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# Effect of Immediate Dentin Sealing and Surface Conditioning on the Microtensile Bond Strength of Resin-Based Composite to Dentin

CRG van den Breemer • M Özcan • MS Cune AP Almeida Ayres • B Van Meerbeek • MMM Gresnigt

### **Clinical Relevance**

For partial indirect restorations, immediate dentin sealing is recommended, as bond strength remains stable over time.

## SUMMARY

This study evaluated the microtensile bond strength ( $\mu$ TBS) of resin-based composite (RBC) to dentin after different immediate dentin sealing (IDS) strategies and surfaceconditioning (SC) methods and on two water storage times. Human molars (n=48) were randomly divided into eight experimental groups involving four different IDS strategies—IDS-1L with one layer of adhesive, IDS-2L with two layers of adhesive, IDS-F with one layer of adhesive and one layer of flowable RBC, and DDS (delayed dentin sealing) with no layer of adhesive (control)—and two different SC methods—SC-P with pumice rubbing and SC-PC with pumice rubbing followed by tribochemical silica coating. The  $\mu$ TBS test was

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performed after one week and after six months of water storage, being recorded as the "immediate" and "aged" µTBS, respectively. **Composite-adhesive-dentin** microspecimens (0.9×0.9×8–9 mm) were stressed in tension until failure to determine the µTBS. Failure mode and location of failure were categorized. Twoway analysis of variance was applied to analyze the data for statistically significant differences between the experimental groups (p < 0.05). Two-way analysis of variance revealed no significant differences between the one-week µTBS specimens for IDS strategy (p=0.087) and SC methods (p=0.806). However, the interaction of IDS strategy and SC methods appeared statistically significant (p=0.016). The six-month specimen evaluation showed no significant difference in µTBS for SC (p=0.297) and SC/IDS interaction (p=0.055), but the *µTBS* of the IDS strategies differed significantly among them (p=0.003). For tribochemical silica-coated IDS, no significant effect of aging on  $\mu$ TBS was recorded (p=0.465), but there was a highly significant difference in  $\mu$ TBS depending on the IDS strategy (p < 0.001). In addition, the interaction of IDS and aging was borderline statistically significant (p=0.045). The specimens failed mainly at the adhesive-dentin interface for all experimental groups. Dentin exposure during clinical procedures for indirect restorations benefits from the application of IDS, which was shown to result in higher bond strength. No significant differences were found between cleaning with solely pumice or pumice followed by tribochemical silica coating.

#### INTRODUCTION

In restorative dentistry, one of the primary goals is to preserve tooth tissue. Removing large amounts of dental structure adversely affects the pulp and may lead to pulp damage.<sup>6,20</sup> When it comes to restoring posterior teeth with large cavities, partial indirect restorations in glass-ceramic or feldspathic porcelain may be indicated. The literature reveals that such restorations have a survival rate of 91% in 10 years.<sup>24</sup> The cause of failure involves fractures (4%), endodontic complications (3%), secondary caries (1%), and debonding (1%).<sup>24</sup> Fractures and debonding are often seen in cases where restorations are bonded to dentin.<sup>13</sup> Creating a strong bond to dentin that is durable over time is more challenging than creating a bond to enamel because of dentin's intrinsic hydrophilic nature.<sup>3</sup> An inadequate seal of dentin by the adhesive may cause postoperative sensitivity, marginal staining, and recurrent caries. Hence, the survival and success of (partial) indirect restorations is often related to the remaining quantity and quality of enamel.<sup>15</sup>

In order to improve bond strength to dentin, Pashley and others introduced the so-called dual bonding technique, which consists of the application of two layers of adhesive resin onto dentin.<sup>26</sup> Applying an adhesive layer directly after crown preparation protects the pulp from bacterial invasion, reduces postoperative sensitivity, and increases bond strength. Other studies revealed that multiple adhesive layers can further improve bond quality and strength.<sup>8,17,19,27</sup> The purpose of sealing dentin directly after preparation is to avoid surface contamination during the temporary phase and to protect dentin by hybridization, thus avoiding sensitivity and preventing water uptake. This requires that the adhesive be light cured immediately, which is commonly not recommended at the time of cementation to avoid restoration fit problems.<sup>32</sup>

In 2005, this concept evolved to immediate dentin sealing (IDS). Prior to luting in the second visit, one commonly recommends decontaminating the IDS by tribochemical silica coating.<sup>21,22</sup> This not only microroughens the surface, thereby improving micromechanical interlocking, but also cleans the surface and enables chemical copolymerization of the resinbased cement with the IDS.<sup>1,33,37</sup> Falkensammer and others<sup>14</sup> concluded that polishing and airborne particle abrasion with silica-coated alumina (Al<sub>2</sub>O<sub>3</sub>) and glycine are equally efficient methods of conditioning IDS surfaces. Other studies showed that soft air abrasion,<sup>34</sup> airborne particle abrasion with Al<sub>2</sub>O<sub>3</sub>,<sup>11,22,23</sup> or fluoride-free pumice paste systems<sup>5,12,21</sup> resulted in the highest bond strength. However, it is unknown which method is most suitable for conditioning IDS prior to cementation.

Results from a recent systematic review indicated that the effect of IDS on bond strength is tested mainly by using a microtensile bond strength ( $\mu$ TBS) approach.<sup>38</sup> The  $\mu$ TBS test is generally accepted as one of the most valid bond-strength tests because it is performed perpendicular to the adhesive interface.<sup>10,35</sup> Using a  $\mu$ TBS test, a more favorable stress distribution is achieved, resulting from the small specimen size.

The objective of this study was to compare different IDS applications and surface conditioning

Table 1: Brand, Manufact Order)	turer, Main Chemical Composition, and Batch Number of the Different Products Us	ed (in Alphabetical	
Product (Manufacturer)	Composition	Batch Number	
CoJet sand (3M, Seefeld, Germany)	Aluminum oxide (Al_2O_3) particles coated with silica (particle size: 30 $\mu\text{m})$	446317/534151	
Durelon (3M)	Powder: zinc oxide, stannous fluoride, tin dioxide	514362	
	Liquid: water, polyacrylic acid	510198	
Enamel plus HFO UD2 (Micerium, Avegno, Italy)	1,4-Butandioldimethacrylate, urethane dimethacrylate, Bis-GMA	2015007203	
ESPE-Sil (3M)	Ethyl alcohol, 3-methacryloxypropyl- trimethoxysilane, methyl ethyl ketone	598868	
Glycerin Gel K-Y, (Johnson & Johnson, Sezanne, France)	Purified water, glycerin, methylparaben, propylparaben, propylene glycol, hydroxyethylcellulose, dissodium phosphate, sodium phosphate, tetrasodium EDTA	2744V	
Grand IO Flow (VOCO, Cuxhaven, Germany)	1,6-Hexanediylbismethacrylate, BIS GMA, triethylene glycol dimethacrylate	1512472	
Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein)	Ethanol, 3-trimethoxysilsylpropyl-methacrylate, methacrylated phosphoric acid ester	T07775	
Optibond FL (Kerr,	Primer: HEMA, GPDM, PAMM, ethanol, water, photoinitiator	5534310	
Orange, CA, USA)	Adhesive: TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, photoinitiator	5594053	
Total Etch (Ivoclar Vivadent)	37% phosphoric acid (H <sub>3</sub> PO <sub>4</sub> )	L049	
Abbreviations: Bis-GMA, bispheno acid dimethacrylate; PAMM, phtha	ol A-glycidyl methacrylate; EDTA, Ethylenediaminetetraacetic acid; HEMA, Hydroxyethylmethacrylate; GF Ilic acid monomethacrylate;TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate	<sup>o</sup> DM, glycerophosphoric	

(SC) methods by determining the bond strength to dentin with and without water storage aging. The null hypotheses tested were that (1) there is no significant difference in  $\mu$ TBS among the four IDS strategies investigated, (2) among the two SC methods, and (3) for the "immediate" (one-week) and "aged" (six-month)  $\mu$ TBS.

## **METHODS AND MATERIALS**

## **Specimen Preparation**

Human molars (n=48) were collected, stored in distilled water, and used at maximum one month postextraction. Each specimen was embedded in gypsum to facilitate handling. The occlusal coronal third of the crown was removed with a diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), thereby exposing a flat midcoronal dentin surface. The dentin surfaces were verified for the absence of enamel and/or pulp tissue exposure using a stereomicroscope (Wild M5A, Wild, Heerbrugg, Switzerland).

# **Study Design**

The flattened specimens were randomly divided into a total of eight experimental groups involving four different IDS strategies—IDS-1L with one layer of adhesive, IDS-2L with two layers of adhesive, IDS-F with one layer of adhesive and one layer of flowable resin-based composite (RBC), and DDS (delayed dentin sealing) with no layer of adhesive (control)— and two different SC methods—SC-P with pumice rubbing and SC-PC with pumice rubbing followed by tribochemical silica coating. The  $\mu$ TBS test was performed after one week and after six months of water storage, being recorded as the "immediate" and "aged"  $\mu$ TBS, respectively.

# IDS

The brand, manufacturer, main chemical composition, and batch number are detailed in Table 1 for the different products used in this study.

Regarding IDS-1L, the flat dentin was etched for 15 seconds with 37% phosphoric acid (Total Etch, Ivoclar Vivadent, Schaan, Liechtenstein) and rinsed thoroughly with a water-air spray for 15 seconds. The surface was air-dried but not desiccated for 3 seconds, after which a primer (Optibond FL Primer, Kerr, Orange, CA, USA) was applied with a light brushing motion for 15 seconds and gently air-dried for 10 seconds and suction dried for 15 seconds. A thin layer of heated (40°C) adhesive resin (Optibond FL Adhesive, Kerr) was applied onto the surface using a light brushing motion for 15 seconds and photopolymerized for 10 seconds (Bluephase Style, Ivoclar Vivadent) (>1000 mW/cm<sup>2</sup>). Regarding IDS-2L, the same procedures were performed with the addition that a second layer of adhesive was likewise applied as described above for the first adhesive layer. Regarding IDS-F, the same procedure was performed as for IDS-1L with the addition that a flowable RBC (Grand IO Flow, VOCO, Cuxhaven, Germany) was additionally applied in a thin layer of about an average thickness of 1 mm and separately light cured for 40 seconds. Finally, in all groups, glycerin gel (K-Y, Johnson & Johnson, Sezanne, France) was applied, and light curing was repeated for 40 seconds.

The DDS was considered the control; the dentin was not sealed directly after preparation.

# **Temporary Restoration**

After dentin preparation, a temporary restoration (Protemp 4, 3M, Seefeld, Germany) was cemented onto the flat dentin surface using a zinc-carboxylate cement (Durelon, 3M). The temporary phase consisted of three weeks of water storage.

## Surface Conditioning and Composite Buildup

After three weeks, the temporary restorations were removed with a scaler (H5 Anterior Scaler, Hu-Friedy, Chicago, IL, USA). Next, the surfaces of half of the specimens for each of the four IDS groups were cleaned solely by pumice rubbing (SC-P), while those of the other half of the specimens were cleaned by pumice rubbing and additionally tribochemically silica coated (SC-PS) at a distance of 10 mm following a 45° angulation (2 bar; CoJet sand,  $SiO_2$ , 3M). All specimens were subsequently rinsed thoroughly with water and air-dried for 15 seconds, after which a silane coupling agent (ESPE-SIL, 3M) was applied and left to dry (five minutes) according to the method of Ozcan and others.<sup>25</sup> The primer (Optibond FL Primer, Kerr) was then applied onto all specimen surfaces with a light brushing motion for 15 seconds, gently air-dried for 10 seconds, and then suction dried for 15 seconds. A thin layer of heated (40°C) adhesive resin (Optibond FL Adhesive, Kerr) was next applied onto the surface with a light brushing motion for 15 seconds and air-dried for 10 seconds without light curing. An RBC buildup ( $\pm 6$  mm) was subsequently constructed (HFO composite, Micerium, Avegno, Italy) on the prepared surfaces by filling a transparent silicon mold in three to four layers. Caution was taken to avoid air bubbles between the adhesive and RBC layers. The buildup was photopolymerized through the silicon mold using the LED light-curing unit from above and after the last layer it was lightcured for 40 seconds from each of the four sides and from the top; this was repeated after having removed the mold and having applied Glycerin Gel (Johnson & Johnson) at the RBC surface outside.

# Aging and µTBS Testing

After one week of storage in 0.5% chloramine T solution at 37°C, microspecimens were cut for µTBS testing. Per experimental group, 24 microspecimens were tested immediately to measure the one-week µTBS, while another set of 24 microspecimens were tested after six months of storage to measure the sixmonth aged µTBS. The bond strength to dentin was determined using a standardized µTBS protocol.<sup>31</sup> In order to obtain 12 rectangular microspecimen sticks per experimental group  $(0.9 \times 0.9 \times 8-9 \text{ mm})$ , the restored teeth were sectioned perpendicular to the interface using an automated water-cooled precision diamond saw (Accutom-50, Struers, Ballerup, Denmark).<sup>30,40</sup> The dimensions of the sticks were precisely measured by means of a digital caliper (CD-15CPX, Mitutoyo, Kanagawa, Japan) from which the cross-sectional area was calculated ( $\sim 0.9$  $mm^2$ ). The microspecimens were fixed to a modified µTBS testing jig<sup>31</sup> using cyanocrylate glue (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan) and tested in tension mode at a crosshead speed of 1.0 mm/min using an LRX testing machine (Llovd, Hampshire, UK) equipped with a load cell of 100 N. The bond strength values were calculated in MPa by dividing the imposed force (in N) at the time of fracture by the bonded area (in mm<sup>2</sup>). Specimens that failed before actual testing (pretesting failure) were explicitly noted, counted as 0 MPa in further analyses, and thus taken into account for the calculation of the  $\mu$ TBS means.

# **Failure Pattern Analysis**

The failure modes were evaluated using a stereomicroscope (Wild M5A, Wild) at a magnification of up to  $50\times$  and classified as follows: failure in dentin, failure at the adhesive-dentin interface, failure in the adhesive resin, failure at the adhesive-composite interface, and failure in the composite. Representative specimens in each group were selected for further ultrastructural characterization using scanning electron microscopy (SEM). The latter specimens were sputter coated using a 3-nm-thick layer of gold (80%) and palladium (20%) (90 seconds, 45 mA; Balzers SCD 030, Balzers, Liechtenstein) prior to

Table 2: Mear (SC)	e 2: Mean Microtensile Bond Strength (MPa) for the Different Immediate Dentin Sealing (IDS) and Surface-Conditioning (SC) Strategies at One Week and After Six Months of Aging <sup>a</sup>								
	IDS-1L	IDS-2L	IDS-F	DDS					
1 wk									
SC-P	29.8 (13.3) aA	26.4 (11.2) aA	29.1 (13.7) aA	30.2 (17.1) aA					
SC-PS	35.2 (19.0) aA	39.2 (15.2) aB	28.1 (13.6) aA	16.0 (13.5) bB					
6 mo									
SC-P	29.5 (11.6) aA	37.1 (13.9) aA	27.9 (14.3) aA	21.1 (17.0) bA					
SC-PS	35.6 (7.3) aA	29.3 (13.7) aA	40.6 (14.9) aA	21.4 (10.3) bA					
<sup>a</sup> Mean (standard de	viation); same lowercase letters indicate	e absence of statistically significant diffe	erence in the rows, and same uppercas	e letters indicate absence of					

<sup>a</sup> Mean (standard deviation); same lowercase letters indicate absence of statistically significant difference in the rows, and same uppercase letters indicate absence of statistically significant difference in the columns (p<.05); IDS-1L, IDS with one layer of adhesive; IDS-2L, IDS with two layers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC; DDS, delayed dentin sealing (control); SC-P, surface conditioning with pumice; SC-PS, SC followed by tribochemical silica coating.

being examined using a cold field-emission SEM (LEO 440, Leo Electron Microscopy, Cambridge, UK).

#### **Statistical Analysis**

All data were analyzed using a statistical software package (SPSS 22, PASW statistics 18.0.3, Quarry Bay, Hong Kong, China). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test for a normal distribution of the data. As the data were normally distributed, the data were divided in two experiments according to aging (one week, six months) and surface conditioning (SC-P, SC-PS). Two-way analysis of variance and *post hoc* testing were applied to verify possible differences among the groups for the parameters of IDS, SC, and aging on  $\mu$ TBS. In all tests, p < 0.05 was considered to be statistically significant.

#### RESULTS

The IDS strategy (F[1, 217.3]=2.265, p=0.087,  $\eta^2=0.072$ ) and type of surface conditioning (SC) (F[1, 217.3]=0.061, p=0.806,  $\eta^2=0.001$ ) did not produce statistical difference after one week of water aging (Table 2). However, the interaction of IDS and



SC strategy was statistically significant (F[3, 217.3]=3.649, p=0.016,  $\eta^2$ =0.111). Hence, the magnitude of the difference between the two SC methods after one week of water aging depends on the IDS strategy that was applied. Silica-coated (SC-PS) specimens from IDS-2L showed the highest mean one-week µTBS. The mean µTBS was significantly higher (Figure 1) when silica coating (SC-PS) was used for this particular IDS strategy (F[1, 217.3]=4.556, p=0.036,  $\eta^2$ =0.049). In contrast, following a DDS strategy, SC-PS achieved significantly lower µTBS than SC-P (F[1, 217.3]=5.630, p=0.020,  $\eta^2$ =0.060).

After six months of water aging, SC did not significantly affect µTBS (*F*[1, 173.6]=1.099, p=0.297,  $\eta^2=0.012$ ), while the IDS strategy significantly influenced µTBS (*F*[3, 173.6]=5.110, p=0.003,  $\eta^2=0.148$ ). The interaction of IDS and SC was not significant (*F*[3, 173.6]=2.631, p=0.055,  $\eta^2=0.082$ ). Post hoc tests revealed that regardless of the type of SC, all IDS strategies differed significantly (p=0.001 to p=0.004) from the DDS group but not from each other (Table 2; Figure 2).

Table 2 shows that the aging time (F[1, 200.3]=0.000, p=0.997,  $\eta^2=0.000$ ) and IDS strategy

Figure 1. Box plot of microtensile bond strength (MPa) (left) and estimated marginal means of microtensile bond strength (MPa) (right) for the different immediate dentin sealing (IDS) and surface-conditioning strategies at one week. IDS-1L, IDS with one layer of adhesive; IDS-2L, IDS with two layers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC; DDS, delayed dentin sealing (control); SC-P, surface conditioning with pumice; SC-PS, SC followed by tribochemical silica coating.



(*F*[3, 200.3]=0.765, p=0.517,  $\eta^2$ =0.025) did not produce statistical difference for the SC-P groups. There was no interaction of these factors as well (*F*[3, 200.3]= 1.989, p=0.121,  $\eta^2$ =0.064). However, in general, the SC-P groups demonstrated stable µTBS means in time without significant differences among groups (Figure 3).

For the SC-PS specimens, the aging factor produced no significant difference (F[1, 190.6]=0.538,p=0.465,  $\eta^2=0.006$ ). However, there was a highly significant difference in mean µTBS among the IDS strategies (F[3, 190.6]=8.097, p < 0.001,  $\eta^2 = 0.216$ ). DDS surfaces conditioned with silica (SC-PS) exhibited the significantly lowest  $\mu$ TBS (p < 0.001 for all pairwise comparisons). The aging/IDS strategy interaction was also statistically significant (F[3,190.6]=2.801, p=0.045,  $\eta^2=0.087$ ). Hence, for SC-PS specimens, the magnitude of the difference between the two aging evaluations relied on the strategy of IDS employed. Interestingly, the former was caused mainly by a positive effect of aging on the µTBS means of silica-coated (SC-PS) specimens from the IDS-F group (F[1, 190.6] = 4.886, p = 0.020, $\eta^2 = 0.053$ ) (Figure 4), which ultimately led to the highest µTBS reported (Table 2; 40.6±14.9 MPa).



Figure 2. Box plot of microtensile bond strength (MPa) (left) and estimated marginal means of microtensile bond strength (MPa) (right) for the different immediate dentin sealing (IDS) and surface-conditioning strategies after six months of aging. IDS-11, IDS with one layer of adhesive; IDS-2L, IDS with two layers of adhesive; IDS-F, IDS with two layers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC; DDS, delayed dentin sealing (control); SC-P, surface conditioning with pumice; SC-PS, SC followed by tribochemical silica coating.

Failure was observed mainly at the adhesivedentin interface for all the evaluated groups. The fewest failures were seen at the adhesive-composite interface and within dentin or composite (Figure 5). The majority of pretesting failures were related to the DDS groups (Table 3).

## DISCUSSION

Glass-ceramic posterior restorations have a good survival rate; however, their prognosis is highly dependent on the strength of the adhesive interface. The weakest link of the interface is the connection of the adhesive to dentin.<sup>10,13,15</sup> In order to increase the adhesive strength of resin-based materials to dentin in indirect restorations, the concept of IDS was introduced. It was shown in an *in vitro* study that ceramic laminate veneers could benefit when large surfaces of dentin were exposed.<sup>15</sup> Freshly exposed dentin is the ideal substrate for dentin bonding.<sup>28,29</sup> How to apply IDS and how to clean or condition the IDS layer during the luting phase has not been studied to date.<sup>32</sup>

After one week of aging, the magnitude of the difference between the two SC methods depended on

Figure 3. Box plot of microtensile bond strength (MPa) (left) and estimated marginal means of microtensile bond strength (MPa) (right) for the different immediate dentin sealing (IDS) strategies and two evaluation times (1-wk, at one week; 6-m, after six-month aging) when the surface was conditioned with pumice (SC-P). IDS-1L, IDS with one layer of adhesive; IDS-2L, IDS with two layers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC; DDS, delayed dentin sealing (control).



the type of IDS used. This may be explained by the assumption that with only a thin layer of IDS, tribochemical silica coating, which also involves sandblasting, may largely remove the IDS layer, thereby decreasing bond strength. Hence, a thicker IDS layer and silica coating appeared more favorable. This confirms the observations by Stavridakis and others.<sup>36</sup> They demonstrated the risk of reexposure of dentin after conditioning the preparation, which may be reduced by using a filled bonding agent like Optibond FL (Kerr). In the present study, the three-step etch-and-rinse adhesive Optibond FL was chosen because this adhesive is known for its high filler load and high mechanical strength resulting in higher µTBS.<sup>10</sup> However, after six months, this particular effect was not observed. All different IDS strategies obtained higher bond strengths than the control group (DDS), independent of the SC method used. Therefore, the adhesive application to dentin directly after preparation is important to achieve higher bond strength. Hashimoto and others concluded that bond strength Figure 4. Box plot of microtensile bond strength (MPa) (left) and estimated marginal means of microtensile bond strength (MPa) (right) for the different immediate dentin sealing (IDS) strategies and two evaluation times (1-wk, at one week; 6-m, after six-month aging) when the surface was conditioned with pumice followed by tribochemical silica-coating (SC-PS). IDS-1L, IDS with one layer of adhesive; IDS-2L, IDS with two layers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC: DDS. delayed dentin sealing (control).

increased with each adhesive coating up to four layers.<sup>17</sup> Ito and others concluded that simply applying more layers of adhesive could improve the bond strength and the quality of dentin adhesion, especially if the layers were light cured separately.<sup>19</sup> Multiple layers of adhesive resin are thought to create a thicker adhesive layer without affecting the hybrid-layer quality. The increased bond strength results from an improved stress distribution and increased elasticity of the adhesive layer.4,39 However, others claim that each adhesive has its own ideal thickness and that this should be respected in this multilayering technique.<sup>7</sup> The adhesive system was heated before the application because unpublished data from laboratory studies indicate that it improves µTBS.

In contrast to the results of our study, others have observed that bond strength decreased over time,<sup>10,16</sup> which was attributed to hydrolytic degradation of the adhesive interface.<sup>2,25</sup> Our results demonstrate that by using IDS, the adhesive interface was stable over time; we could not find a



View field: 37.95 µm Det: SE 10 µm SEM MAG: 6.66 kx PC: 9



View field: 94.80 µm Det: SE SEM MAG: 2.67 kx PC: 9

Figure 5. Scanning electron micrographs of fracture surfaces from representative specimens after microtensile bond strength testing. (a): Adhesive failure at the adhesivecomposite interface (IDS-1L, SC-PS, 6-m). (b): Adhesive failure at the adhesive-dentin interface (DDS, SC-PS, 6-m). Note the exposed dentin tubuli. IDS. immediate dentin sealing: IDS-1L, IDS with one layer of adhesive: IDS-2L. IDS with two lavers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC; DDS, delayed dentin sealing (control); 1-wk, at one week; 6-m, after six-month aging.

Table 3:Overview of Pretesting Failures (ptf), Total Number of Specimens (n), and Failure Analysis (%) <sup>a</sup>										
	ptf	n	S	I	В	B/C	С			
1 wk										
IDS-1L + SC-P	0	12	11	67	22					
IDS-1L + SC-PS	0	12		17	17		67			
IDS-2L + SC-P	0	12		67			33			
IDS-2L + SC-PS	0	12	50	33			17			
IDS-F+SC-P	1	12		63	25	13				
IDS-F+SC-PS	0	12	11	78			11			
DDS + SC-P	1	12		100						
DDS + SC-PS	4	12		100						
6 mo										
IDS-1L + SC-P	0	12	9	91						
IDS-1L + SC-PS	0	12		80		20				
IDS-2L + SC-P	0	12		76			25			
IDS-2L + SC-PS	0	12	25	75						
IDS-F+SC-P	1	12		33	67					
IDS-F+SC-PS	0	12		67			33			
DDS + SC-P	3	12		80	20					
DDS + SC-PS	1	12		50	50					
<sup>a</sup> IDS, immediate dentin sealing; IDS-1L, IDS with one layer of adhesive; IDS-2L, IDS with two layers of adhesive; IDS-F, IDS with one layer of adhesive and one layer of flowable RBC; DDS, delayed dentin sealing with										

adhesive and one layer of flowable RBC; DDS, delayed dentin sealing with no layer of adhesive (control group); SC, surface conditioning; SC-P, surface conditioning with pumice rubbing; SC-PC, surface conditioning with pumice followed by tribochemical silica coating; 1-wk, immediate; 6-m, aged; ptf, pretesting failure; S, failure in dentin; I, failure at the adhesive-dentin interface; B, failure in adhesive; B/C, failure at the adhesive-composite interface; C, failure in composite.

significantly different effect between the one-week and six-month water storage data. Further investigation is needed to find out if this could be due to an improved resin impregnation associated with an IDS application. According to Magne and others, good bond strength of the definitive restoration to the sealed dentinal surface can be achieved even up to an extended provisionalization phase of 12 weeks.<sup>23</sup> In this study, the provisionalization phase was three weeks, which, according to Magne and others, could not have affected bond strength.<sup>23</sup>

When surfaces were cleaned using pumice, a stable  $\mu$ TBS over time was recorded without a significant difference among the IDS strategies. Surfaces conditioned with tribochemical silica coating demonstrated a more positive effect after six months of aging than the solely pumice-rubbed surfaces. A higher mean bond strength was obtained with a thicker IDS layer compared to a DDS strategy. High mean  $\mu$ TBS values and small standard deviations were seen in the IDS-1L group, when specimens were silica coated, after one week

 $(35.2\pm19.0 \text{ MPa})$  and six months  $(35.6\pm7.3 \text{ MPa})$  of aging. Others attributed the improved bond strength recorded using tribochemical silica coating to the additional chemical bonding of the applied silane coupling agent to the silica-coated surface.<sup>1,33,37</sup>

Most failures in the one-week and six-month aged groups were seen at the adhesive-dentin interface: it might be interesting to see whether there would be more differences in bond strength after a longer period of aging. Pretesting failures were seen mainly in the DDS groups, probably due to water uptake of the dentin. In the DDS group, no adhesive interface was created (directly after preparation), while this adhesive interface was necessary to prevent water uptake from the tooth (in the temporary crown phase).<sup>18</sup> A disadvantage of the µTBS is not only that the actual "bond" strength measured but also that the strength of the complete assembly, including dentin and composite,<sup>9</sup> possibly results in a higher strength that surpasses the interfacial strength. Yet µTBS is generally accepted as one of the most valid bond strength tests.<sup>35</sup> According to a review by Qanungo and others<sup>32</sup> and based on the results of this study, significant differences have been demonstrated between IDS and DDS, this being more in favor of IDS.

#### CONCLUSIONS

From this study, the following can be concluded:

- 1. The  $\mu$ TBS recorded for specimens prepared with any type of the three IDS strategies investigated was higher compared to DDS.
- 2. Cleaning with pumice only or with additional tribochemical silica coating did not affect  $\mu$ TBS. When using a silica-coating technique, a thick IDS layer is recommended.
- 3. A one-layer IDS and surface conditioning involving pumice rubbing with additional tribochemical silica coating appears to be an effective, consistent, durable, and relatively less time consuming IDS procedure.

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#### **Regulatory Statement**

This study was conducted in accordance with all the provisions of the local human subjects oversight committee

guidelines and policies of Rijksuniversity Groningen in the Netherlands.

#### **Conflict of Interest**

The authors did not have any commercial interest in any of the materials used in this study.

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