THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Effect of interim cement application on bond strength between resin cements and dentin: Immediate and delayed dentin sealing

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ABSTRACT

Statement of problem. Despite the advances in materials and techniques, adhesion to dentin is challenging because of the complex composition of dentin's mineral, organic, and fluid phases.

Purpose. The purpose of this in vitro study was to evaluate the bond strength of 2 different resin cements (conventional and self-adhesive) with or without previous dentin sealing and the effect of interim cement.

Material and methods. Forty-five molars were embedded into acrylic resin blocks and a flat dentin surface was exposed. Twenty teeth (n=5 per group) were treated with the conventional resin cement associated with etch-and-rinse or self-etch adhesive approaches, applied before (immediate dentin sealing) or after (delayed dentin sealing) the application/removal of interim cement. Another 25 teeth (n=5, per group) were treated with self-adhesive resin cement with (self-etch mode [immediate dentin sealing or delayed dentin sealing]) or without adhesive application. Furthermore, in the self-adhesive resin cement group, the application of polyacrylic acid for dentin etching before cementation was evaluated. Composite resin blocks were cemented onto flat, treated dentin surfaces, and the assemblies were sectioned into bar-shaped specimens for microtensile bond strength testing. The data were subjected to 1-way ANOVA followed by the post-hoc Tukey test (α=.05). The failure patterns were classified as cohesive, adhesive, or mixed.

Results. The application of adhesive before interim cement (immediate dental sealing) promoted the highest values of bond strength for both resin cements (P<.001). For self-adhesive resin cement, polyacrylic acid-enhanced bond strength after the application of interim cement.

Conclusions. The application of dental adhesive immediately after tooth preparation (immediate dentin sealing) and before the use of an interim cement promoted the highest values of bond strength to dentin with the resin cements tested. (J Prosthet Dent 2017;117:792-798)

Since the advent of adhesive dentistry, the composition of the materials and the clinical methods used for adhesion has changed.[1] Several studies have evaluated different adhesive materials and techniques, and the results of these reports are fairly consistent.[2-6] However, adhesion to dentin is challenging because of the complex composition of dentin's mineral, organic, and fluid phases.[4,7]

The conventional bonding technique includes a 3-step etch, prime, and bond protocol followed by conventional resin cement. These systems are reported to achieve the highest bond strength values.[4] The superior bonding may be attributed to optimal dentin hybridization and the formation of a complete hybrid layer. The adhesive infiltrates into the collagen fibrils, forming a biocomposite;[8] therefore, the resin matrix is reinforced by these fibrils. These adhesives have achieved consistently good bond strength values in in vitro tests.[4,7-9] However, in clinical practice, techniques with

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Clinical Implications

In addition to possibly decreasing postoperative sensitivity, the application of adhesive after tooth preparation (immediate dentin sealing) and before the application of interim cement presents advantages regarding bond strength to resin cements.

several steps are considered more sensitive and prone to error.[4,5,10]

Self-adhesive resin cements such as RelyX Unicem 2 (3M ESPE) may eliminate some of the problems with multistep systems. According to the manufacturer's instructions and reported studies,[11-15] pretreatment of either the tooth or the restoration is unnecessary. The adhesive properties of these cements are based upon acidic monomers that demineralize and infiltrate the tooth substrate, resulting in micromechanical retention.[16,17] Chemical adhesion to the hydroxyapatite is provided by secondary reactions.[3,17,18] In addition, so-called universal adhesives have been released to incorporate all possible techniques for dentin/enamel/restoration surface treatment in 1 product[19,20] and are used together with a resin cement.

The appropriate timing of adhesive application is a concern. Applying dental adhesive before definitive impression making, the so-called immediate dentin sealing (IDS) technique,[5,21] provides adhesion to a freshly cut and uncontaminated dentin, which is ideal for bonding.[22,23] The IDS technique provides higher values for bond strength,[21] and the adhesive layer applied before impression making does not interfere with the complete seating of the restoration.[21] The IDS technique, cavity design optimization, and cervical margin relocation are classified as modern treatment concepts, combining simpler techniques with more predictable and durable results.[24] When the adhesive is applied only at the moment of definitive cementation, the approach is known as delayed dentin sealing (DDS).[25]

Interim restorations are luted to the prepared tooth with interim cement to avoid infection, tooth sensitivity, or tooth movement.[26] However, eliminating all of the interim material from the dentin surface before definitive cementation is difficult to, and the penetration of adhesive monomers into the tooth substrate may be impaired.[22,23]

Because little information is available regarding the IDS technique associated with universal adhesives and self-adhesive resin cements, the purpose of this study was to evaluate the bond strength of 2 different resin cements (conventional and self-adhesive) with regard to previous dentin sealing and the effect of interim cement. The null hypothesis was that the values of bond strength of IDS would not be higher than those of DDS.

MATERIAL AND METHODS

This study evaluated the microtensile bond strength between dentin and a conventional or a self-adhesive resin cement by investigating the effect of the timing of adhesive application and the use of interim cement. It was approved by the Council of Ethics in Research of the University of Taubate, protocol 423,189.

The different steps involved in preparing the specimens from each group are illustrated in Figures 1 and 2, and described in Table 1. The roots of 45 sound, recently extracted (up to 7 days) human third molar teeth were embedded in acrylic resin blocks up to the cementoenamel junction. Their occlusal surfaces were abraded with silicon carbide paper (180 to 600 grit) in a polishing machine (Aropol E; Arotec S.A. Ind & Com) until a flat dentin surface was exposed. The smear layer formed was expected to be uniform for all teeth.

Two different resin cements were used: conventional resin cement (RelyX ARC; 3M ESPE) and self-adhesive resin cement (RelyX U200; 3M ESPE). In the conventional resin cement group (C) (Fig. 1), the adhesive (Scotchbond Universal/Dual Cure Activator; 3M ESPE) was applied in both self-etch (S) and etch-and-rinse (E) mode at 2 different times: immediately after abrading the dentin (IDS technique), or the adhesive was applied after the application/removal of the interim cement (DDS) and immediately before application of the resin cement (CST-DDS and CET-DDS). For the IDS groups, the interim cement (T) (RelyX Temp; 3M ESPE) was applied after application of the adhesive, (CST-IDS and CET-IDS).

In the self-adhesive (Sa) resin cement group, 3 subgroups were tested without adhesive and 2 with adhesive (Fig. 2): (Sa) control group, no surface treatment; (SaT) application/removal of interim cement; (SaTPa) application/removal of interim cement followed by dentin etching with 11.5% polyacrylic acid for 30 seconds (Vidrion Dentin Etch; S.S. White); (SaT-IDS) application of adhesive (Scotchbond Universal/Dual Cure Activator; 3M ESPE) immediately after abrading the dentin (IDS technique), interim cement applied after adhesive; or (SaT-DDS) application of adhesive (Scotchbond Universal/Dual Cure Activator; 3M ESPE) after the application/removal of interim cement (DDS) and immediately before application of the resin cement.

Groups with interim cement application (T) received an interim restoration fabricated from an autopolymerizing acrylic resin (9×9×5 mm, Dencôr; Classico), simulating an interim restoration cemented with a eugenol-free interim resin cement (RelyX Temp; 3M

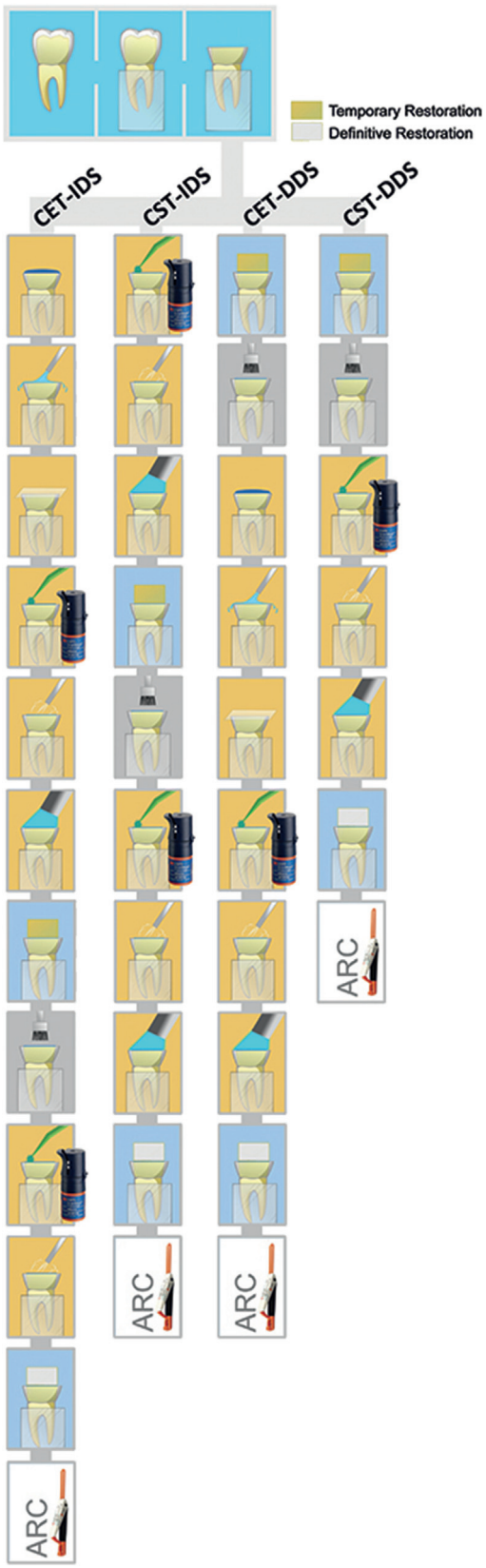

Figure 1. Specimen fabrication steps. Conventional resin cement.

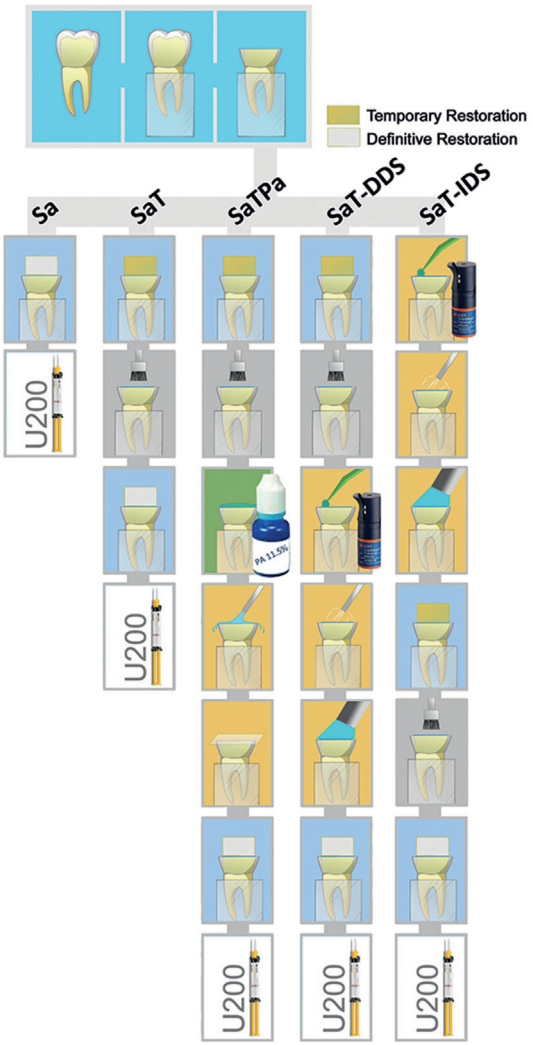

Figure 2. Specimen fabrication steps. Self-adhesive resin cement.

ESPE). These restorations and remaining interim cement were removed after 1 week, using a dental excavator, and the preparation was cleaned with a rotary brush attached to a handpiece associated to pumice.

To model definitive cementation, composite resin blocks (Z100, Incisal Shade; 3M ESPE) were bonded with the respective resin cement to the occlusal surface of the molars after the described procedures. The composite resin blocks were airborne-particle abraded with 50-μm aluminum oxide particles for 10 seconds at a distance of 20 mm, cleaned in an ultrasonic bath for 5 minutes, and air-dried. A thin layer of the adhesive system was applied on the cementation surface of the block. The respective resin cement was applied and the resin block was seated on the treated dentin under finger pressure. Excess material was removed and the cement light activated (Bluephase; Ivoclar Vivadent AG) for 40 seconds.

Table 1. Testing of groups[a]

Resin Cement	Dental Adhesive		Interim Cement/ Restoration (T)	Group	n
Conventional (C)	Universal adhesive	Etch-and-rinse (E)	After adhesive (IDS)	CET-IDS	5
			Before adhesive (DDS)	CET-DDS	5
		Self-etch (S)	After adhesive (IDS)	CST-IDS	5
			Before adhesive (DDS)	CST-DDS	5
Self-Adhesive (Sa)	No	No	No	Sa	5
			Yes	SaT	5
			Yes	SaTPa[b]	5
	Universal Adhesive	Universal Adhesive	Yes, after adhesive (IDS)	SaT-IDS	5
			Yes, before adhesive (DDS)	SaT-DDS	5

C, conventional resin cement; CET, conventional resin cement, etch-and-rinse approach, interim cement; CST, conventional resin cement associated to self-etch adhesive and application of interim cement; DDS, delayed dentin sealing: interim cement applied before adhesive; E, etch-and-rinse approach; IDS, immediate dentin sealing; Pa, polyacrylic acid. S, self-etch approach; Sa, self-adhesive resin cement; T, interim cement.
[a]Groups were tested according to resin cement applied, adhesive and mode of application of adhesive, and previous cementation of interim restoration. [b]Application of polyacrylic acid after removal of interim cement.

After specimens were stored in distilled water at 37°C for 7 days, the resin-cement-dentin specimens were longitudinally sectioned into bar-shaped specimens, using a cross-sectional area of 0.9 ±0.1 mm^2 (Isomet; Buehler). The dimensions were verified with digital calipers (Starrett 727; L.S. Starrett).

The specimens were individually attached to a microtensile bond strength (μTBS) testing apparatus with cyanoacrylate adhesive (Super Bonder Gel; Loctite/Henkel) and tested to failure in a universal testing machine (DL 1000; EMIC) at a crosshead speed of 0.5 mm/min.

Fractured specimens were examined by using stereomicroscopy (Olympus SZ-CTV; Olympus) at magnification ×50 to determine the mode of failure as adhesive, mixed, or cohesive. Representative images were recorded.

Data obtained from microtensile testing (MPa) were tested for normal distribution with the Kolmogorov-Smirnov test and subjected to 1-way analysis of variance (ANOVA) and Tukey post hoc tests (α<.05); data were evaluated separately for each cement tested.

RESULTS

For both conventional and self-adhesive resin cements, the IDS technique (groups CET-IDS, CST-IDS, and SaT-IDS) promoted significant highest values for μTBS (*P*<.001), despite the mode of adhesive application (Fig. 3). For self-adhesive resin cement, the application of adhesive (SaT-IDS and SaT-DDS) promoted higher values (*P*<.001) of bond strength than groups without adhesive; and when no adhesive was used, as indicated by the manufacturer, the pretreatment of dentin with polyacrylic acid (SaTPa) enhanced bond strength (Fig. 3).

The main failure mode found for the conventional resin cement was mixed failure with fracture of the composite resin; and the main failure mode found for the self-adhesive resin cement tested was adhesive failure at the cement-dentin interface (Table 2, Figs. 4, 5). All failures in the groups with the lowest values of bond strength (Fig. 3, Sa and SaT) were classified as adhesive failures (Table 2).

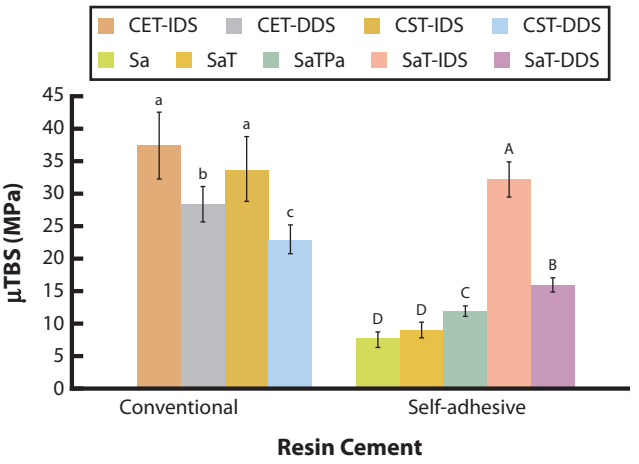

Figure 3. Bond strength of each cementation method. Different letters indicate statistical difference (*P*<.001). Lowercase letters refer to conventional cement. Uppercase letter refers to self-adhesive cement. C, conventional resin cement; DDS, delayed dentin sealing: interim cement applied before adhesive; E, etch-and-rinse approach; IDS, immediate dentin sealing: interim cement applied after adhesive; Pa, polyacrylic acid. S, self-etch approach; Sa, self-adhesive resin cement; T, interim cement.

Table 2. Failure mode according to each tested group

	% Failure Mode		
Group	Adhesive	Mixed	Cohesive
CET-IDS	37.5	62.5	0.0
CET-DDS	20.0	80.0	0.0
CST-IDS	25.0	62.5	12.5
CST-DDS	41.2	58.8	0.0
Sa	100.0	0.0	0.0
SaT	100.0	0.0	0.0
SaTPa	76.0	24.0	0.0
SaT-IDS	64.0	31.0	5.0
SaT-DDS	47.0	40.0	13.0

C, conventional resin cement; DDS, delayed dentin sealing: interim cement applied before adhesive; E, etch-and-rinse approach; IDS, immediate dentin sealing: interim cement applied after adhesive; Pa, polyacrylic acid. S, self-etch approach; Sa, self-adhesive resin cement; T, interim cement.

DISCUSSION

This study evaluated the influence of the timing of adhesive application before (IDS) or after application of interim cement (DDS) on microtensile bond strength between the resin cement and the dentin. IDS resulted in the highest values for μTBS for both cements tested, irrespective of the application method used for the adhesive (self-etch or etch-and-rinse), rejecting the null hypotheses. Freshly cut dentin, immediately after tooth preparation and before impression making, is

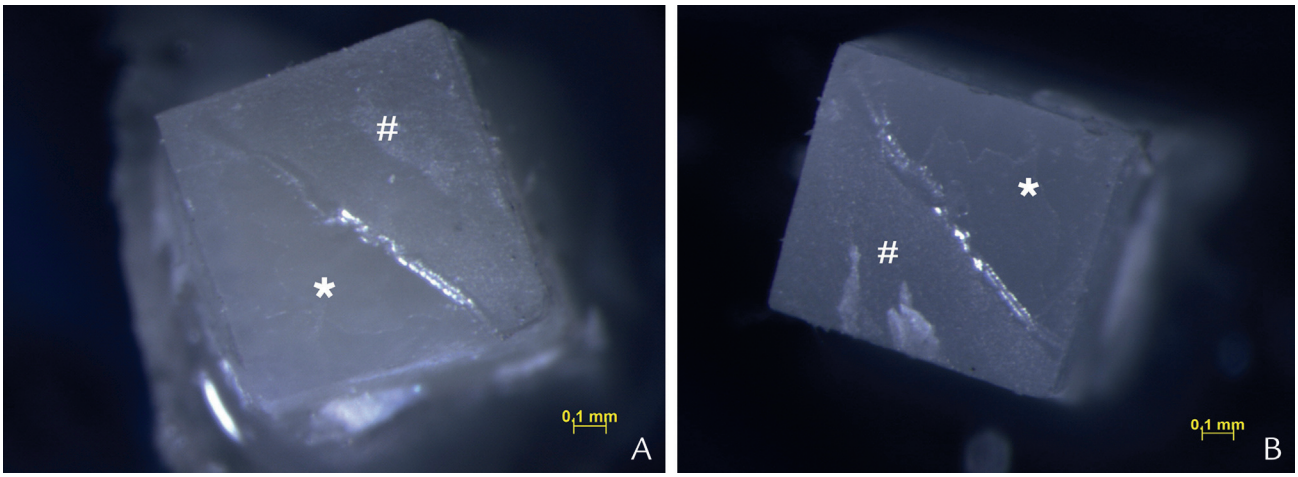

Figure 4. Representative image (original magnification ×50) of failure classified as mixed-cohesive failure of composite resin (*) associated to adhesive failure between conventional resin cement and dentin (#) (group CST-IDS). CST, conventional resin cement associated to self-etch adhesive and application of interim cement; IDS, immediate dentin sealing.

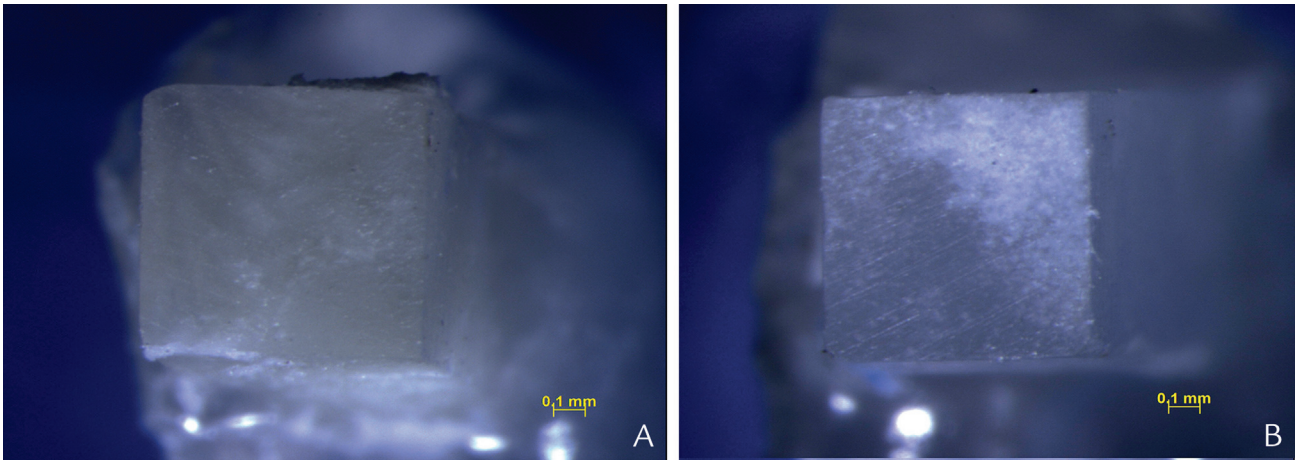

Figure 5. Representative image (original magnification ×50) of failure classified as adhesive – failure at self-adhesive resin cement (left)/dentin (right) interface (group Sa). Sa, self-adhesive.

considered ideal for bonding.[14] When it is freshly cut, the dentin fibrils are not collapsed and dentin is also not affected by dehydration or contaminated. Thus, the penetration of dental adhesives after demineralization is facilitated, and the formation of the hybrid layer is optimized.[14]

Bond strength results decreased after contamination with interim cements (DDS) compared with those observed with the IDS technique ($P<.001$). Previous studies[5,23] have demonstrated the same effect of interim cement on bond strength values. The removal of interim cement can be difficult. In the current study, the use of polyacrylic acid before applying self-adhesive resin cement (SaTPa group) improved the bond strength compared with the unetched group (SaT group) (Fig. 3). Polyacrylic acid is used as a dentin conditioner to enhance the adhesion of glass ionomer cements. It partially removes the smear layer[6] and probably any remaining interim cement, leaving free calcium and phosphate ions on the dentin surface, resulting in a better chemical reaction with self-adhesive cements.[2,6] Higher bond strength was also observed when dentin was etched with phosphoric acid after the application/removal of interim cement (CET-DDS group) when compared with self-etch adhesive (CST-DDS).

For the self-adhesive resin cement, the use of adhesive-enhanced bond strength values after the application of interim cement (SaT-DDS > SaT). The similarity between Sa and SaT may be explained by the initial acidity of self-adhesive cement (pH=2 in the first minute after mixing according to the manufacturer's information). It may decontaminate the dentin surface after removal of the interim cement. Even so, the high viscosity of the resin cement and the neutralizing effect

between cement and dentin may limit the demineralization and penetration of the cement into dentin,[16] resulting in adhesive failure at this interface (Table 1, Fig. 5). Thus, the application of an adhesive (SaT-IDS/DDS) improves the bond strength in this situation. The self-adhesive resin cement does not use the etch-and-rinse approach, which would increase leakage[13] and decrease bond strength[16]: the etched dentin is unable to increase the pH of acidic resin from self-adhesive resin cement, impairing resin cement conversion.

Another advantage of IDS is the efficient sealing of dentin,[14] which may provide protection of the pulp from microleakage. When the enamel is removed during preparation, 1 to 2 million dentin tubules are exposed,[15] and both interim and definitive restorations allow microleakage. Thus, sealing dentin is a preventive procedure, preserving pulp vitality.[9] In addition to these advantages, the IDS technique increases the fracture resistance of lithium disilicate veneers when bonded to dentin.[25]

The present study found that with a conventional resin cement, if an interim restoration is cemented before application of the adhesive, then an etch-and-rinse system is preferable to a self-etch system. The CET-DDS group presented higher bond strength values than the CST-DDS group (Fig. 3). Preliminary etching promotes demineralization of the dentin substrate, allowing resin monomers to penetrate into the collagen fibrils and creating a homogeneous hybrid layer and stable bonds.[10] The improved bond strength after dentin etching with polyacrylic or phosphoric acid may also be due to the rinsing after the etching. Rinsing removes contaminants from the interim cement, decreasing the surface energy and improving adhesion to dentin. In self-etch adhesives, the adhesive monomers are acidic and simultaneously demineralize and penetrate the dentin, incorporating the smear layer.[10] In the first situation, the application and rinsing of acid may contribute to the removal of any remaining interim cement. In the second situation, remaining interim cement is not removed and may impair bond strength.

The results of the present investigation should not be extrapolated to the cementation of endodontic posts. Inside the root canal, other factors are involved regarding adhesive application, including the application of adhesive to the entire root dentin, acid etching, and polymerization difficulties within the root canal and the reduced number of dentin tubules.[11] In these situations, self-adhesive resin cement without the application of adhesives is indicated.[12]

The geometric form of abutment, vitality of the tooth, and pulpal pressure were not simulated in the present study but may be factors of influence in clinical situations. Long-term clinical trials are needed to determine the optimal timing of adhesive application and the influence of interim cement.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Immediate dentin sealing led to the highest bond strength values between resin cement and crown dentin for both conventional and self-adhesive resin cements.
2. The use of interim cement before adhesive application reduces adhesion to dentin.

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