




RESEARCH ARTICLE

# Significance of immediate dentin sealing and flowable resin coating reinforcement for unfilled/lightly filled adhesive systems

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## Abstract

**Background:** Immediate dentin sealing implies applying an adhesive system to dentin directly after tooth preparation, before impression. The technique is universal (inlays, onlays, veneers, crowns) and well documented clinically and experimentally. Different types of dentin bonding agents (DBAs) are available on the market. Major differences lie in the thickness of the hybrid layer and overlaying adhesive resin (filled vs. unfilled/lightly filled adhesives).

**Objective:** The objective of this work is to provide precise clinical instructions and present new experimental data about the bond strength of five DBAs (Optibond FL, Scotchbond MP, Single Bond Plus, Clearfil SE Bond, and Scotchbond Universal) used conventionally (dentin sealed at the time of restoration delivery) or with immediate dentin sealing, as well as with an additional flowable resin coating.

**Methods:** Seventy-five human molars were selected, restored/tested according the microtensile bond strength method. Fifteen groups (n=5) were obtained from the combination of the five DBAs and three application modes: delayed dentin sealing, immediate dentin sealing and immediate dentin sealing with flowable resin coating.

**Results:** It appears that immediate dentin sealing was confirmed to significantly improve the bond strength of all tested adhesives. The use of a flowable resin coating reinforcement after immediate dentin sealing increased the microtensile bond strength of all unfilled/lightly filled adhesives (from 233% of increase for ScotchBond MP, up to 560% for Clearfil SE Bond) and maintained the performance of the 3-step golden standard adhesive. Optibond FL used with (52.51 MPa) or without (54.75 MPa) additional flowable resin coating and Clearfil SE Bond (45.64 MPa) used with flowable resin coating provided the best results.

**Clinical Significance:** The original immediate dentin sealing (IDS) technique implies the use of a filled DBA. With unfilled/lightly filled adhesives, it is suggested to reinforce IDS with an additional flowable resin coating. This seems especially paramount to the performance of simplified adhesive systems to protect the thin bonding interface from oxygen inhibition and preserve IDS layer during predelivery cleaning of the preparation. The clinical reinforcement of unfilled/lightly filled IDS with flowable resin composite is encouraged for more predictable bonding.

## 1 | INTRODUCTION

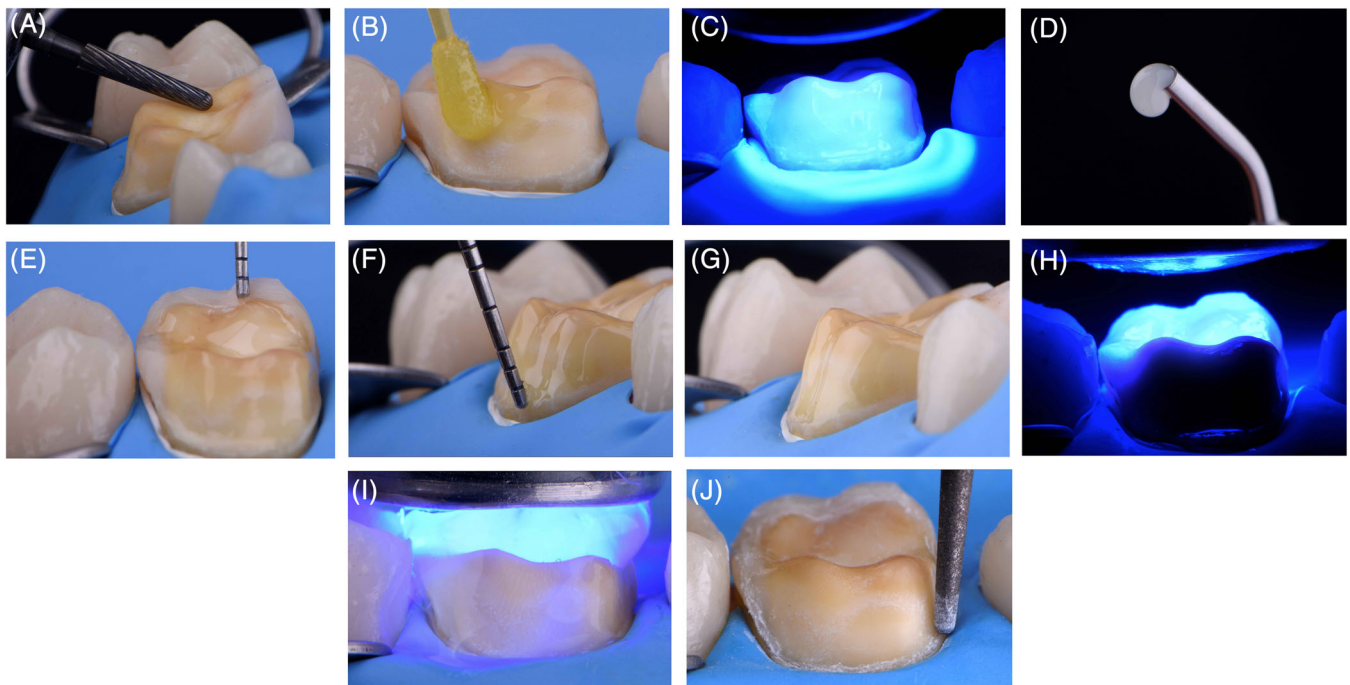
Immediate dentin sealing (IDS) is an optimized dentin bonding mode and has been used since the mid-1990s and represents the best dentin bonding strategy when using indirect or semi-(in)direct bonded restorations.<sup>1</sup> There are over 20 reasons to justify the use of IDS, among which the decrease in bacterial leakage, postoperative sensitivity and gap formation, increase in bond strength, mechanical resistance of the overlaying restoration, reinforcement of tooth structure, as well as many practical advantages.<sup>2-30</sup> Altogether, those elements have a significant impact on the clinical performance of the restorations<sup>27</sup> and increased survival rates have been demonstrated for porcelain veneers bonded to large dentin surfaces.<sup>31</sup>

IDS consists of applying the dentin bonding agent (DBA) immediately after dentin preparation, hence avoiding its contamination with oral fluids and impression or provisional materials.<sup>2,20,32-37</sup> The original technique called resin coating<sup>38,39</sup> used an unfilled/lightly filled adhesive and aimed at sealing both enamel and dentin.<sup>40</sup> In the early 1990s, Pashley et al<sup>33</sup> suggested to seal crown preparations with a DBA for biological reasons. But they warned about the problem of pooling of adhesive onto the preparation shoulder, hence the necessity to apply the DBA before making impressions. Air-thinning the

adhesive is not an efficient solution to the problem of resin accumulation because thin adhesives do not polymerize properly and then interfere with the polymerization of the impression material.<sup>41</sup> Therefore some combinations of resin liners and impression materials are not compatible with the IDS technique.<sup>41,42</sup>

All those problems are avoided in the original IDS technique because a filled adhesive system such as Optibond FL (Kerr) is used. The more uniform thickness of the flowable resin coating<sup>43</sup> radiopacity, and outstanding bond strength<sup>2,20,32,44</sup> make this filled 4th generation 3-step etch-and-rinse adhesive the perfect DBA for IDS. Some clinicians, however, feel more comfortable not etching deep dentin and prefer self-etch DBAs instead, which are simpler, easier and faster to apply, potentially generating less errors (reduced technique sensitivity)<sup>45</sup> and postoperative sensitivity.<sup>46-48</sup> However, simplified adhesives have inferior mechanical performance.<sup>44,49-51</sup> Particularly when used in the IDS technique, there is a higher risk of removing the thin adhesive and re-exposing the dentin when cleaning the preparation just before final delivery.<sup>20,41,43</sup> This might explain why a number of studies fail to show the benefit of IDS.<sup>36,52-54</sup>

A possible solution to increase the dentin bond strength of these simplified adhesives is to supplement IDS with an layer of flowable resin composite<sup>5,7,10,55,56</sup> to protect and strengthen the hybridized dentin,<sup>57-59</sup> the so-called “reinforced IDS” approach (Figure 1).



**FIGURE 1** “Reinforced IDS” technique for lightly filled DBA, Clearfil SE Bond. (A) After guided preparation, rubber dam isolation is preferred for moisture control. Dentin is refreshed with carbide bur at low speed for decreased smear layer. (B) Self-etch primer is actively applied on dentin for 20 s (“dentin spa”) then solvents are evaporated with gentle air-drying for 5 s. (C) Adhesive resin is also actively applied on dentin for 20 s. The lightly filled adhesive is air-thinned for 5 s and light-polymerized for 15 s. (D) Filled hydrophobic resin (flowable composite resin) is used to protect hybrid layer, reinforcing the immediately sealed dentin. (E) Thin coat of flowable composite resin is applied over entire IDS area with aid of a microbrush or a periodontal probe. (F) Excess of resin on chamfers are removed with a clean microbrush or a probe. (G) Aspect of reinforced IDS layer (flowable resin coating) after resin excess removal. (H) Light-polymerization of the reinforced IDS layer (resin coat) for 20 s in each area of the preparation. (I) Air-blocking barrier is used to avoid the oxygen-inhibited layer and extra 10 s of light-polymerization is added to each area of the preparation. (J) Enamel margins are refined in low speed to remove resin excesses. Dentinoenamel junction should be left untouched

**TABLE 1** Material, commercial name, manufacturer, and composition for each material used

Material	Commercial name	Manufacturer (location)	Composition
Dentin bonding agent	Optibond FL	Kerr (Orange, CA)	Primer: Hydroxyethyl methacrylate, glycerolphosphate dimethacrylate, phthalic acid monoethyl methacrylate, ethanol, water, photo-initiator. Adhesive: Triethylene glycol dimethacrylate, urethane dimethacrylate, glycerolphosphate dimethacrylate, hydroxyethyl methacrylate, bis-phenol A glycol dimethacrylate, filler (48%wt), photo initiator.
	Scotchbond Multi-Purpose	3M ESPE (St. Paul, MN)	Primer: Water, 2-Hydroxyethyl Methacrylate (HEMA), copolymer of acrylic and itaconic acids. Adhesive: Bisphenol A Diglycidyl Ether Dimethacrylate (BISGMA), 2-Hydroxyethyl Methacrylate (HEMA)
	Single Bond Plus (Single Bond 2)	3M ESPE (St. Paul, MN)	BISGMA, Silane Treated Silica, HEMA, Copolymer of Acrylic and Itaconic Acids, Glycerol 1,3 Dimethacrylate, UDMA, Water, Diphenyliodonium Hexafluorophosphate.
	Clearfil SE Bond	Kuraray Noritake Dental (Tokyo, Japan)	Self-etch primer: 10-MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, water Adhesive: 10-MDP, bis-GMA, HEMA, hydrophilic dimethacrylate, camphorquinone, silanated colloidal silica
	Scotchbond Universal	3M ESPE (St. Paul, MN)	2-Hydroxyethyl methacrylate, BISGMA, 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide, ethanol, water, 2-propenoic acid, 2-methyl-, 3-(trimethoxysilyl) propyl ester, reaction products with vitreous silica, copolymer of acrylic and itaconic acid, camphorquinone, dimethylaminobenzoate(-4), (dimethylamino)ethyl methacrylate
Resin composite	Filtek Z100	3M ESPE (St. Paul, MN)	Silane Treated Ceramic, TEGDMA, BISGMA, 2-Benzotriazolyl-4-Methylphenol
Flowable resin composite	Filtek Bulk Fill Flow	3M ESPE (St. Paul, MN)	Silane treated ceramic, UDMA, Substituted Dimethacrylate, Ytterbium Fluoride, BISGMA, BISEMA-6, TEGDM.
Provisional restoration	Revotek LC	GC Corporation (Tokyo, Japan)	Urethane dimethacrylate (UDMA), trimethacrylate, amorphous silicon dioxide, butylated hydroxytoluene (BHT), titanium dioxide, iron(III) oxide.
Impression material	Express XT	3M ESPE (St. Paul, MN)	Quartz silica, vinyl polydimethylsiloxane (VPS), dimethyl methyl hydrogen silicon fluid; silane treated silica, chromium oxide, poly(dimethylsiloxane).
Etchant	Potenza Attacco	PHS (Joinville, Brazil)	35% phosphoric acid
Air abrasion powder	Aluminum Oxide	Bioart (São Carlos, Brazil)	50 $\mu$ m aluminum oxide particles ( $Al_2O_3$ )
Artificial saliva	Artificial Saliva	Pharmacy School. Federal University of Goiás. Brazil. (Goiânia, Goiás)	Sorbitol 4.27%, saccharin 0.03%, Potassium chloride 0.062%, sodium chloride 0.096%, magnesium chloride 0.012%, calcium chloride 0.007%, monobasic potassium phosphate 0.27%, nipagine 0.2%, distilled water 1000QSP

Hence, an increase in bond strength of the reinforced IDS approach is expected.<sup>5,7,55</sup> In practice, there are countless numbers of simplifies adhesives on the market with different compositions and prices, which could benefit from a reinforced IDS approach.

The objective of this study was to evaluate the microtensile bond strength of unfilled/lightly filled DBAs (Scotchbond Multi-Purpose, Single Bond Plus, Clearfil SE Bond, and Scotchbond Universal) compared to the golden standard filled Optibond FL with and without IDS as well as with and without reinforcement with a flowable resin coating. The

null hypotheses of the study were that there are no differences between 1) the three different application modes (with and without IDS and with reinforced IDS), and 2) the five types of DBAs.

## 2 | MATERIAL AND METHODS

This experiment was approved by the Ethical Committee from Federal University of Goiás (CAAE: 96380418.6.0000.5083). Seventy-five

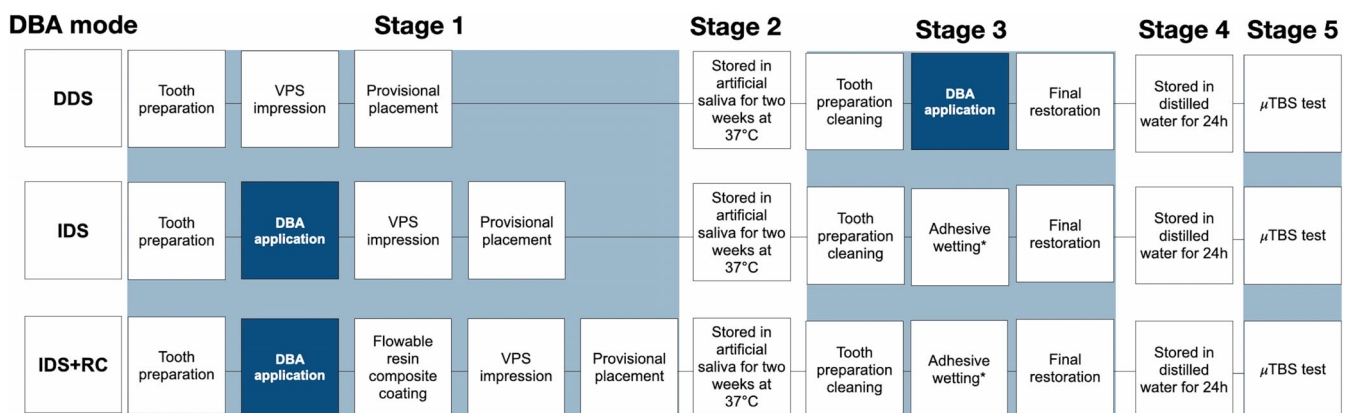
**TABLE 2** Technical procedures for each dentin bonding agent used in the study

Dentin bonding agent	Technical procedure
Optibond FL-OBFL	Etchant: 35% phosphoric acid application for 15 s, rinse for 20 s; gentle air-drying for 3 s (without desiccation). Primer: active application for 15 s; gentle air-drying for 5 s for solvent evaporation. Bond: active application for 15 s. No air-thinning. Light-polymerization for 15 s.
Scotchbond Multi-Purpose-SBMP	Etchant: 35% phosphoric acid application for 15 s, rinse for 20 s; gentle air-drying for 3 s (without desiccation). Primer: active application for 15 s; gentle air-drying for 5 s for solvent evaporation. Bond: active application for 15 s; gentle air drying for 5 s for air-thinning. Light-polymerization for 15 s.
Single Bond Plus-SBP	Etchant: 35% phosphoric acid application for 15 s, rinse for 20 s; water excess removal with cotton pellet. Single bottle: two times active application for 15 s each; gentle air-drying for 5 s for solvent evaporation and adhesive air-thinning. Light-polymerization for 15 s.
Clearfil SE Bond-CFSE	Primer: active application for 20 s; gentle air-drying for 5 s for solvent evaporation. Bond: active application for 20 s; gentle air-drying for 5 s for air-thinning. Light-polymerization for 15 s.
Scotchbond Universal-SBU	Single bottle: active application for 20 s; gentle air-drying for 5 s for solvent evaporation and adhesive air-thinning. Light-polymerization for 15 s.

recently extracted and caries free third molars from young patients were collected. The teeth were cleaned and stored in thymol solution 0.2%. The crowns were sectioned and prepared for microtensile bond testing according a method described elsewhere.<sup>60</sup> The specimens were randomly distributed into three main groups (25 teeth each): delayed dentin sealing (DDS), IDS, and immediate dentin sealing with additional flowable resin coating (IDS + RC). Within each group, five different DBAs were used ( $n = 5$ ): Optibond FL (OBFL), Scotchbond Multi-Purpose (SBMP), Single Bond Plus (SBP), Clearfil SE Bond (CFSE), and Scotchbond Universal (SBU), for a total of 15 groups. All the materials are given in Table 1. Each DBA was used according to the manufacturer's instructions and the exact sequence is presented in Table 2.

The tooth preparations in DDS specimens were not sealed but immediately submitted to impression with vinyl polydimethylsiloxane (VPS) (Express XT, 3 M ESPE, St Paul, MN), followed by provisional restoration placement (Revotek LC, GC, Tokyo, Japan) for 2 weeks and immersion in artificial saliva at 37°C. Following that period, the provisional restoration was removed, the preparation cleaned with 50  $\mu$ m aluminum oxide air-abrasion (5 s at 1.5 cm distance and 2 bar) and the DBA applied but not polymerized. Two increments (2 mm each) of resin composite (Filtek Z100, 3 M ESPE, St Paul, MN) were placed and polymerized for 30 s in the occlusal face and 15 s each side face, totalizing 90 s (1200 mW/cm<sup>2</sup>, Radii-Cal, SDI, Bayswater, Australia). Air-blocking barrier (K-Y Johnson & Johnson, New Brunswick, NJ) was added for 10 s of additional polymerization.

For IDS specimens, tooth preparations were subjected to dentin sealing (following the DBA manufacturers' instructions, Table 2) prior to impression. The polymerized adhesive surface was blocked with glycerin jelly and polymerized for an additional 10 s, rinsed and cleaned with pumice and water using an ultra-soft brush prior to VPS impressions. Unlike DDS specimens, provisionalization was preceded by isolation with petroleum jelly (Vaseline, Unilever, London, UK) to prevent adherences. The samples were stored for 2 weeks immersed in artificial saliva at 37°C.

**FIGURE 2** Study flowchart. μTBS, microtensile bond strength; DBA, dentin bonding agent; VPS, vinyl polydimethylsiloxane. \*wetting of preparation surface with adhesive resin of corresponding dentin bonding agent

**TABLE 3** Mean microtensile bond strength (in MPa) and standard deviation (DP) of all 15 experimental groups

		DBA				
		OBFL	SBMP	SBP	CFSE	SBU
DBA mode	DDS	13.31 ± 2.54 <sup>A,a</sup>	10.70 ± 3.45 <sup>A,a</sup>	12.72 ± 3.78 <sup>A,a</sup>	6.91 ± 2.25 <sup>A,a</sup>	7.19 ± 1.58 <sup>A,a</sup>
	IDS	54.75 ± 11.21 <sup>B,a</sup>	22.06 ± 5.34 <sup>B,b</sup>	16.68 ± 3.54 <sup>A,b</sup>	17.67 ± 5.45 <sup>A,b</sup>	15.26 ± 4.27 <sup>A,b</sup>
	IDS + RC	52.51 ± 5.85 <sup>B,a</sup>	35.65 ± 7.68 <sup>C,b</sup>	37.02 ± 5.29 <sup>B,b</sup>	45.64 ± 8.92 <sup>B,ab</sup>	35.05 ± 6.89 <sup>B,b</sup>

Note: Values in MPa with different letters indicate a statistically significant difference ( $p < 0.05$ ). Uppercase letters compare columns and differences among the three DBA modes of application. Lowercase letters correspond to rows and differences among five dentin bonding agents: OBFL, SBMP, SBP, CFSE, and SBU. Application modes: DDS; IDS; IDS + RC.

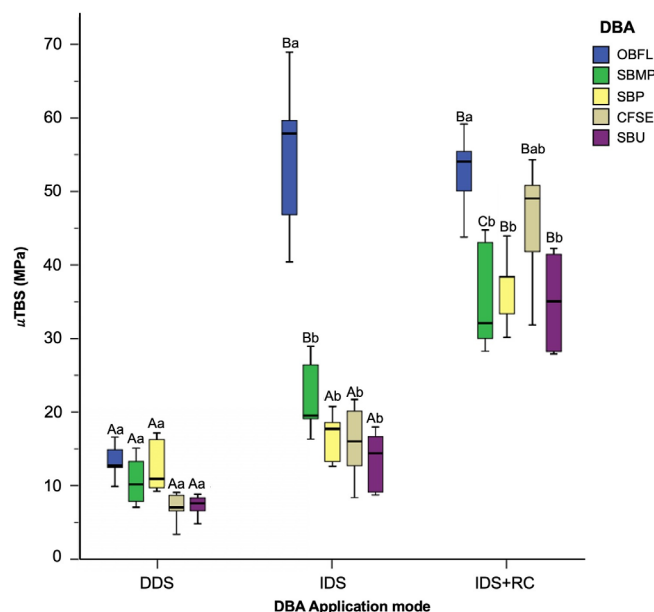
Abbreviations: CFSE, Clearfil SE Bond; DBA, dentin bonding agents; DDS, delayed dentin sealing; IDS + RC, immediate dentin sealing with flowable resin coating; IDS, immediate dentin sealing; OBFL, Optibond FL; SBMP, Scotchbond Multi-Purpose; SBP, Single Bond Plus; SBU, Scotchbond Universal.

IDS + RC groups were treated similarly to IDS ones except for the addition and polymerization of a layer of flowable resin composite (Filtek Bulk Fill Flow, 3 M ESPE, St. Paul, MN) over the polymerized DBA.

For IDS and IDS + RC, the provisional was removed after 2 weeks, the preparation was cleaned with 50  $\mu$ m aluminum oxide airborne-particle (5 s at 1.5 cm and 2 bar) and phosphoric acid (15 s, rinsed and dried) and covered with a layer of adhesive resin corresponding to the DBA. This adhesive layer was not polymerized, so that the final restoration can be fully seated. Restoration consisted of two increments (2 mm each) of resin composite (Filtek Z100, 3 M ESPE, St. Paul, MN) as previously described for DDS groups.

All specimens were stored in distilled water at room temperature for at least 24 h before the microtensile bond test. Dentin-resin beams were obtained according a method described elsewhere,<sup>60</sup> attached to the grips of a universal testing machine (5965 Universal Testing System, Instron, Norwood, MA) using a cyanoacrylate adhesive (Super Bonder, Loctite, Düsseldorf, Germany) and tested in tension at a crosshead speed of 0.5 mm/min until failure. Maximum tensile load (software BlueHill 2, version 2.23) expressed in units of stress (MPa) was obtained by division of the force (N) and specimen cross-sectional area, measured with a digital caliper (Absolute Origin, iGaggin, San Clemente, CA). Ten beams were randomly selected from each restored tooth, and the average value for each tooth was used in the calculations. After the test, the failure mode of each beam was determined under stereoscopic microscope (DSM 300, Kozo, Nanjin, China). Failure was classified as an adhesive failure if the fracture was located entirely between the adhesive layer and dentin (hybrid layer failure) or if the fracture continued from the adhesive into either the resin composite (adhesive interface failure) or flowable resin coating (resin coating interface failure), and as a cohesive failure if the fracture occurred exclusively within the resin composite or dentin. Cohesive failures were censored and not included in the calculation of bond strength or failure mode, as they do not account for the real adhesive properties (bond strength).

Bond strength data were obtained from the 15 experimental groups (mean microtensile bond strength testing from the 10 beams used as a single measurement, yielding 5 measurements per group). Shapiro–Wilk tests were used to test normal distribution of the data. The homogeneity of variance was assessed using the Levene test. As



**FIGURE 3** Box plot of microtensile bond strength ( $\mu$ TBS) in MPa. Microtensile bond strength values with different letters indicate a statistically significant difference ( $p < 0.05$ ). Uppercase letters compare differences among the three modes of application within same dentin bonding agent (DBA). Lowercase letters correspond to differences among five DBAs within same mode of application. DBAs: Optibond FL (OBFL), Scotchbond Multi-Purpose (SBMP), Single Bond Plus (SBP), Clearfil SE Bond (CFSE) and Scotchbond Universal (SBU). Application modes: delayed dentin sealing (DDS); immediate dentin sealing (IDS); immediate dentin sealing with flowable resin coating (IDS + RC)

the data were normally distributed, two-way analysis of variance (DBA and application mode) were applied to analyze possible differences between the groups using a statistical software program ( $\alpha = 0.05$ ) (SPSS statistics 23, IBM, Armonk, NY). Tukey's post hoc test was applied to compare the differences between groups where the microtensile bond strength was the dependent variable while DBA application mode (3 levels) and DBA itself (5 levels) were independent variables.

Both dentin and resin sides of typical fractured beams from each group were air dried, sputter coated with gold/palladium, and

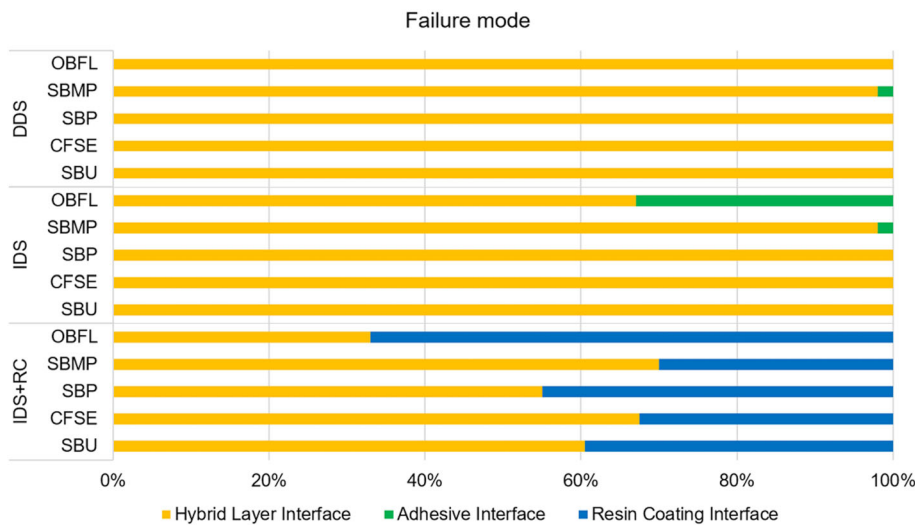


examined using a scanning electron microscopy (SEM) in different magnifications. One additional sample of each DBA was selected to have half of its surface resin coated after IDS. Sealed surfaces were cleaned with pumice, air-abraded and etched as described prior to final restoration, and examined using SEM in order to observe the effect of predelivery cleaning on filled and unfilled/lightly filled DBA and flowable resin composite (resin coating). The flowchart of the study is presented in Figure 2.

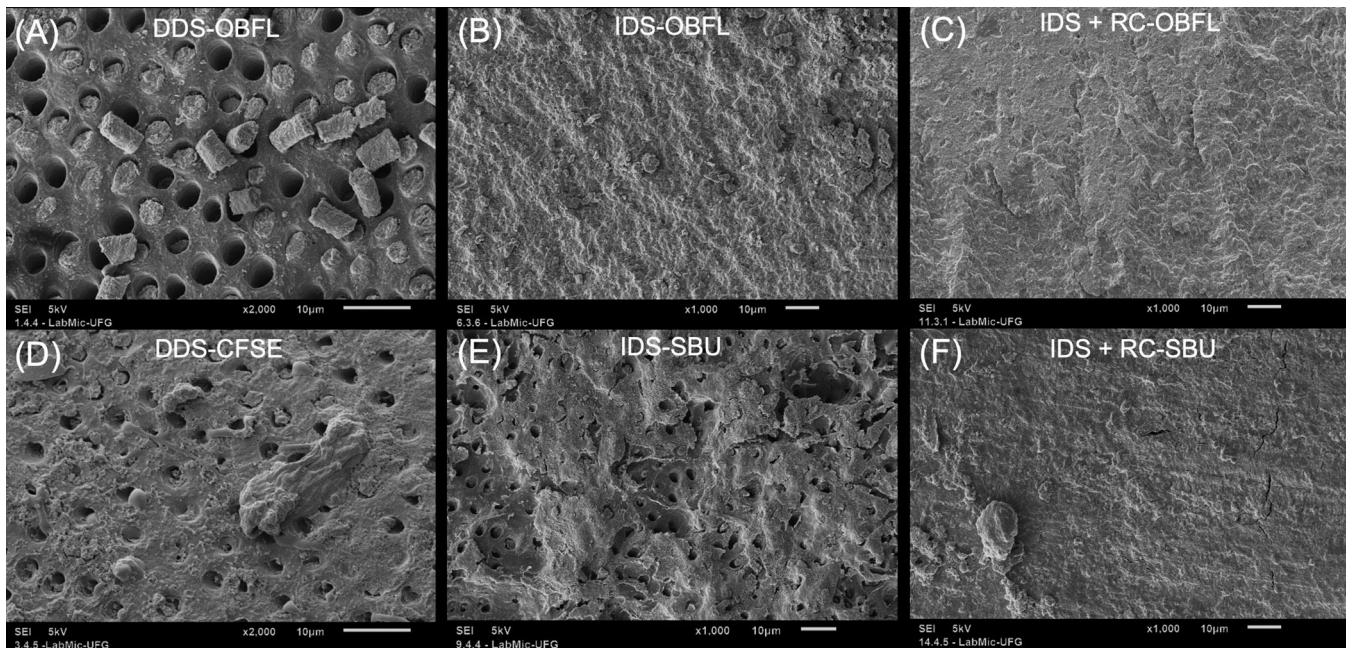
### 3 | RESULTS

#### 3.1 | Microtensile bond strength test

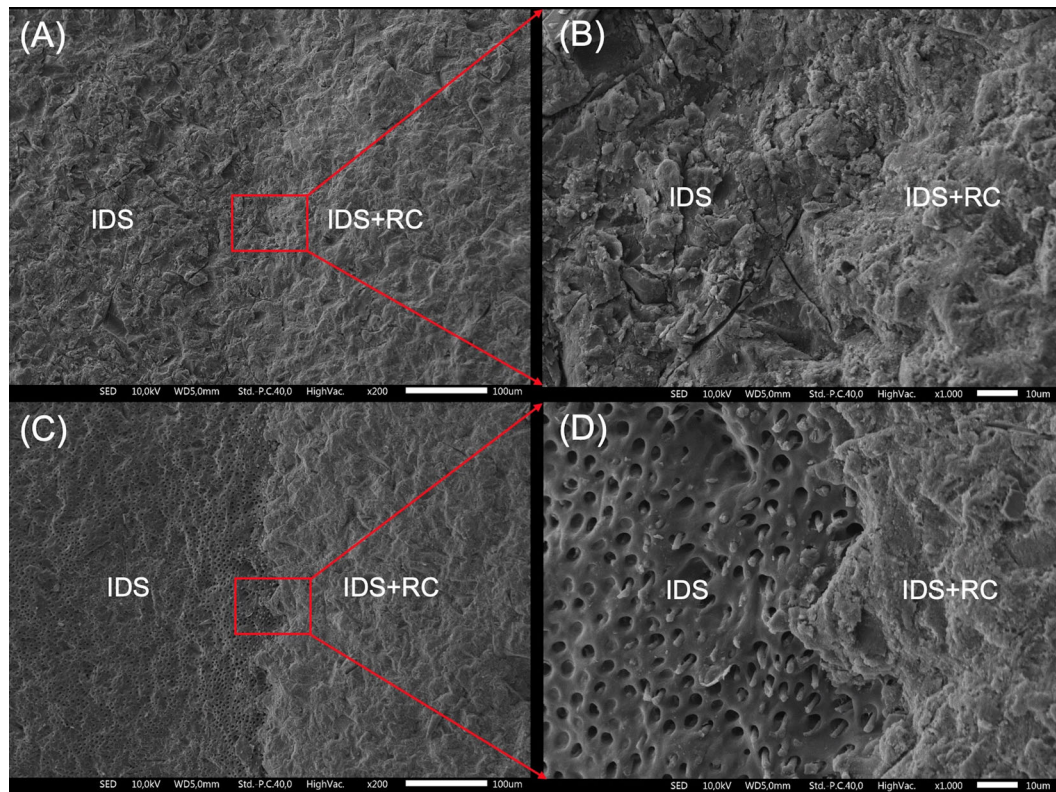
The  $\mu$ TBSs are shown in Table 3 and Figure 3. Two-way ANOVA revealed that bond strength was influenced by the DBA application mode ( $F = 178,893$ ,  $p < 0.0001$ ) and DBA itself ( $F = 31,123$ ,  $p < 0.0001$ ), and the interaction between application mode and DBA



**FIGURE 4** Failure mode. Interfacial failure between dentin and adhesive: hybrid layer interface. Interfacial failure between adhesive and composite: adhesive interface. Interfacial failure between flowable resin coating and composite: resin coating interface. Dentin bonding agents: Optibond FL (OBFL), Scotchbond Multi-Purpose (SBMP), Single Bond Plus (SBP), Clearfil SE Bond (CFSE) and Scotchbond Universal (SBU). Application modes: delayed dentin sealing (DDS); immediate dentin sealing (IDS); immediate dentin sealing with resin coating (IDS + RC)



**FIGURE 5** Scanning electron microscopy of beams after failure. (A) DDS-OBFL with failure at hybrid layer (hybrid layer interface), same as DDS-CFSE (D). (B) IDS-OBFL presenting failure between adhesive and resin composite (adhesive interface). (C) IDS + RC-OBFL presenting failure between flowable resin coating and resin composite (resin coating interface), same as IDS + RC-SBU (F). (E) IDS-SBU with hybrid layer interface failure. CFSE, Clearfil SE Bond; DDS, delayed dentin sealing; IDS + RC, immediate dentin sealing with resin coating; IDS, immediate dentin sealing; OBFL, Optibond FL; SBU, Scotchbond Universal



**FIGURE 6** Scanning electron microscopy of IDS and IDS + RC after predelivery cleaning. Optibond FL in IDS mode on the left half and in IDS + RC mode on the right half at 200 times (A) and 1000 times (B). The hybrid layer is preserved either on IDS and IDS + RC modes. Single Bond Universal in IDS mode on the left half and in IDS + RC mode on the right half at 200 times (C) and 1000 times (D). The hybrid layer was partially removed during predelivery cleaning, exposing dentin tubules, and the same pattern was observed in all unfilled/lightly filled DBAs (SBMP, SBP, CFSE, and SBU). CFSE, Clearfil SE Bond; DBA, dentin bonding agents; IDS + RC, immediate dentin sealing with resin coating; IDS, immediate dentin sealing; SBMP, Scotchbond Multi-Purpose; SBP, Single Bond Plus; SBU, Scotchbond Universal

( $F = 10,129$ ,  $p < 0.0001$ ). IDS increased the bond strength for OBFL from 13 to 55 MPa ( $p < 0.001$ ) and 11 to 22 for SBMP ( $p < 0.05$ ) when compared to the DDS application mode. For all other DBAs the increase in IDS was not statistically significant in comparison to DDS. OBFL outperformed all other DBAs in IDS application mode.

The reinforced IDS (IDS + RC) increased the bond strength of all unfilled/lightly filled DBA (SBMP, SBP, CFSE, and SBU) when compared either to DDS or IDS application modes. For the filled DBA (OBFL) the flowable resin coating did not increase the bond strength. There was no statistical difference between OBFL and CFSE on the IDS + RC application mode, although OBFL outperformed SBMP, SBP, and SBU.

### 3.2 | Failure mode

The failure modes are shown in Figure 4. SEM pictures of typical fracture modes are shown in Figure 5. For DDS and IDS groups, failure was mainly at the level of the hybrid layer (dentin/adhesive interface), and was associated with lower microtensile bond strength for all DBAs. In the IDS + RC groups, a significant amount of the failures were found at the flowable resin coating/resin composite interface.

Additional specimens for each DBA in IDS and IDS + RC modes were submitted to SEM analysis, to observe the effect of predelivery surface cleaning on the integrity of the hybrid layer (Figure 6). For the IDS mode, the hybrid layer of highly filled DBA (OBFL) in the IDS mode was maintained, and dentin was still sealed. The hybrid layer of unfilled/lightly filled DBAs (SBMP, SBP, CFSE, and SBU) was partially removed by predelivery cleaning with pumice and soft brush, air-abrasion and etching. The damage of the hybrid layer explains the poor microtensile bond strength results of unfilled/lightly filled DBAs. For the IDS + RC mode, the hybrid layer was not damaged regardless of the DBA type.

## 4 | DISCUSSION

The microtensile bond strength of unfilled/lightly filled DBAs (Scotchbond Multi-Purpose, Single Bond Plus, Clearfil SE Bond, and Scotchbond Universal) was compared to the golden standard filled Optibond FL with and without IDS as well as with and without the reinforcement with a flowable resin composite coating. The first null hypothesis of the study was rejected in part because significant differences were noted among the three different application modes (with

and without IDS and with reinforced IDS), even though there was not difference between DDS and IDS for the simplified DBAs. The second null hypothesis was also rejected in part because of the numerous differences among the five types of DBAs. The present data suggest the different DBAs and the IDS technique will not only yield different bond strengths but will also likely be improved when adding a thin layer of flowable resin composite over the hybrid layer. The IDS + RC technique, so called "reinforced IDS", substantially increased the bond strength of all adhesives compared to the IDS technique except for OBFL, and there was statistical difference between the DBAs for each mode, except for DDS.

There is significant evidence showing the use of additional flowable resin coating on the performance of DBAs, however, this work is the first one to consider adhesives from four different generations combined with three application modes in the same study. The research protocol also included artificial saliva as a contaminating factor during storage of the specimens, as well as impression, temporary restoration, and surface conditioning steps prior to final restoration. Existing studies found no improvement on bond strength when using IDS, maybe due to the fact the DDS workflow in those studies did not simulate either impression, temporization, or storage.<sup>36,52-54</sup>

Minimally invasive dentistry and intact dental tissue conservation through adhesion is the cornerstone of the biomimetic approach in restorative dentistry.<sup>61,62</sup> However, studies show that the interface between dentin and adhesive material is the one with the greatest fragility in the restoration.<sup>63</sup> The model for resin-dentin adhesion should be the simulation of the dentinoenamel junction (DEJ). The human microtensile biologic strength of the DEJ was estimated at 51 MPa.<sup>64</sup> This is the minimum reference that should be met with resin bonding systems. It can be argued that even higher bond strength should be obtained to offset the unavoidable degradation of the hybrid layer over time.<sup>65</sup> In view of these considerations, OBFL is the only system that has proven to not only exceed the bond strength of the DEJ but also the one displaying the highest stability over time.<sup>44,60,66-68</sup> This study adds a trophy to that list by demonstrating that this product is also the most appropriate for IDS, as originally stated when proposing this nomenclature.<sup>37</sup> The most prominent finding is the fact that OBFL provided the best performance without requiring an additional flowable resin coating. This is because its elastic modulus is close to that of a flowable resin composite with 48% wt radiopaque filler (silica particles and barium glasses). The SEM image (Figure 6(B)) clearly show the stable and resistant adhesive surface with OBFL even after storage time, provisional restorations and cleaning of the surface.

The benefits of IDS, however, have been questioned because of some studies, which did not demonstrate its superiority over the traditional approach.<sup>36,52,53</sup> It appears from the results of the present study that IDS only increased the bond strength of the etch-and-rinse 3-steps DBAs (OBFL and SBMP), whereas the filled DBA (OBFL) outperformed the unfilled/lightly filled DBA (SBMP). The other systems did not show statistical difference between DDS and IDS application modes. This is explained in Figure 6(C,D) as the predelivery surface cleaning removed the thin IDS layer and re-exposed the dentin. In fact, it is generally

recommended to avoid thick layers of unfilled/lightly filled adhesive resins that could affect the cohesive strength of the interface.<sup>69-72</sup> For this reason, those unfilled/lightly filled DBAs are usually air-thinned, which was done even for the IDS groups in the present study. In a similar study,<sup>66</sup> CFSE was not air-thinned for IDS and resulted in a significant improvement of bond strength compared to DDS. Stavridakis et al.<sup>43</sup> demonstrated that the adhesive layer thickness also depends on geometry and that the filled adhesive provides a more uniform thicknesses (70–180 microns) compared to unfilled/lightly filled ones that tend to pool in concavities (>300 microns) and present reduced thicknesses in convexities (<30 microns). Those thin areas will likely be abraded away during predelivery cleaning of the preparation. The present study demonstrates that the reinforced IDS mode of application (IDS + RC) allows for most DBAs to have the same higher potential as OBFL. A similar result could be observed in another study,<sup>6</sup> where it is concluded that the association of flowable resin composite (resin coating) to the CFSE adhesive system significantly increased the bond strength. The present study extends this conclusion to the most recent multimode universal adhesives. The fracture analysis in Figure 4 demonstrates that failures that originally occurred at the hybrid layer interface in the IDS groups started to happen between the flowable resin coating and the final restoration in the IDS + RC groups, hence showing that the seal of the tubular region is potentially maintained. Self-etching systems have the advantage of being less technique-sensitive<sup>44,63,73,74</sup> and two-bottle systems seems to be reliable and have excellent clinical performance.<sup>63</sup> However, based on this study, they should be applied in conjunction with an additional flowable resin coating when using IDS. This may not be ideal when dealing with thin restorations such as anterior or occlusal ceramic veneers where additional restorative clearance would need to be prepared to make space for the flowable liner. In this specific instances, OBFL represents the best choice because of its ideal combination of thickness (ca. 80  $\mu$ m) and viscosity.<sup>1</sup> In addition, there are no known interactions between oxygen-inhibited OBFL and VPS materials, however, inherently thin unfilled/lightly filled adhesives show significant inhibition effects or adherences on both VPS and polyethers.<sup>41,42</sup> Hence a final benefit of flowable resin coating is the prevention of significant interactions between thin adhesives and impression materials.<sup>20,75</sup>

Several predelivery cleaning methods have been previously analyzed in the literature to establish a reliable resin-to-resin bond at the surface of the IDS layer.<sup>37,53,76</sup> In the present study, the sealed surfaces were cleaned with pumice and a soft brush, then air-abraded with 50  $\mu$ m aluminum oxide followed by phosphoric acid etching for degreasing.<sup>77,78</sup> Wetting the preparation with a layer of adhesive resin (without polymerization) is recommended when using highly filled luting materials (such as preheated restoratives) while flowable luting agents can be applied directly to the preparation.<sup>79,80</sup>

The findings of the present study open the doors for the use of multiple DBAs for IDS, providing the clinicians with more choices in addition to providing access to a technique that would be otherwise difficult to apply if limited to a single DBA. Further clinical trials will allow to take into account pulpal pressure, aging, bacterial contamination, biocorrosion, which will certainly affect the relevance of the present in vitro results.



## 5 | CONCLUSION

The conclusion of this study was:

- IDS improved the microtensile bond strength ( $\mu$ TBS) to dentin when a filled DBA was used and when using SBMP;
- IDS did not increase the  $\mu$ TBS when unfilled/lightly filled DBAs were used except when using SBMP;
- Unfilled/lightly filled DBAs should be reinforced with a flowable resin coating to improve the  $\mu$ TBS to dentin for IDS.

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## DISCLOSURE

The authors do not have any financial interest in the companies whose materials are included in this article.

## DATA AVAILABILITY STATEMENT

Data available on request from the authors.

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