the Journal of CONTEMPORARY DENTAL PRACTICE at www.thejcdp.com
Volume 4 Number 4 November 15, 2003 Fall Issue

Immediate and 24-Hour Bond Strengths of Two Dental Adhesive Systems to Three Tooth Substrates

Yousef Fouad Talic, BDS, MS, FICOI, DICOI, DASO

Abstract

Bond strengths of bonded composite resins to tooth substrates vary depending on when they were measured. Most bond strengths reported in the literature are a result of one hour, 24-hour, or longer periods of time that do not simulate actual clinical practice when occlusal adjustment and finishing and polishing procedures are performed within seconds after restoration placement.

There are many different ways to measure the bond strength of direct esthetic restorations to various dental substrates. This research uses a method published previously that compares immediate and 24-hour bond strengths of a single-bottle dental adhesive and a self-etching primer adhesive to prepared enamel, unprepared enamel, and prepared dentin substrates.

Significant differences were found between immediate and 24-hour bond strengths, but there were essentially no differences between substrates or adhesives.

Keywords: Immediate bond strength, 24-hour bond strength, esthetic composite restorations, *in vitro*, enamel bonding, dentin bonding

Citation: Talic YF. Immediate and 24-Hour Bond Strengths of Two Dental Adhesive Systems to Three Tooth Substrates. J Contemp Dent Pract 2003 November;(4)4:028-039.

Introduction

"Curing" direct esthetic restorations is widely misunderstood since the "curing" light merely initiates a chemical process that technically takes infinite time to complete. As a consequence, the bond strengths of bonded esthetic restorations to tooth substrates will vary depending on when they were measured. Most *in vitro* bond strengths reported in the literature are a result of one hour, 24-hour, or longer periods of time that do not simulate actual clinical practice when occlusal adjustment and finishing and polishing procedures are performed within seconds after restoration placement.

The purpose of this study was to compare the immediate and 24-hour *in vitro* bond strengths of two popular dental adhesive systems when applied to three tooth substrates.

Literature Review

Different bond strengths of dental adhesive products are reported nearly every day.[1-7] Because of the variety of testing methods used, it is not always clear how the products actually compare. This is further complicated by the number of new products introduced to the market and by the lack of a precise definition of many independent variables associated with these products. The most recent entries into the fray have been self-etching/primer dental adhesives. These products have stimulated a great deal of discussion within the profession. Anecdotally, there are a number of advantages, chief among them being virtually no post-operative sensitivity.[8] Also, ease and speed of manipulation are important advantages. There are disadvantages, however, that make this category of materials fairly controversial.

Because the etching produced by these materials is not as aggressive as that produced by phosphoric acid gels, their use on uncut enamel has been suspect especially for esthetic incisal, occlusal, and interproximal restorations where restorative materials are often extended over prepared margins for proper blending to achieve invisible margins.

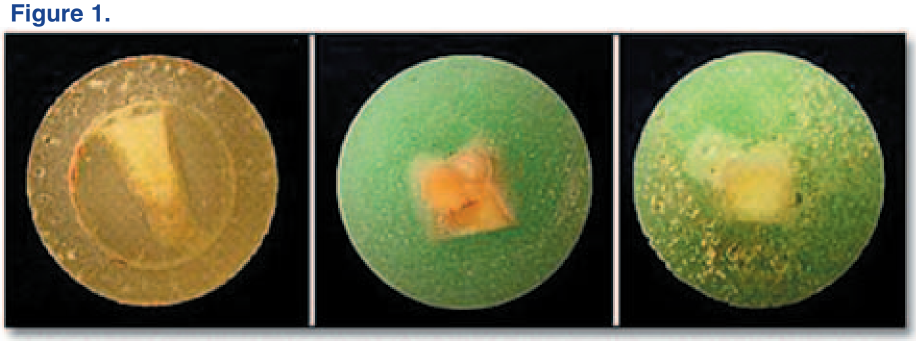
Further, the extent to which these materials can modify the smear layer on cut enamel and dentin surfaces and, thus, achieve higher bond strengths has been studied with varying results.[9-14]

Material and Methods

Two dental adhesive systems were used for this study. The first, OptiBond Solo™ plus (Kerr, USA), is a single-bottle system. It contains ethyl alcohol as a solvent. The second, Clearfil™ SE Bond (Kuraray, Japan) is a two-bottle system that includes a self-etching primer. Both systems have been highly rated by an independent esthetics research group.[15]

Three human tooth substrates were mounted in acrylic as previously reported (Figure 1).[16,17] The first substrate was clean, unprepared enamel. Relatively flat coronal areas of central and lateral incisors were cleaned with a pumice flour slurry and rubber cup for 5 seconds and rinsed for 5 seconds. No other preparation was performed prior to etching. The second and third substrates were prepared enamel and dentin. These specimens were prepared using a model trimmer with coarse and fine silicon carbide wheels and rinsed for 5 seconds.[16] Specimens were cleaned with a pumice flour slurry and rubber cup for 5 seconds and rinsed with an air/water syringe for 5 seconds.

Figure 1.



Left: Sound enamel substrate. Center: Prepared enamel substrate. Right: Dentin substrate.

The embedding mold used for this method is made of polycarbonate and does not stick to methylmethacrylate resins. Therefore, no separating medium is required. The mold (Ultradent Products Inc., USA) can make 15 cylindrical specimens, each 1 inch in diameter and up to 1 inch in height. Care should be taken to remove any air pockets around the edges of the specimen holes to prevent leakage of the embedding resin. Each tooth substrate is positioned within its specimen hole using a small portion of sticky wax, if necessary, to secure uncut specimens as well as the exposed adhesive on the cellophane tape.

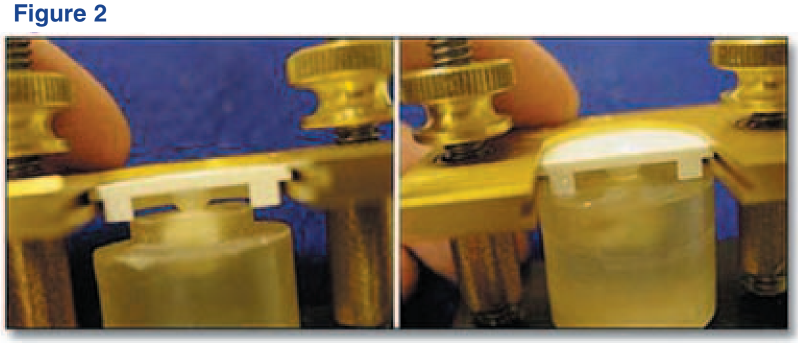
The precise orientation of each substrate can be observed through the clear cellophane tape. This enables specific substrate orientations to be achieved for various test procedures. Because of the design of the polyethylene insert used to “restore” the tooth substrate, a 1 inch diameter rubber washer should be placed into the specimen hole when uncut specimens are to be embedded. Once the washer is removed after embedding, the additional space makes it easier to get close adaptation of the polyethylene insert to the more irregular uncut surfaces.

Clear embedding resin is mixed to a fluid consistency. Clear, pink Orthoresin (Dentsply De Trey, Germany) mixed in 1 part powder/4 parts liquid provides ample pouring time and allows for specimen observation. Some resins do not shrink as much as others and may make it difficult to remove specimens from the mold. Some resins bloat and overflow when mixed at a 1:4 ratio. Some opaque resins work well but prevent specimen observation. These resins are to be avoided. Once specimens have hardened, they may be removed with simple finger pressure. Because of the surface irregularities of the unprepared specimens, a slightly different embedding procedure was required as noted in the previous report (Figure 2).[16]

For the OptiBond Solo™ plus specimens, 37.5% phosphoric acid gel (Kerr Gel Etchant, sds Kerr, USA) was applied and agitated for 15 seconds and rinsed with an air/water syringe for 5 seconds. Excess water was removed by gentle air drying for 1 second. The surface was shiny with water, but puddles of water were removed. The bottle of OptiBond Solo™ plus was shaken. One drop was placed in a mixing well. The adhesive was lightly brushed onto the tooth surface with a flexible, disposable applicator (Kerr Applicators™, sds Kerr, USA) every 5 seconds for a period of 20 seconds. After water was cleared from the air/water syringe, light air was used to thin the adhesive and evaporate the solvent for 5 seconds. The adhesive was light-initiated for 20 seconds with the light-curing unit (ESPE Elipar Highlight, Seefeld, Germany).

For the Clearfil™ SE Bond specimens, the SE Primer bottle was shaken. One drop was placed in a mixing well. The primer was lightly brushed onto the tooth surface with a disposable brush applicator and allowed to sit undisturbed for 20 seconds. After water was cleared from the air/

Figure 2

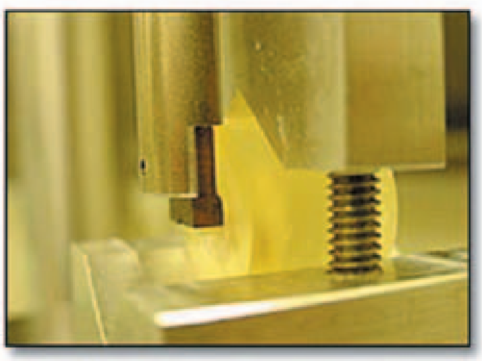


Left: A gap is created using a rubber washer during embedding so the mold insert can be more closely adapted to the surface irregularities of the unprepared enamel surfaces.
Right: When the smooth surfaces were created for the prepared enamel and dentin and a close adaptation is required, a gap is not necessary.

water syringe, light air was used to dry the primer for 5 seconds. The Clearfil™ SE Bond bottle was shaken. One drop was placed in a mixing well. The adhesive was lightly brushed onto the tooth surface with a disposable brush applicator. After water was cleared from the air/water syringe, light air was used to thin the adhesive and evaporate the solvent for 5 seconds. The primer/adhesive was light-initiated for 10 seconds.

All specimens were "restored" according to the previously reported method.[17] Enamel A2 shade composite (XRV™ Herculite®, Kerr, USA) was used to bulk fill the specimen mold. The composite was light-initiated for 40 seconds. Specimens for immediate bond strength testing were placed in an Instron machine according to the previously reported method within 1 minute of light-initiation.[16] Specimens for 24-hour bond strength testing were stored in distilled water until testing. The Instron crosshead speed was 0.5mm/min (Figure 3).

Figure 3



Specimen positioned in the Instron machine, ready for testing.

Results were analyzed using ANOVA and Bonferroni tests with $\alpha = 0.05$.

Results and Discussion

The results of this study are presented in Tables 1 and 2. Table 1 shows the bond strengths in MPa measured within one minute of initiation. The means and standard deviations are shown at the bottom of the table. Statistically, there were no differences between any of the groups except for between sound enamel substrates and dentin substrates and between prepared enamel substrates and prepared dentin substrates with OptiBond Solo™ plus. Although the variability is greater with the sound enamel specimens, this is to be expected with the somewhat curved surfaces found with incisor enamel. For the same reason, the values are not significantly higher ($\alpha = 0.05$) for the sound enamel substrates mostly because surface irregularities introduce compressive and tensile factors to an essentially sheer type of test. It may be speculated that cleaning the uncut enamel substrates with pumice slurry improved results, but this was not studied. Despite what many clinicians believe, the bond strength to dentin was higher than the bond strength to enamel but only a few significant instances were noted.

Table 2 shows the bond strengths in MPa measured 24-hours after initiation. The means and standard deviations are shown at the bottom of the table. Again, the expected variability with the uncut enamel substrates was seen. Again statistically, no differences were found between any of the groups except between dentin substrates with Clearfil™ SE Bond and dentin substrates with OptiBond Solo™ plus.

While there were no differences between the bond strengths of either bonding agent on uncut enamel substrates, there were highly significant differences between one minute and 24-hour specimens. In 24 hours, the average bond strength using Clearfil™ SE Bond increased from 25.7MPa (±6.6MPa) to 42.5MPa (±4.4MPa). In 24 hours, the average bond strength using OptiBond Solo™ plus increased from 24.9MPa (±3.6MPa) to 44.2MPa (±6.9MPa).

Although there were no differences between the bond strengths of either bonding agent on prepared enamel substrates, there were highly or mostly very highly significant differences between one minute and 24-hour specimens. In 24 hours, the average bond strength using Clearfil™ SE Bond increased from 25.6MPa (±3.9MPa) to 42.5MPa (±3.4MPa). In 24 hours, the average bond strength using OptiBond Solo™ plus increased from 24.6MPa (±3.3MPa) to 45.9MPa (±3.1MPa).

Interestingly there were no differences between the bond strengths of either bonding agent on cut dentin substrates, but there were highly significant differences between one minute and 24-

Table 1. Bond Strengths (MPa) Measured within 1 Minute of Initiation					
Clearfil™ SE Bond			OptiBond Solo™ plus		
Uncut Enamel	Cut Enamel	Cut Dentin	Uncut Enamel	Cut Enamel	Cut Dentin
19.7	25.4	32.5	25.2	24.0	32.1
34.0	21.7	28.4	22.1	30.3	29.7
25.6	18.9	33.1	18.7	21.0	34.7
16.5	30.7	19.6	26.6	19.9	34.8
15.5	26.0	30.9	27.9	23.6	30.6
26.9	29.8	29.7	18.9	28.7	29.9
34.1	22.6	27.9	25.9	27.9	31.2
22.4	27.4	33.2	28.9	23.5	33.0
25.6	23.5	28.8	25.1	26.1	28.4
29.6	31.0	31.5	24.9	22.7	32.0
32.4	24.8	27.9	29.4	22.8	32.9
25.7	25.6	29.4	24.9 [a]	24.6 [b]	31.8 [ab]
6.6	3.9	3.8	3.6	3.3	2.0

[a] significant difference
[b] highly significant difference

Table 2. Bond Strengths (MPa) Measured 24-Hours After Initiation					
Clearfil™ SE Bond			OptiBond Solo™ plus		
Uncut Enamel	Cut Enamel	Cut Dentin	Uncut Enamel	Cut Enamel	Cut Dentin
47.0	42.8	38.5	45.3	50.7	51.3
36.3	44.5	43.7	50.6	46.3	41.9
46.2	38.6	39.4	27.1	49.6	55.0
45.7	43.7	41.2	42.4	44.4	49.0
41.1	43.9	38.3	48.7	42.4	49.9
39.9	41.2	37.6	46.7	44.7	54.4
40.0	36.5	43.9	38.6	48.9	42.0
50.1	46.3	41.0	44.8	39.9	50.3
37.4	47.7	41.0	42.1	45.8	46.5
39.5	39.0	38.7	49.3	46.2	44.4
43.8	43.7	36.6	51.0	45.7	45.6
42.5	42.5	40.0 [c]	44.2	45.9	48.2 [c]
4.4	3.4	2.4	6.9	3.1	4.5

[c] highly significant difference

hour specimens. In 24 hours, the average bond strength using Clearfil™ SE Bond increased from 29.4MPa (±3.8MPa) to 40.0MPa (±2.4MPa). In 24 hours, the average bond strength, using OptiBond Solo™ plus, increased from 31.8MPa (±2.0MPa) to 48.2MPa (±4.5MPa).

Conclusion

There was no significant difference between the bond strengths of the two bonding systems either measured immediately or at 24 hours. There was a significant increase in bond strength for both products between immediate bond strengths and 24 hour bond strengths. It appears cleaning the substrate surfaces prior to etching may be very important, especially for self-etching products.

References

1. Barkmeier WW, Erickson RL. Shear bond strength of composite to enamel and dentin using Scotchbond Multi-Purpose. Am J Dent. 1994 Jun;7(3):175-9.
2. Bruke FJ, McCaughey AD. The four generations of dentin bonding. Am J Dent. 1995 Apr;8(2):88-92. Review.
3. Cardoso PE, Braga RR, Carrilho MR. Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems. Dent Mater. 1998 Nov;14(6):394-8.
4. Leirskar J, Oilo G, Nordbo H. In vitro shear bond strength of two resin composites to dentin with five different dentin adhesives. Quintessence Int. 1998 Dec;29(12):787-92.
5. Kanca J 3rd, Sandrik J. Bonding to dentin. Clues to the mechanism of adhesion. Am J Dent. 1998 Aug;11(4):154-9.
6. Van Meerbeek B, Braem M, Lambrechts P, et. al. Evaluation of two dentin adhesives in cervical lesions. J Prosthet Dent. 1993 Oct;70(4):308-14.
7. Van Meerbeek B, Braem M, Lambrechts P, et. al. Two-year clinical evaluation of two dentine-adhesive systems in cervical lesions. J Dent. 1993 Aug;21(4):195-202.
8. Ibsen R, Ouellet D, Strassler H. Clinically successful dentin and enamel bonding. Am J Dent. 1989 Jul;2 Spec No:125-31.
9. Chigira H, Yukitani W, Hasegawa T, et. al. Self-etching dentin primers containing phenyl-P. J Dent Res. 1994 May;73(5):1088-95.
10. Harada N, Nakajima M, Pereira PNR, et. al. Tensile bond strengths of a newly developed one-bottle self-etching resin bonding system to various dental substrates, Dentistry in Japan 36: 47-53.
11. Tay FR, Carvalho R, Sano H, et. al. Effect of smear layers on the bonding of a self-etching primer to dentin. J Adhes Dent. 2000 Summer;2(2):99-116.
12. Duke ES, Robbins JW, Snyder DS. Clinical evaluation of a dentinal adhesive system: three-year results. Quintessence Int. 1991 Nov;22(11):889-95.
13. Elhabashy A, Swift EJ Jr, Boyer DB, et. al. Effects of dentin permeability and hydration on the bond strengths of dentin bonding systems. Am J Dent. 1993 Jun;6(3):123-6.
14. Gwinnet AJ, Kanca JA 3rd. Micromorphology of the bonded dentin interface and its relationship to bond strength. Am J Dent. 1992 Apr;5(2):73-7.
15. REALITY 2000, Number 14, REALITY Publishing Company, Houston TX, USA, 2000. {This is a pamphlet}
16. Talic YF. Method for immediate measurement of in vitro bond strength of bonded direct esthetic restorations. J Contemp Dent Pract 2003 August;(4)3:011-023.
17. Talic YF. Comparative bond strengths of five dental adhesives on dentin. J Pak Dent Assoc 2003; 12(2):69-76.

Acknowledgement
This research was supported in part by The College of Dentistry Research Center, King Saud University, Projects 1734, 1736, 1737, 1738.

About the Author

Yousef Fouad Talic, BDS, MS, FICOI, DICOI, DASO

Dr. Talic is an Associate Professor in the Department of Prosthetic Dental Sciences at King Saud University College of Dentistry, Riyadh, Kingdom of Saudi Arabia and serves as the Director of Postgraduate Dental Education. He is the former Chairman of the Department of Prosthetic Dental Sciences and former Program Director of the Prosthodontics Graduate Program of the same University. His major fields of interests are implants, temporomandibular disorders, denture base resins, composite restorations, and dental adhesive systems.

e-mail: ytalic@ksu.edu.sa