Microtensile Bond Strength, Bonding Interface Morphology, Adhesive Resin Infiltration, and Marginal Adaptation of Bulk-fill Composites Placed Using Different Adhesives

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Purpose: This study evaluated dentin bond strength, failure mode, interface morphology, adhesive infiltration into dentin, and marginal adaptation of bulk-fill composites used with different adhesives.

Materials and Methods: Third molars received occlusal class I cavities (4 mm x 4 mm x 4 mm) that were bulk-filled with Admira Fusion x-tra (Voco) or SonicFill 2 (Kerr) using four adhesives (Scotchbond Multipurpose, 3M Oral Care; Clearfil SE Bond, Kuraray Noritake; OptiBond All-In-One, Kerr; Futurabond U, Voco). Scotchbond was used with acid-etching, while the remaining adhesives were applied in self-etch mode. Sixty-four teeth were selected for the microtensile bond strength test (n = 8). Failure modes were analyzed with scanning electron microscopy (SEM). Interface morphology and adhesive infiltration (n = 3) were investigated using confocal laser scanning microscopy (CLSM). Marginal adaptation (n = 3) was also evaluated using SEM. Bond strength, failure mode, and adhesive infiltration data were analyzed for distribution and homocedasticity, followed by appropriate statistical analyses ($\alpha = 0.05$).

Results: Regarding bond strength, no differences were found among adhesives for SonicFill; Clearfil showed a significantly lower mean value than did Scotchbond ($p \leq 0.05$) for Admira; the two composites did not differ. Adhesive and mixed failures were observed for all groups. Scotchbond led to thicker hybrid layers with deeper adhesive infiltration as opposed to Futurabond. The groups Admira+Futurabond, SonicFill+Clearfil, and SonicFill+Futurabond presented the highest marginal discontinuity.

Conclusion: The tested bulk-fill composites did not affect dentin bonding. Scotchbond and Clearfil seem to be reliable for bonding SonicFill 2 to dentin. The performance of Futurabond was questionable, given its poor-quality interface and higher percentages of marginal gaps.

Keywords: dentin, composite resin, ultrasonics, dental etching, dental bonding, adhesive.

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The use of resin-based composites as direct restorative materials for posterior teeth has increased in recent decades.[2,12] The clinical success of direct restorations made with composites depends on several factors, namely patient selection, cavity location and size, material choice, composite placement technique, and light curing.[47] Although the avail-

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able dental literature reports long-term clinical service for composite restorations, undesirable polymer-inherent side-effects, such as polymerization shrinkage and low wear resistance of some types of composites, still limit the use of these materials.[12] In fact, polymerization shrinkage-induced stresses at the tooth-restoration interface may result in enamel cracks, postoperative sensitivity, poor marginal adaptation, marginal discoloration, microleakage (which can lead to recurrent carious lesions), and ultimately, failed restorations.[18]

Some modifications in the composition of resin composites have been suggested, such as alterations in monomeric composition and photoinitiators, as well as changes in the amount of monomers, shape, type, and/or superficial treatment of filler particles.[26] Consequently, low polymerization-shrinkage composites, known as bulk-fill, were developed. These materials enable clinicians to perform direct restorations by placing a single composite layer up to 4-5 mm thick (depending on manufacturer's instructions) all at once, as they generate lower shrinkage stress while presenting higher reactivity to light curing.[32] Among the advantages of bulk-fill composites, reduced incorporation of defects into restorations and faster clinical restorative procedures are particularly interesting.[8,41]

Admira Fusion x-tra (Voco; Cuxhaven, Germany) is a bulk-fill composite based on organically modified ceramics (ormocers). Ormocers are hybrid polymers characterized by a siloxane network selectively modified by the incorporation of organic groups (polysiloxanes with light-curable methacrylates covalently bonded to silica).[35] This special composition renders the monomer molecules larger and, consequently, might reduce polymerization shrinkage, wear, and leaching.[7] According to the manufacturer, this ormocer-based material would reduce the volumetric shrinkage to an extremely low level (1.25% by volume), causing very low shrinkage stress (3.87 MPa).[54] SonicFill 2 (Kerr; Orange, CA, USA) is the second generation of a high-viscosity bulk-fill composite that requires the use of an ultrasonic handpiece for application. The energy delivered to the material by sonic activation should produce a significant reduction in its viscosity, turning it into a flowable composite-like material, which would improve its adaptation to the cavity walls and margins.[20] Once the sonic energy is removed, the composite would gradually return to its initial high viscosity, assuring adequate mechanical properties for the restoration.[38] Additionally, ultrasonic application should also reduce void formation in the bulk material.[25]

Similar to non-bulk-fill composites, the hybridization process is responsible for bonding bulk-fill composites to the dentin substrate.[40,59] This process is characterized by the interdiffusion of adhesive monomers into dentin, creating a hybridization zone (layer of resin-reinforced dentin) that bonds the restorative material to the substrate.[36] Conventionally, in order to create a hybrid layer, the dentin surface needs to be etched with phosphoric acid to expose collagen fibrils.[43] Hence, the adhesive resin will infiltrate into the collagen-fibril network, creating micromechanical interlocking to retain the restoration in position.[39]

Adhesives can be classified regarding how they interact with the substrate and their number of clinical application steps.[61] There are etch-and-rinse adhesives, which usually involve two or three application steps, and self-etch adhesives, commonly applied in one or two steps.[53] Etch-and-rinse bonding agents require a separate etching step with phosphoric acid before their application to remove the smear layer and demineralize the dentin surface.[37] Conversely, self-etch adhesives do not need previous etching with phosphoric acid, as they contain acidic monomers capable of mildly etching and infiltrating dentin simultaneously, in addition to chemically reacting and bonding with calcium from dentin hydroxyapatite.[62] Recently, "universal" adhesives, designed to be used in either etch-and-rinse or self-etch approaches, depending on the preference of the operator, have also been introduced on the market.[49]

The bond strength created by the adhesive should be able to withstand the polymerization shrinkage stress of the composite, helping ameliorate its consequences. In fact, a recent publication demonstrated that the bonding strategy (etch-and-rinse or self-etch) significantly influenced the mean values of shrinkage vectors in class-I cavities, which might have occurred because of the lower bond strength achieved with the self-etch approach, according to the authors.[29] Furthermore, the rapid rise and evolution of several types of bulk-fill composites, as well as the paucity of studies dealing with the interaction between these restorative materials and the dentin bonding approaches currently available (3-step etch-and-rinse, 2-step self-etch, and 1-step self-etch) should be considered. Thus, this study aimed to analyze the influence of different adhesives on the bonding interfaces created in class I cavities filled with two currently available bulk-fill composites (Admira Fusion x-tra and SonicFill 2). In order to achieve this objective, dentin microtensile bond strength, failure mode, morphology of the bonding interface, adhesive resin infiltration, and marginal adaptation were evaluated.

The null hypotheses were: 1. there would be no statistically significant differences in dentin bond strength between the tested composites when the same adhesive was used; 2. there would be no statistically significant differences in dentin bond strength between the tested adhesives when the same composite was used; 3. there would be no statistically significant differences in the proportion of failure mode types within each of the tested groups; 4. marginal discontinuity along the cavity walls would not differ between the investigated composites or between the tested bonding agents; 5. the morphology of the bonding interface would not differ between the tested bonding agents; 6. there would be no statistically significant differences in adhesive resin infiltration between the tested bonding agents.

MATERIALS AND METHODS

Teeth Selection and Preparation

One hundred twelve sound human third molars were collected and utilized according to a protocol approved by the

Table 1 Commercial name (manufacturer, shade), composition, and batch number of the materials used in the present study

Material (manufacturer, shade)	Composition	Batch number
Admira Fusion x-tra Nano-hybrid ORMOCER restorative material (Voco, shade A2)	Organically modified silicic acid, barium-aluminum-silica-glass, silica nanoparticles	1840062
SonicFill 2 Single-Fill Bulk Fill Dental Composite System (Kerr, shade A2)	Silicon dioxide, glass, oxide, chemicals, poly(oxy-1,2-ethanediyl), α,α'-[(1-methylethylidene)di-4,1-phenylene]bis[ω-[(2-methyl-1-oxo-2-propen-1-yl) oxy]-, ytterbium trifluoride, 2,2'-ethylenedioxydiethyl dimethacrylate	6964456
Adper Scotchbond Multi-Purpose Adhesive (3M Oral Care)	Primer: water, 2-hydroxyethyl methacrylate, copolymer of acrylic and itaconic acids Adhesive: bisphenol A diglycidyl ether dimethacrylate, 2-hydroxyethyl methacrylate	N876946
Clearfil SE Bond (Kuraray Noritake)	Primer: 2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophilic aliphatic dimethacrylate, dl-camphorquinone, water, accelerators, dyes Bond: bisphenol A diglycidylmethacrylate, 2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aliphatic dimethacrylate, colloidal silica, dl-camphorquinone, initiators, accelerators	CMo165
OptiBond All-In-One Single Component Self-Etch Dental Adhesive (Kerr)	Acetone, 2-hydroxyethyl methacrylate, ethanol, 2-hydroxy-1,3-propanediyl bismethacrylate	LL01577
Futurabond U Dual-curing universal adhesive (Voco)	Liquid 1: bisphenol A diglycidyl ether dimethacrylate, 2-hydroxyethyl methacrylate, 1,6-hexanediylbismethacrylate, acidic adhesive monomer, urethanedimethacrylate, catalyst Liquid 2: ethanol, initiator, catalyst	1748169
Ultra-Etch Etchant (Ultradent)	Phosphoric acid, dimethicone	BFDJ8

Ethics Committee in Research of the Piracicaba Dental School – University of Campinas (CAAE #86272518.3.0000.5418). All teeth were cleaned by hand scaling with a periodontal curette (SS White Duflex; Juiz de Fora, MG, Brazil), and polished with a paste of pumice and water. Afterwards, they were stored in thymol solution (Labsynth; Diadema, SP, Brazil) at 4°C for no longer than three months.

Dentin Microtensile Bond Strength (μTBS) Test

Sixty-four teeth were selected and randomly assigned into eight groups (n = 8) according to bonding strategy (three-step etch-and-rinse, two-step self-etch, or one-step self-etch) and bulk-fill composite (Admira Fusion x-tra, Voco; SonicFill 2 Single-Fill Composite System, Kerr). Table 1 describes the materials used in this study, and Table 2 presents the different experimental groups and their corresponding acronyms. Using a diamond saw (Buehler; Lake Bluff, IL, USA) under water cooling, the teeth were sectioned 2 mm below the cementoenamel junction to remove their roots, and the occlusal enamel was flattened by grinding the teeth down on silicon carbide paper (600-grit). Then, standardized 4 mm x 4 mm x 4 mm occlusal class I cavities were prepared with a diamond bur (#1016, KG Sorensen; Barueri, SP, Brazil) by a single operator (MRS) who had been previously trained and calibrated by one of the authors with extensive clinical experience (MG).

Four different adhesives were used: a three-step etch-and-rinse bonding agent (3E&R, Adper Scotchbond Multipurpose, 3M Oral Care; St Paul, MN, USA); a two-step self-etch bonding agent (2SE, Clearfil SE Bond, Kuraray Noritake; Osaka, Japan); a one-step self-etch bonding agent (1SE, Optibond All-in-One, Kerr), and a universal bonding agent (U, Futurabond U, Voco). Although Adper Scotchbond Multipurpose is not considered the gold standard for etch-and-rinse adhesives, it is still a literature-proven, widely available, three-step etch-and-rinse adhesive. Many reports have shown that Adper Scotchbond Multipurpose provides adequate dentin bonding,[13,17,34] and therefore its use in the present study as a control group was very unlikely to hinder the results. Clearfil SE Bond, on the other hand, was chosen because it is the gold standard for self-etch adhesives, and one of the main objectives of this study was to compare its performance with the simplified self-etch/universal bonding agents recommended by each manufacturer of the tested bulk-fill composites. Optibond All-in-One and Futurabond U were selected not only because they are both self-etch products recommended by the manufacturers of the tested composites, but also because they present a similar composition, which is based on a mixture of methacrylates and bis-methacrylates.

For the 3E&R adhesive, enamel and dentin were both etched with 35% phosphoric acid gel (Ultra-Etch, Ultradent; South Jordan, UT, USA) for 30 and 15 s, respectively. In regard to the remaining adhesives, selective enamel etching was carried out for 30 s with the same phosphoric acid gel mentioned above, while dentin was treated using the self-etch approach. All adhesives were applied and light cured according to their respective manufacturers' instruc-

Table 2 Experimental groups

		Adhesive	
Group acronym	Composite	Commercial name	Classification
ADM + SB	Admira Fusion x-tra	Scotchbond Multipurpose	3E&R
ADM + CF	Admira Fusion x-tra	Clearfil SE Bond	2SE
ADM + OB	Admira Fusion x-tra	OptiBond All-In-One	1SE
ADM + FU	Admira Fusion x-tra	Futurabond U	U
SF + SB	SonicFill 2	Scotchbond Multipurpose	3E&R
SF + CF	SonicFill 2	Clearfil SE Bond	2SE
SF + OB	SonicFill 2	OptiBond All-In-One	1SE
SF + FU	SonicFill 2	Futurabond U	U

Abbreviations: 3E&R (three-step etch-and-rinse); 2SE (two-step self-etch); 1SE (one-step self-etch); U (universal).

Table 3 Mean (±SD) dentin microtensile bond strength in MPa, and [pre-test failures/total number of specimens], according to adhesive and composite

	Composite	
Adhesive	Admira Fusion x-tra	SonicFill 2
Adper Scotchbond Multipurpose	33.29 (± 7.4) [0/54] ^{Aa}	33.27 (± 3.7) [4/36] ^{Aa}
Clearfil SE Bond	24.98 (± 7.0) [2/47] ^{Ab}	27.51 (± 9.5) [2/26] ^{Aa}
OptiBond All-In-One	30.49 (± 5.2) [2/44] ^{Aab}	30.37 (± 6.3) [0/27] ^{Aa}
Futurabond U	27.87 (± 2.6) [1/38] ^{Aab}	27.39 (± 6.1) [0/22] ^{Aa}

Means followed by the same superscript letters are not statistically significantly different ($p > 0.05$). Uppercase letters compare different composites within the same adhesive (rows), while lowercase letters compare different adhesives within the same composite (columns).

tions. Subsequently, the cavities were bulk-filled by the selected composites using single 4-mm-thick increments, which were light cured with a multiple-peak LED light-curing unit (Valo, Ultradent) for 20 s. Before starting the experimental procedures, the light-curing unit was checked using a spectroradiometer (MARC-PS, BlueLight Analytics; Halifax, NS, Canada) to ensure the delivery of a radiant exposure of at least 16.8 J/cm^2.[50] Admira Fusion x-tra was applied using an insertion instrument, while SonicFill 2 was placed into the cavities by means of uni-dose capsules attached to an ultrasonic applicator. Similar to cavity preparation, a single operator (BOS), previously trained and calibrated by the more experienced author (MG), performed all adhesive and restorative procedures to avoid inter-operator variability. Then, the restored teeth were stored at 37°C for 24 h in sealed vials containing water-soaked cotton at the bottom in order to keep them in a humid environment without submerging the composite material in water.

Thereafter, the teeth were serially sectioned in lingual-buccal and mesial-distal directions using a high-speed diamond saw (Buehler; Lake Bluff, IL, USA) under water-cooling to obtain stick-shaped specimens with a cross-sectional area of approximately 1 mm^2. Each restored tooth resulted in 4-9 specimens, and therefore a total of 294 specimens was obtained.

Specimens were tested in a microtensile device attached to a universal testing machine (EZ Test, Shimadzu; Kyoto, Japan). Each specimen was fixed to the device with a cyanoacrylate-based glue (Super Bonder Gel, Henkel/Loctite, Diadema; SP, Brazil), and submitted to the μTBS test at a constant speed of 1 mm/min until failure. After testing, specimens were measured with a digital caliper (Mitutoyo; Kanagawa, Japan) to determine their cross-sectional area. The peak tensile load at the moment of fracture was divided by the cross-sectional area of each specimen to obtain the dentin μTBS in MPa. Then, the mean bond strength values for each group were registered using each tooth as an experimental unit. Pre-test failures were recorded, but not included in calculating the μTBS means.

Scanning Electron Microscopy (SEM) Analysis of Failure Mode

The failure mode of each specimen submitted to the μTBS test was analyzed by SEM (JSM 5600LV, JEOL; Tokyo, Japan). The tested samples were mounted on metal stubs with the fractured surfaces facing upwards, sputter-coated with gold (SDC 050 Sputtercoater, Bal-Tec; Balzers, Liechtenstein), and submitted to microscopic analysis at 100X and 400X magnifications. Specimens were classified into seven categories, according to a previously published study:[52] type I: cohesive failure in resin composite; type II: adhesive failure between resin composite and bonding agent; type III: adhesive failure between dentin and bonding agent; type IV: mixed failure (dentin, bonding agent, and resin composite can be observed on the same fractured surface); type V: cohesive failure in the bonding agent; type VI: cohesive failure in the hybrid layer; and type VII: cohesive failure in dentin.

Confocal Laser Scanning Microscopy (CLSM) Analysis of Adhesive-Dentin Bonding Interface Morphology

The occlusal enamel of 24 third molars was ground down using 600-grit silicon carbide paper, and standardized 4 mm x 4 mm x 4 mm occlusal class I cavities were prepared. Rhodamine B dye (Sigma-Aldrich; St. Louis, MO, USA), which has a pinkish-red color, was added to the adhesives at a concentration of 0.1 wt%.[30] Teeth were then randomly assigned to the experimental groups described in Table 2 (n = 3), and restorative procedures were carried out as described above for the μTBS test. Samples were stored in vegetable oil at 37°C for 24 h to avoid water loss and/or dye dissolution. Afterwards, teeth were sectioned into 1-mm-thick slices. These slices were manually polished with 2000-grit silicon carbide paper for 30 s.

Subsequently, samples were analyzed by CLSM (TCS SP5, Leica Microsystems; Mannheim, Germany). The excitation energy provided by the argon (488 nm) and He-Ne (453 nm) lasers and the photomultipliers amplification were constant throughout the whole investigation. A layer approximately 10 μm below the surface of the sample was observed, and the CLSM micrographs were obtained in fluorescent and transmission modes with an oil-immersion objective (63X magnification, 3X zoom, numeric aperture of 1.3, pinhole of 5.5 μm). At least three sets of micrographs were obtained for each group, which comprised (1) an image of dye detection in fluorescent mode, (2) a gray-scale image of the sample surface in transmission mode, and (3) an image formed by overlapping the micrographs of fluorescent and transmission modes.

For the bonding interface morphology analysis by CLSM, visual differences among experimental groups were analyzed regarding the presence and thickness of the hybrid layer, and resin tags formation. The general appearance of the slices from the three repetitions of each group was used to characterize trends observed in different test conditions. Additionally, a quantitative analysis of adhesive resin infiltration into dentin was carried out using the ImageJ software (National Institute of Health; Bethesda, MD, USA). Three measurements of the integrated density (pixels/μm^2) of the fluorescent signal observed in the micrographs were obtained for each image, from the top of the hybrid layer to 15 μm into the dentin substrate.

Internal Marginal Adaptation Analysis by SEM

Additional teeth (n = 3) were prepared for each group as described above, and transversally sectioned into halves. A polyvinyl siloxane with materials of light and heavy consistencies (Express XT, 3M Oral Care) was used to make impressions of the half-restored surfaces after polishing. Epoxy resin (EpoxiCure, Buehler) was then poured into the impressions, and the resulting polymeric replicas were sputter-coated with gold (SCD 050, Bal-Tec; Balzers, Liechtenstein). Then, the presence of internal interfacial gaps was analyzed in a scanning electron microscope (JEOL, JSM-5600LV; Tokyo, Japan). Approximately 40 images were obtained for each specimen at 200X magnification in order to observe the entire length of the bonded interfaces. ImageJ software was used to precisely measure the length of debonded areas along the internal perimeter of the restorations. The scale bar of SEM images was used for distance calibration, and individual measurements of debonded segments were obtained in millimeters. These were converted to percentage of the total length of the bonding interface, resulting in marginal discontinuity measurements for each group.[44]

Statistical Analysis

Data were tested for normal distribution (Shapiro-Wilk and Kolmogorov-Smirnov test) and homoscedasticity (Levene's test). Microtensile bond strength data was analyzed with two-way ANOVA, followed by Bonferroni's post-hoc test, while failure mode data were submitted to Pearson's chi-squared test. Results of integrated density were analyzed with the Kruskal-Wallis test, followed by the Mann-Whitney post-hoc test. CLSM and SEM images were descriptively analyzed through visual comparison between groups by two independent evaluators (MS and RBEL), who consulted with a third researcher (MG) in case of disagreement regarding their conclusions. Statistical analyses were performed using SPSS 21.0 (IBM SPSS, v. 21.0, IBM; Armonk, NY, USA), with a significance level set at 5%.

RESULTS

Microtensile Bond Strength Test

Mean (± SD) dentin bond strengths are presented in Table 3, along with the proportion of pre-test failures for each group (pre-test failures/total number of specimens per group). Two-way ANOVA showed a significant influence of the type of adhesive on bond strength ($p = 0.013$). On the other hand, no significant influence of the composite ($p = 0.764$) or significant interaction between type of adhesive and composite ($p = 0.901$) were found.

The tested bulk-fill composites did not differ from each other when the same adhesive was used ($p > 0.05$). In regard to the Admira Fusion x-tra results, Adper Scotchbond Multipurpose showed higher bond strength than did Clearfil SE Bond ($p = 0.016$), whereas both OptiBond All-In-One and Futurabond U presented intermediate values that did not differ from the other adhesives ($p > 0.05$). As for SonicFill 2, no statistically significant differences in bond strength means were found between the adhesives ($p > 0.05$).

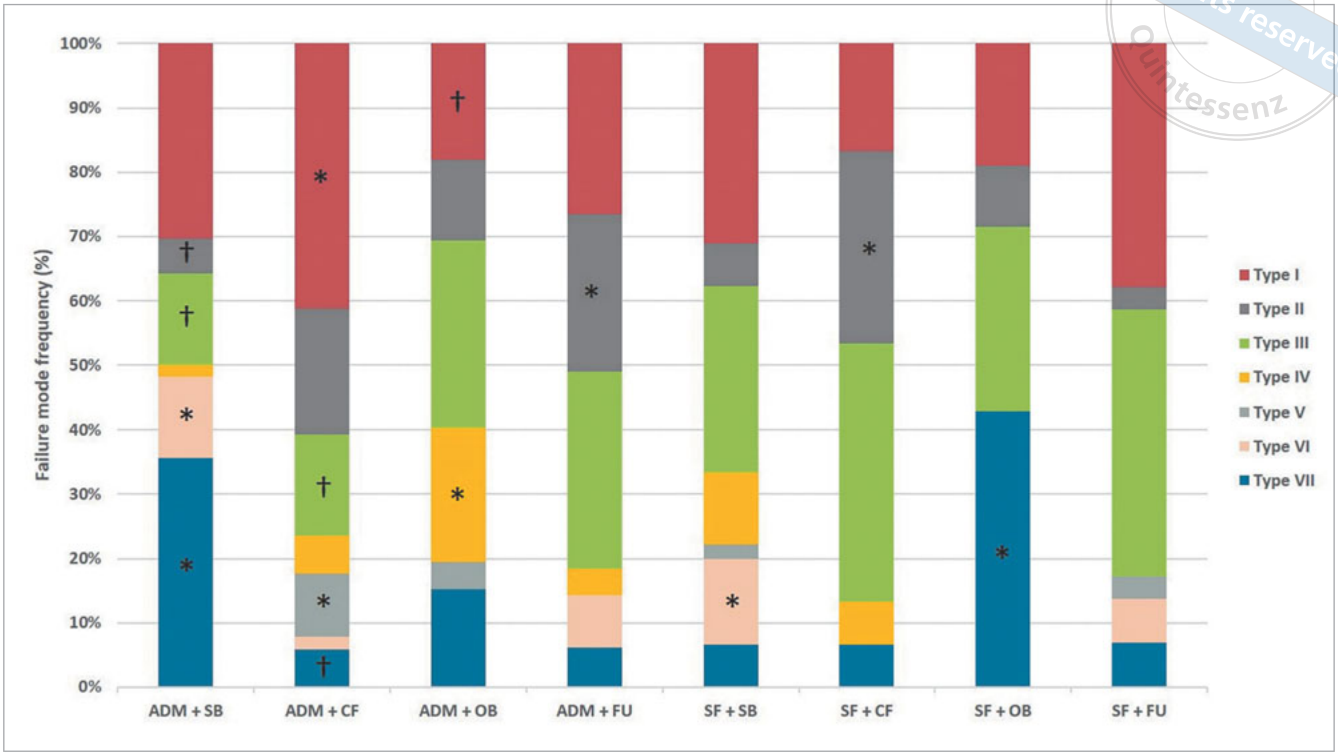

Fig 1 Failure modes (in %) according to each group. Type I: cohesive failure in composite; type II: adhesive failure between bonding agent and composite; type III: adhesive failure between bonding agent and dentin; type IV: mixed failure; type V: cohesive failure at the adhesive layer; type VI: cohesive failure in the hybrid layer; type VII: cohesive failure in dentin. *Represents a statistically significantly higher trend of occurrence of a certain failure mode. †Represents a statistically significantly lower occurrence of a certain failure mode.

Failure Mode Analysis by SEM

Figure 1 depicts the frequency of occurrence of each failure mode. Pearson's chi-squared test indicated that the association between adhesive and composite statistically significantly influenced the failure mode distribution [X^2(42 degrees of freedom) = 129.87; $p < 0.001$]. A lower tendency of occurrence (adjusted residual < -1.96) was observed for the following failure modes within the tested groups: Admira Fusion x-tra + Adper Scotchbond Multipurpose (types II and III); Admira Fusion x-tra + Clearfil SE Bond (types III and VII); Admira Fusion x-tra + OptiBond All-in-One (types I and VI). Conversely, a higher tendency of occurrence (adjusted residual > 1.96) was found for the following fracture patterns within the evaluated groups: Admira Fusion x-tra + Adper Scotchbond Multipurpose (types VI and VII); Admira Fusion x-tra + Clearfil SE Bond (types I and V); Admira Fusion x-tra + OptiBond All-in-One (type IV); Admira Fusion x-tra + Futurabond U (type II); SonicFill 2 + Adper Scotchbond Multipurpose (type VI); SonicFill 2 + Clearfil SE Bond (type II); and SonicFill 2 + OptiBond All-in-One (type VII).

Adhesive-Dentin Bonding Interface Morphology Analysis by CLSM

Figure 2 shows representative images of the interface morphology analysis performed by CLSM. For both composites, thick hybridization zones (over 6 µm) and multiple, deep resin tags were observed when used with the adhesive Scotchbond Multipurpose. In the Admira Fusion x-tra + Adper Scotchbond Multipurpose group, a clear differentiation between resin composite and adhesive layer was not possible. The self-etch adhesive Clearfil SE Bond led to hybrid layer formation for both composites, although a thicker hybrid layer and shorter resin tags were observed in the Admira Fusion x-tra + Clearfil SE Bond group compared with its counterpart (SonicFill 2 + Clearfil SE Bond). Hybrid zones and resin tags were also found in the micrographs of the OptiBond All-In-One groups. On the other hand, Futurabond U resulted in thin hybrid layers (less than 1.5 µm), without any resin tags and thick adhesive layers.

The integrated density (pixels/μm^2) of the fluorescent signal obtained by confocal microscopy was used to calculate adhesive resin infiltration and is presented in Fig 3. The nonparametric Kruskal-Wallis test showed that the type of adhesive significantly influenced the results for both Admira Fusion x-tra (X^2(3 degrees of freedom) = 25.869, $p = 0.001$), and SonicFill 2 (X^2(3 degrees of freedom) = 8.378, $p = 0.039$). When the composite Admira Fusion x-tra was used, Scotchbond Multipurpose presented higher adhesive resin infiltration compared to Clearfil SE Bond (adjusted p-value = 0.001) and Futurabond U (adjusted p-value = 0.001),

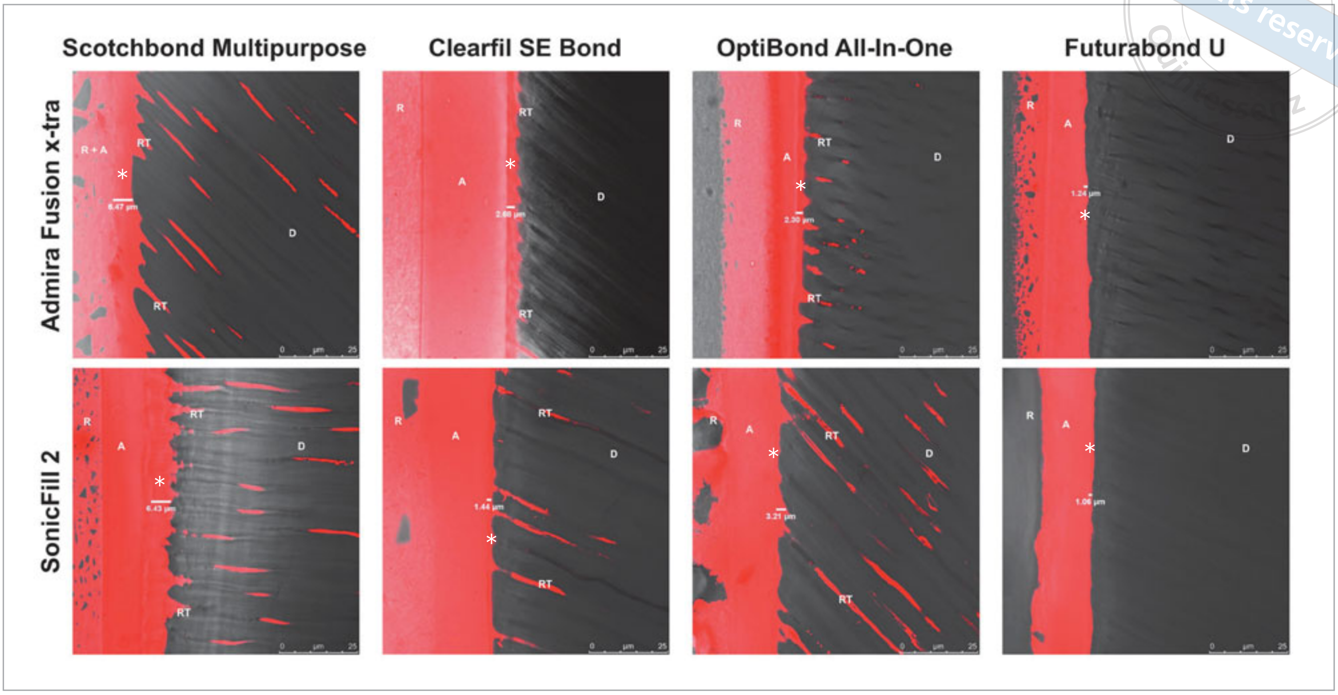

Fig 2 CLSM images (overlap of fluorescent and transmission modes) of the bonding interface for the tested groups. The top row presents micrographs of Admira Fusion x-tra, while the bottom row shows micrographs of SonicFill 2. The columns present micrographs of the tested adhesives, from left to right: Scotchbond Multipurpose, Clearfil SE Bond, OptiBond All-In-One, and Futurabond U. *Indicates hybrid zones. R: resin composite; A: adhesive layer; RT: resin tags; D: dentin.

but it did not differ from OptiBond All-In-One (adjusted p-value > 0.05). Futurabond U also showed significantly lower adhesive resin infiltration than OptiBond All-In-One (adjusted p-value = 0.014). For SonicFill 2, OptiBond All-In-One presented higher integrated pixel density than did Futurabond U (adjusted p-value = 0.05), while the remaining adhesives did not differ between each other (adjusted p-value > 0.05).

Internal Marginal Adaptation Analysis by SEM

Figure 3 shows representative SEM images of the internal adaptation of the restorative material related to the cavity angle and pulpal wall of the tested groups. Overall, Admira Fusion x-tra presented adequate marginal adaptation at both sites (cavity angles and pulpal wall), with low percentages of marginal discontinuity for Scotchbond Multipurpose (5.3%), Clearfil SE Bond (9.4%), and OptiBond All-In-One (8.4%). The group Admira Fusion x-tra + Futurabond U showed higher marginal discontinuity (40.0%). Conversely, SonicFill 2 led to more defects along the adhesive-dentin interfacial perimeter of the restoration, with few occasional small gaps for Scotchbond Multipurpose and OptiBond All-In-One (26.9% and 39.3% marginal discontinuity, respectively), and longer, wider gaps for Clearfil SE Bond and Futurabond U (62.1% and 69.2% marginal discontinuity, respectively).

DISCUSSION

Shrinkage stresses are an unavoidable consequence of the polymerization process that occurs in a confined space (tooth cavity) due to bonding to enamel/dentin walls or other composite layers.[24] These internal stresses are transferred to the bonding interface as tensile forces, which might result in stress-relieving gaps if they exceed the local bond strength.[16] Therefore, the marginal and internal integrity of composite restorations might be compromised by debonding and premature gap formation, which could translate into clinical complications, such as marginal leakage, marginal staining, post-operative sensitivity, and/or recurrent caries.[18,33] Thus, the analysis of the bonding interface quality when using different adhesives in association with low-shrinkage bulk-fill composites is of the utmost importance in the current context of restorative dentistry.

As observed in Table 3, the different composition and application procedures of the tested restorative materials did not affect dentin µTBS, since no statistical differences were found between composites for any of the investigated bonding agents. Hence, the first null hypothesis was accepted. Scarce and conflicting data about the tested composites are available in dental literature. Abbasi et al[1] analyzed the volumetric polymerization shrinkage of six composites, including

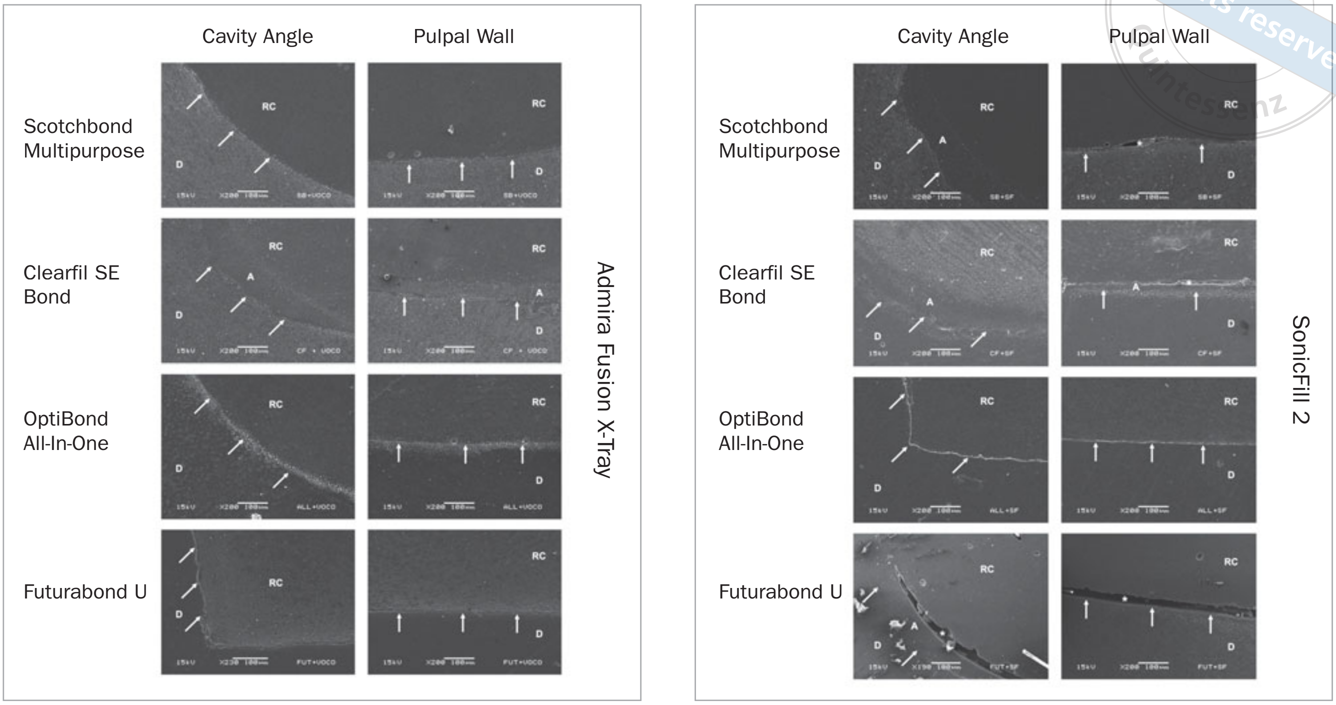

Fig 3 Representative SEM images of internal marginal adaptation for the tested groups at 200X magnification. Arrows indicate the bonding interface, and * indicates gap areas. RC: resin composite; D: dentin; A: adhesive layer.

SonicFill 2, which did not differ from a microhybrid, non-bulk-fill material. Their findings agreed with other publications which stated that the SonicFill restorative system led to similar volumetric polymerization shrinkage compared to non-bulk-fill composites.[6,28] As for Admira Fusion x-tra, a recent study showed its shrinkage stress to be lower than a methacrylate-based, non-bulk-fill composite when larger resin increments of 24 mm^3 were used, which was not the case for 12-mm^3 samples.[48]

Another study demonstrated Admira Fusion x-tra and SonicFill presented lower linear shrinkage and shrinkage force than the non-bulk-fill control group, while Admira Fusion x-tra also showed superior results compared to SonicFill.[14] On the other hand, no differences in cuspal deflection measurements between Admira Fusion x-tra and the SonicFill restorative system have been reported. However, the same study found that both bulk-fill composites performed better than a methacrylate-based, non-bulk-fill material.[55] The bond strength and morphology of the dentin-adhesive interface, as well as the internal adaptation results of the present study, coupled with the volumetric polymerization shrinkage and shrinkage stresses previously reported for Admira Fusion x-tra and SonicFill,[1,6,14,28,48,55] could lead to a better understanding of the behavior of these materials in class I cavities.

However, µTBS results should be cautiously interpreted because there is significant variability between the tested composites regarding composition and application mode. One might argue the only way to achieve a precise, trustworthy comparison between composite-related variables is by using a model, standardized composition. Thus, the target study variable could be modified to isolate its influence on the results by, for example, having the same monomer blend and photoinitiator system for all groups, but with different filler content/ratio,[4,22] or by having different types of monomers between groups, but all with the same filler content and photoinitiator system.[15,19] Consequently, compositional variability is an inherent limitation seen in many in vitro studies evaluating commercially available composites.[21] Another variable with respect to the tested composites would be their mode of application, since SonicFill 2 was applied with an ultrasonic device, while Admira Fusion x-tra was manually inserted into the cavity using a spatula. However, applying Admira Fusion x-tra with the same ultrasonic device would be interesting to evaluate the effect of mode of application in comparison with SonicFill 2. This procedure was not possible because the manufacturer of the SonicFill restorative system has developed its own ultrasonic applicator that is not compatible with the uni-dose capsules of Admira Fusion x-tra. Additionally, an in-depth evaluation of this variable was not within the scope of the present study, and Admira Fusion x-tra is not recommended for ultrasonic application by its manufacturer.

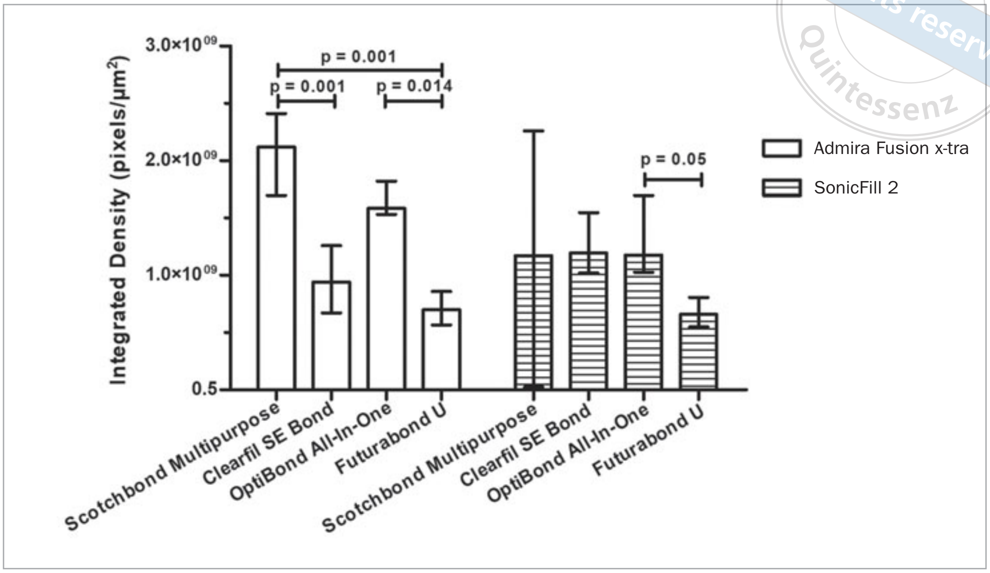

Fig 4 Median and interquartile integrated density (pixels/µm^2) of adhesive resin infiltration into etched dentin for the tested groups. Horizontal bars connecting different groups represent statistically significant differences (adjusted p-value < 0.05; Mann-Whitney test).

The creation of a high-quality hybrid layer, the interaction between dentin and adhesive resin, and the presence of a hydrophobic adhesive layer have been suggested as requirements to minimize the main consequence of shrinkage stresses, ie, the disruption of the contact between resin composite and cavity walls.[6] Even so, the bond strength of SonicFill 2 did not seem to be affected by the different dentin bonding approaches (3E&R with Scotchbond Multipurpose, 2SE with Clearfil SE Bond, 1SE with OptiBond All-In-One or Futurabond U). All the tested adhesives probably established a tight bond between the composite and the dental structure, which could mask the influence of polymerization shrinkage stress, unless the samples are submitted to loading, which allows residual stresses at the restoration margins to weaken the bonding interface.[28]

In contrast, Admira Fusion x-tra presented lower bond strength when used with Clearfil SE Bond compared to Scotchbond Multipurpose, while no other statistical difference was observed between bonding agents for this composite (Table 3), requiring the rejection of the second null hypothesis. Nevertheless, this group had the highest number and a statistically significant higher trend of resin composite cohesive failures (Fig 1). The occurrence of cohesive failures implies that the resin-dentin bond strength could be stronger than the cohesive strength within the composite or the dentin substrate.[11] Hence, this fracture pattern might lead to underestimating the true dentin-adhesive bond strength value.[9] Consequently, the performance of Clearfil SE Bond cannot be questioned solely based on the present results, especially since this bonding agent is regarded as the gold standard for self-etch adhesives, with proven clinical performance.[27,45] Moreover, a comparison between an ormocer-based bonding agent and a non-specific self-etch adhesive, both used with an ormocer composite, showed significantly better interfacial morphology and microleakage results when the ormocer was used with an specific, chemically similar bonding agent.[23] Therefore, the clinical performance of Clearfil SE Bond with Admira Fusion x-tra should be investigated in comparison with non-ormocer composites to determine whether the less favorable results of this study could be composite-dependent due to potential chemical incompatibility between the adhesive resin and the ormocer resin matrix.

Statistical analysis showed higher trends toward adhesive failures (between bonding agent and composite), cohesive failures within the hybrid layer, and mixed failures among the tested groups (Fig 1). Therefore, the third null hypothesis also had to be rejected. Adhesive failures between simplified bonding agents and composites might occur due to chemical reactions between acidic adhesive monomers and tertiary amines from the resin composite, mainly when the uncured acidic adhesive layer and composite remain in contact for a prolonged period.[56] Failures within the adhesive-composite interface might also occur due to regions of poor co-polymerization between the adhesive resin and the lining composite; ideally, the bonding agent should bind to the underlying composite through a process of co-polymerization of residual double bonds (–C=C–) in the oxygen inhibition layer.[60] Cohesive failures within the hybrid layer imply that a defective hybridization zone was formed, and this region fractured because it was the weakest area of the bonding interface.[3] In contrast, mixed failures suggest the presence of a strong bond, with a fracture path probably starting in composite and reaching dentin through the bonding interface.[63] Theoretically, mixed failures are preferable, as they indicate that the structures involved in dentin bonding acted as a single unit rather than separate layers.[31,52]

SEM showed that Clearfil SE Bond + SonicFill 2 and Futurabond U + SonicFill 2 were the groups with the highest percentages of marginal discontinuity (Fig 3), also resulting in high numbers of adhesive failures. Altough the dentin bond strength of these two groups was not statistically different than that of Adper Scotchbond Multipurpose + SonicFill 2 or OptiBond All-in-One + SonicFill 2, the latter adhesives showed more cohesive failures, which might suggest their hybridization zones were not as sensitive to tensile stresses generated by composite polymerization shrinkage as were Clearfil SE Bond or Futurabond U. Consequently, the fourth null hypothesis was rejected. Additionally, microtensile bond strength specimens produced from gap areas were more prone to manipulation errors and were not included in the mean bond strengths, which is in keeping with the guidelines of the Academy of Dental Materials.[5] This might have increased the bond strength means for Clearfil SE Bond and Futurabond U, despite the higher percentages of marginal gaps, especially because these adhesives presented reasonably higher standard deviations than their Admira Fusion x-tra counterparts (Table 3).

Low adhesive resin infiltration into dentin and poor interaction between the adhesive and dentin were observed for Futurabond U, as shown in the CLSM images (Fig 2). Based on the information previously discussed, the results for Futurabond U combined with SonicFill 2 might be due to incompatibility between the acidic adhesive resin and the composite,[56] since the use of an ultrasonic applicator would prolong the contact of the adhesive with SonicFill 2, resulting in type-II adhesive failures. However, this does not apply to the other group with a higher proportion of type II failures (almost 30% for the Clearfil SE Bond + SonicFill 2 group), because Clearfil SE Bond is a 2SE adhesive that requires application of a hydrophobic layer of adhesive resin over the acidic dentin primer, preventing its contact with the composite.[58] Thus, we speculate that the higher probability of adhesive-composite failures in this group might have been due to localized regions of insufficient co-polymerization between the adhesive resin of Clearfil SE Bond and the ultrasonically applied composite. SonicFill 2 is a reasonably new material and, as far as can be told from the current scientific literature, its compatibility with other polymer-based restorative products has not been studied yet.

Another interesting finding derived from the present results concerns the performance of the two 1SE adhesives. Regardless of the bulk-fill composite used, Optibond All-in-One and Futurabond U did not differ in regard to dentin bond strength or adhesive infiltration into dentin. Still, Futurabond U seemed to generate higher marginal discontinuity than Optibond All-in-One for all of the tested resin-based restorative materials. In the case of Admira Fusion x-tra, although Futurabond U showed higher marginal discontinuity than did Optibond All-in-One, gap formation was observed mostly at the axial walls of the cavities. Consequently, these localized regions of marginal disruption were very unlikely to affect bond strength results, which might explain the similar mean values obtained for the two 1SE adhesives. Conversely, SEM analysis revealed longer and wider gaps at the pulpal wall of the cavities of SonicFill 2 when this composite was coupled with Futurabond U compared to Optibond All-in-One. The difference between the 1SE bonding agents regarding marginal discontinuity, despite their similar bond strength, further supports our hypothesis that dentin bond strengths were not affected by gap formation due to the impossibility of testing specimens obtained from these regions, since they would debond during cutting or manipulation.

Figure 2 depicts the morphological features of the bonding interfaces investigated in the present study. A clear correlation between hybrid layer thickness and bond strength has not been successfully demonstrated,[46] although a hybridization zone of at least 0.5 µm, which would be roughly five collagen fibrils deep, has been suggested as the minimum threshold to create an adequate bonding interface in intertubular dentin.[57] The present CLSM results are in accordance with the bond strengths obtained, since even the thinnest hybrid layer (1.06 µm for Futurabond U + SonicFill 2) was well over 0.5 µm thick. Another interesting finding regarding bonding interface morphology was that some of the groups presented noticeably short or no resin tags (Clearfil SE Bond + Admira Fusion x-tra, Futurabond U + Admira fusion x-tra, and Futurabond U + SonicFill 2). However, the presence of deep resin tags does not contribute much to bond strength, as the surface adhesion and the entanglement between adhesive resin and intertubular collagen fibrils are the two main factors contributing to the overall resin-dentin bond.[51] Also, the cross-sectional area of resin tags decreases along their depth, which reduces their contribution to bonding.[42] Thus, the fifth null hypothesis was not accepted.

Quantitative analysis of integrated density (pixels/μm^2) led to the rejection of the sixth null hypothesis, as it revealed higher adhesive resin infiltration for the etch-and-rinse approach compared to the remaining adhesives for Admira Fusion x-tra (except OptiBond All-In-One), while Futurabond U had lower adhesive resin penetration compared to OptiBond All-In-One for both composites (Fig 4). These results were expected, since the separate etching step used by Scotchbond Multipurpose causes deeper demineralization of around 5-10 µm,[51] leaving a larger area needing to be filled by the bonding agent. Furthermore, CLSM images imply that Futurabond U had a lower ability to demineralize and infiltrate dentin compared to the other self-etch bonding agents, which corroborates previous results in the literature, showing a thin hybrid layer and larger tracer-infused water-rich zones for Futurabond U compared to other self-etch adhesives.[10]

The present results may give a general idea about important aspects to consider when using the tested composites in combination with different dentin bonding approaches in clinical dentistry. The type of bulk-fill restorative material (ormocer-based or ultrasonically applied) does not seem to play a major role in the final quality of dentin-adhesive bonds. Nonetheless, bond strengths and bonding morphology characteristics suggest that the adhesives Adper Scotchbond Multipurpose and Clearfil SE Bond still remain the more reliable options for bonding ultrasonically applied SonicFill 2 to dentin. However, the chemical compatibility of Clearfil SE Bond with the new composites tested should be further investigated, since the Admira Fusion x-tra specimens bonded with this self-etch adhesive presented lower bond strengths than did Scotchbond Multipurpose, while higher numbers of failures between the adhesive resin and the tested bulk-fill composites were detected when it was combined with SonicFill 2. The adhesive Futurabond U presented lower-quality bonding interfaces and higher percentages of internal gaps, especially when associated with SonicFill 2. Therefore, the long-term performance of this bonding agent is questionable, and it needs to be thoroughly studied in future investigations.

CONCLUSIONS

- The type of bulk-fill composite (Admira Fusion x-tra or SonicFill 2) did not affect dentin microtensile bond strength, regardless of bonding approach.
- The dentin bond strength of Admira Fusion x-tra was affected by the type of adhesive used, although this effect should be interpreted with caution, considering failure mode analysis.
- The etch-and-rinse approach led to thicker hybrid layers with high adhesive infiltration into the dentin substrate.
- SonicFill 2 yielded higher marginal discontinuity compared to Admira Fusion x-tra.

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Clinical relevance: The results provide a general idea about dentin bonding quality when the tested bulk-fill composites are used with different bonding agents. Scotchbond Multipurpose seems a reliable option for bonding bulk-fill composites to dentin, while Futurabond U was not able to prevent gap formation.