Scanned

Shear Bond Strength of Total Etch Two-Step Primer/Adhesive Systems with Dual and Chemical-Cure Resin Cements.

Research Report

Graduate Resident:

Marianna Pasciuta, DDS.

Thesis Advisor:

Deborah Cobb, DDS, MS.

Thesis Committee Members:

Murray Bouschlicher, DDS, MS.
Gerald Denehy, DDS, MS.
Deborah Haselton, DDS, MS.
Marcos Vargas, DDS, MS.

College of Dentistry | Department of Operative Dentistry, Iowa City, Iowa 52242-1001 | 229 Dental Science S | 319/335-7207, Fax 319-337267

2,09,0

INTRODUCTION

- Traditionally, multiple-step adhesive systems, have been used for the cementation of indirect restorations (inlays/onlays/crowns/bridges) and posts. However, with advances in modern dentistry, clinicians are demanding efficient alternatives to multiple-step adhesive systems. The two-step primer/adhesive systems represent a simplified alternative in bonded restorative procedures.
- Recent studies indicate that these light-cure two-step primer/adhesive systems do not adequately bond to chemical-cure resin composite core buildup materials.
- Although dual cure resin cements are typically used for resin and ceramic inlays and onlays, in deep cavity preparations the light may be unable to penetrate through the restoration to adequately polymerize the resin cement at the pulpal or axial interface. Therefore, polymerization would depend upon the chemical cure catalyst in the cement and a similar problem of inhibition between the visible light cure two-step primer/adhesive and the chemical cure resin cement could occur.
- Recently many primer/adhesive systems have added a separate chemical cure catalyst in an attempt to improve the degree of polymerization and bond strengths for indirect applications. It is uncertain if this catalyst enhances bonding of indirect esthetic restorations, particularly in the deeper areas inaccessible to light.
- While the convenience of simplified adhesive systems is desirable, it is important to determine if they provide adequate adhesion at the tooth-restorative interface when used in conjunction with chemical-cure and dual-cure resin cements in light inaccessible situations for the cementation of indirect restorations and posts.

PURPOSE

The purpose of this in vitro study was to compare the shear bond strength of total etch two-step primer/adhesives, total etch dual cure two-step primer/adhesives and a conventional total etch three-step adhesive, when used with a dual and a chemical-cure resin cement. The predominant mode of failure at the adhesive joint, quantified as adhesive, cohesive or mixed was determined by scanning electron microscopy analysis. Additionally, the acidity of all adhesives was measured and associated to the shear bond strength values obtained with the use of a dual and chemical-cure resin cements.

MATERIALS AND METHODS

One hundred sixty non-carious, intact, extracted human molars were used for the study. Teeth were obtained from the Dows Institute for Dental Research at the University of Iowa and were refrigerated for up to 6 months in 0.01% thymol solution to prevent bacterial growth. All specimens were debrided with Gracey curettes 11-12 and 13-14 and transferred to distilled water for at least 48 hours before beginning the experiment.

Teeth were sectioned from their roots using a water-cooled dual wheel model trimmer (Whip Mix Corp.). Buccal surfaces were ground using the trimmer prior to mounting to obtain a flattened enamel surface. Buccal surfaces were used since anatomically they provide a flatter enamel bonding substrate.

Since adhesion to enamel is more predictable and less technique sensitive than adhesion to dentin, enamel was selected as the bonding substrate for the current study to control for variability in the bonding procedures. To specifically test the adhesive-cement interface, the cement was directly bonded onto enamel surfaces, thus reducing the components of the adhesive joint.

Teeth were mounted in a 15-hole specimen teflon mold (25.3 mm diameter x 25.9 mm depth/hole) using a self curing acrylic resin (Harry J. Bosworth Fastray Blue Powder & Liquid, Skokie, IL.) with the flattened buccal surface face down and held in place using double stick tape. Two layers of acrylic resin were used in order to diminish the heat produced by the acrylic polymerization. The first layer was more fluid than the second one in order to reduce the amount of air void formation in the specimen-bonding surface.

Once the acrylic had hardened, specimens were removed from the teflon mold and, exposed enamel buccal surfaces of specimens were ground using a custom rotating-squaring jig (Ultradent Products, Utah) attached to a water-cooled dual wheel trimmer to obtain an even flat enamel surface, then polished with 600 grit silicon carbide grinding paper (Carbimet, Buehler, Lake Bluff, IL.) for a final surface finish in a Ecomet V Polisher (Buehler, Lake Bluff, IL). The teeth were examined with a 30X dissecting light microscope (Bausch & Lomb) to insure that bonding surfaces were on enamel.

Specimen Distribution

Twenty teeth were randomly assigned to one of the following eight adhesive's groups:

Total Etch Two-Step Primer/Adhesives

Single Bond (SB) [3M Dental Products]

One Step (OS) [Bisco]

OptiBond Solo Plus (Solo) without catalyst [Kerr Corporation]

Total Etch Dual-Cure Two-Step Primer/Adhesives

OptiBond Solo Plus + catalyst without light activation of the two components (Solo+C).

OptiBond Solo Plus (light activated) + catalyst (without light activation) (Solo+LC+C).

IntegraBond Dual Cure (IB) [Premier]

Prime & Bond NT Dual Cure (PB) [Dentsply Caulk].

Total Etch Three-Step Adhesive and Control

All Bond 2 (AB2) [Bisco].

Ten teeth in each adhesive group were randomly assigned to a chemical-cure resin cement, C&B Luting Composite (CB) [Bisco]. The other ten specimens were assigned to a dual-cure resin cement, Calibra (Cal) [Dentsply Caulk]. The cements were bonded directly onto the flattened enamel surfaces of each specimen.

Bonding Procedures

A special positioning jig [Ultradent Products, Utah] was used to standardize the bonding procedure. The jig included a vertical sliding upper member and a polyethylene (2.38 mm diameter x 2.5 mm height) mold, which was held against the specimen via two knurled thumbscrews. Enamel bonding surfaces were acid-etched for fifteen seconds, rinsed and air-dried. After the adhesive was applied and the solvent was evaporated following manufacturers instructions, the upper member was slid upward and the specimen was carefully oriented into the jig. Once correctly aligned, the upper member was slid down into position and the two knurled thumbscrews were lightly tightened to secure the polyethylene mold against the bonding surface. For those adhesives requiring light polymerization, the adhesive was then light-cured through the opening in the mold as per manufacturers instructions using an Optilux 500 visible light curing unit (Kerr-Demetron Research Corporation). The intensity of the light was consistently in the range of 550-600 mw/cm^2 and was monitored with a curing radiometer model 100 every 5 specimens (Kerr-Demetron Research Corporation, Danbury, CT).

After adhesive application, the cements were mixed following manufacturers instructions, loaded into a Centrix syringe (Mark IIIp Speed Slot Syringe & Accudose Disposable Tubes. Centrix, Inc. Shelton, CT) and placed directly into the opening of the cylindrical polyethylene mold. Special care was taken to avoid packing the cements above the 45 ° angle of the mold. Any excess cement was removed with an applicator tip (Kerr Applicators, Kerr). To simulate light inaccessible situations, neither of the two cements used were light cured. After the chemical cure setting time for each cement elapsed the bonded specimens were carefully removed from the jig using a condenser to firmly hold down the cement cylinder while the upper member of the jig was slid upward.

All the specimens were stored in distilled water at 37 °C in a humidity chamber (Precision Thelco Model 4) set at 37 °C / 95% for 96 hours. After water storage, all specimens were thermocycled 300 times in a thermocycling machine (Willytec,Haake DC1, Germany) in water baths maintained at 5 °C and 55 °C. The dwell time in each bath was twenty seconds and the transfer time from one bath to the other was ten seconds.

pH Measurement of Adhesives

0.25 ml of each adhesive was mixed and stirred with 0.375 ml of EtOH-H_2O (70:30) solution. The pH of all adhesives without diluting them in ethanol (full strength) was also measured. The pH of all components of all adhesives was measured at ambient temperature (20-25°C) using pH indicator strips non-bleeding pH 0-6 (Colorphast, Darmstadt-Germany). For those systems that require the application of a mixture of two or more components, the ph of the mixture was also determined. pH measurement are reported within a 0.5 range (Table 4).

Shear Bond Testing

Prior to the test, any excess flash of resin cement and/or adhesive was removed from around the cement button using a straight edge razor blade. The blade was gently placed at the base of the cement cylinder and was moved in an outward position to avoid putting any pressure on the specimen.

Specimens were secured in a mounting jig and a semi-circular shearing chisel [Ultradent Products, Utah] attached to the crosshead, was used and aligned parallel to the flattened enamel surface to apply a shearing force at 1mm/min until failure using a Zwick material testing machine (Type:144560, Zwick of America Inc., East Windsor, Connecticut; last calibrated in 7/20/00 under the certification #: NC920336). Shear bond strengths (SBS) were calculated in megapascals (MPa) using the Zwick computer software.

Scanning Electron Microscopy

After shear bond testing, the debonded cement cylinders of all specimens were mounted on aluminum SEM stubs using cyanocrylate. The stubs were gold sputter coated. Failure modes were observed with an Amray 1820 scanning electron microscope (Boston, MA). All specimens were evaluated at 30, 300, 500 and 1000X. The initiation-fracture site was recorded for all specimens and the predominant mode of failure assessed for the different adhesives and cement types.

RESULTS

Shear Bond Strength Test

The table below shows the two-way ANOVA for the main effects (cement, adhesive and the interaction of cement and adhesive). All main effects were significant at $p < .0001$.

Table 1. Two-way ANOVA for the main effects

Main effects	DF	SS	F	Pr>F
Adhesive	7	8069.18	15.61	< .0001
Cement	1	1221.00	16.53	< .0001
Adhesive∗ Cement	7	3101.38	6.00	< .0001
Error	142	10486.71		

Because the interaction of adhesive-cement was significant, other statistical tests were performed to further explain the differences between the cements and the adhesives. Student's t-test showed that only the adhesives, OptiBond Solo Plus ($t = 3.04$, $p = .007$) and OptiBond Solo Plus (without light activation) plus the dual cure component (Solo +C) ($t=3.79$, $p = .0013$) were significantly different when used with the chemical cure vs the dual cure cement.

OptiBond Solo Plus (Solo) and OptiBond Solo Plus (without light activation) plus the catalyst component (Solo+C) had significantly lower bond strength values when used with the chemical cure resin cement C & B compared to the dual cure resin cement, Calibra (Table 3)

Table 2 shows mean shear bond strength (MPa), standard deviations (SD) and sample observations (N) of the adhesives/cements used in this study, listed by adhesive categories.

One way ANOVA for mean shear bond strength (MPa), standard deviation (SD) and Duncan's Multiple Range Test is displayed in Table 3.

<u>For the chemical cure resin cement, C & B Luting Composite, the following results were obtained</u>:

1. IntegraBond (IB), Single Bond (SB) and Prime & Bond NT (PB) had the highest and comparable SBS. IntegraBond and Single Bond had significantly higher shear bond strength than the other adhesives.
2. Prime & Bond NT (PB), demonstrated significantly higher shear bond strength than All Bond (2 AB2), OptiBond Solo Plus+ Catalyst (Solo+ C) and OptiBond Solo Plus (Solo), but was not significantly different from OptiBond Solo Plus (light cured)+ Catalyst (Solo+ LC+C) and One Step (OS).
3. OptiBond Solo Plus (Solo), showed significantly lower SBS than all other adhesives except for OptiBond Solo Plus and the catalyst (Solo+ C).

For the dual cure resin cement, Calibra, the following results were obtained:

1. IntegraBond (IB), OptiBond Solo Plus (light activated) plus the catalyst (Solo+ LC+ C), Prime & Bond NT (PB), OptiBond Solo Plus and the catalyst (Solo + C), Single Bond (SB) and OptiBond Solo Plus (Solo) had similar shear bond strengths and all had significantly higher bond strengths than OptiBond Solo Plus (Solo).
2. IntegraBond (IB), OptiBond Solo Plus (light activated) plus the catalyst (Solo+ LC+ C), Prime and Bond NT (PB) and OptiBond Solo Plus and the catalyst (Solo+ C) had significantly higher shear bond strength than One Step (OS) and All Bond (AB2).
3. OptiBond Solo Plus (Solo) showed significantly lower shear bond strength than all other adhesives except for OptiBond Solo Plus and the catalyst (Solo+ C), which is comparable.

Adhesives pH Measurement

The pH measurement values, for each component of all adhesive systems and the mixed components for those systems that require the mixture of two components, are reported in Table 4.

The most acidic adhesive among all adhesive systems used in this study was OptiBond Solo Plus followed by OptiBond Solo Plus and the catalyst component (Solo+C) and Prime & Bond NT. Single Bond, One Step and All Bond 2 all last three adhesive showed equal acidity. IntegraBond was the least acidic adhesive tested.

Pearson's correlation risk revealed no statistically significant correlation between the adhesives acidity and shear bond strength (probabilility 0.47>0.06).

Scanning Electron Microscopy Examination

Modes of failures reported as adhesive (between enamel/adhesive or between adhesive/cement), mixed adhesive (a combination of adhesive failures between enamel/adhesive and adhesive/cement) and cohesive (in enamel) were reported in Table 5.

All OptiBond Solo Plus groups (Solo, Solo + C, Solo+ LC+ C) when used with the chemical or dual cure resin cement showed a similar pattern of mixed adhesive failure between enamel/adhesive and adhesive/cement, with "voids" in the adhesive and cement surfaces.
The predominant mode of failure for all adhesives/cement combinations was adhesive.

Table 2. Mean Shear Bond Strength (MPa), Standard Deviations (SD), Sample Observations (N): Listed by Adhesive Categories

		Resin Cements			
		C & B		Calibra	
Adhesive Category	Adhesive	Mean SBS±SD (MPa)	N	Mean SBS±SD (MPa)	N
Two-Step Primer/Adhesives	(SB)	32.06 ± 5.20	10	28.60 ± 5.24	10
	(OS)	22.43 ± 3.41	10	23.04 ± 3.23	9*1
	(Solo)	7.41 ± 12.03	3*2	24.79 ± 13.48	8*3
Dual-Cure Two-Step Primer/Adhesives	(Solo+LC+C)	22.60 ± 16.70	7*4	31.55 ± 8.17	10
	(Solo+C)	8.02 ± 13.05	10	29.69 ± 12.51	10
	(IB)	38.39 ± 4.48	10	36.18 ± 5.92	10
	(PB)	29.86 ± 2.66	10	30.75 ± 3.19	10
Three-Step Adhesive [Control]	(AB2)	16.08 ± 2.67	10	16.75 ± 3.39	9*5

*1 =One sample broke accidentally before shear bond testing.

*2 =Seven samples debonded after the cementation procedure while removing the jig before shear bond testing.

*3 = Two samples debonded after the cementation procedure while removing the jig before shear bond testing.

*4 = Three samples debonded after the cementation procedure while removing the jig before shear bond testing.

*5 = One sample broke accidentally before shear bond strength testing.

Note: When the cements are compared within each cement, C&B Luting Composite had ($F= 14.99$, $p< .001$) and Calibra had ($F=5.28$, $p= <.0001$).

Table 3. One Way ANOVA For Mean Shear Bond Strength (MPa), Standard Deviation (SD) and Duncan's Multiple Range Test

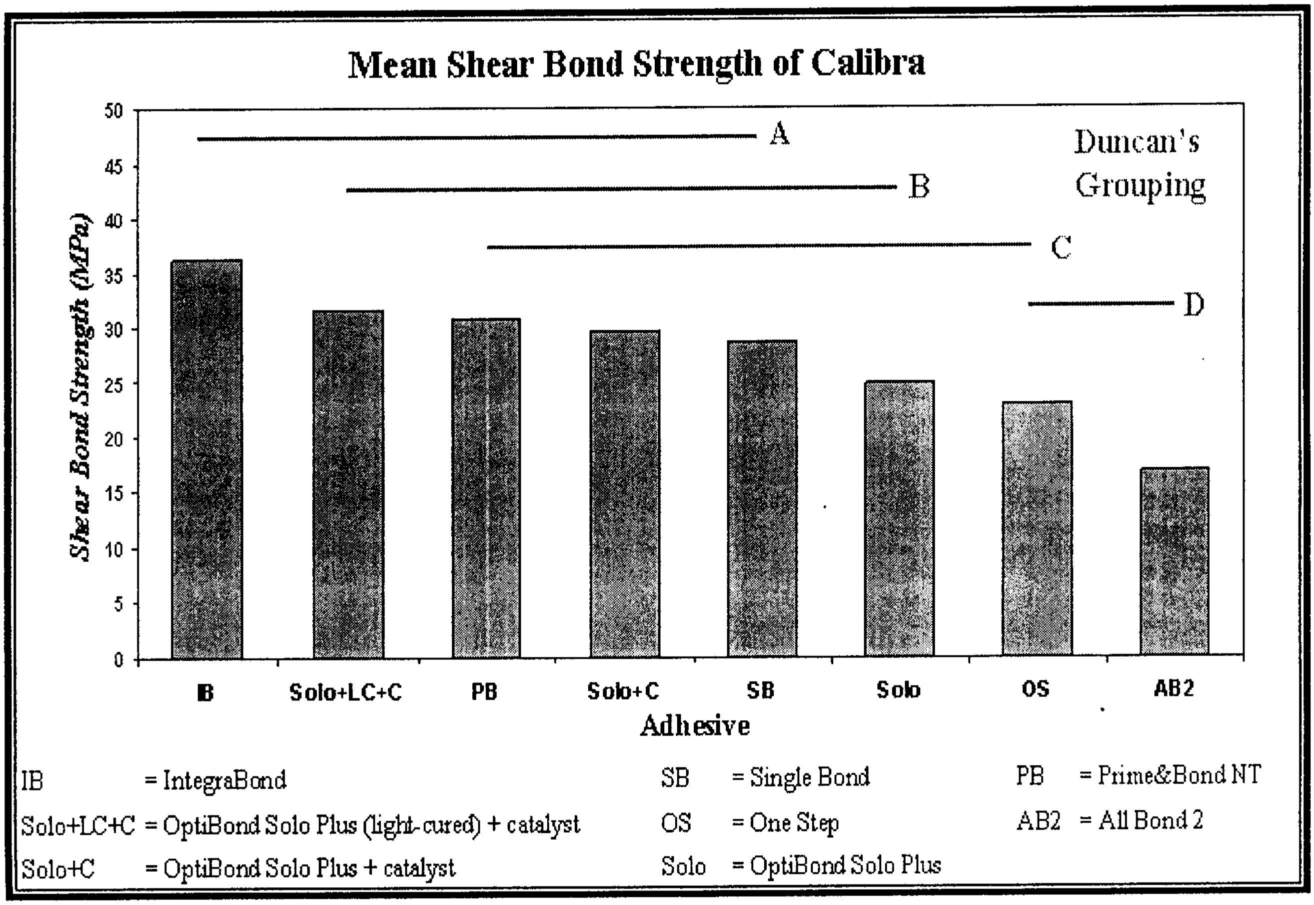
	C & B (Chemical Cure Cement)		Calibra (Dual Cure Cement)		
Adhesive	Mean SBS±SD (MPa)	Duncan's Grouping	Adhesive	Mean SBS±SD (MPa)	Duncan's Grouping
*IntegraBond	38.39 ± 4.48	A	*IntegraBond	36.16 ± 5.92	A B
□Single Bond	32.06 ± 5.20	A	(Solo+LC+C)	31.55 ± 8.17	A B
*Prime&Bond	29.86 ± 2.66	A B	*Prime&Bond	30.75 ± 3.19	A B
* Solo+LC+C	22.60 ± 16.70	B C	**(Solo+C)**	**29.69** ± **12.51**	A B
□ One Step	22.43 ± 3.41	B C	□Single Bond	28.60 ± 5.24	A B C
•All Bond 2	16.08 ± 2.67	C D	**(Solo)**	**24.79** ± **13.48**	B C
*** Solo+C**	**8.02 ± 13.05**	D E	□ One Step	23.04 ± 3.23	C D
□ **Solo**	**7.41** ± **12.03**	E	•All Bond 2	16.75 ± 3:39	D

□ Two-step Primer/Adhesive

* Dual Cure Two-Step Primer/Adhesive

• Three-Component Adhesive

Duncan's Grouping for the Dual Cure Resin Cement Calibra



Duncan's Grouping for the Chemical Cure Resin Cement C & B

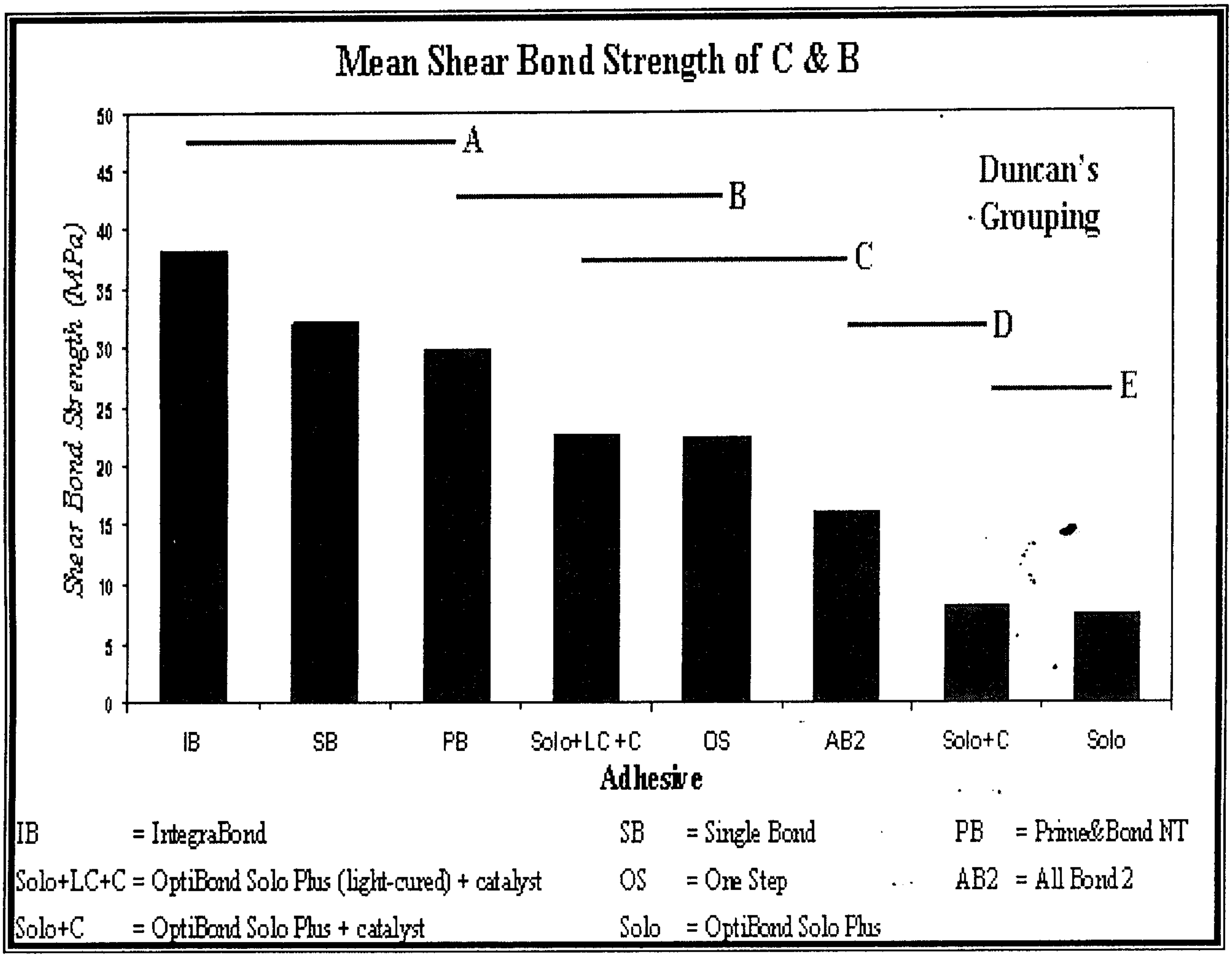


Table 4. pH Values of Adhesives used in this Study

Adhesives	Adhesive+EtOH+H_2O	Pure Adhesive	Other references
Solo	3.0 – 3.5	5.0 – 5.5	2.81*1 / 2.1*3
Solo + Catalyst	3.5 – 4.0	5.0 – 5.5	—
Solo Catalyst	4.5 – 5.0	5.0 – 5.5	—
Prime & Bond NT	4.0 – 4.5	4.5 – 5.0	2.68*1
PB Catalyst	4.5 – 5.0	4.5 – 5.0	—
PB + Catalyst	4.5 – 5.0	4.5 – 5.0	—
Single Bond	4.5 – 5.0	4.5 – 5.0	3.60*1
One Step	4.5 – 5.0	5.0 – 5.5	4.60*1 / 4.55*2
AB 2 Primer A	5.5 – 6.0	5.0 – 5.5	—
AB 2 Primer B	4.5 – 5.0	5.0 – 5.5	—
Primer A + B	4.5 – 5.0	4.5 – 5.0	—
AB 2 D/E	4.5 – 5.0	4.5 – 5.0	—
AB2 Prebond	4.5 – 5.0	4.5 – 5.0	—
AB2 D/E + Pb	4.5 – 5.0	4.5 – 5.0	—
IntegraBond	5.0	5.0	—
IB Catalyst	5.0	5.5	—
IB + Catalyst	5.0	5.0	—

*1: Sanares et al., (2000) *3: Van Meerbeek (unpublished)

*2: Suh et al., (2001)

Table 5. Scanning Electron Microscopy Analysis

Adhesive/Cement	Adhesive Failure (A/E)	Mixed Failure(A/C)	Cohesive Failure (E)
Single Bond+ C&B		100%	
Single Bond+ Cal.		100%	
One Step+ C&B	57.14%	42.85%	
One Step+ Cal.		100%	
Solo+ C&B		100%	
Solo+ Cal.		100%	
Solo+Catalyst+C&B		100%	
Solo+Catalyst+ Cal.		100%	
Solo(LC)+Cat+C&B		100%	
Solo(LC)+Cat+Cal.	30%	70%	
IntegraBond+ C&B	90%		10%
IntegraBond+ Cal.	100%		
P&B NT + C&B	44.4%	44.4%	11.11%
P&B NT + Cal.	55.5%	44.4%	
All Bond 2+ C&B	75%	25%	
All Bond 2+ Cal.	100%		

CONCLUSIONS

For the Chemical Cure Resin Cement C & B

1. A dual cure two-step primer/adhesive IntegraBond, and a two-step primer/adhesive system Single Bond, showed significantly higher shear bond strength than all other adhesives except for a dual cure two-step primer/adhesive (Prime & Bond NT).
2. OptiBond Solo Plus (Solo) had lower shear bond strength than all other adhesives except for OptiBond Solo Plus + Catalyst (Solo+ C).
3. The best results for OptiBond Solo Plus were obtained when using OptiBond Solo Plus (light activated) plus the catalyst component. For the dual-cure cement Calibra no significant difference in shear bond strength was found among the three Optibond Solo Plus groups.

For the dual cure resin cement Calibra:

1. For the dual cure resin cement Calibra, four dual cure two-step primer/adhesives: IntegraBond, Solo (light cured) + catalyst, Prime & Bond NT dual cure, OptiBond Solo Plus + Catalyst; and a two-step primer/adhesive Single Bond, had comparable shear bond strength and all but Single Bond had significantly higher shear bond strength than the two-step primer-adhesive One Step and the control All Bond 2.
2. The three-step adhesive system All Bond 2 showed significantly lower shear bond strength than all other adhesives except for One Step, which was comparable when used in conjunction with the dual cure cement.