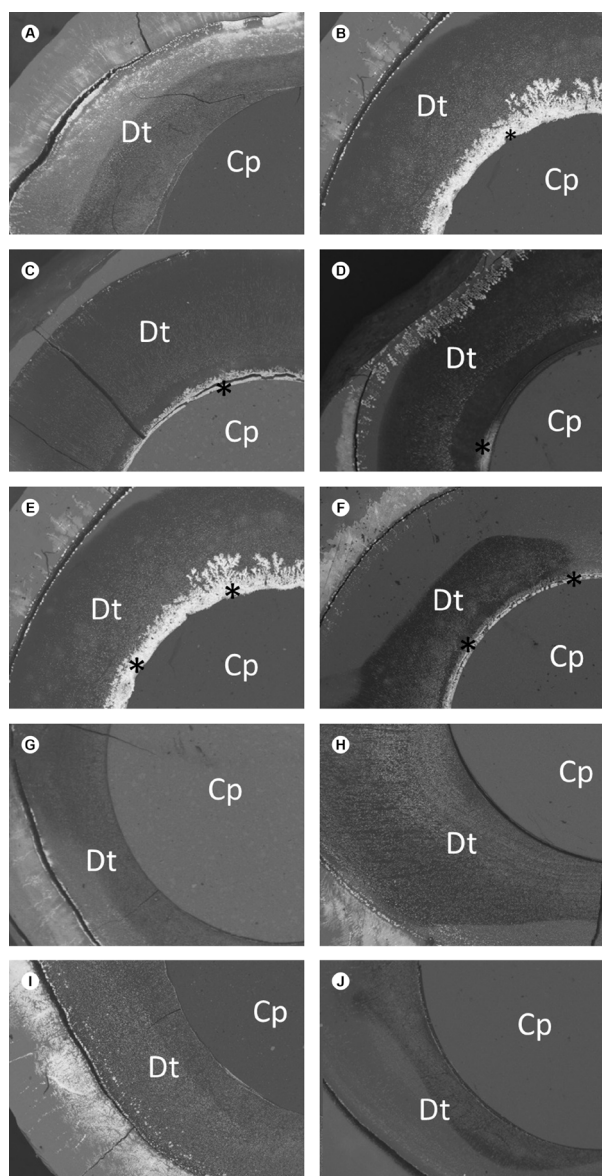


Figure 2. Increased penetration of silver nitrate (*) was found at the bottom surface (A) compared to the top surface (B) for all composites; Aura Bulk Fill (C) and Filtek Bulk Fill (D) had less nanoleakage expression (*) than their traditional correspondents Aura (E) and Filtek Z250 XT (F) at the bottom surface, whilst at the top surface Aura Bulk Fill (G) and Filtek Bulk Fill (H) showing lower nanoleakage than expression for all composites Aura (I) and Filtek Z250 XT (J). Cp: composite; Dt: Dentin.

Table 4. Mean \pm standard deviation of the gaps percentage according to types of composite and surfaces

Composite	Surface	
	Top	Bottom
Filtek Z250XT	16.15 \pm 1.7 cB	30.72 \pm 3.6 cA
Filtek Bulk Fill	19.96 \pm 1.9 bB	38.30 \pm 3.8 bA
Aura	19.80 \pm 1.2 bB	42.65 \pm 5.5 bA
Aura Bulk Fill	37.62 \pm 2.9 aB	55.74 \pm 6.7 aA

Different lowercase letters indicate significant differences between the resins for the same region of the specimens. Different uppercase letters indicate significant differences between specimen regions of the same resin.

a good substitute of human dentin in adhesion test and was previously used to measure bond strength of bulk fill composites (12). Although the bond strength of bulk fill composites can also be analyzed using microtensile tests after filling in posterior cavities (15), specimen preparation using diamond saws to obtain the beams can transfer external stress to the tooth/composite interface, leading to an underestimation of bond strength values. In contrast, the push-out method allows the measurement of bond strength without this external stress, so that differences found between materials are due only to their intrinsic characteristics such as chemical composition (organic matrix, photoinitiators, and filler particles), translucency, and shrinkage stress. Although slight differences between adhesive behaviors of composites might be found in some points, the bond strengths of the global interface area were similar. The fact that all specimens showed adhesive failures can strengthen the reliability of the test used, once adhesive failures commonly result from a proper distribution of forces at the specimen interface during the bond strength test (16).

However, although all composites tested presented similar adhesion behavior in dentin cavities, bottom/top hardness ratio, marginal adaptation, and nanoleakage were different among groups. The lower bottom/top hardness ratio showed by Aura Bulk Fill in comparison with the other composites may indicate lower polymerization depth and might be related to the composition of the resin matrix. It has been observed that Bis-EMA ((2,2-bis[4-(2-methacryloxy)ethoxy]phenyl)propane) is able to restrict the mobility of UDMA (diurethane dimethacrylate) monomers and decrease their reactivity and conversion value (17). Although Aura also contains Bis-EMA and UDMA, it was polymerized in two successive 2-mm increments, differently from the single 4-mm increment done with Aura Bulk Fill. Thus, it is likely that decreased radical mobility of UDMA monomers in the presence of Bis-EMA (18) associated with light attenuation in the 4-mm increment caused the lower bottom/top hardness ratio by Aura Bulk Fill composite. In addition, the universal shade of Aura Bulk Fill might be more opaque than the other composites, which have A2 and E2 shades. However, further optical analysis would be necessary to confirm this supposition. In fact, a poorly polymerized composite resin may be associated with the presence of residual monomers, which are cytotoxic and can affect the clinical success of the restoration (18).

Other important parameters that affect clinical

success of composite restorations are marginal adaptation and nanoleakage in the adhesive interface. The presence of gaps in the margins leads to the appearance of stains, recurrent caries, oversensitivity, and pulp pathologies (19). Nanoleakage is a leakage that occurs in nanometer-scale spaces, allowing the penetration of bacterial products or oral fluids across the interface, which compromises the stability of the resin-dentin bond through the hydrolytic breakdown of the adhesive resin or presence of collagen in the hybrid layer (20).

The fact that the bottom surface showed more marginal gaps and nanoleakage than the top surface may be attributed to the decreased degree of conversion of the adhesive system at the bottom of the cavity because of light attenuation, since the tip of the curing device was 4 mm apart from the lower surface. Light attenuation decreases the energy density that reaches the adhesive system at the bottom of the cavity, which can decrease the degree of conversion of the adhesive system (20). A poorly polymerized self-etching adhesive system, as the universal system used in this study, may affect bonding performance, since residual acidic monomers can retain their etching potential, thus jeopardizing adhesion (21) and increasing nanoleakage (22).

Aura Bulk Fill obtained the lowest percentage of adapted margins, while Filtek Z250 XT obtained the highest one, especially on the bottom surface. The decreased polymerization depth of Aura Bulk Fill associated with the probably low degree of conversion of the adhesive system at the bottom surface may have produced a weaker composite/adhesive interface on the bottom surface, potentiated by non-converted monomers of the composite. This might have contributed with increased formation of marginal voids, favoring the occurrence of gaps in the margin (23).

Nanoleakage expression was more prevalent between the adhesive system and dentin, and at the bottom surface, which can be justified by the probably poorer polymerization of the adhesive system due to the light attenuation and reduction of energy density at the bottom of the cavity. If a poor deep polymerization of the Filtek Z250 XT, Filtek Bulk Fill, and Aura composites had been the reason for increased nanoleakage in the lower surfaces, nanoleakage should be expressed at the composite/adhesive system interface (24). Indeed, the fact that some composites tend to debond in deep areas of preparations rather than in superficial ones (24) may also have contributed to the higher prevalence of

nanoleakage in the bottom surface.

Although Filtek Z250 XT showed better adapted margins than the other composites, the bulk fill composites Filtek Bulk Fill and Aura Bulk Fill showed less nanoleakage than traditional composites at the bottom surface. This finding probably occurred because nanoleakage indicates the occurrence of nanospaces that are not seen with marginal adaptation analysis due to their small sizes.

Although the conventional technique to evaluate nanoleakage expression by means of 2D scanning electron microscopy image projections may be dependent on direction, position, and inclination of the chosen field-of-view, it is still been used in recently studies (25). Since our images were obtained in an only inclination of the field-of-view, discrepancies due to artifacts could be avoided. However, further investigations should be performed to analyze the mechanical properties of regular viscosity bulk fill composites in aged specimens, which we did not investigate. Also, other composites available in the market should be evaluated to verify their quality.

The tested regular viscosity bulk fill composites (Filtek Bulk Fill and Aura Bulk Fill) performed similar to or better than traditional composites (Filtek Z250 XT and Aura), and Filtek Bulk Fill performed better than Aura Bulk Fill regarding bottom/top hardness ratio, marginal adaptation, and interfacial nanoleakage.

Resumo

O objetivo deste estudo foi avaliar a resistência de união, razão base/topo de microdureza, adaptação marginal e nanoinfiltração interfacial de resinas compostas bulk fill (BF) e tradicional (RT) de viscosidade regular. Duas BF (Filtek Bulk Fill e Aura Bulk Fill) e duas RT (Filtek Z250XT e Aura) foram testadas. Quarenta cavidades cônicas (4,8x2,8x4,0) foram preparadas em dentina bovina e restauradas com os compósitos (n=10). Depois de 24 h em água, a adaptação marginal foi avaliada por meio do corante com detector de cárie. As faces de topo e base das restaurações cônicas foram coradas por cinco segundos e a porcentagem de fendas na interface compósito/dentina foi determinada utilizando-se imagens analisadas num programa (ImageTool). A microdureza Vickers foi mensurada e a razão base/topo de microdureza (B/T) foi determinada. A resistência de união por push-out foi executada numa máquina de ensaios universal (0,5mm/min) e o padrão de fratura avaliado num estereomicroscópio (20x). Espécimes adicionais (n=3) foram confeccionados para avaliação da nanoinfiltração interfacial. Os dados foram analisados por meio de ANOVA a dois fatores e teste de Tukey ($\alpha=0,05$). A porcentagem de fendas foi maior na base que no topo. A B/T da Aura Bulk Fill foi estatisticamente menor que os demais compósitos. A resistência de união por push-out foi similar entre os compósitos. As BF apresentaram menos nanoinfiltração que as RT na base das restaurações cônicas e não houve diferenças entre os materiais no topo. Conclui-se que a Filtek Bulk Fill comportou-se melhor que a Aura Bulk Fill considerando-se as propriedades analisadas.

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