

Flexural Strength, Elastic Modulus, and pH Profile of Self-etch Resin Luting Cements

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Abstract

Purpose: To determine the flexural strength, modulus of elasticity, and 24-hour pH profile of three self-etching resin luting cements and to obtain comparative data for representative conventional resin and resin-modified glass ionomer luting cements.

Materials and Methods: Three self-etching resin luting cements [RelyX Unicem (3M ESPE), Maxcem (Kerr), Embrace Wetbond (Pulpdent)] were tested and compared with two conventional resin cements [RelyX ARC (3M ESPE), Linkmax (GC)] plus two resin-modified glass ionomer luting cements [Fuji Plus (GC), RelyX Luting Plus (3M ESPE)]. Flexural strength and modulus of elasticity were determined using bar-shaped specimens ($2 \times 2 \times 25 \text{ mm}^3$) at 24 hours, using an Instron universal testing machine. Setting pH was measured using a flat-surface pH electrode at 0, 2, 5, 15, and 30 minutes and 1, 2, 4, 6, and 24 hours after mixing. Testing was performed under both dual-cured and self-cured conditions for all dual-cure cements. Data analysis included ANOVA and Tukey's test (p < 0.05).

Results: The self-etching cements showed similar flexural strength to the conventional resin cements, except for Embrace Wetbond self-cured, which was considerably lower. Modulus of elasticity results were both higher and lower than for conventional resin cements. All photopolymerized conventional and self-etch dual-cure cements showed markedly higher flexural strength and modulus than when solely self-cured. The resinmodified glass ionomer cements were characterized by lower flexural strength and elastic modulus. Self-etching resin cements showed lower initial pH (2.0 to 2.4) than conventional resin cements (4.8 to 5.2) and a wide range of final pH values (3.9 to 7.3) at 24 hours. One self-etching cement (Unicem) revealed a unique pH profile characterized by a more rapid rise in pH to neutrality both when dual-cured (15 minutes) and when auto-cured (1 hour).

Conclusions: The self-etching resin cements evaluated in this study displayed disparate properties and cannot be considered a homogeneous group. Flexural strength properties were most uniform and were similar to those of the conventional resin cements, whereas moduli of elasticity showed greater variation. Setting pH profiles differed, depending on the brand and mode of cure, even within the same category of luting cement. All cements with dual-cure capability, both conventional and self-etch, showed significantly superior properties when photopolymerized.

Resin luting cements are necessary for the cementation of the vast majority of esthetic porcelain, ceramic, and indirect composite restorations. Resin cements generally have superior mechanical properties,^{1,2} provide increased retention in low-retention clinical situations,³ and can increase the fracture resistance of overlying ceramic materials;⁴ however, the use of resin luting agents with the related bonding requirements is technique-sensitive⁵ and is associated with a higher incidence of postoperative sensitivity. Polymerization shrinkage may lead to cuspal deformation, bond failure, microleakage, and postoperative sensitivity.^{6,7} Multipurpose resin luting agents with self-etch adhesive capability have relatively recently been introduced as an alternative to conventional luting cements. The self-etch capability eliminates the need for separate etching, priming, and bonding steps in an attempt to simplify the cementation procedure, reduce technique sensitivity, and improve clinical success. The number of such proprietary self-etching cements on the market is currently increasing, and independent studies of basic material properties are necessary to characterize these new materials. Although some information is available,^{1,8} there are currently no published studies that have investigated the properties of these self-etching cements as a group. Comparative information, in relation to the properties of known conventional resin cements, would help characterize this new group of cements and provide some necessary information for the clinician.

Flexural strength and modulus of elasticity are useful basic parameters for the assessment of mechanical characteristics of dental materials. The failure potential of a cemented restoration under applied forces is related to the mechanical properties of the individual parts, and flexural strength and elastic modulus are important properties with regard to the ability of the cement to resist stress without fracture and/or permanent deformation. It is also considered of value to know the pH change of the cements with time during setting. The incorporation of acidic, self-etching capability changes the customary physicochemical characteristics of a resin cement and deserves some consideration with relation to biocompatibility and resin conversion.

The objective of this study was to provide information on mechanical and physical properties of self-etching resin cements by comparing their flexural strength, modulus of elasticity, and pH profiles with those of representative commercial examples of conventional resin and resin-modified glass ionomer cements. The hypothesis that self-etching universal resin luting cements would have different physical and mechanical properties compared to the traditional luting cements was tested. Differences in physical and mechanical properties of cements may impact the clinical performance of luting agents, and the knowledge of such differences will aid in material selection.

Materials and methods

The materials used in this study are listed in Table 1. Selfetch resin, conventional resin, and resin-modified glass ionomer luting cements were compared. To maximize standardization, one person prepared the specimens and conducted the testing. The materials were mixed and dispensed in accordance with the manufacturer's instructions. GC Fuji Plus (2:1 powder: liquid by weight) and RelyX Luting Plus were mixed for 20 seconds. Conventional resin cements RelyX ARC and Linkmax were mixed for 10 seconds. RelyX Unicem was enclosed in capsules (maxicaps) and mixed using an amalgam triturator (Vari-Mix II, Dentsply Caulk, Milford, DE) at high setting for 15 seconds. Mixing tips were used to dispense Maxcem. Embrace Wetbond contained in standard syringes was mixed with a spatula for 20 seconds.

Flexural strength

Flexural strength was determined according to ISO Standard-4049, except that the stainless steel molds were replaced with equivalent molds made from silicone impression material (Aquasil LV, Dentsply Caulk) to facilitate specimen demolding without specimen fracture. Rectangular bar-shaped specimens $(25 \times 2 \times 2 \text{ mm}^3)$ were prepared by placing the freshly-mixed luting cement into the silicone mold on a glass slide lined with a transparent polyester film. A second microscope glass slide lined with clear film was placed over the mold, and the whole unit clamped securely. Eight specimens were prepared for each cement/shade (n = 8). The self-etch and conventional resin cement specimens were either allowed to "self-cure" or were photopolymerized for the "dual-cure" mode. For the selfcure specimens, an opaque plastic card was placed between the polyester film and the glass slide to block out the ambient light. All self-cure specimens were stored in the dark at 37°C for a period of 60 minutes before removal from the molds. Dual-cure specimens were exposed to intense photopolymerization using a halogen light source (Triad 2000 light cure unit, Dentsply International, York, PA), at a distance of approximately 4 cm for 1 minute. The photopolymerized specimens were removed from the molds 15 minutes following the exposure to the light source. The specimens were checked for visible defects and, if necessary, polished using 320-grit abrasive paper to remove any flash. The specimens were stored in distilled water in the dark at 37°C until testing. After 24 hours, the height and width of the specimens were measured to an accuracy of 0.01 mm using a digital micrometer (Mitutoyo digimatic caliper 500-136, Kawasaki, Japan). The mean of three measurements was noted.

Table	1	Lutina	cements	used
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Commercial name	Туре	Manufacturer	Shade	Lot number
GC Fuji Plus	Resin-modified glass ionomer	GC America, Alsip, IL		Powder 0406031 Liquid 0406041
RelyX Luting Plus	Resin-modified glass ionomer	3M ESPE, St Paul, MN		AP4AM
RelyX ARC	Dual-cure conventional resin	3M ESPE	A3	20050519
Linkmax	Dual-cure conventional resin	GC America	Clear	0406092
			A3	0406111
Unicem	Dual-cure self-etch resin	3M ESPE	Translucent	202316, 209177
			A3	202491
Maxcem	Dual-cure self-etch resin	SDS Kerr, Orange, CA	Clear	423532, 428596
			Yellow	429303, 432541
Embrace Wetbond	Dual-cure self-etch resin	Pulpdent, Watertown, MA	Universal	040112, 050415

The 3-point-bend flexural strength test was performed using a universal uni-axial servo-mechanical testing machine (Model 3401, Instron Corp., Canton, MA). The apparatus consisted of two lower rods mounted parallel and 20 mm apart and a third centered upper rod for load application. The load was applied at a crosshead speed of 0.75 mm/min. A chart plotter traced the load-deformation profile. Flexural strength, F, was determined using the following equation:

$$F = 3P_f L/2WH^2$$

where P_f is the measured load exerted on the specimen at the point of fracture, L is the distance between the supports on the tension surface (20 mm), W is the mean specimen width, and H is the mean height of the specimen. The data were analyzed statistically using analysis of variance (ANOVA) and Tukey's test (p < 0.05).

Modulus of elasticity

Modulus of elasticity, E, was determined from the loaddeformation profiles generated during the 3-point-bend flexural strength testing as described previously using the following equation:

$$\mathbf{E} = (\Delta \mathbf{F} / \Delta \mathbf{Y}) \times (\mathbf{L}^3 / 4\mathbf{W}\mathbf{H}^3)$$

where $\Delta F/\Delta Y$ is the change in force (ΔF) per unit change in deflection of the center of the specimen (ΔY), L is the distance between the supports on the tension surface (20 mm), W is the mean specimen width, and H is the mean specimen height. The data were analyzed statistically using ANOVA and Tukey's test (p < 0.05).

pН

Each material was mixed and placed into plastic wells approximately 13 mm in diameter and 2 mm deep. Four samples were tested for each cement. A combination electrode, (catalogue number 13-620-83, Fisher, Pittsburgh, PA) was used with an Accumet 620 pH/mV meter (Fisher) to record pH measurements. The electrode was placed directly into the setting material. For set material, a pipette was used to dispense a small amount distilled water $(0.06 \pm 0.01 \text{ ml})$ to wet the cement surface before the electrode was placed on it. The addition of water is a standard method used for pH measurements of solid materials, and results are considered reliable and valid if the same small amount of water is used. For the self-cure specimens, the first pH measurement was taken immediately after mixing (t = 0 minutes), followed by readings at 2, 5, 15, and 30 minutes, and at 1, 2, 4, 6, and 24 hours. For dual-cure specimens, the first measurement was taken 2 minutes after mixing (1 minute after photopolymerization). Between measurements, the electrode was removed and the specimens were stored in a humidity chamber (37°C, >80% RH). The pH data was analyzed statistically using ANOVA repeated measures procedure (p < 0.05). Tukey's test was used for multiple comparisons at each time period (p < 0.005).

Results

Flexural strength

The results for 24-hour flexural strength are depicted in Figure 1 and tabulated in Table 2. There were significant differences in flexural strength among the cements (p < 0.0001). Resin-modified glass ionomers had significantly lower flexural strength. Self-etching cements and conventional resin cements had comparable flexural strength within the same mode of cure, with the exception of Embrace Wetbond in self-cure mode, which was more similar to resin-modified glass ionomers. For resin cements, especially Embrace Wetbond, dual-cure specimens had significantly higher flexural strength than self-cure specimens.

Modulus of elasticity

The results for 24-hour modulus of elasticity are depicted in Figure 2 and tabulated in Table 2. There were significant differences in modulus of elasticity among the cements (p < 0.0001). Self-cured and dual-cured RelyX Unicem translucent and A3 cements and dual-cured Maxcem clear cement had significantly higher modulus of elasticity values than resin-modified glass ionomers and conventional resin cements within the respective



Figure 1 Flexural strength of self-etching conventional resin and resin-modified glass ionomer cements in self-cure and dual-cure modes (n = 8).

Table 2 Flexural strength and modulus of elasticity of self-etching resin conventional resin and resin-modified glass ionomer cements in self-cure and dual-cure modes

	Self	f-cure	Dual-cure		
Cement	Flexural strength (MPa)	Modulus of elasticity (GPa)	Flexural strength (MPa)	Modulus of elasticity (GPa)	
Fuji Plus	36.8 (8.8) ^a	3.9 (0.8) ^b	NA	NA	
RelyX Luting Plus	35.2 (2.4) ^a	3.7 (0.3) ^b	NA	NA	
RelyX ARC	96.1 (14.3) ^{cde}	5.5 (0.9) ^{bcd}	131.2 (21.7) ^{gh}	9.6 (1.0) ^{fg}	
Linkmax clear	78.9 (8.7) ^{bcd}	5.2 (0.9) ^{bc}	135.0 (13.9) ^{ghi}	9.2 (1.1) ^{fg}	
Linkmax A3	79.5 (7.3) ^{bcd}	5.3 (0.4) ^{bc}	126.7 (13.7) ^f	10.0 (1.4) ^g	
RelyX Unicem translucent	75.6 (11.4) ^{bcd}	8.9 (1.3) ^{efg}	130.6 (19.6) ^{gh}	16.5 (1.7) ⁱ	
RelyX Unicem A3	70.2 (5.9) ^b	7.8 (1.0) ^{ef}	99.4 (18.2) ^{dg}	13.0 (3.0) ^h	
Maxcem clear	104.0 (9.5) ^{ef}	6.8 (1.1) ^{cde}	158.2 (10.8) ⁱ	12.7 (1.1) ^h	
Maxcem yellow	76.3 (8.3) ^{bc}	4.2 (0.6) ^b	139.1 (17.7) ^{ghi}	10.4 (1.4) ^g	
Embrace Wetbond	45.3 (10.3) ^a	1.2 (0.3) ^a	150.7 (10.3) ^{hi}	7.4 (0.7) ^{def}	

Superscript indicates groups with statistically similar values (p = 0.05).



Figure 2 Modulus of elasticity of self-etching conventional resin and resin-modified glass ionomer cements, in self-cure and dual-cure modes (n = 8).

cure modes. The other self-etching cements had modulus values that were not significantly different from those of resinmodified glass ionomer or conventional resin cements. Photopolymerization had a significant positive effect on modulus of elasticity for all the cements.

pН

The pH profiles for all the cements over a 24-hour period are depicted in Figure 3. ANOVA yielded a significant effect, and time, cement, and polymerization mode were significant interacting factors (p < 0.0001). Self-etching cements exhibited significantly lower initial pH than the conventional resin cements. The conventional resin cements had the highest initial pH of all the cements types. Immediately after photopolymerization (t = 2 minutes), the cure mode had no significant effect on the cement pH; however, at t = 30 minutes, the effects of photopolymerization on self-etch resin cements were significant. At t = 30 minutes to 24 hours, the dual-cure cements had significantly higher pH than their self-cure counterparts, with

one exception. RelyX Unicem displayed a unique rapid rise in pH in both self-cure and dual-cure modes. By t = 1 hour, no significant difference in pH was observed between self-cure and dual-cure Unicem specimens. The cement also generally exhibited the highest final pH values, which were achieved within one hour.

Discussion

Self-etching cements are a relatively new category of resin luting agents. Although described as multipurpose luting cements, they are essentially resin cements, and it is necessary to define whether they are capable of a performance equal in all respects to that of conventional resin cements. Conventional resin luting cements currently provide the greatest bonding capability and highest strength for clinical attachment of indirect restorations;^{4,9} however, self-etching resin cements greatly simplify the cementation technique and have the potential to decrease technique and postoperative sensitivity. Knowledge of



Figure 3 (A) pH profiles of the self-etching cements over 24-hour period (n = 4). (B) pH profiles of the conventional resin and resin-modified glass ionomer cements over 24 hour period (n = 4).

their comparative performance with respect to bonding ability, sealing efficacy, strength, solubility, pulpal tolerance, and clinical performance is necessary. The current study was designed to investigate basic mechanical and physical parameters. Flexural strength, modulus of elasticity, and pH profiles were determined in relation to standard examples of both conventional resin and resin-modified glass ionomer cements. Flexural strength and modulus of elasticity are considered useful in the characterization of cements and are indicative of the cements' ability to resist high masticatory forces, preventing prosthesis dislodgement, and/or microleakage.¹⁰

For the resin-modified glass ionomer cements, the determined flexural strengths were similar to those provided by the manufacturer as well as those previously reported.^{11,12} Such cements are characterized as relatively low- to moderate-strength, general-purpose cements with high fluoride content. They are not recommended for high-strength clinical situations. Greater variability exists in the literature for flexural strength of conventional resin luting cements with studies reporting higher,^{1,8,13} similar,^{1,2,14} and lower values^{13,15} than those determined in this study. Flexural strengths of the self-etching cements were in the same range as the conventional resin cements tested and for the first commercial material (Rely X Unicem), flexural strengths in both self-cure and dual-cure modes were substantially higher than those reported in the literature.^{1,8} With the exception of one self-etch cement (Embrace Wetbond), which had a self-cure value only 30% that of the dual-cure mode, all cements met the ISO 4049 standard for minimum flexural strength (50 MPa) as type 2, class 3 luting materials.¹⁶

The elastic moduli determined for the resin-modified glass ionomer cements were relatively low and equivalent to reported values.¹² There were no significant differences in modulus of elasticity among the three conventional resin cements. Similar modulus of elasticity results have been reported in the literature for one of the standard conventional cements tested (RelyX ARC) in self-cure mode;^{13,17} however, the values for dual-curing in this study were higher than reported previously.^{2,15} Yoshida et al¹³ and Lu et al¹⁴ also reported modulus values between the self-cure and dual-cure values obtained in this study for the other standard conventional resin cement tested (Linkmax). The elastic moduli of the self-etching cements showed great variability. The modulus observed for one (RelyX Unicem translucent) was particularly high when dual-cured. This may be attributable to the quantity and type of inorganic filler present or to the degree of polymer conversion. It has been suggested that the preferred modulus of elasticity

of a luting cement is one that falls between the modulus of the dentin and the restorative material to provide similar deformation under loading.¹⁸ As the modulus of dentin is 12 to 20 GPa, the cements with higher modulus results are considered more desirable. A higher elastic modulus allows the cement to resist elastic deformation in regions of high biting forces or in longer span prostheses.¹⁰

All conventional and self-etch resin cements in this study showed markedly higher strengths and elastic moduli in dualcure mode than in self-cure mode. The same findings have been reported in a number of previous studies on resin cements.^{2,15,19} Reported flexural strengths of self-cured cements were determined to be approximately 80% of the value obtained for dual-cure mode.^{1,2,19} In comparison, the flexural strengths of self-cured cements in the current study were generally 60 to 70% of the value obtained for dual-cure mode. The relatively greater improvement in mechanical properties of the dual-cured over the self-cured cements of this study could be attributed to the intense photopolymerization used. The results of this study suggest that the self-cure conversion of double bonds is insufficient to provide optimal mechanical properties. It is recommended that all dual-cured resin cements receive maximum photopolymerization to achieve superior conversion and optimum material properties wherever clinically possible.

The most notable differences between self-cure and dual-cure modes for all properties tested were shown by one self-etching resin cement (Embrace Wetbond). The self-cure modulus and strength results for this cement were only 16 to 30% of the dual-cure results. The pH profile showed little change in acidity over time in the self-cure mode, whereas photopolymerization provided a more substantial pH rise during setting. These results indicate intrinsic inadequate self-cure chemistry. Furthermore, this material exhibited an unset surface layer similar to an oxygen-inhibited layer even after dual-curing and complete surface coverage by the mold assembly. This observation highlights the reduced polymerization capability of this material. This self-etch cement should only be used in the dual-cure mode under optimal polymerization conditions.

The self-etching resin cements, as expected, revealed low initial pH values, which were comparable to one of the resinmodified glass ionomer cements; however, at 24 hours, the pH values were very variable, displaying both the lowest and highest final pH values. While one (Rely X Unicem) showed a very rapid rise in pH and highest final pH values in both self-cure and dual-cure modes, both Maxcem and Embrace Wetbond in self-cure modes showed only modest increase. Although this observation could be attributed partially to the inability of these cements to self-cure fully while exposed to air, Rely X Unicem revealed a unique chemistry that allowed a rapid rise despite the mode of cure. For this cement, neutrality was achieved only 15 minutes after dual-curing and 1 hour in self-cure mode. It has been suggested that the prolonged acidity, particularly the period below pH 3 may be a factor in pulp inflammation and postoperative sensitivity with luting cements.²⁰ All the photopolymerized dual-cure conventional and self-etching resin cements recorded a minimum of pH 5 by 15 minutes; however, the period of lower pH was extended for resin cements in the self-cure mode. The significance of an extended period of lower than neutral pH has not been determined; however it is known that adequate polymerization is an important prerequisite for stability and biocompatibility of any material.¹⁹

New proprietary self-etching resin cements are currently being introduced into the market. Based on this study, self-etching cements have the potential to provide mechanical and physical characteristics similar to those of conventional resin cements. In addition, they offer a simpler, less technique-sensitive, one-step approach to resin luting and bonding; however, due to the variability between the various brands tested it cannot be assumed that all the cements in this new group are synonymous. Significant differences exist, and individual product performance requires assessment. Further investigation of additional properties and performance will be required for these and other new materials of this type as they enter the marketplace.

Conclusions

- The self-etching resin cements evaluated in this study displayed disparate properties and cannot be considered a homogeneous group. There was a tendency for these selfetch cements to have similar flexural strength and equal or higher elastic moduli than the conventional resin cements, but individual differences were also apparent.
- 2. All dual-cured luting cements, both self-etch and conventional, showed highly superior properties when photopolymerized.
- 3. As expected, the pH profile during setting of self-etch cements differed from conventional resin with high initial acidity and gradual pH rise. One self-etch cement revealed a unique rapid rise to neutrality in both dual-cure and self-cure modes.

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