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Dentin Bonding Agent Volatility in Relation to Shear Bond Strength



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LtCol John Brewster

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Abstract

Objective: Bonding agents are subject to differing degrees of solvent volatility depending on the type and amount of solvent used. The purpose of this study is to compare the evaporation of DBA solvents as a function of time and determine the impact of mass lost on shear bond strength (SBS) of a commonly used resin-composite bonded to dentin.

Methods: The following DBA systems were utilized: OptiBond Solo Plus (Kerr Corp.), OptiBond FL (Kerr Corp.), Clearfil SE Bond 2 (Kuraray America), Scotchbond Universal (3M) and AdheSE Universal (Ivoclar Vivadent). The resin-composite used for SBS testing was Filtek Supreme Ultra (3M). Each of the DBAs were dispensed, then massed at 10-minute intervals: immediate usage, 10-, 20- and 30-minute exposure. 240 extracted human third molars were sectioned to remove root structure and mounted in acrylic. Dentin bonding surfaces were prepared following ISO 29022 instructions for grinding/polishing of specimens and stored in 0.2M phosphate buffered saline (PBS). The teeth were randomly divided into 20 groups of 12, based on the use of 5 different DBAs at 4 different time periods. The bonding agent was applied and cured per manufacturer instructions to the dentin surface of each specimen. Each specimen was placed into a jig and the resin-composite material was applied and light cured. Specimens were stored for 24 hours in PBS at 37 degrees Celsius and then tested until failure using a universal testing machine at a rate of one millimeter per minute. Failure loads were analyzed using Kruskal-Wallis/Dunn's post hoc at a 95 percent level of confidence ($\alpha=0.05$).

Results: Minimal mass lost was seen over time concerning the adhesives of OptiBond FL and Clearfil SE Bond 2. While the greatest mass loss was seen in the primer for OptiBond FL. No statistical difference was found comparing SBS of individual DBAs over time. Statistical differences in SBS were noted at each time interval when comparing DBAs.

Conclusions: Under the limitations of this study, bonding agent solvent dissolution does not appear to influence SBS of resin-composite to dentin. Bonding agents containing ethanol as a solvent experience

more volatility than those with water as a solvent. Bonding with self-etch systems also resulted in greater SBS than etch-and-rinse systems. Further research should be considered for a greater understanding of this relationship.

Manuscript

Introduction:

An area of constant research and development in the field of dentistry is that of dentin bonding agents (DBA).^{1,2,3} Advances in this field provide for greater handling properties⁴ and enhanced bonding strengths of both direct resin-composite restorations as well as bonded fixed restorations.⁵ While amalgam restorations continue to have a greater prognosis when compared to bonded resin-composites in both the short-term and long-term, this discrepancy is narrowing.^{6,7} The majority of resin-composite restorations failures are due to degradation of, and microleakage at the DBA hybrid layer, resulting in recurrent caries along the restoration margin.^{8,9}

In order to achieve optimum bond strength between a restoration and tooth structure, immaculate use of DBAs must be implemented.¹⁰⁻¹⁵ Historically speaking, 3-step etch-and-rinse and 2-step self-etch adhesive systems have been viewed as the gold-standard in dentin bond durability.¹⁶ Most recently, universal adhesive systems released, and although long-term clinical studies are still needed, initial research presents with mixed results in terms of bond durability.¹⁷ Those containing methacryloyloxydecyl dihydrogen phosphate (MDP) have performed with a greater and more stable resin-dentin bond strength and reduced marginal nanoleakage.¹⁸⁻²⁰

Clinicians routinely face challenges that can potentially reduce optimum bond strength of a DBA. It has been researched and documented that DBA bond strength is impaired by a number of clinical situations, including: contamination with saliva,¹⁰ contamination with heme¹¹ or a hemostatic agent,¹² contamination with water,¹³ the presence of cervical sclerotic dentin,¹⁴ and the inability to completely light-cure the restoration.¹⁵

Dentin bonding agents, their primers especially, contain solvents that help displace water from dentin, allowing resin penetration. Evaporation of these solvents can occur if left open over time²¹ and it has been discovered that this loss of solvent can lead to a decrease in the dentin bond strength.²² Repeated opening

and closing of a single bottle adhesive system (one-step etch and rinse) resulted in solvent evaporation and ultimately impaired marginal seal of class 5 restorations.²³

This study was conducted to determine how the shear bond strength (SBS) of a resin-composite restoration to dentin is affected by the volatility and dissolution of DBA components. The null hypotheses were that there would be no difference in SBS between:

1. The control group of each bonding agent used immediately, and the experimental groups of bonding agents allowed to undergo 10, 20, or 30 minutes of dissolution
2. Bonding agents compared to one-another

Materials/Methods:

Massing (n=5):

Five dental bonding agents: OptiBond Solo Plus (Kerr Corp.), OptiBond FL (Kerr Corp.), Clearfil SE Bond 2 (Kuraray America), Scotchbond Universal (3M) and AdheSE Universal (Ivoclar Vivadent) were used for both the massing and bonding portions of this experiment. To mass, dual-well plastic dappen dishes were initially weighed empty. Each bonding agent was then dispensed into the bonding trays and re-weighted immediately, then re-weighted after a delay of 10, 20, and 30 minutes. For non-simplified DBA systems, primer and adhesive components were massed independently. They were left open to ambient air during these time periods (22.2C, 53%RH). Percentage change of mass was calculated for each specimen.

Bonding (n=12):

One hundred twenty freshly extracted, caries-free human third molars were used in this study. They were extracted for routine clinical indications and collected from local oral and maxillofacial surgery clinics following protocol defined by the 81st Medical Group Institutional Review Board (IRB). Tooth roots were removed by sectioning at the cemento-enamel junction with a dental lab handpiece. The crowns were then sectioned in half along the central groove in a mesio-distal plane to produce two

specimens, a facial and lingual surface. Each specimen was mounted in a self-curing tray resin (Bosworth Fastray, Gibbstown, NJ, USA) with facial or lingual external surface facing outwards. All specimens were stored in water at room temperature before surface preparation.

Dentin bonding surfaces were prepared for shear testing, following specifications of ISO 29022, using a two-step sequential finishing process under running water. Course silicon carbide grinding paper, $125 \pm 1 \mu\text{m}$ (CarbiMet 120, Buehler), was used to access dentin and create a flat bonding area large enough to place a resin-composite button of diameter 2.38 mm. Then fine silicon carbide grinding paper, $35 \pm 1 \mu\text{m}$ (CarbiMet 400, Buehler) was used to refine the dentin surface until it was smooth upon visual inspection. Specimens were stored in distilled water at room temperature until bonding protocols were completed; within 4 hours of surface preparation.

Prepared specimens were then randomly divided into 20 groups (n=12) as shown in the table below.

OptiBond Solo Plus	OptiBond FL	Clearfil SE Bond 2	Scotchbond Universal	AdheSE Universal
Immediate bond	Immediate bond	Immediate bond	Immediate bond	Immediate bond
10-minute delay	10-minute delay	10-minute delay	10-minute delay	10-minute delay
20-minute delay	20-minute delay	20-minute delay	20-minute delay	20-minute delay
30-minute delay	30-minute delay	30-minute delay	30-minute delay	30-minute delay

Specimens were rinsed and dried with oil-free compressed air for 5-10 seconds. Bonding agents were dispensed into plastic dappen dishes and the immediate application groups were bonded immediately after dispensing. Timers were utilized to achieve appropriate time delays for delayed application groups, 10, 20 and 30 minutes after initial dispensing. Bonding agents were applied following manufacturer recommendations and polymerized utilizing a PolyWave® visible curing light (BluePhase G2, Ivoclar Vivadent, Amherst, NY, USA). Each specimen was then mounted in a microshear bonding jig with a

polytetrafluoroethene insert (Ultradent, South Jordan, UT, USA) centered over the dentin bonding surface. The polytetrafluoroethene inserts have a 2.38 mm diameter cylindrical space to serve as a mold for a restorative material; inserts were replaced after 6 uses. A nanofilled resin-composite (Filtek Supreme Ultra (shade A2B), 3M ESPE, St. Paul, MN, USA) was bonded to the specimen through the jig insert; dimensions of 2.38 mm internal diameter and 2mm height. The resin was then photopolymerized for 20 seconds using a visible curing light (BluePhase G2, Ivoclar Vivadent) and the specimen was carefully removed from the apparatus. Excess resin-composite was removed when necessary with a scalpel blade with the diameter ascertained with a digital caliper as close to the tooth surface as possible. This process was completed utilizing each bonding agent at each time increment, following manufacturer instructions as seen in the table below.

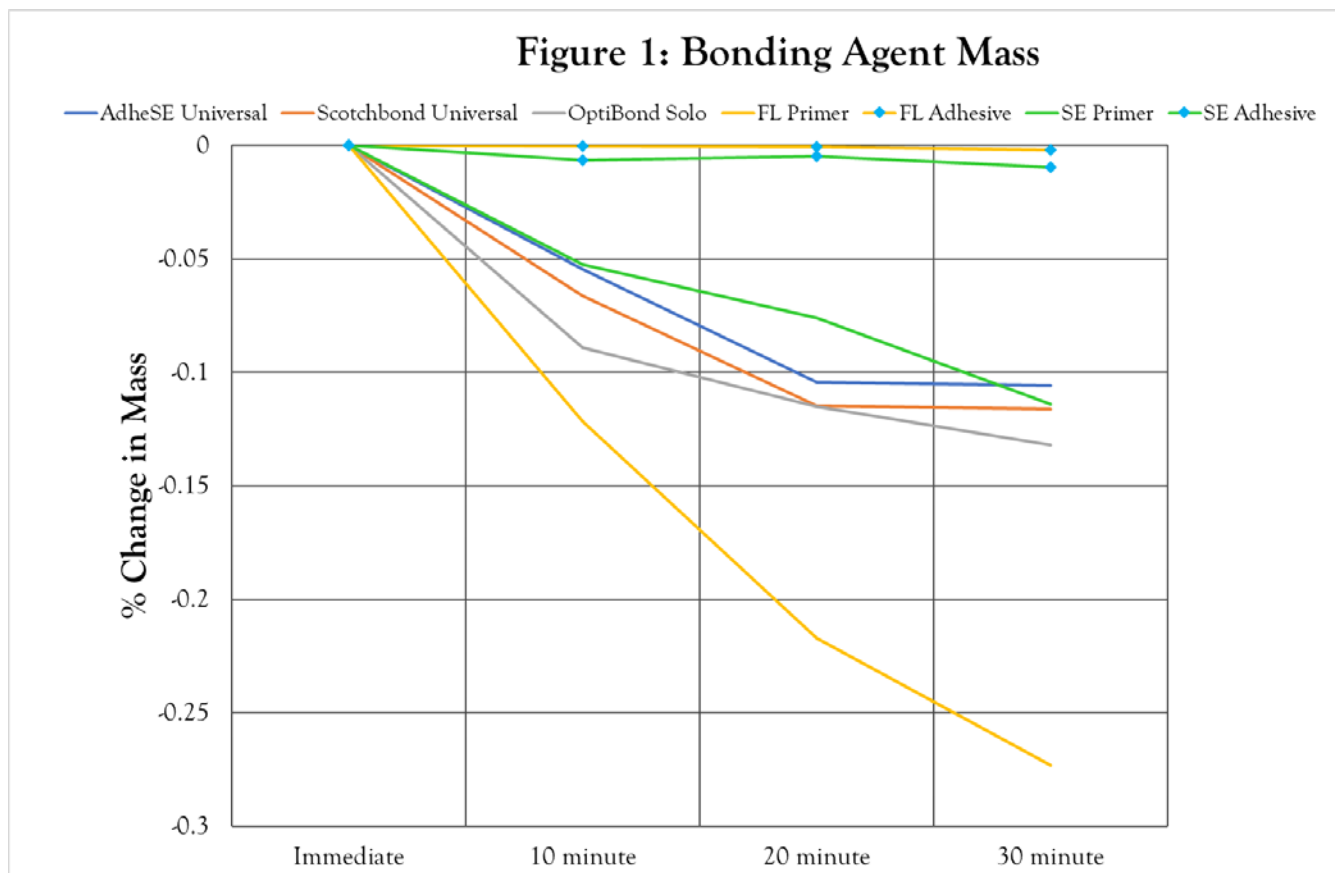
Bonding Agent	Acid Etch	Primer	Adhesive	Light Cure
Clearfil SE2		Apply 20s Dry 5s or doesn't move	Apply 10s Air thin 3s	10s
OptiBond FL	Etch 15s Rinse, dry 5s	Apply 15s Dry 5s	Apply 10s Air thin 3s	20s
OptiBond Solo Plus	Etch 15s Rinse, dry 5s	Apply 15s Air thin 3s		20s
Scotchbond Universal		Apply 20s Air thin 5s or doesn't move		10s
AdheSE Universal		Apply 20s Air thin until no movement		10s

Restored specimens were stored in 0.2M PBS for 24hrs at 37C and then mounted in a universal testing machine (Ultratester, Ultradent, South Jordan, UT, USA) and tested in shear until failure at a crosshead speed at 1mm/min with the calculated failure stress recorded with mean values recorded. For massing and SBS, the data was subjected to the Shapiro-Wilk and Bartlett's test. Massing data for individual bonding agents overtime showed normal distribution and were further analyzed utilizing a repeated measures ANOVA with Fisher's post hoc when comparing a single bonding agent over time. Both data for comparing mass across DBA groups and all SBS data failed to meet the assumptions for parametric analysis; Thus,

Kruskal-Wallis and Dunn's post hoc test was used to compare results across the dentin bonding agents tested and for SBS data. All of tests were at a 95 percent level of confidence ($\alpha = 0.05$). Statistical analysis was performed using SPSS 21 (IBM/SPSS, Chicago, IL, USA).

Results:

The massing results are presented in Figure 1 and Table 1.



Percent Mass Change				
Bonding Agents	Immediate	10 minute	20 minute	30 minute
AdheSE Universal	0 A	-0.0545 (0.01) B c	-0.1045 (0.03) C cd	-0.1059 (0.02) C c
Scotchbond Universal	0 A	-0.0661 (0.01) B c	-0.1147 (0.01) C d	-0.1159 (0.02) C cd
OptiBond FL Primer	0 A	-0.1216 (0.04) B d	-0.2171 (0.06) C d	-0.2732 (0.07) D e
OptiBond FL Adhesive	0 A	-0.0003 (0.00) A a	-0.0006 (0.00) A a	-0.0020 (0.00) A a
Optibond Solo Plus	0 A	-0.0893 (0.01) B d	-0.1150 (0.02) C d	-0.132 (0.01) C d
Clearfil SE Primer	0 A	-0.0525 (0.02) B c	-0.0760 (0.03) C c	-0.1141 (0.00) D c
Clearfil SE Adhesive	0 A	-0.0064 (0.01) A b	-0.0047 (0.02) A b	-0.0094 (0.02) A b

*n=12; Capital letters annotate statistically similar groups per row (ANOVA repeated measures with Fisher's LDS)
 Lowercase letters annotate statistically similar groups per column (Kruskal-Wallis Test with Dunn's post hon)
 Negative values indicate reduced mass from Immediate/baseline*

Percentage mass loss was most predominately seen in simplified DBAs and multi-step primers. Minimal mass loss was seen in the adhesive component of OptiBond FL and Clearfil SE Bond 2 systems.

Shear bond strength testing results are shown in Figures 2, 3 and Table 2 below.

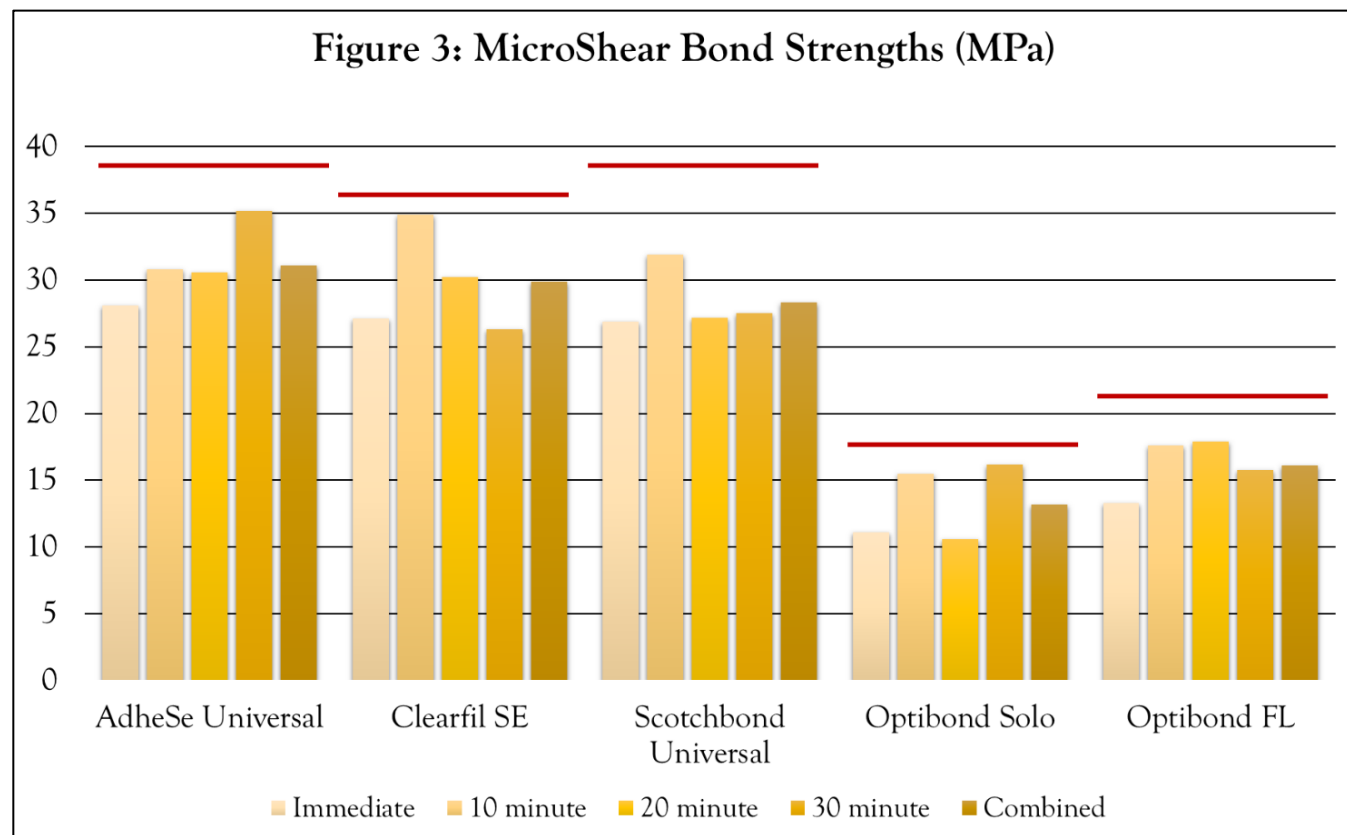
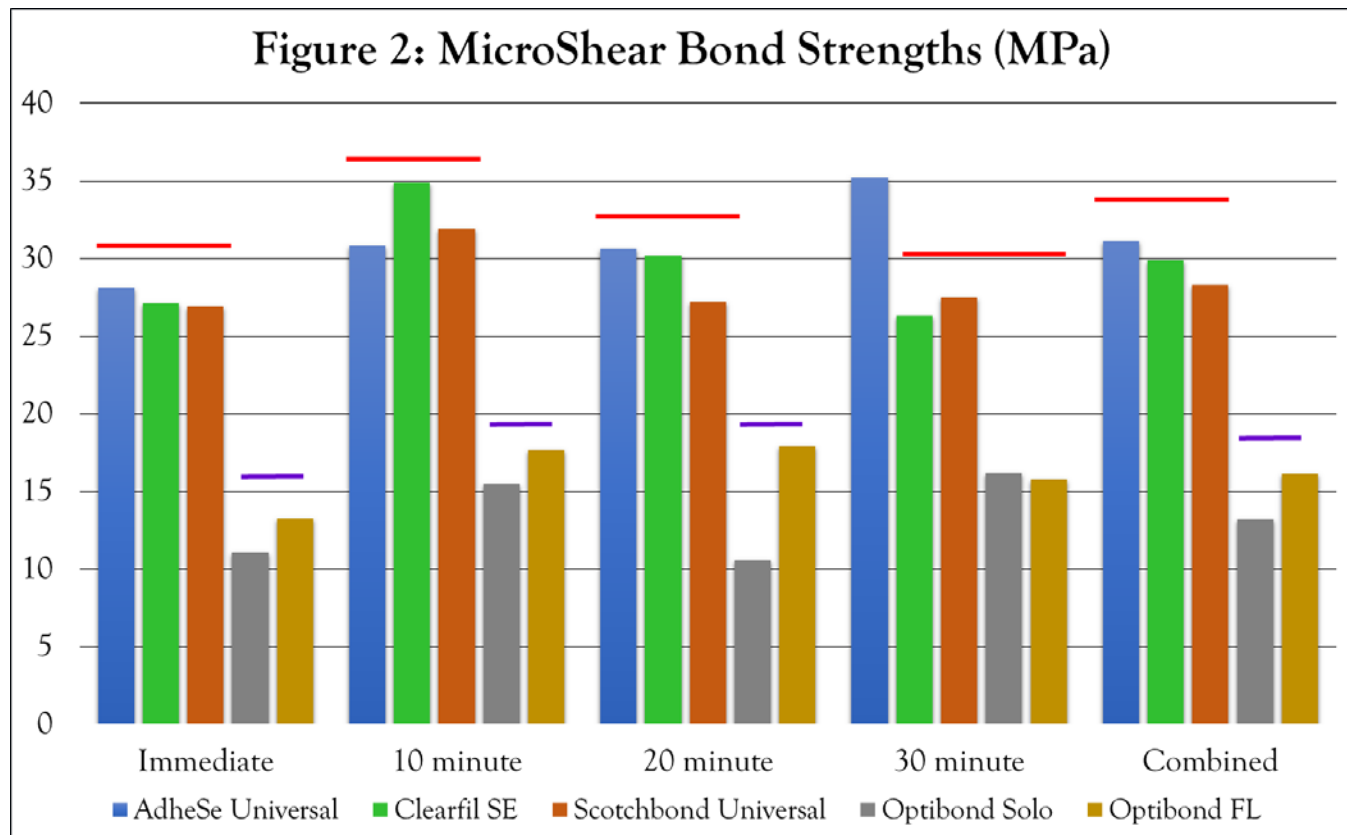


Table 2

Bonding Agents	MicroShear Bond Strenghts (Mpa)				
	Immediate	10 minute	20 minute	30 minute	Combined
AdheSe Universal	28.1 (4.8) A a	30.8 (7.3) A ab	30.6 (7.0) A a	35.2 (7.1) A a	31.1 (6.9) a
Clearfil SE	27.1 (14.5) A a	34.9 (14.9) A a	30.2 (8.2) A a	26.3 (12.6) A ab	29.9 (12.9) a
Optibond FL	13.3 (4.9) A b	17.6 (7.3) A b	17.9 (7.9) A b	15.8 (5.2) A b	16.1 (6.5)b
Optibond Solo	11.1 (8.8) A b	15.5 (11.5) A c	10.6 (13.1) A b	16.2 (12.4) A b	13.2 (11.4) b
Scotchbond Universal	26.9 (5.7) A a	31.9 (8.3) A a	27.2 (10.0) A a	27.5 (6.8) A ab	28.3 (7.9) a

*n=12; Capital letters annotate statistically similar groups per row; Lowercase letters annotate statistically similar groups per column;
Kruskal-Wallis Test with post stepwise step-down test*

In each of the previous figures, solid lines above the data indicate statistical similarities. For all time periods other than at 30 minutes, AdheSE Universal, Scotchbond Universal and Clearfil SE Bond 2 outperformed OptiBond FL and OptiBond Solo Plus. At 30 minutes, AdheSE Universal showed a significantly greater SBS than all other bonding agents tested. But when the bonding agents were compared against themselves across time periods (Figure 3) no significant difference was shown to exist. Figure 4 and Table 3 below illustrate the mean SBS for each bonding agent tested across the entire experiment, regardless of performance at specific time intervals. The table also illustrates the significantly superior performance of each Clearfil SE Bond 2, AdheSE Universal and Scotchbond Universal compared to OptiBond FL and OptiBond Solo Plus.

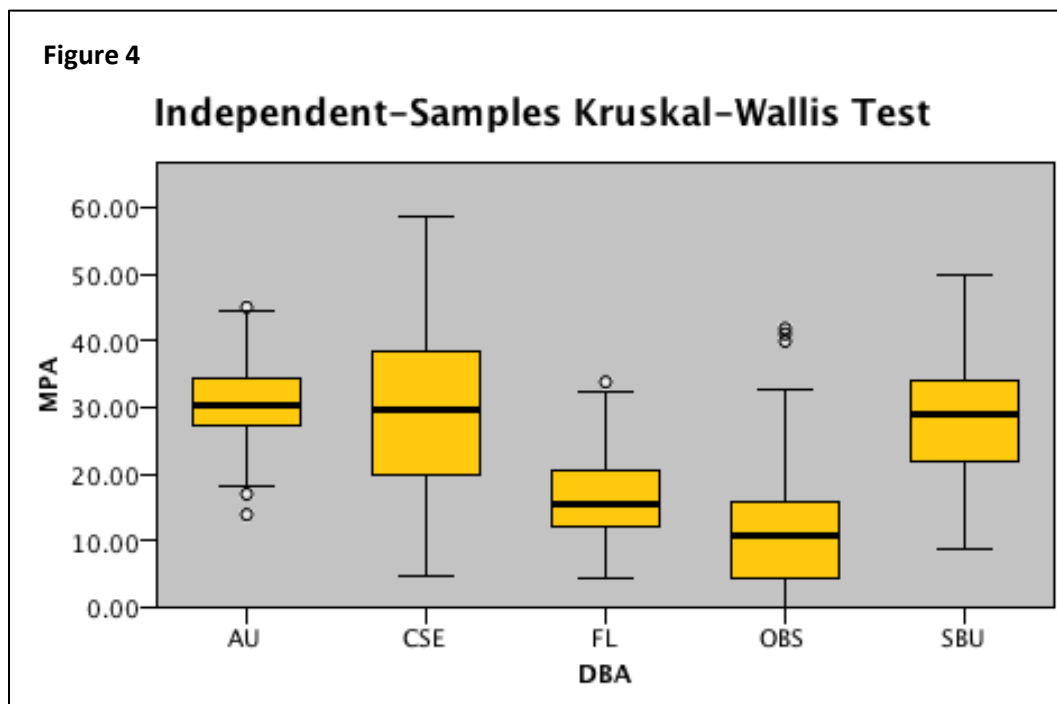


Table 3

Sample 1-Sample2	Test Statistic	Std. Error	Std. Test Statistic	Sig.	Adj.Sig.
OBS-FL	12.022	14.058	.855	.392	1.000
OBS-SBU	-86.808	13.986	-6.207	.000	.000
OBS-CSE	87.028	14.133	6.158	.000	.000
OBS-AU	103.001	14.058	7.327	.000	.000
FL-SBU	-74.786	13.832	-5.407	.000	.000
FL-CSE	75.006	13.980	5.365	.000	.000
FL-AU	90.979	13.904	6.543	.000	.000
SBU-CSE	.219	13.908	.016	.987	1.000
SBU-AU	16.192	13.832	1.171	.242	1.000
CSE-AU	15.973	13.980	1.143	.253	1.000

Discussion:

Dentin bonding agents are formulated by the combining of multiple ingredients, akin to that of a bartender creating a cocktail. Much chemistry is involved in the successful blending of these ingredients to allow them to provide the desired clinical outcome, a resilient bond of restorative material to tooth substrate.²⁴ Among the components is the bonding agent solvent. The solvent plays a very important role: it is hydrophilic, allowing penetration into the dentinal tubules, absorbing and displacing water.²⁴ This, in conjunction with the additional chemistry of a single bottle bonding system or the primer of a multi-step system, helps maintain collagen integrity, allowing micromechanical interaction by resin monomers, with the minimal presence of residual water.²⁵

Bonding agent solvents are usually either ethanol, butanol, acetone, water or a combination of the mentioned.²⁵ Certain solvents, such as ethanol, are commonly coupled with water allowing them to form an azeotrope.^{26,27} This allows both ethanol and water to dissolve at essentially the same rate, potentially minimizing residual water and resulting in an ultimately greater bond strength.²⁸ Acetone though, has previously been shown to be very volatile, with the evaporation rate often exceeding that of water.²⁹

Acetone wet-bonding is more technique sensitive and can result in a tooth too dry or too wet, leading to hydrolytic degradation of the dentin-restorative material interface.³⁰

The solvent is ideally driven off following application to tooth substrate. This allows for maximum penetration of the hydrophobic resin, resulting in the greatest potential bond strength. Increased hydrophilicity of bonding agents (mainly simplified systems) has been shown to result in statistically greater amounts of water sorption after cure.³¹ A previous study by Carvalho et al showed that increased duration of air drying during bond application resulted in increased bond strength regarding acetone-based systems, but showed no difference concerning ethanol-based bonding agents.³² The opposite has also been illustrated, that differing levels of air drying duration (regardless of solvent type) does not lead to differences in microtensile bond strength.²⁸ Our study did not examine increased periods of air application for solvent dissolution, but rather ambient dissolution of solvent. Similar results were shown: that ethanol-based systems performed similarly, regardless of degree of solvent dissolution.

Concerns with long term bond stability of universal adhesives still exist^{33,34}, as they are relatively new on the market. As demonstrated in studies previously, simplified adhesives, especially two-step etch-and-rinse systems tend to have decreased performance compared to their three-step etch-and-rinse or two-step self-etch counterparts.¹⁶ Two universals were included in this study, Scotchbond Universal and the recently-released AdheSE Universal. Our study showed similar results between both the Universals and a gold standard two-step self-etch adhesive, Clearfil SE Bond 2. Even though these three bonding agents performed better than both OptiBond FL and OptiBond Solo Plus, they performed statistically similar to each other throughout the experiment, without any differences seen across increased periods of dissolution. This is in slight contradiction to a previous dissolution study by Pongprueksa et al, which specifically examined varying dissolution of Scotchbond Universal. After allowing maximum dissolution, it was seen that dentin microtensile bond strength had statistically decreased compared to bonding agent with half solvent and maximum solvent concentration.²² It was reported that maximum dissolution was

obtained after a period of 14 days. In a similar study, universal adhesives utilized in an etch-and-rinse modality, showed increased microtensile bond strength when air drying during application was increased from 5 seconds to 25 seconds.³⁵ These significant results were not echoed concerning self-etch adhesive systems. Both results differ from our study most likely due to the fact they were essentially controlling the level of dissolution compared to a more ambient manner of evaporation.

Regarding the sub-optimal performance of OptiBond FL three-step etch-and-rinse adhesive system, it is hypothesized that SBS results would normalize after extended periods of artificial ageing. There is much documented success of this bonding agent and it continues to be viewed as a gold-standard in the field.¹⁶

Limitations of this study include: difficulty obtaining morphologically similar third molar specimens, resulting in variability of dentin preparation depth to achieve the required bondable dentin surface; the limited artificial ageing storage period of only 24 hours; and the inaccuracies and potential errors that are associated with ISO 29022. Future studies should be designed to account for some of these by spending more time collecting ideal specimens and ageing for more than 24 hours (thermal cycle and/or long-term storage).

Conclusions:

This study evaluated the effects of ambient dissolution over 3 time periods on the SBS of 5 commonly used dentin adhesives. Under the conditions, it was found that increased dissolution, verified by mass change, did not result in significant differences of SBS. Differences did exist between bonding agents, with Clearfil SE Bond 2, AdheSE and Scotchbond Universals outperforming OptiBond FL and OptiBond Solo Plus, independent of extent of dissolution. Clinical impact:

- 1.) Use of DBA immediately or shortly after distribution produces similar SBS results
- 2.) Use of Universal adhesives have performed well but continue to require longer term studies

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