Eur. J. Prosthodont. Rest. Dent., Vol.16, No. 4, pp 166-170
Printed in Great Britain

Bonding of Crown and Bridge Adhesive Resins to Dentine

Saad A Osman*, John F. McCabe† and Angus W.G Walls‡

Abstract - *The shear bond strength of three adhesives, Panavia 21, Superbond, All Bond C&B Cement, and Variolink (a dual cure resin) to various dentine depths were determined. Fifteen human fully erupted permanent first and second molars were wet ground using 500 and then 800 grit abrasive papers to expose the superficial, middle and the deep dentine, for each adhesive tested. Five samples were prepared for each dentine depth. The adhesives were bonded to the samples using gelatine capsules and were matured for 24 h in water at 37^{0}C. The samples were debonded in shear using tensile testing machine at a cross-head speed of 1 mm/ min. The data were analysed using ANOVA and the Tukey test. The fracture surfaces were examined by optical microscopy. The bond strength of Superbond to dentine was significantly higher (P< 0.05) than any of the materials tested. The bond strength of all materials tested was shown to be affected by dentine depth, except for Superbond. Fractured dentine specimens showed that the samples of Superbond are almost cohesive (> 90%), and the samples of other adhesives are mostly adhesive (> 70%). These results confirm that Superbond is capable of forming a bond at various dentine depths.*

KEY WORDS: Bonding, adhesive, tensile test

INTRODUCTION

The bond strength to tooth structure, which depends on the enamel etch technique, has become a routine and reliable aspect of modern restorative dentistry. In crown and bridge applications, however, particularly when dentine has been exposed, the potential for achieving an effective bond is limited[1,2]. An effective bond is essential to offset polymerisation-induced stress[3] encountered in many crown and bridge situations.

The preparation of posterior teeth for crown and bridge applications usually exposes the occlusal dentine, or results in the reduction of significant amounts of occlusal dentine. Studies have shown that occlusal dentine tends to provide lower bond strengths than proximal and buccal dentine[4,5]. Accordingly, in clinical crown and bridgework, the ability of a luting resin to establish an effective bond at different occlusal dentine depths would be of considerable clinical value.

Recently, significant improvements in polymer chemistry have led to the development of several adhesive materials but these adhesives are different in their compositions and their methods of attachment to the tooth surface. The objectives of this work was to investigate and compare the shear bond strength of several adhesive luting agents to dentine, including superficial, middle and deep levels, and to determine the mode of failure using a light stereomicroscope. The adhesive system that provided the highest shear bond strength at various dentine depths was also determined

* MDS, MSc, PhD
† BSc, PhD, DSc
‡ BDS, PhD, FDSRCS

MATERIALS AND METHODS

Shear test specimens were prepared by bonding each of the four adhesive luting agents, Superbond, Panavia 21, All-Bond Crown and Bridge cement and Variolink (Table 1), to dentine.

Twenty extracted noncarious human fully erupted permanent first and second molars were used. After extraction, the teeth were cleaned and stored in 0.5% chloramine, before use. To prepare the dentine surface, the occlusal surface of the molar teeth was removed using 180-grit wet paper on a Metaserv rotary pre-grinder by hand until the dentine surface was almost exposed, leaving only a small amount of enamel showing. The teeth were then mounted in polyester resin, and the polyester was placed in a fume cupboard for 24h until the resin was fully set. For the preparation of the samples for the superficial dentine bond strength measurements, the occlusal dentinal surface of each sample was ground by hand using 500 then 800 grit wet and dry paper, and the surface was examined under a light stereomicroscope to ensure that no enamel remnants were left on the dentine surfaces. The specimens were divided into four groups of five teeth, for bonding with the four adhesives under consideration. After debonding, subsequent tests were performed following the removal of a further 0.5 mm of dentine by grinding with 500 then 800 grit wet paper in accordance to the procedure outlined above to provide five bond strength values for 'middle' dentine and then finally 'deep' dentine making a total 15 samples for each adhesive. A circular hole, 5 mm in diameter was punched in adhesive tape (made from PVC) backed with cellulose acetate sheet, and the tape was attached centrally to the ground surface of the adherend to demarcate the bonding area. The margins of the tape were punched to ensure adaptation of the tape to the surface.

Table 1. *Adhesives used in this study*

Adhesive	*Composition*	*Bat. No*
All Bond C&B Cement	BIS-GMA self-cured Luting composite and Quartz filler *	049297
All Bond	Etchant: 10% H_3PO_4 Primer A: NTG-GMA Primer B: BPDM Adhesive: BIS-GMA, UDMA, HEMA	880016
Panavia 21	MDP monomer, Catalyst, Inorganic Fillers Tooth Primer (Liquid + Liquid B)	41205
Superbond	Monomer (liquid): MMA, 4-META Polymer (powder): PMMA Catalyst: TBB Green Activator: an aqueous solution of 10% citric acid + 3% ferric chloride	60802
Varilink	Dimethacrylate, Fine Particle, Glass filler	814578 (Catalyst) 803085 (Base)
	Email Preparator: etching gel.ntac Primer: TEGDMA	811339
	Syntac Adhesive: PEGDMA	810826
	Heliobond: BIS-GMA	814325
	TEGDMA	803798

BIS-GMA: Bis-phenol glycidyl methacrylate;
NTG: Nitroglycerine
BPDM: Bi-Phenyl dimethacrylate
UDMA: Urethane dimethacrylate
HEMA: 2-Hydroxyethyl methacrylate
MDP: Methacryloyloxydecyl dihydrogen phosphate
MMA: Methyl methacrylate;
4-META: Methacryoxyethyl trimellitate anhydride;
PMMA: Polymethyl methacrylate
TBB: Tri-n-Butyl borane;
TEGDMA: Tetra ethylene glycol dimethacrylate
PEGDMA: Poly (ethylene glycol) dimethacrylate

Table 2. *The shear bond strength to dentine for all adhesives tested and the mode of failure (N=15)*

Adhesive	*Dentine Depth*	*Shear Bond Strength (MPa)*		*Mode of Failure (N=15)*				
		Mean ±SD	*Mean Combined Value*	*A*	*C/D*	*C/R*	*Mixed*	*Mixed Area (%) for Combined Results. Mean ±SD*
Superbond	S	21.3 ± 3.4						
	M	21.3 ± 5.3	20.6 ± 6.1	1	14			
	D	19.2 ± 9.2						
Panavia 21	S	14.5 ± 2.9						
	M	7.3 ± 3.9	10.3 ± 4.5	12			3	48.3 ± 16.1 C/D 51.7 ± 16.1 A
	D	6.9 ± 6.0						
All Bond C&B	S	13.1 ± 1.7						
	M	9.3 ± 5.6	9.6 ± 5.5	11		1	3	40.0 ± 10.0 C/D 60.0 ± 10.0 A
	D	8.5 ± 4.5						
Variolink	S	4.6 ± 2.0		5				
	M&D	0.0 +						

Means that are not significantly different [P > 0.05 (ANOVA and Tukey)] are indicated by vertical lines. Dentine Depth: S: superficial, M: middle, D: deep.

Mode of failure: A: Adhesive, C/D: Cohesive in dentine, C/R: Cohesive in resin. Adhesive marked by + failed before it could be tested.

The test specimens were prepared using #5 gelatine capsules (Eli Lilly and Company Indianapolis, USA) of 4.6 mm diameter. The capsules were vented to allow air to escape and pre-loaded with a visible light cured composite as a space filler (any brand - time expired was used for this purpose) to about two-third of their length, leaving a 1 mm space for the experimental adhesive resin system. This composite was light-cured before the capsule was finally filled with the mixture of the resin luting agent under consideration. All adhesives were used in accordance with the manufacturer's instructions, including conditioning, rinsing and drying of the prepared tooth surfaces. Details of the compositions are provided in Table 1.

After the capsule was seated on the bonding site, a vertical loading apparatus was used to hold the specimens under a constant pressure, and, more importantly, to prevent the specimen from sliding away from the bonding site during polymerisation of the resin. All Bond 2, plus All Bond C & B Cement were used as follows: the dentine surface was etched for 15 s with 'All-Etch' (10% phosphoric acid), then rinsed for 5s and the air syringe was used for 2s to remove excess surface water. One drop each of Primer A and B were mixed and five coats of the mixed primer were applied to the surface and dried for 5s. Pre-bond resin was then applied over the primed surfaces of the dentine with a brush and light-cured for 20s. Finally, the gelatine capsule was filled with All-Bond C&B Cement, after being mixed for 15s, and placed on the prepared surface for bonding. For Superbond, the dentine surface was treated with a solution of 10% Citric Acid and 3% Ferric Acid using a brush. After 10s, the surface of the dentine was rinsed thoroughly and dried with filtered compressed air. To apply Superbond to the dentine 4 drops of monomer, 1 drop of catalyst and a small spoon of polymer were mixed and stirred for 10s. The capsule was then filled with resin and applied to the prepared surface and the excess was removed. For Panavia 21, one drop each of liquids A and B were stirred for 5s using a sponge pledged. The mixture was applied to the bonding surface for 60s and then dried with filtered compressed air. Equal amounts of Panavia 21 resin catalyst and universal pastes were mixed for 30 seconds, and the capsule was filled with the mixture, which in turn was positioned on the bonding site. The excess cement was then removed, and the oxygen-inhibiting gel (Oxygard) was placed around the bonding circle to promote polymerisation of Panavia 21 resin cement. For the Variolink, the dentine surface was etched with Email Preparator (37% phosphoric acid), and then left for 15s prior to rinsing and drying with filtered compressed air. Syntac as a bonding primer was used to condition the dentine with a brush and left for 20s and then light-cured for 20s using a Visilux 2 lamp (power > 800 mW/cm^2) after being dried. A second coat of Syntac was applied to the surface, immediately dried and light-cured for 20s. Syntac Adhesive was applied to the dentine for 15s and air dried, and then after 10s. Heliobond was applied to the dentine, and light cured for 20s. Finally, equal amounts of Variolink catalyst and paste were mixed for 10s and the capsule was filled with the mixture before being applied to the dentine surface and the excess was removed. Following the above procedure, the dentine specimens were allowed to come to room temperature, after being storage for a period of 24 h at 37° C. To determine the shear bond strength, each test specimen was positioned in a shear test jig, to provide a base for testing and debonded using the Instron testing machine at a cross head speed of 1 mm/min and full scale deflection of 1 KN. The data on the mean and the standard deviation was received by the computer after the fracture of the specimens occurred. The shear bond strength was calculated (in MPa) from the load required to cause fracture of the bond (in Newtons) divided by the nominal area of the bonding circle (mm^2). The values of the shear bond strength for each material tested at superficial, middle and deep dentine (5 samples each), and combined dentine depths (15 samples each) were obtained. These data were subjected to a one-way analysis of variance (ANOVA) and Tukey's pairwise comparisons testing, using the Minitab Statistic Computing Package. The Tukey test was used to provide confidence intervals for all pairwise comparisons between mean values for all specimens tested in this study. Test results at levels of $P > 0.05$ were regarded as non-significant. All fractured specimens were examined with a light stereomicroscope to determine whether failures were adhesive (at the adhesive/tooth interface) or cohesive (within adhesive or tooth) or a combination (mixed) of both types of failures. For specimens showing mixed failures, the proportion of adhesive and cohesive fractures was estimated visually.

RESULTS

Mean shear bond strengths and standard deviations (MPa ± SD) for each adhesive group bonded to 'superficial', 'middle' and 'deep' dentine are summarised in Table 2. The data for the mean of combined values of bond strength, irrespective of dentine depth, are also given in Table 2. Statistical data showed that the bond strength of Superbond to various and combined dentine depth was significantly higher than of the other adhesives tested ($P < 0.05$). The mean values of the bond strength among the depths of dentine were not significantly different ($P > 0.05$) for all materials tested, with exception Panavia 2. The values of the bond strength of this adhesive at superficial (14.5 ± 2.9 MPa) and middle (7.3 ± 3.9 MPa) dentine had shown to be significantly different from each another. Fractured dentine specimens showed that the samples of Superbond are almost cohesive (> 90%), and the samples of the other adhesives are mostly adhesive (> 70%).

DISCUSSION

This study showed that the bond strength of all adhesives tested to dentine tended to decrease as the depth of the dentine increase for all the adhesives tested with the exception the Superbond. Previous reports had shown that the bond strength of adhesive resin materials to dentine tended to decrease with increasing dentine depth[6-9]. The consistent high bond strengths to various dentine depths, produced by Superbond in the present study were also reported previously[10-12]. However, it is difficult to compare the results of this study directly with those reported by other workers due to the fact that the bond strength results to dentine can be influenced not only by the material used and the experimental methods[5], but also by other factors. These factors include the tooth storage time[13], storage media, the dentine level and the dentine characteristics[14-16]. The presence of moisture, and the relative humidity[17] can also have a significant influence on the bond strength.

The shear bond strength of 20 MPa that is routinely obtained for enamel bonding has been the goal for dentine bonding for many years[18]. In the present study, the value of 20.6 ± 6.1 MPa for Superbond is similar to the value obtained for enamel[19] and above the value of 16.8 MPa. The latter value has been described as the minimum value required to prevent the formation of gaps between the resin and tooth structure[20]. These gaps are caused by the stresses induced by the polymerisation shrinkage of resin-bond material[21]. It has been suggested that certain monomers, such as 4-META may penetrate and infiltrate into the intra- and intertubular dentine, and combine with the collagen and hydroxyapatite to form a hybrid layer[22].

The inability of All-Bond C&B Cement and Panavia 21 to provide consistently high shear bond strengths to dentine depths is probably related to different factors. Firstly, the resin monomers may have failed to wet fully and penetrate the conditioned dentine surface[23,24]. Secondly, in addition to the differences in chemical composition among the adhesives tested, there may be differences in the quality and morphology of the hybrid layer[25]. These differences in the hybrid layer may also have a significant effect on the bond between these adhesives and the dentine surface[26]. According to the scientific documentation of Variolink in 1993, the bond strength of Variolink in combination with Syntac to dentine is within the range of 5.6 ± 3.0 MPa. The value of 4.6 MPa reported for the bond strength of Variolink to dentine (Superficial dentine), in the present study is within the range of those reported in the scientific documentation. Variolink failed to produce any effective adhesion to middle and deep dentine. The possible explanation for this failure is that Syntac as a primer failed to sufficiently demineralise the peritubular dentine[27]. Thus, the initial wetting process, penetration of the adhesive, and the bond seems to be confined solely to the dentine surface.

In the present study, most of the debonded specimens of Superbond (> 90%) showed cohesive failures within the dentine, where a substantial amount was chipped off, thus requiring a higher shear load to fracture. This is possibly due to the formation of the hybrid layer, which is essential for producing a strong bond between the resin and the dentine[28]. With the exception of Superbond, the proportion of cohesive failures and the bond strengths reduced as dentine depth increased, for all adhesives tested. There was a tendency for the proportion of cohesive failures to increase with increasing bond strength. This is in agreement with previous findings[10] where it was reported that many cohesive failures of resin or dentine in the bond strengths occur over 12 – 15 MPa.

Although clinical recommendations can be made, there are also certain limitations to the test methods that should be considered. The oral environment has many variables and is much more complex than can be replicated in a simple shear test. Tensile, rotational and cyclic forces are also placed on teeth and cast restorations. Temperature changes, as well as other factors in the mouth, may affect the properties of adhesive materials. A further limitation is the sensitivity of the technique in handling and manipulation of adhesive materials, particularly when bonding a resin to dentine. Bonding resins to dentine often produces a wide variance within studies and standard deviations from the mean bond strength (Table 2).

The overall findings of this study was that Superbond was produced a high bond strength that had exceeded the cohesive strength of dentine. This could conclude that the bond strength of adhesive resin luting agents to dentine, particularly when a large amount of dentine has been removed, e.g. in some clinical bridgework, may depend on the choice of adhesive material.

MANUFACTURER'S DETAILS

- All Bond C&B Cement. Bisco Inc., Itasca, Illinois, USA
- All Bond 2. Bisco Inc., Itasca, Illinois, USA
- Panavia 21. Kuraray Co., Osaka, Japan
- Superbond. Sun Medical Co. Kyoto, Japan
- Variolink. Vivadent Ets, Schaan, Liechtenstein
- #5 gelatine capsule. Eli Lilly and Company, Indianapolis, USA

ADDRESS FOR CORRESPONDENCE

John F. McCabe, Professor of Dental Materials Science, School of Dental Sciences, Newcastle University, Newcastle upon Tyne, NE2 4BW, UK. E-mail:j.f.mccabe @ ncl.ac.uk

REFERENCES

1. Aboush, Y.E. and Jenkins, C.B. The bonding of an adhesive resin cement to single and combined adherends encountered in resin-bonded bridge work: an in vitro study. *Br. Dent. J., 1991*; **171**: 166-69.
2. Triolo, P.T., Kelsey, W.P. and Barkmeier, W.W. Bond strength of an adhesive resin system with various dental substrates. *J. Prosthet. Dent.*, 1995; **74**: 463-68.
3. Feilzer, A.J., de Gee, A.J. and Davidson, C.L. Setting stress in composite resin in relation to configuration of the restoration. *J. Dent. Res.*, 1987; **66**: 1636 – 39.
4. Causton, B.E. In vitro assessment of dentine bonding agents: *To Day Dentist* (special Issue)., 1987; 3: 18-27.
5. Oilo, G. and Olsson, S. Tensile bond strength of dentin adhesives: a comparison of materials and methods. *Dent. Mater.*, 1990; **6**: 138-44.
6. Stanford, I.W. A comparison of the effectiveness of dentine bonding agents. *Inter. Dent J.*, 1985; **35**: 139-44.
7. Mitchem, J.C. and Gronas, D.g. Effects of time after extraction and depth of dentine on resin dentine adhesives. *J. Am. Dent. Assoc.*, 1986; **113**: 585-87.
8. McCabe, J.F. and Rusby, S. Dentine bonding agents--characteristic bond strength as a function of dentine depth. *J. Dent.*, 1992; **20**: 225-30.
9. Panighi, M.M., Allart, D., Jacquot, B.M., Camps, J. and G'Sell. Christian. Influence of human tooth cryopreservation on dentine bond strength. *Dent. Mater.*, 1997; **13**: 56-61.
10. Tagami, J., Tao, L. and Pashley, D.H. Correlation among dentin depth, permeability, and bond strength of adhesive resins. *Dent. Mater.*, 1990; **6**: 45-50.
11. Tao, L., Tagami, J. and Pashley, D.H. Pulp pressure and bond strengths of Superbond and Gluma. *Am. J. Dent.*, 1991; **4**: 73 – 6.
12. Pashley, E.L., Tao, L., Matthews, W.G. and Pashley, D.H. Bond strengths to superficial, intermediate and deep dentin in vivo with four dentin bonding systems. *Dent. Mater.*, 1993; **9**: 19-22.
13. Goodis, H.E., Marshall, G.W., White, J.M, Gee, L. and Marshall, S.J. Storage effect on dentine permeability and shear bond strengths. *Dent. Mater.*, 1993; **9**: 79-84.
14. Suzuki, T. and Finger, W.J. Dentin adhesives: site of dentin vs. bonding of composite resins. *Dent. Mater.*, 1988; **4**: 379-83.
15. Perinka, L., Sano, H., Hosoda, H. Dentine thickness, hardness,

and Ca concentration vs. bond strength of dentine adhesives. *Dent. Mater.*, 1992; **8**: 229-33.
16. Bouillaguet, S., Ciucchi, B., Sano, H. and Pashley, D.H. (1994): Influence of dentine thickness and region on tensile bond strength. *J. Dent. Res.*, 1994; 73: 1522. 296 (Abst).
17. Burrow, M.F, Taniguchi, Y., Nikaido T., et al. (1995): Influence of temperature and relative humidity on early bond strengths to dentine. *J. Dent.*, 1995; **23**: 41-5.
18. Barkmeier, W.W., Suh. B.I. and Cooley. R.L. (1991a): Adhesive resin bond strength to dentine and Ni Cr Be alloy. *J. Dent. Res.*, 1991a; 70: 526. 2076 (Abst).
19. Barkmeier, W.W., Cooley, R.L. and Douville, CJ (1991b): Shear bond strength to dentin and Ni-Cr-Be alloy with the All-Bond. *J. Aesthet. Dent.*, 1991b; **3**: 148-53.
20. Munksgaard, E.C. and Asmussen, E. Dentine-polymer bond promoted by Gluma and various resins. *J. Dent. Res.*, 1985; **64**: 1409-11.
21. Davidson, C.L., de Gee, A.J. and Feilzer, A.J. The competition between the composite-dentine bond strength and the polymerization contraction stress. *J. Den. Res.*, 1984; **63**: 1396-99.
22. Nakabayashi, N., Nakamura, M. and Yasuda, N. Hybrid layer as a dentine-bonding mechanism. *J. Aesthet. Dent.*, 1991; **3**: 133-38.
23. Eick, J.D., Cobb, CM., Chappell R.P, et al The dentinal surface: its influence on dentinal adhesion. Part I. *Quintessence. Int.*, 1991; **22**: 967-77.
24. Van Meerbeek, B., Inokoshi, S., Braem, M., Lambrechts, P. and Vanherle G. Morphological aspects of resin-dentine interfussion zone with different dentine adhesive systems. *J. Dent. Res.*, 1992; **71**: 1530-40.
25. Prati, C., Ferrieri, P., Galloni, C., Mongiorgi, R. and Davidson, C.L. Dentine permeability and bond quality as affected by new bonding systems. *J. Dent.*, 1995; **23**: 217-26.
26. Nakabayashi, N., Kojima, K. and Masuhara, E. The promotion of adhesion by the infiltration of monomers into tooth substrates. *J. Biomed. Mater. Res.* 1982; **16**: 265-73.
27. Chappel, R.P., Spencer, P. and Eick, J.D. The effect of current dental adhesives on the dental surface. *Quintessence. Int.*, 1994; **25**: 851-59.
28. Triolo, P.T. and Swift, E.J. Shear bond strength of ten adhesive systems. *Dent. Mater.*, 1992; **8**: 370-74.