

Effect of Mode of Polymerization of Bonding Agent on Shear Bond Strength of Autocured Resin Composite Luting Cements

(Effet de la méthode de polymérisation de la résine de liaison sur la résistance adhésive, en cisaillement, des résines de scellement autopolymérisées)

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S o m m a i r e

Objectif : Des rapports isolés ont fait état d'une faible résistance adhésive avec les matériaux à résine composite autopolymérisée combinée, notamment quand on utilise les résines photopolymérisées où on retrouve l'apprêt et l'adhésif dans une même bouteille. L'objectif de la présente étude est de déterminer si la méthode de polymérisation de la résine adhésive influe sur la résistance d'adhésion à la dentine des ciments à résine composite autopolymérisée.

Méthodologie : La résistance adhésive, en cisaillement, de 2 résines de scellement, Calibra et RelyX ARC, autopolymérisées, combinées à 4 résines adhésives différentes, Scotchbond Multipurpose Plus, Prime & Bond NT, IntegraBond et Single Bond, polymérisées à de la dentine bovine par photopolymérisation, autopolymérisation ou les 2, a été définie. Le pH de chaque résine adhésive et de ses composants a été mesuré. Une analyse de la variance à 2 facteurs a permis d'évaluer l'effet du ciment et de l'adhésif sur la résistance d'adhésion en cisaillement. Pour chaque résine, la variable de l'adhésif comprenait les facteurs de la marque du produit et de la méthode de polymérisation. Étant donné une interaction significative entre les variables susmentionnées, les moyennes des moindres carrés des 16 combinaisons de ciment et d'adhésif ont été comparées.

Résultats : Il n'existait aucun lien cohérent entre la résistance adhésive et la méthode de polymérisation de la résine adhésive. Les différences significatives notées dans la résistance étaient spécifiques à la marque de la résine adhésive – le pH de la résine variant suivant la formulation du fabricant et la résistance étant proportionnelle au pH.

Conclusions : La faible résistance adhésive notée *in vitro* avec certaines combinaisons de résine de liaison et de résine de scellement pourrait être significative du point de vue clinique.

Mots clés MeSH : dental bonding/methods; dentin-bonding agents/chemistry; resin cements/chemistry

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The placement of all-ceramic restorations to meet the esthetic demands of patients has increased. This has led to greater use of adhesive resin cements to provide strength for all-ceramic restorations and to ensure secure attachment to the tooth.^{1,2} Cements with adhesive properties have a distinct advantage because of their potential to reduce microleakage and associated sequelae. Porcelain veneers,

ceramic inlays and onlays, and adhesive fixed partial dentures became predictable treatment options only with the development of resin cements. The retention of conventional indirect restorations and fixed prostheses can be improved with the use of adhesive resin cements.^{3,4} Such cements provide better retention for short crown³ and short post⁴ preparations *in vitro* when adjunctive retentive design features are inadequate.

Resin luting cements also exhibit low solubility. Numerous proprietary resin cements and bonding agents are available, and in vitro assessment is essential to determine optimal performance.

There have been anecdotal reports^{5,6} of low bond strength with autocured (AC) resin composite materials, particularly when light-cured (LC) bonding agents that combine primer and adhesive in a 1-bottle preparation are used. These reports led to speculation that some bonding agents and resin cements may be incompatible and prompted further research⁷⁻¹¹ in this area. Because of the chemical similarities between these materials, problems encountered with the use of resin composite core buildup materials may also apply to resin composite luting cements. One in vitro study found that different modes of primer polymerization affected the bond strength of each resin cement differently.¹⁰ However, little is known about the efficacy of the attachments between resin cements and bonding agents achieved with different methods of polymerization.

Both resin cements and bonding agents can be formulated to allow LC, AC or dual-cure polymerization.¹² LC polymerization occurs when a diketone photoinitiator, such as camphoroquinone, absorbs light in the 400- to 500-nm range and interacts with an organic amine accelerator, such as *N,N*-dimethylaminoethylmethacrylate, producing free radicals that initiate the polymerization. AC or self-curing polymerization is possible when a peroxide initiator and a tertiary amine accelerator are combined; their reaction produces free radicals at room temperature. Dual-cure formulations consist of reagents for both AC and LC polymerization.

It is critical that all the components at the adhesive interface are able to undergo maximum polymerization to ensure optimal bond strength, as well as maximum physical and biological properties. The degree of conversion of monomer to polymer is defined as the percentage of carbon double bonds that are consumed. In clinical situations polymerization is seldom complete, and the degree of conversion ranges from 50% to 70%.¹² Conversion depends on the resin composition and the quantity of free radicals for polymerization that are generated. The degree of conversion from monomer to polymer is comparable between LC and AC resin composites with the same monomer formulations, provided that effective LC polymerization is achieved. Clinicians often encounter situations in which the resin cement must undergo AC polymerization because of inaccessibility to the visible LC source.

Table 1 Bonding agents used in a study of shear bond strength with autocured resin luting cements

Manufacturer	Bonding agent
3M Dental Products St. Paul, Minn.	Scotchbond Multipurpose Plus Single Bond
Dentsply/Caulk Milford, Del.	Prime & Bond NT Prime & Bond Self Cure Activator
Premier King of Prussia, Penn.	IntegraBond IntegraBond Auto-Cure Activator

However, the bonding agent is always accessible to light, and it has been demonstrated in vitro that separate LC polymerization of the bonding agent leads to greater bond strength.¹³ The clinical significance of inadequate polymerization at the adhesive interface includes decreased retention, increased marginal leakage, negative pulpal response and reduced longevity of the restoration. Better adhesion resulting from precuring the bonding resin must be balanced with the potential risk of excessive film thickness and incomplete seating of the indirect restoration.

The purpose of this study was to determine if the mode of polymerization of the bonding agent influences the strength of the attachment of AC resin luting cements to dentin.

Materials and Methods

The 2 resin luting cements, Calibra (Dentsply/Caulk, Milford, Del.) and RelyX ARC (3M Dental Products, St. Paul, Minn.), were selected because they can be subjected to

Table 2 Protocol for various bonding agents

Bonding agent	Protocol
Scotchbond Multipurpose Plus	
Light cure	<ul style="list-style-type: none"> Apply 2 coats primer, then wait 20 seconds before air drying Apply 1 coat adhesive, then wait 20 seconds before air drying Light cure for 10 seconds
Autocure	<ul style="list-style-type: none"> Apply 1 coat activator, then wait 5 seconds before air drying Apply 2 coats primer, then wait 20 seconds before air drying Apply 1 coat catalyst
Prime & Bond NT	
Light cure	<ul style="list-style-type: none"> Apply 1 coat adhesive, then wait 20 seconds before air drying Light cure for 10 seconds
Autocure	<ul style="list-style-type: none"> Apply 1 coat adhesive plus activator mixture, then wait 20 seconds before air drying
Dual cure	<ul style="list-style-type: none"> Apply 1 coat adhesive plus activator mixture, then wait 20 seconds before air drying Light cure for 10 seconds
IntegraBond	
Light cure	<ul style="list-style-type: none"> Apply first coat, then wait 20 seconds before air drying Light cure for 20 seconds Apply second coat, air dry, then light cure for 20 seconds
Autocure	<ul style="list-style-type: none"> Apply 2 coats adhesive plus activator mixture Air dry each coat separately
Single Bond	<ul style="list-style-type: none"> Apply 2 consecutive coats, wait 20 seconds, then air dry Light cure for 10 seconds

AC polymerization. Calibra has high AC ability.¹⁴ RelyX ARC is promoted by the manufacturer as allowing easy removal of excess cement.

The bonding agents used in this study are listed in **Table 1**. Scotchbond Multipurpose Plus (3M Dental Products), Prime & Bond NT (Dentsply/Caulk) and IntegraBond (Premier, King of Prussia, Penn.) were selected because they are available in both LC and AC formulations. Single Bond (3M Dental Products) is formulated for LC polymerization only and is a 1-bottle preparation made by the same manufacturer as Scotchbond Multipurpose Plus.

Bovine teeth, stored frozen in distilled water before the study and refrigerated in distilled water during the study, were mounted in AC polymethyl methacrylate using moulds 2.5 cm in diameter and 2.0 cm in depth. Before bonding, the buccal superficial dentin was exposed by grinding with 180-grit silicon carbide paper mounted on a grinding wheel under running water. Each tooth was polished manually on wet 600-grit silicon carbide paper¹⁵ and rinsed just before the bonding procedure. After preparation and analysis with the first cement, the teeth were reused for testing the second cement by manually polishing the dentin surface with wet 320-grit silicon carbide paper followed by wet 600-grit silicon carbide paper.

Manufacturers' instructions were used as guidelines in the bonding protocol for each agent (**Table 2**). A 35% phosphoric acid conditioner (3M Dental Products), used for all treatment groups, was applied to the broad dentin surface with a brush tip, left for 15 seconds and rinsed thoroughly. If the manufacturer's instructions did not state that the 2 coats of bonding agent were to be applied consecutively, the first layer was air-dried before application of the second coat. If bald spots were observed on the dentin surface, additional bonding agent was applied. Where applicable, the bonding agent was subjected to LC polymerization with a conventional quartz halogen LC unit (Spectrum 800, Dentsply/Caulk) with a light intensity of at least 500 mW/cm². The adhesive area was demarcated by a coni-snap #4 natural-snap gelatin capsule (Wiler Fine Chemicals Ltd., London, Ont.) supported by an impression putty mould. Equal amounts of catalyst and base pastes were mixed within 10 seconds. The resin cement was loaded into the gelatin capsule with a ball burnisher to a height of 2–3 mm. The Calibra cement was allowed to cure for 10 minutes and the RelyX ARC cement for 20 minutes before immersion in distilled water. The RelyX ARC cement was allowed to cure for a longer period because the surface was still tacky after 10 minutes. The specimens were stored for 24 hours at 37°C in distilled water before being subjected to the shear bond strength test. Ten specimens were prepared for each of the 16 combinations of bonding agent and resin cement.

The method for testing shear bond strength followed 1994 ISO Technical Specification No. 11405.¹⁶ The shear force was applied with a knife-edged rod attached to an Instron universal testing machine (model 4301, Instron, Canton, Mass.) at a crosshead speed of 1 mm/min. The shear bond strength was

calculated by dividing the peak failure loads by the bonding area (17.34 mm²). Mode of failure was determined by visual examination. Visible fracture of the dentin or resin cement was recorded as cohesive failure. All other failures were recorded as adhesive failure.

To determine if pH might affect the bond strength of the bonding agent – resin cement combinations, the pH of the bonding agents was measured with a Ag/AgCl reference electrode (catalogue no. 13-620-83, Fisher Scientific Canada, Nepean, Ont.) and an Accumet pH meter (model 620, Fisher Scientific Canada). The pH meter was calibrated with solutions of pH 7.0 and 4.0. The pH of the cured and uncured bonding agents as well as that of the individual components was measured by dispensing the bonding agent onto a mixing pad and bringing the electrode into direct contact. Measurements for set LC and AC materials involved placing a drop of distilled water between the material and the electrode. The pH of all AC bonding agents was measured after 6 minutes. The pH of all LC bonding agents was measured 1 minute after curing the material for 20 seconds. pH measurements were made in triplicate.

Statistical Analyses

Two-way analysis of variance was used to test the effect of cement and adhesive on shear bond strength. For each bonding agent, the adhesive variable combined the factors product brand and mode of polymerization. With significant interaction among the above variables, the least square means of the 16 combinations of resin cement and adhesive were then compared. The computer program SAS version 8.2 (SAS Institute Inc., Cary, NC) was used for all statistical analyses.

Results

The mean bond strengths (with standard deviations and standard errors) for combinations of bonding agent and resin cement are listed in descending order of shear bond strength for the Calibra cement in **Table 3** and for the RelyX ARC cement in **Table 4**. There were significant interactions among the 16 combinations of bonding agent and resin cement ($p < 0.001$).

The frequency of cohesive failure in dentin was higher in pairs with high bond strengths (**Tables 3 and 4**). Similarly, the depth at which cohesive failure in dentin occurred was greater with increasing bond strength.

The pH of uncured and cured bonding agents and their components are listed in **Table 5**. The pH values reflect the presence of acidic components in the bonding agent formulations.

Discussion

Numerous methods are available for evaluating the adhesion of dental materials to teeth. In vitro measurement of bond strength is the most common method of evaluating the presence and extent or value of the adhesive bond. The shear bond strength test involves loading the adhesive surface in shear until fracture occurs. The advantage of this test method is that it is relatively simple with respect to specimen preparation, equipment required and test setup. The main criticism is that

Table 3 Mean bond strength and cohesive failure rate for dentin and resin cement for teeth prepared with Calibra resin luting cement

Bonding agent	Bond strength (MPa)			Cohesive failure (%)	
	Mean*	SD	SE	Dentin	Resin cement
Scotchbond Multipurpose Plus, autocure	13.15 a	2.83	0.90	90	0
Single Bond	10.53 b	1.83	0.58	40	10
IntegraBond, light cure	10.52 b	3.26	1.03	50	0
Scotchbond Multipurpose Plus, light cure	8.13 c	1.03	0.32	50	50
Prime & Bond NT, dual cure	5.46 d	1.31	0.41	50	0
IntegraBond, autocure	4.41 d,e	2.35	0.74	20	0
Prime & Bond NT, autocure	3.77 d,e	0.79	0.25	0	0
Prime & Bond NT, light cure	3.11 e	1.37	0.43	10	0

SD = standard deviation, SE = standard error.

*Means followed by different letters are significantly different ($p < 0.001$).

Table 4 Mean bond strength and cohesive failure rate for dentin and resin cement for teeth prepared with RelyX ARC resin luting cement

Bonding agent	Bond strength (MPa)			Cohesive failure (%)	
	Mean*	SD	SE	Dentin	Resin cement
Scotchbond Multipurpose Plus, light cure	12.99 a	3.77	1.19	90	0
Prime & Bond NT, dual cure	11.33 a,b	4.22	1.33	50	10
IntegraBond, light cure	11.20 a,b	2.66	0.84	50	20
Single Bond	9.24 b	3.64	1.15	50	10
Scotchbond Multipurpose Plus, autocure	9.24 b	2.24	0.71	30	0
IntegraBond, autocure	6.89 c	2.86	0.90	10	0
Prime & Bond NT, light cure	4.43 d	3.31	1.05	10	0
Prime & Bond NT, autocure	2.97 d	1.30	0.41	0	0

SD = standard deviation, SE = standard error.

*Means followed by different letters are significantly different ($p < 0.001$).

it measures the cohesive strength of the material being bonded or the substrate (or both), rather than the bond strength of the adhesive interface.¹⁷ This is a problem because failure in this situation does not simulate the clinical mode of failure (i.e., failure of the adhesive between the restoration and the tooth). Cohesive failure in the dentin was observed in this study and is recognized as a limitation of the study; however, the shear bond strength test was used to screen for potentially large differences among the combinations of bonding agent and resin cement.

Although there is a lack of literature on the bond strength of adhesives used with resin luting cements, numerous studies have measured the bond strengths of bonding agents in combination with resin composite restorative materials. Scotchbond Multipurpose has consistently performed well during in vitro studies,¹⁸⁻²² as it did in this study. This agent was used here as a control material in which the primer and adhesive are provided separately. The 3 other bonding agents were formulated with the primer and adhesive combined in one bottle. The fact that the 1-bottle and 2-bottle variants from a single manufacturer yielded similar bond strength suggests that the problem of low bond strength between the bonding agent and the resin cement cannot be attributed to the manner of delivery of the bonding agent.

Some combinations of bonding agent and resin cement yielded low bond strength (Tables 3 and 4). Application of the Prime & Bond NT and IntegraBond agents is sensitive to technique: the dentin should not be too wet or too dry.²³ Both of these bonding agents contain acetone as the solvent for the hydrophilic resins. The strong air blast recommended by the manufacturer of IntegraBond probably further aggravated the technique sensitivity of this material by desiccating the dentin and creating bald spots on the bonding surface. Water-based primers are less sensitive than acetone-based primers to the degree of dentin moisture,²³⁻²⁵ as demonstrated with Scotchbond Multipurpose and Single Bond.

Insight into the formulation of the bonding agents was gained by measuring the pH of the individual components of each bonding agent system. There has been speculation²⁶ that the acidity of the bonding agent may affect the degree of conversion of the bonding agent or the AC resin cement. The dipentaerythritolpentaacrylate phosphate ester (PENTA) molecule used in Prime & Bond NT is an obvious difference in chemical composition between this agent and all the other bonding agents in this study. The known acidity of the PENTA molecule was confirmed by the low pH of the Prime & Bond LC component (Table 5). The bond strength of the Prime & Bond NT dual-cure component was higher than that

Table 5 pH of uncured and cured components and mixtures of bonding agents

Bonding agent	pH \pm SD	
	Uncured	Cured
Scotchbond Multipurpose Plus		
Primer	3.8 \pm 0.04	NA
Light-cure adhesive	5.9 \pm 0.24	5.6 \pm 0.49 ^a
Activator	6.6 \pm 0.23	NA
Catalyst	3.7 \pm 0.08	NA
Activator, primer and catalyst combined	4.1 \pm 0.00	4.0 \pm 0.29 ^a
Prime & Bond NT		
Light-cure component	2.1 \pm 0.31	1.7 \pm 0.20 ^a
Autocure component	7.1 \pm 0.08	NA
Light-cure and autocure components combined	4.2 \pm 0.19	2.8 \pm 0.14 ^a
IntegraBond		
Light-cure component	5.5 \pm 0.08	5.3 \pm 0.16 ^a
Auto-cure component	4.2 \pm 0.07	NA
Light-cure and autocure components combined	4.9 \pm 0.15	4.3 \pm 0.24 ^a
Single Bond (light cure only)	4.2 \pm 0.04	3.4 \pm 0.47 ^a

SD = standard deviation, NA = not applicable.

^aValues for the bonding agent when it was used with the resin luting cement.

of other Prime & Bond versions, despite the fact that it has the same formulation as the Prime & Bond AC mixture. This suggests that the efficiency of the AC activator and pH may affect dentin bond strength, because LC polymerization was necessary to increase bond strength. A microtensile bond strength study showed that the bond strengths of all 4 LC 1-bottle bonding agents in combination with an AC composite produced significantly low bond strengths.⁹ There was a positive correlation between the acidity of the bonding agents and resulting bond strength with the AC composite. Ultra-structural observations provided evidence to support the authors' hypothesis that the residual acidic resin monomers from the 1-bottle bonding agent interacted with the binary peroxide-amine catalytic components often present in AC resin composites.

Attachment to tooth structure appears to depend on the formulation of the bonding agent. The different chemical formulations of proprietary bonding agents probably contributed to the differences in bond strengths observed in this study. In a previous study, low shear bond strengths of an autopolymerizing core buildup composite bonded to dentin with 9 dentin bonding agents were attributed to material incompatibilities.⁷ In another recent study, 2 of 5 bonding agents tested with an AC resin composite produced low bond strengths.¹¹ One study of adhesion of resin composite core materials to dentin⁸ concluded that low bond strengths observed with certain combinations of bonding agent and resin composite might have been due to the incompatibility of components of different manufacturers' resins; alternatively, it was suggested that the air-inhibited layer of the bonding agent

might have contributed to inadequate polymerization at the interface between resin core and bonding agent. Differences in formulation might also include type of solvent, film thickness, degree of oxygen inhibition, proportion of hydrophilic to hydrophobic components and efficiency of the initiator system. AC polymerization of IntegraBond produced low bond strengths with both resin cements, even though this bonding agent was not unusually acidic, so the efficiency of the AC component must be questioned.

Although the studies available so far do not provide data to inform clinicians about the clinical performance of most combinations of bonding agent and resin cement, making clinicians aware of potential clinical problems and encouraging prudence in the selection of dental materials are the first steps in achieving predictable long-term clinical results.

Conclusions

No consistent relationship was found between shear bond strength of dentin and mode of polymerization of bonding agents when AC resin luting cements were used. Similarly, no relationship was found between shear bond strength of dentin and type of bonding agent (separate or combined primer and bonding agent application) when AC resin luting cements were used. However, there were significant differences in bond strength specific to the proprietary brand of bonding agent. Prime & Bond NT and IntegraBond in combination with either Calibra or RelyX ARC cement produced the lowest mean bond strengths. Finally, the acidity of the bonding agent and the efficiency of AC polymerization may be associated with low bond strengths when AC resin cements are used. ♦

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