Comparison of the Flexural Strength of Five Adhesive Resin Cements

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Statement of Problem: The increased use of adhesive resin cements in bonded prosthetic restorations has led to restorations debonding under function.

Purpose: This investigation evaluated the differences in the flexural strength of new adhesive resin cements as a function of specimen age and storage condition.

Materials and Methods: Four new dual-cure cements were compared to C/B Metabond. Twenty specimens of each of the five cements were prepared in a rectangular glass mold (25 × 2 × 2 mm). The new cements were light-activated with a 550 mW/cm^2 lamp for 80 seconds on both the top and bottom surfaces. The auto-cured cement was allowed to set according to manufacturer's directions. Half the specimens were tested immediately after curing while the other half were stored in distilled water at 37°C for 30 days. A three-point bending test was performed using an Instron at a crosshead speed of 1 mm/min. The results were analyzed by analysis of variance and Scheffé tests ($p < 0.05$) to examine the effect of specimen age and storage condition.

Results: RelyX ARC exhibited a significantly higher flexural strength compared with Calibra and Panavia F when tested immediately. The standard cement, C/B Metabond, deformed and did not fracture at the immediate test time. After storage, the flexural strength had significantly improved from the immediate test time for Calibra, Cement-It, Panavia F, and C/B Metabond. However, there were no significant differences in the flexural strength among the cements when tested after 30 days in water at 37°C.

Conclusion: Immediately after curing, these new adhesive resin cements are not equivalent, as evidenced by the significant variability in the measured flexural strength. The distinctions among the cements diminish after aging in water, which may be due to residual polymerization or a plasticizing effect from water absorption.

Clinical Significance: When light-cured, all the new adhesive resin cements have greater early strengths than the auto-cured cement; however, the wide variation in immediate bending strength suggests that some cements may be more appropriate for use in high-stress clinical situations such as resin-bonded fixed partial dentures.

J Prosthodont 2007;16:18-24.

INDEX WORDS: dual-cure resin cements, resin bonded fixed partial dentures, auto-cured cement

THE ADVENT OF esthetic dentistry has led to numerous applications for adhesive resin cements. According to Diaz-Arnold et al,[1] these cements, based on resin composite technology, have been used for retaining crowns on short tapered preparations and metal resin-bonded fixed partial dentures (FPDs). They are also the adhesive of choice for esthetic ceramic or composite restorations including inlays, onlays, veneers, crowns, FPDs, and fiber-reinforced composite restorations. Chemically polymerized adhesive resin cements have been clinically used for bonding resin-bonded FPDs for many years.[2] Li and White[3] studied the mechanical properties of luting agents and suggested that their successful use of this application was due in part to superior adhesion, but may also be due to high toughness measured from energy absorption tests. Elastic as well as plastic deformation under a compressive load can

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Accepted September 15, 2005.

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1059-941X/06

doi: 10.1111/j.1532-849X.2006.00151.x

be considered an important failure mechanism of the retentive ability of luting cements.[4] White and Yu reported C/B Metabond demonstrated "flexibility" during testing at early set times and low bond strength during the early gel-like phase.[2] They suggested that C/B Metabond should not be used as a luting agent in fixed prosthodontics due to considerable plastic deformation that adversely lowered its early compressive strength comparable to conventional luting agents.[2]

The basic composition of most modern resin cements is similar to that of resin composite filling materials. Polymerization is achieved by a peroxide-amine induction system or light activation. A few systems use both mechanisms and are referred to as "dual-cure" cements.[5] Darr and Jacobsen[6] evaluated the conversion of various dual-cure and chemical-cure luting cements under test conditions of light-cure only. Their results suggested that the luting agents were inefficiently cured by either mechanism during the early stages of restoration placement. Wide variations in physical and mechanical properties are found among the commercially available adhesive resin cement products. In addition to the effects of the different setting mechanisms, the properties of adhesive resin cements vary considerably due to compositional differences such as the type and amount of the diluent monomer and the type, size, and quantity of filler particles.[7]

One of the most widely studied properties is bond strength. Several test methods have been used to measure bond strength at different interfaces, such as between the resin cement and the dental alloys or resin composites and between the adhesives and the tooth structure, either enamel or dentin. Many tests are conducted with the specimen stressed in shear.[8] Investigations have tested the bonds in tension.[9] Neither of these testing modalities reflects the true clinical situation because of the differences between geometry of the test specimens and the actual dimensions of the clinical application, the ramifications of working on extracted teeth, the impact of specimen preparation for the test, and the influence of the testing apparatus.[10]

As a general rule, bond strength values overestimate the actual bond strengths manifested in the clinical application.[11] The flexural properties of many materials may actually be more important than their tensile, shear, or compressive strengths, because resin-bonded FPDs are more likely to be subjected to bending forces than to the other types of stresses.[12] Large differences in flexural strength have been reported for luting cements.[3] The flexural strength values reported for light-cured composites tested in accordance with International Organization for Standardization (ISO) specification 4049 have been reported to be between 86 ± 5 and 155 ± 7 MPa.[13,14] Since an adhesive resin cement's ability to resist bending under an applied flexural load may be an indicator of its potential for success in the oral environment, the differences observed in these cements may have clinical implications.[3,15] Resin cements as a group are virtually insoluble in the oral environment.[16] Prior to testing, specimens are typically stored in water and undergo thermocycling to simulate the oral environment.[11] The effect of these storage conditions has been a significant decrease in bond strength when subjected to a longer storage time.[17] A study by Diaz-Arnold et al[17] evaluated the tensile bond strength of three adhesive resin cements with storage conditions of 2 versus 30 days, and thermocycled versus nonthermocycled storage. The results indicated strong bond strengths for all three materials, but observed that the longer storage time significantly decreased the bond strengths of two of the materials tested.

Ferracane et al[18] studied dental composites and the effect of water, aging, changing degree of conversion, filler volume, and filler/matrix coupling. The results demonstrated that the initial properties of composites are significantly influenced by the three variables: degree of cure, filler volume, and percentage of silane-treated filler. The flexure strength of the composites generally increased with degree of cure and, to a lesser extent, with increased filler volume and the percentage of silane-treated filler. Results of this study indicated that long-term aging in water had little influence on flexure strength.

Kim et al[19] looked at filler morphology and loading on the mechanical properties of composites. The composites with the highest filler by volume exhibited the highest flexural strength.

Ferracane's[18] study showed that long-term aging in water slightly but significantly reduced fracture toughness for all of the composites and reduced microhardness for about half the composites, but had less of a long-term effect on elastic modulus and flexural strength. There is only limited degradation of composites in the water medium. Other solvents may be more aggressive and produce different results, especially if cyclic loading were to occur.

Ferracane[18] hypothesized that water sorption causes a softening of the polymer resin component by swelling the network and reducing the frictional forces between polymer chains. Once the network is saturated with water and becomes softened, the composite structure stabilizes and there is no further reduction in properties within the time frame studied. This limited reduction in properties provides evidence that further degradation, such as filler/matrix interfacial hydrolysis or polymer matrix crazing, may be absent or may not continue significantly once the composite has become saturated and remains wet.

The purpose of this in vitro study was to compare the flexural strength of four recently introduced dual-cure adhesive resin cements and a standard autopolymerizing material using the methodology outlined in the ISO 4049[20] specification for resin-based materials. In particular, this investigation sought to determine if the initial strength of the materials after light exposure was equivalent for all the cements and to study what, if any, effects long-term water storage had on the flexural properties of these adhesive resins.

Materials and Methods

The materials selected for the investigation represent four recently introduced adhesive resin cements and an autopolymerizing material to be used as a standard. These adhesive resin cements are listed in Table 1.

A rectangular mold (25 × 2 × 2 mm) was made of glass slides and used to fabricate 20 specimens from each of the five adhesive resin cements. A custom jig was made to facilitate fabrication of multiple accurate rectangular glass molds. A second custom jig was made to support the rectangular mold during specimen fabrication (Fig 1). A water soluble separator[21] was used to lubricate the mold for ease of specimen removal. The molds were prepared in advance and maintained at a temperature of 37°C to simulate mouth temperature. The manufacturers' mixing directions for the cements were followed. Adhesive resin cement was mixed, placed in the rectangular portion of the mold, covered with a glass slide, then cured for 80 seconds from the top. The mold was removed from the supporting jig and cured for 80 seconds from the bottom to ensure adequate cure.

Once a specimen was cured, the glass slides could be separated with a #21 blade, the specimen retrieved, and any excess material carefully removed. All specimens were visually inspected to verify the absence of voids. Any specimen exhibiting voids that could be reasonably assumed to adversely affect load values was eliminated from strength calculations. All specimens with voids were removed prior to testing so the sample size remained at 20. Ten of the finished specimens were immediately placed in a container of distilled water and stored at 37°C for 30 days; the other ten specimens were tested immediately upon removal from the mold. One operator made all the specimens. To allow for any effect of a handling bias, only three to five specimens were made from a cement at any lab period. The operator then switched to another cement until 20 specimens were completed. Thus, the order of making the test specimens was randomized.

The polymerization reaction for light-cured composites is affected by the intensity of the light.[22,23] The output of the two curing lights (Demetron Optilux 500, Kerr Corp., Danbury, CT) was checked prior to beginning the specimen preparation and frequently throughout the fabrication process by the curing light's built-in digital radiometer. The output of each curing light was 550 mW/cm^2. The lights were used in alternating fashion to ensure consistent light output.

A three-point bending device was custom fabricated according to ISO specification 4049[20] (Fig 2). Two platforms were fabricated in wax and cast in Vitallium (Austenal, Chicago, IL). The platforms were evaluated for parallelism on a standard parallelometer (Stanley Tools Group, New Britain, CT) to ensure uniformity. The lower platform is a base measuring 35 × 10 × 3 mm and has two round bars, 2 mm in diameter spaced 20 mm apart. The upper platform has the same base with a single rod centered halfway between and parallel to the rods in the lower platform. Three white reference dots were placed at half the width of the lower platform, at each end and center, to allow for fast visual alignment of the specimen in the testing apparatus. The test apparatus was attached to an Instron Universal Testing Machine, Model 1011 (Instron Corp., Canton, MA) that was calibrated prior to each testing session. A compressive load was applied to the specimen at a crosshead speed of 1 mm/min using a 50-kg load cell.

The light-cured, immediately tested specimens were made as previously described. Immediately after autopolymerizing for 10, 20, 30, and 60 minutes, C/B Metabond specimens were tested in the specimen testing apparatus to load to failure.

The stored specimens were removed from water and tested wet, after blotting dry. A black reference dot was placed at each end of the specimen to indicate the top for subsequent reorientation during evaluation. The bending data were recorded as load to failure. After fracture, the thickness and width of all specimens were measured with a digital micrometer. The flexural

Table 1. Materials Used

Cement	*Manufacturer*	*Batch Number*	*Components*	*Setting Reaction*
C&B Metabond	Parkell, Farmingdale, NY	990561	Base—MMA monomer, inhibited; powder—PMMA; dentin activator—ferric chloride solution; citric acid; polyvinyl alcohol; water	Auto-cure
Calibra	Dentsply Caulk, Milford, DE	9906223-9905072	Bis-GMA/triethylene glycol dimethacrylate resins and titanium dioxide; silica fume	Dual-cure
Cement-It	Jeneric/Pentron Wallingford, CT	22767	Base and catalyst-resins: Bis GMA; UDMA; HDDMA; silane fillers: barium glass, inorganic fluoride, borosilicate glass, silica zirconia; amine and inorganic pigments—BASE only; benzoyl peroxide-catalyst; UV stabilizers-base and catalyst	Dual-cure
Panavia F	Kuraray Co., Ltd., Osaka, Japan	61132	A Paste: silanated silica, colloidal silica, hydrophobic aromatic dimethacrylate, 10-methacryloxydecyl dihydrogen phosphate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, benzoyl peroxide B Paste: silanated barium glass, silanated titanium oxide, sodium fluoride, colloidal silica, hydrophobic aromatic dimethacrylate, hydrophilic dimethacrylate, hydrophobic aliphatic dimethacrylate, n,n'-diethanol-p-toluidine, sodium aromatic sulfinate	Dual-cure
RelyX ARC	3M ESPE, Minneapolis, MN	19990616	A Paste: zirconia silica filler 65-75% by wt., triethylene glycol dimethacrylate, bisphenol A diglycidyl methacrylate, dimethacrylate polymer, pigments B Paste: zirconia silica filler 60-70% by wt., triethylene glycol dimethacrylate, bisphenol A diglycidyl methacrylate, dimethacrylate polymer	Dual-cure

strength was calculated from the fracture load and specimen dimensions according to the following formula:[7]

$$\text{Stress} = \frac{3 \times \text{fracture load} \times \text{length}}{2 \times \text{width} \times \text{thickness}^2}.$$

At fracture, all specimens were visually inspected to verify the absence of voids. Any specimen exhibiting voids that could be reasonably assumed to adversely affect load values was eliminated from the study. New specimens were made and aged to keep the number of specimens constant.

Statistical Analyses

The mean and standard deviation were calculated for each group. For the two experimental groups, light-cured/immediately tested versus light-cured/stored, a

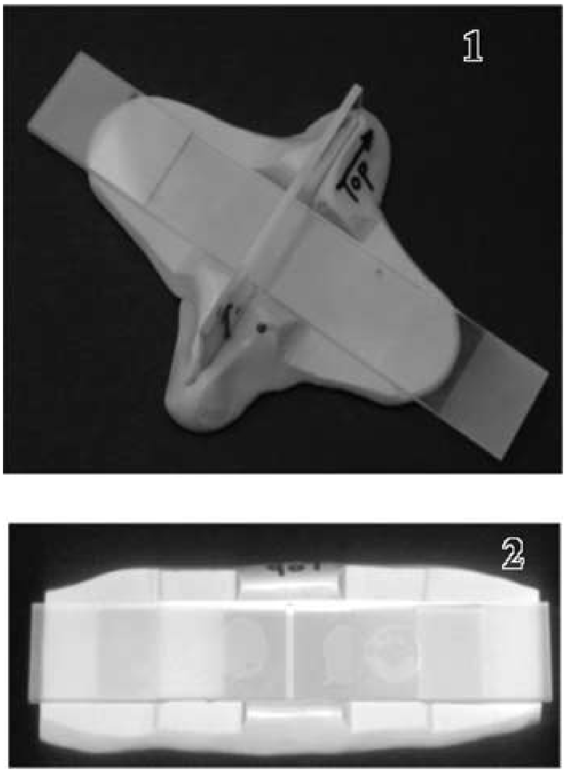

Figure 1. A custom jig was made to facilitate fabrication of multiple accurate rectangular glass molds. A second custom jig was made to support the rectangular mold during specimen fabrication.

two-way analysis of variance (ANOVA) was performed to determine if significant differences ($p < 0.05$) existed within and between the groups. The Scheffé multiple comparison test was completed to determine which materials were similar ($p < 0.05$). C/B Metabond was excluded from this evaluation because the material failed to fracture after 1 hour from mixing.

In addition, the results from the new cement specimens that had been stored were compared to the autopolymerizing cement specimens stored under identical conditions. A one-way ANOVA was performed to determine if significant differences ($p < 0.05$) existed among materials.

Results

Results comparing the immediate test condition and the stored test condition for all four light-cured cements are listed in Table 2. C/B Metabond specimens tested immediately after autopolymerizing for 10, 20, 30, and 60 minutes exhibited bending rather than fracture. Thus,

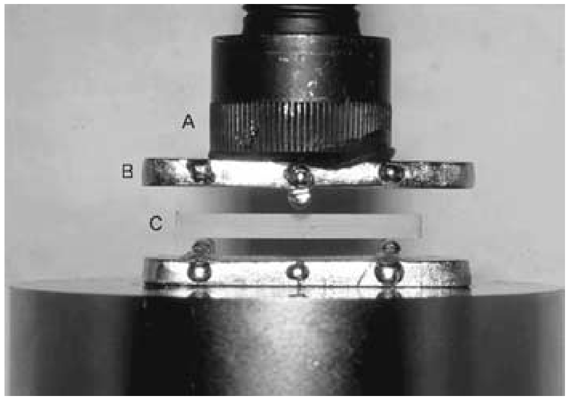

Figure 2. Three-point bending apparatus. (*A*) Instron Model 1011, crosshead speed 1 mm/min using a 50-kg load cell. (*B*) Three-point bending device, 35 × 10 × 3 mm base with 2 mm bars cast from Vitallium. (*C*) 25 × 2 × 2 mm rectangular cement sample. Mold used to fabricate sample makes a line in sample.

results from this cement were not included in this test condition. The two-way ANOVA ($p < 0.05$) and Scheffé test showed that there was a high significant difference between the flexural strength of RelyX ARC (137 ± 15 MPa) and two of the other dual-cured cements, Calibra (100 ± 19 MPa) and Panavia F (94 ± 15 MPa), when the materials were light-cured and tested immediately. However, under the immediate test conditions, there was no significant difference in the flexure strength values obtained for Calibra, Cement-It, and Panavia F.

The five cements that were stored in water for 30 days at 37°C, when C/B Metabond was tested (flexural strength 135 ± 20 MPa) and analyzed with the one-way ANOVA ($p < 0.05$), demonstrated no significant difference among any of the cements. There was no significant difference in

Table 2. Results of Flexure Strength Testing

	Immediately After Light-Curing (MPa)	*Stored 1 Month After Light-Curing (MPa)*
Materials		
RelyX ARC	137 ± 15^{a}	163 ± 16^{c}
Cement-It	$107 \pm 19^{a,b}$	$166 \pm 39^{c,*}$
Calibra	100 ± 19^{b}	$150 \pm 19^{c,*}$
Panavia F	94 ± 15^{b}	$135 \pm 19^{c,*}$

Lowercase superscript letters indicate no significant difference among values within treatments.
*Indicates significant difference between values of different treatments.

the flexural strength among any cement tested after 30 days in water at 37°C. RelyX ARC was the only cement that was not significantly affected by storage in water at 37°C for 30 days. Calibra, Cement-It, and Panavia F exhibited a significant increase in flexural strength as an effect of storage in water at 37°C for 30 days.

Discussion

Tested under the immediate loading conditions, C/B Metabond did not exhibit enough early strength to be loaded until fracture; the specimen deformed until it made contact with the testing apparatus base. Thus, results from this cement were not included in the immediate test condition.

The wide variability in flexural strength (94 ± 15 to 137 ± 15 MPa) observed for the adhesive resin cements in our study compare favorably with the flexural strength values reported for light-cured composites (86 ± 5 to 155 ± 7 MPa) tested in accordance with ISO specification 4049.[20] The differences detected in the range of values could be due to the differences in composition and, in particular, the filler content between these materials. White and Yu reported increased filler content to be related to increased compressive and diametral strengths.[2]

The results of our study would tend to confirm that C/B Metabond exhibits considerable plastic deformation during early stages of setting. Large differences in flexural behavior among cement types may have clinical implications, considering that resin composite cements are used for adhesive-bonded restorations where ultimate strength and energy absorption are essential, according to Li and White.[3] From our study, the cement that exhibited significantly different flexural strength at the immediate test condition suggests that RelyX ARC, which had the greatest early strength may be a more appropriate material for adhesive bonded restorations that have minimal resistance and retention form. Attar et al[24] studied mechanical and physical properties of five contemporary dental luting agents and found that photopolymerization of the resin-based cements was necessary to maximize strength and rigidity; therefore areas where light cannot reach all of the cement may reduce mechanical properties. Further study of these new adhesive resin cements is necessary to consider all the properties that affect the clinical situation.

In this study, the flexural strength of RelyX ARC was not significantly influenced by changes due to long storage; however, the other three new adhesive resin cements, Calibra, Cement-It, and Panavia F, did exhibit a significant increase in flexural strength after storage for 1 month at 37°C. One explanation could be that significant residual or continued polymerization occurred for these cement specimens. This assumption would also seem to be true for the autopolymerizing material, C/B Metabond. Ferracane et al's[18] study on aging and composites also found that initial properties of composites are significantly influenced by three variables: degree of cure, filler volume, and percentage of silane-treated filler. The flexure strength of the composites generally increased with degree of cure, and to a lesser extent, with increased filler volume and the percentage of silane-treated filler.[18]

There were no significant differences among flexural strengths for all the cements following storage in water at 37°C for 30 days. Ferracane et al[18] found that that long-term aging in water had little influence on flexure strength. RelyX ARC and Cement-It exhibited greater flexural strength than C/B Metabond; RelyX ARC exhibited greater flexural strength than Panavia F, but these differences were not significant under the testing conditions. This observation would tend to suggest that as the adhesive resin cements age, any difference in regard to clinical implications would appear to be less critical; however, in clinical situations where bending forces are anticipated, adhesive resin cements exhibiting a high immediate flexural strength might be preferred. A clinical trial comparing the behavior of adhesive resin cements and bending forces would have to be undertaken to conclusively say that high immediate flexural strength is preferred.

Conclusions

Within the conditions of this in vitro study, one may conclude the following: in vitro, RelyX ARC had the highest immediate flexural strength. Immediately after curing, these new adhesive resin cements are not equivalent, as evidenced by the significant variability in the measured flexural strength. The distinctions among the cements diminish after aging in water, which may be due to residual polymerization or a plasticizing effect from water absorption.

Clinical Significance

When light-cured, all the new adhesive resin cements have greater early strengths than the autocured cement; however, the wide variation in immediate bending strength suggests that some cements may be more appropriate for use in high-stress clinical situations, such as resin-bonded FPDs.

Acknowledgments

The authors would like to express their gratitude to the following: Dentsply/Caulk, Milford, DE for Calibra; Jeneric/Pentron Wallingford, CT for Cement-It; Kuraray Co., Ltd., Osaka, Japan for Panavia F; Parkell, Farmingdale, NY for C/B Metabond; 3M ESPE, Minneapolis, MN for RelyX ARC. This study was supported by the ACP 3M ESPE Fellowship in Geriatric Prosthodontics.

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