

Bond Durability of Different Resin Cements to Caries-Affected Dentin Under Simulated Intrapulpal Pressure

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Clinical Relevance

Resin cement with self-etch adhesive containing methacryloyloxydecyl dihydrogen phosphate shows promise in obtaining homogenous and durable bonding to normal and caries-affected dentin.

SUMMARY

Objective: To evaluate the durability of the bond of different resin cement systems to normal dentin (ND) and caries-affected dentin (CAD) with and without simulated intrapulpal pressure (IPP).

Methods and Materials: Molars with midcoronal caries were used. Occlusal enamel was cut to expose both dentin substrates (ND and CAD). Dentin substrates were differentiated

using visual, tactile, caries-detecting dye, and dye-permeability methods. Prepared crown segments were equally divided according to the tested resin cement systems: etch-and-rinse resin cement, self-etch resin cement containing methacryloyloxydecyl dihydrogen phosphate (MDP), and self-adhesive resin cement. In addition to the dentin substrates and the resin cement types, the effect of application/storage conditions (with or without simulated IPP and with or without thermocycling) were tested. A microtensile bond strength test was done using a universal testing machine. Failure modes were determined using a scanning electron microscope.

Results: Etch-and-rinse resin cement strength values were significantly affected by the difference in the dentin substrates as well as the different application/storage conditions. Self-etch adhesive containing MDP bonded equally to ND and CAD and remained stable under all tested conditions. Self-adhesive resin cement revealed a similar bond to ND and CAD; however, its values were the lowest, especially

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when IPP and thermocycling were combined. Mixed failure was the predominant failure mode.

Conclusions: Etch-and-rinse resin cement was sensitive to dentin substrate and application/storage conditions. Resin cement with self-etch adhesive containing MDP revealed more reliable bonding to ND/CAD even when IPP and thermocycling were combined. The bonding of the self-adhesive resin cement could not compete with other resin cements.

INTRODUCTION

Advances in adhesive dentistry have provided solutions to many esthetic challenges faced by clinicians. In certain clinical situations, indirect resin composite restorations represent an alternative to direct ones due to some advantages such as the ease of developing and maintaining occlusal surface anatomy, contours, and contacts.¹ Nevertheless, one of the prime requirements for achieving successful indirect composite restorations is to gain proper bonding to the tooth structure.² Resin cements are increasingly used for bonding indirect restorations due to their better bond strengths, excellent mechanical properties, and improved esthetics when compared with conventional cements.³ Currently, resin cements are used with etch-and-rinse or self-etch adhesives. However, the increased tendency to simplify and reduce the bonding steps has led to combining the adhesive system and the cement in a single application through the use of self-adhesive resin cements.⁴

At the same time, bonding to tooth structure depends not only on adhesive systems but also on the bonding substrates.⁵ Many of the dentin bonding studies were done on normal human dentin. However, this is not the substrate frequently encountered in clinical dentistry.⁶ Caries-affected dentin (CAD) has different structural and compositional characteristics compared with normal dentin (ND). CAD also has shown different permeability with intrapulpal pressure.⁷ Thus, bonding to this dynamic substrate is crucial and appears to influence the long-term durability of adhesive systems. Consequently, it would be of interest to evaluate the bonding performance of the different resin cement systems with different adhesive strategies to ND and CAD after the application of simulated intrapulpal pressure with and without thermocycling.

The null hypotheses were 1) the different types of dentin substrates (ND and CAD) had no impact on

dentin/resin cement bond strength; 2) there was no difference in the microtensile bond strength among the different resin cement systems of different adhesive strategies; and 3) the different application/storage conditions (intrapulpal pressure simulation or thermocycling alone or in combination) had no effect on the bond strength of different resin cements to dentin.

METHODS AND MATERIALS

Specimen Preparation

A total of 120 molars with midcoronal caries (site and size = 1.2) were used.⁸ The collected teeth were stored in phosphate buffer saline containing 0.2% sodium azide at 4°C for not more than two weeks until being used.⁹ The study was accomplished in accord with local human participants' oversight committee guidelines.

Occlusal enamel was trimmed perpendicularly to the long axis of each tooth using a slow-speed diamond-saw sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant to expose flat ND and CAD surfaces. Ground dentin surfaces were examined for any signs of exposure to be discarded. Another cut was made parallel to the occlusal surface, 2 mm below the cemento-enamel junction, exposing the pulp chamber. Remnants of pulp tissue in the pulp chamber were removed using a discoid excavator (Carl Martin GmbH, Solingen, Germany) without touching the walls of the pulp.¹⁰ Each crown segment was mounted on a polymethacrylate plate containing a 19-gauge needle in the center using cyanoacrylate adhesive (Rocket Heavy, Dental Ventures of America, Corona, CA, USA) and subsequently embedded in chemically cured polyester resin (Polyester resin #2121, Hsein, Taiwan) up to the level of the cemento-enamel junction.¹⁰

CAD Identification

CAD was differentiated with the aid of visual and tactile methods.¹¹ In addition, caries-detecting dye¹² and the dye permeability test¹³ were used. In the dye permeability test, 10% methylene blue was permeated into the tooth through the pulp chamber under pressure. Caries-detecting dye appears to stain partially demineralized collagen matrices in CAD.¹⁴ The selective staining of ND with methylene blue was attributed to decreased permeability of CAD in relation to ND due to the presence of peritubular and intertubular crystal formation into the dentinal tubules. Thus, the ND was stained

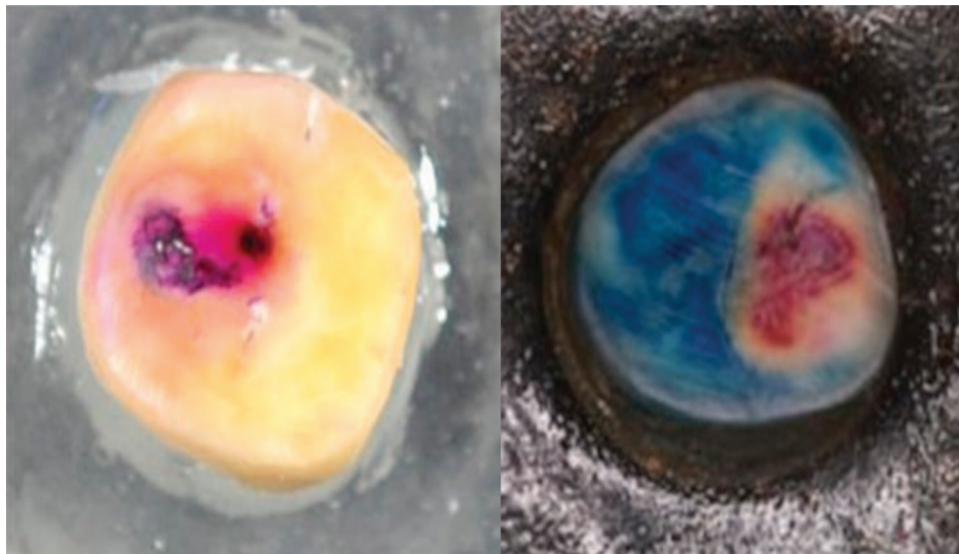


Figure 1. Caries-affected dentin identification using caries-detecting dye and methylene-blue dye permeability test.

blue, whereas the CAD was stained a pale pink (Figure 1). The dentin surface was flattened using a rotary grinding machine under water coolant and wet hand polished using 600-grit silicon carbide paper (MicroCut, 8 inch, Buehler Ltd, Lake Bluff, IL, USA) for 10 seconds to produce a standardized smear layer.¹³

Restorative Procedures

The crown segments with ND and CAD were divided equally according to the type of resin cement system (resin cement with etch-and-rinse adhesive [Variolink II/Adper Scotch Bond Multi-Purpose, VL, 3M ESPE, St Paul, MN, USA], resin cement with self-etch adhesive containing methacryloyloxydecyl dihydrogen phosphate [MDP; Panavia F2.0/ED primer II, PF, Kuraray Medical, New York, NY, USA], or self-adhesive resin cement [RelyX Unicem 2, RX, 3M ESPE]). Table 1 shows materials specifications, manufacturers, compositions, and batch numbers. The prefabricated resin composite blocks (4-mm thickness) were made using microhybrid resin composite (Filtek Z250, 3M ESPE) and light cured for 40 seconds from the top and the bottom using an LED light-curing unit (Guilin Woodpecker Medical Instrument Company, London, UK) with an intensity of 800 mW/cm². The resin composite blocks were subjected to additional light and heat curing at 110°C for seven minutes, using an oven (Coltene/Whaledent, Inc. DI-500, Cuyahoga Falls, OH, USA) to ensure complete polymerization. The bonding surface of each block was sandblasted with 50 µm alumina particles for 20 seconds using the Renfert sandblasting device

(US Dental Depot, Fort Lauderdale, FL, USA). To standardize the distance to 1 cm and the angle to be 90° between the resin composite block and the nozzle handpiece of the sandblasting device, a specially constructed assembly was used. Following sandblasting, chemical surface treatment was done using Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) silane coupling agent. The silane was applied to the sandblasted surface using a microbrush, left for one minute, and then air dried using an air spray syringe.

The cementation procedure was carried out according to manufacturers' instructions (Table 2) while the specimens were still connected to the intrapulpal pressure assembly under 15 mm Hg (P₁), reproducing the effect of the local vasoconstrictor in local anesthetics,¹⁵ or not (P₀). After bonding of the P₁ specimens, the intrapulpal pressure was raised to 20 mm Hg. The specimens of P₀ and P₁ were then inserted in plastic containers containing artificial saliva. The intrapulpal pressure assembly and the specimens in the plastic containers were placed in a specially constructed large incubator at 37°C for 24 hours (Figure 2). After incubation, the specimens were sectioned to obtain multiple sticks (0.9±0.01 mm²). A precise digital caliper (Proficraft, Mebschieber, Germany) was used to check the cross-sectional area and length of the sticks. Sticks (n=2 for each dentin substrate/specimen) of the same cross-sectional area and length were selected. Then, half of the sticks (ND and CAD) were subjected to 10,000 thermocycling cycles between 5°C and 55°C with a dwell time of 30 seconds and a transfer time of 10 seconds¹⁶ (T₁) and the other half were not (T₀).

Table 1: *Material Specifications, Manufacturers, Compositions, and Batch Numbers*

Material Specification	Manufacturer	Composition	Batch No.
Etch-and-rinse resin cement system Variolink II resin cement	Ivoclar Vivadent Schaan, Liechtenstein	Monomer matrix: Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate. Fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide.	39409
Etch-and-rinse adhesive system Adper Scotch Bond MultiPurpose	3M ESPE Dental Products St Paul, MN, USA	Additional contents: catalysts, stabilizers, pigments. The base contains 26.3% wt monomer and 73.4% wt filler, whereas the catalyst (high viscosity) contains 22% wt monomer and 77.2% wt filler. Etchant: 37% phosphoric acid (pH < 1) Primer: ethanol, HEMA and polyalkenoic acid Adhesive: HEMA and Bis-GMA	75405
Self-etch resin cement Panavia F2.0	Kuraray Medical, New York, NY, USA	Paste A: MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aromatic dimethacrylate, silanated silica filler, silanated colloidal silica, di-camphorquinone, catalysts, initiators Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, surface-treated sodium fluoride, catalysts, accelerators, pigments	0256AA
Single-step self-etch adhesive system ED primer II	Kuraray Medical, New York, NY, USA	Liquid A: HEMA, MDP, water, 5-NMSA, accelerators Liquid B: 5-NMSA, water, catalysts, accelerators Oxyguard II: glycerol, polyethyleneglycol catalysts, accelerators, dyes.	0509AA
Self adhesive resin cement RelyX Unicem 2	3M ESPE Dental Products St Paul, MN, USA	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers initiators, stabilizers Catalyst paste: methacrylate monomers, alkaline fillers, silanated fillers, initiators, stabilizers, pigments	424967
Filtek Z250	3M ESPE Dental Products St Paul, MN, USA	Resin: Bis-GMA, UDMA, bis-EMA Fillers: (60%) zirconia/silica with particle size 0.01-3.5 µm	138842

Abbreviations: Bis-EMA, bisphenol ethoxylated dimethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; 5-NMSA, N-methacryloyl-5-aminosalicylic acid; HEMA, 2-hydroxyethylmethacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate.

Microtensile Bond Strength Testing

Each stick was fixed to the modified Academisch Centrum Tandheelkunde Amsterdam (ACTA) micro-tensile strength jig¹⁷ with a cyanoacrylate adhesive (Rocket Heavy) and stressed in tension using a universal testing machine (Lloyd Instruments Ltd, Ametek Company, Bognor Regis, West Sussex, UK) at a cross-head speed of 0.5 mm/min until failure. The tensile force at failure was recorded and

converted to tensile stress in megapascal units using computer software (Nexygen-MT, Lloyd Instruments). Sticks that failed before testing were counted as 0 MPa.^{10,18} Bond strength values were submitted to a three-way analysis of variance (ANOVA) with repeated measures to determine the effect of dentin substrates, the resin cements systems, and application/storage conditions. It was also used to detect any significant interactions among these three variables. One-way ANOVA was

Table 2: Adhesive Systems Application and Resin Cements Manipulation

Resin Cement System	Adhesive Systems Application Steps	Resin Cements Manipulation
Etch-and-rinse resin cement system (Adper Scotch Bond MultiPurpose and Variolink II resin cement)	-Etchant: applied for 15 seconds, rinsed with water spray for 15 seconds, then dried with gentle air flow for 5 seconds. -Adper Scotch Bond MultiPurpose primer: applied, left undisturbed for five seconds, then gently thinned with a mild oil-free air stream for five seconds and at a distance from the dentin surface of 2 cm. -Adper Scotch Bond MultiPurpose adhesive: applied, left undisturbed for 15 seconds, and light cured for 10 seconds	The base and catalyst pastes of Variolink II were applied in a 1:1 ratio and mixed for 10 seconds then applied on treated dentin surface. The resin composite onlay was then cemented under a specified load, and the excess cement was removed using a sponge followed by light curing for 20 seconds.
Single-step self-etch resin cement system (ED primer II and Panavia F2.0 resin cement)	- One drop of liquid A and B of ED Primer was mixed on a glass slab, applied on the dentin surface, and left undisturbed for 30 seconds. -The excess was removed with a sponge, then dried with gentle air flow for five seconds and at distance of 2 cm from dentin surface.	-Equal amounts of paste A and paste B of resin cement was mixed on a glass slab for 20 seconds and applied on the treated surface of composite inlay. -Resin composite onlay was then cemented under a specified load, and the excess cement was removed using a sponge followed by light curing for 20 seconds -Oxyguard II was applied directly to the margins of the restoration, left for three minutes, and then removed.
Self-adhesive resin cement RelyX Unicem 2	No dentin surface pretreatment was done	-Equal amounts of base and catalyst pastes were dispensed, then mixed for 20 seconds. -The mix was applied on the treated dentin surface. -The resin composite onlay was then cemented under a specified load, and the excess cement was removed using a sponge followed by light curing for 20 seconds.

used to test the significant difference among the tested resin cements bonded to each dentin substrate with each application/storage condition as well as for the significant difference among the application/storage conditions for each resin cement bonded to each dentin substrate. Pairwise comparisons were calculated using the Bonferroni test. The *t*-test was used to test the significant difference between the bond strength values to ND and CAD with each resin cement under each application/storage condition. A probability test set at $\alpha=0.05$ was used for statistical significance. All statistical calculations were done using computer program SPSS version 15 (IBM SPSS statistics, Armonk, NY, USA) for Microsoft Windows.

The failure modes of all specimens were evaluated using a scanning electron microscope (SEM) at 100× magnification. Failure modes were classified as type 1: adhesive failure along the dentin side; type 2: cohesive failure in the adhesive layer; type 3: cohesive failure in the resin cement; type 4: mixed

failure (adhesive failure along the dentin side and cohesive failure in the adhesive layer); type 5: mixed failure (adhesive failure along the dentin side and cohesive failure in the resin cement); type 6: mixed failure (adhesive failure along the dentin side, cohesive failure in the adhesive layer and cohesive failure in the resin cement); and type 7: mixed failure (cohesive failure in the adhesive layer and cohesive failure in the resin cement).

RESULTS

The three-way repeated measures ANOVA results showed a statistically significant effect for the dentin substrates (ND and CAD) ($p<0.001$), the resin cement systems (VL, PF, and RX) ($p<0.001$), and the application/storage conditions (P_0T_0 [control], P_1T_0 , P_0T_1 , P_1T_1) ($p=0.021$). The interaction between resin cement systems and application/storage conditions was also significant ($p=0.002$). Nevertheless, the interactions among dentin substrates × resin cement systems, dentin substrates × applica-

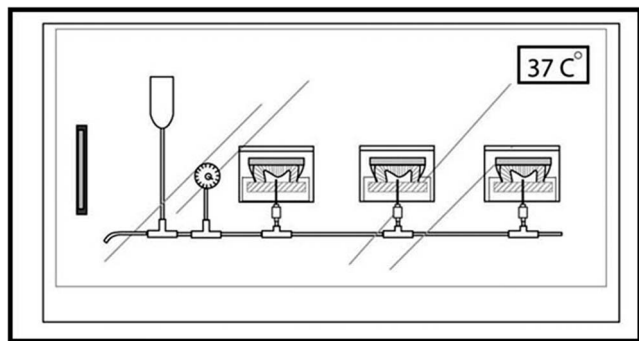


Figure 2. A diagram showing the specimens in the incubator while immersed in artificial saliva and subjected to intrapulpal pressure simulation.

tion/storage conditions, and dentin substrates \times resin cement systems \times application/storage conditions were not significant ($p > 0.05$). Means and standard deviations (SD) of microtensile bond strength (μ TBS) values of all tested variables are presented in Table 3. VL showed higher bond strength to ND compared with CAD after 24 hours with P_0T_0 ($p = 0.02$) and with P_0T_1 ($p = 0.04$); whereas, with the other application/storage conditions no significant differences were recorded. Also, the bond strength values of PF and RX to ND were not significantly different from those to CAD under any application/storage condition.

Regarding the bond strength among the different resin cements with each application/storage condition, one-way ANOVA revealed a statistically significant difference among them whether bonded to ND or to CAD when subjected to P_0T_0 and P_1T_1 conditions (Table 3). For ND with the P_0T_0 condition,

VL bond strength values showed statistically significantly higher value compared with PF and RX, although no statistically significant difference was recorded between PF and RX values (Table 3). For the P_1T_1 condition, RX revealed the lowest value. For CAD with the P_0T_0 and P_1T_1 conditions, RX also revealed the lowest value.

VL bond strength values to ND and CAD were significantly different with the tested application/storage conditions; RX bond strength values significantly decreased after P_1T_1 application/storage condition. Nevertheless, values for PF bond strength to both dentin substrates were not statistically changed with all tested application/storage conditions (Table 3).

Regarding the failure modes, Figure 3 shows the percentages of the recorded failure modes. VL bonded to ND and CAD showed predominately type 6 mode of failure, whereas PF specimens displayed type 5. RX bonded to ND commonly showed type 1 failure, whereas with CAD, type 5 was the most frequent failure mode. Representative SEM micrographs for some failure modes of different resin cements bonded to either ND or CAD are presented in Figure 4.

DISCUSSION

Results of the present study indicate the rejection of the three null hypotheses because the difference in dentin substrates (ND and CAD) has a significant effect on dentin/resin cement bond strength. Also, there was a statistically significant difference among the three types of resin cements. Moreover, the difference in application/storage conditions showed a

Table 3: Mean and Standard Deviation (SD) of the Microtensile Bond Strength (μ TBS) Values (MPa) of the Tested Variables^a

Dentin Substrates	Application/Storage Conditions	Resin Cement Systems			p-Value
		Variolink II	Panavia F2.0	Rely X Unicem 2	
Normal dentin (ND)	Artificial saliva (control) P_0T_0	35.2 \pm 5.8 ^{*Aa}	24.9 \pm 6.1 Ba	20.5 \pm 2.6 Ba	<.01
	IPP simulation P_1T_0	26.3 \pm 6.0 Ab	24.4 \pm 4.6 Aa	19.5 \pm 5.3 Aa	.07
	Thermocycling P_0T_1	26.6 \pm 6.7 ^{**Ab}	26.0 \pm 5.6 Aa	21.2 \pm 7.1 Aa	.26
	IPP simulation/thermocycling P_1T_1	20.3 \pm 4.0 Ac	19.6 \pm 1.9 Aa	16.4 \pm 2.3 Ba	.04
p-value		<.001	.07	.27	
Caries-affected dentin (CAD)	Artificial saliva (control) P_0T_0	28.8 \pm 2.8 ^{*Aa}	23.7 \pm 6.0 Aa	17.9 \pm 4.7 Ba	<.01
	IPP simulation P_1T_0	27.0 \pm 8.0 Aa	20.2 \pm 7.0 Aa	18.6 \pm 7.1 Aa	.10
	Thermocycling P_0T_1	20.3 \pm 3.2 ^{**Ab}	21.4 \pm 3.6 Aa	22.5 \pm 2.5 Aa	.41
	IPP simulation/thermocycling P_1T_1	22.3 \pm 3.0 Ab	22.9 \pm 2.1 Aa	13.7 \pm 3.8 Bb	<.01
p-value		<.01	.57	.02	

Abbreviations: P_0T_0 , specimens stored in artificial saliva for 24 hours, neither subjected to intrapulpal pressure (IPP) simulation nor to thermocycling; P_1T_0 , specimens subjected to intrapulpal simulation (IPP) but not subjected to thermocycling; P_0T_1 , specimens subjected to thermocycling but not IPP simulation; P_1T_1 , specimens subjected to IPP simulation and thermocycling.

^a Different capital letters denote significant differences within rows, whereas different small letters denote significant differences within a column for each dentin substrate. * and ** indicate differences between ND and CAD for the same resin cement subjected to the same storage condition.

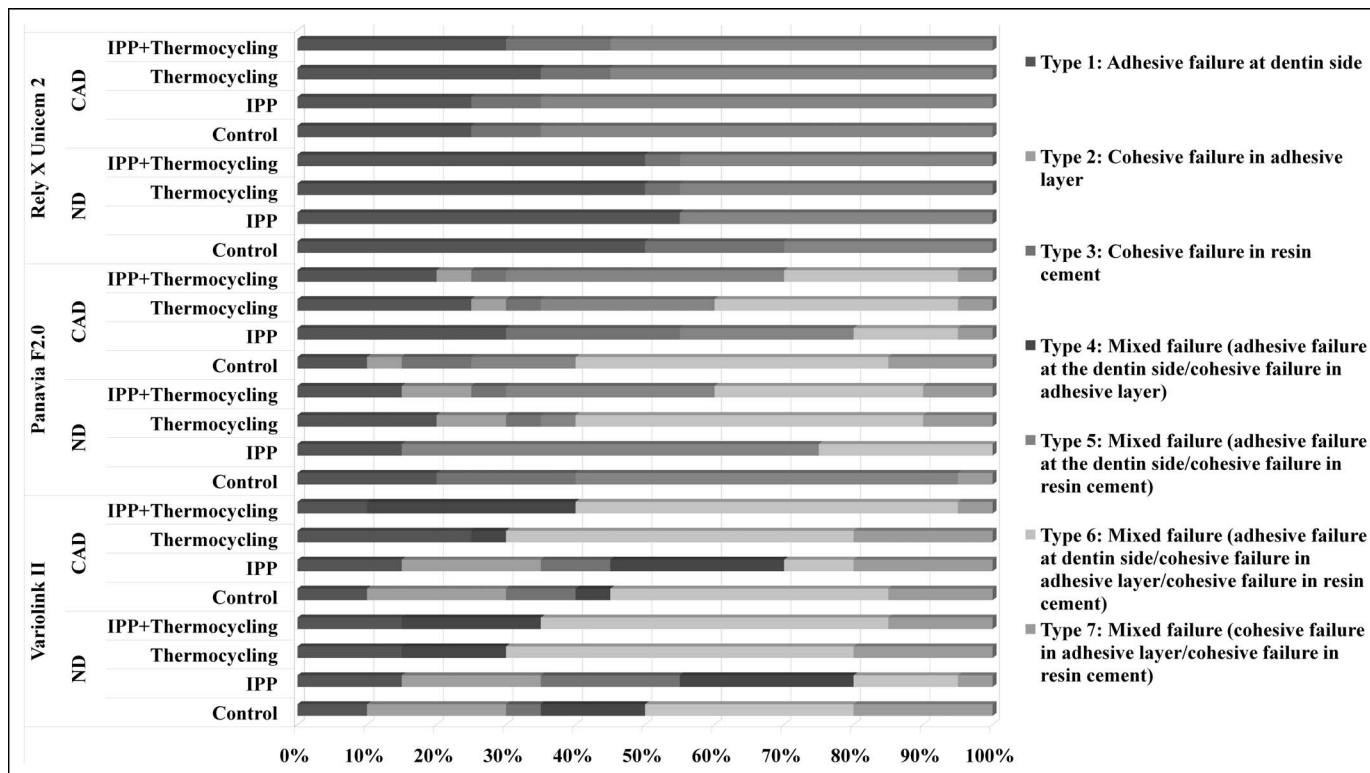


Figure 3. The percentages of the recorded modes of failure.

significant effect on the bond strength to different dentin substrates.

In the present study, the different resin cement systems bonded differently with the different dentin substrates (ND and CAD). The significant effect for the difference in dentin substrates on bonding was recorded by others, although they tested other adhesive systems.¹⁹⁻²²

VL bonded to ND showed significantly higher bond strength values than those of PF and RX resin cement systems. The better bonding of etch-and-rinse adhesive systems over other systems has been previously reported.²³ The superiority of the etch-and-rinse resin cement system in bonding to ND might be attributed to the effect of phosphoric acid etching to dentin, which results in removal of the smear layer, thus allowing proper resin infiltration of the dentin.²⁴ The primer component of the etch-and-rinse adhesive Adper Scotch Bond MultiPurpose used with VL resin cement contains ethanol, which helps to remove the residual water from the collagen matrix and keeps the expanded collagen matrix stiffened, allowing for better resin infiltration.²⁵ Moreover, the hydrophobic bisphenol glycidyl methacrylate (Bis-GMA) and the hydrophilic 2-hydroxyethyl methacrylate (HEMA) components of VL are

soluble in ethanol.²⁶ This allows for more resin infiltration into acid-etched matrices, forming well-hybridized resin tags that contribute to micromechanical retention.³ On the other hand, the lower μ TBS of the RX system compared with VL etch-and-rinse resin cement could be attributed to its mode of adhesion, which counts on smear layer penetration rather than its removal; thus, a weak link is expected to be formed between the resin cements and the underlying dentin.²⁷ Although the RX can form a chemical bond with the smear layer-covered dentin,²⁸ this reaction is superficial with no hybrid layer formation.²⁹ The nonsignificant difference between the PF and RX when bonded to ND was in accordance with De Munck and others,⁴ Abo-Hamar and others,³⁰ and Tonial and others.³¹ However, Abo-Hamar and others³⁰ disagreed with the results of the present study because they found that the bond strength of VL was not significantly different from those of PF and RX. This could be because they used a different etch-and-rinse adhesive system (Syntac) with the VL resin cement. Yang and others³ showed that the bond strength of PF was higher than that of RX, which also contradicted the present study's findings. This can be attributed to the mode of cure used for the RX in their study (self-cure mode) and the dual-cure mode used in the present

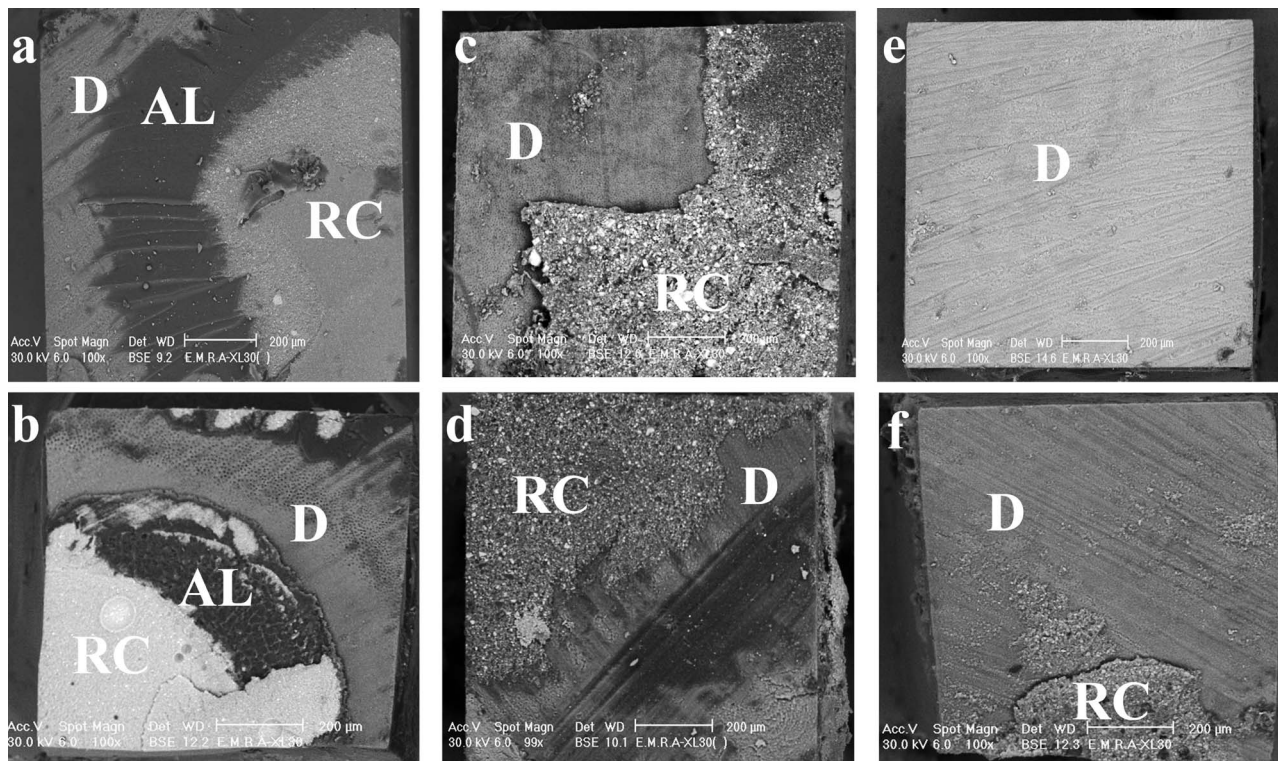


Figure 4. SEM photomicrographs showing the predominant failure modes of Variolink II resin cement (a): bonded to ND and (b): bonded to CAD. Panavia F2.0 resin cement (c): bonded to ND and (d): bonded to CAD. RelyX Unicem 2 resin cement (e): bonded to ND and (f): bonded to CAD. AL, adhesive layer; D, dentin; RC, resin cement.

study. It was reported that the degree of conversion of RX with the self-cure mode was lower than that with either the light- or dual-cure mode.³²

Regarding the CAD, VL (etch-and-rinse resin cement) bond strength values were greatly affected. The decrease in bond strength of etch-and-rinse adhesives bonded to CAD has also been reported by other researchers.^{12,21,22} In CAD, the intertubular dentin has been, to some extent, demineralized due to the caries process before acid etching. After the etching step with the VL resin cement system, the demineralized layer might be too deep to be efficiently infiltrated by the adhesive, including the whole demineralized substrate thickness, resulting in a defective hybrid layer.²¹ In addition, it has been reported that CAD contains deposits of β -tricalcium phosphate (β -TCP; also called whitlockite) in the dentinal tubules. These demineralized/remineralized portions in the same carious lesion cause disproportional and nongradient infiltration of resin monomers into the CAD substrate.³³ The high hydrophilic, unsaturated, methacrylate phosphate ester functional monomer (10-MDP) content in the PF self-etch adhesive resin cement system allows an intense chemical interaction with hydroxyapatite³⁴

that is highly available in CAD. This interaction could promote reliable and better hybridization. As for RX, it incorporates two setting reactions: a dual-cured redox reaction for polymerization of the resinous phase and an acid-base reaction resulting in the formation of calcium phosphates. Bonding with dentin is established by the ionization of phosphoric acid methacrylates of the monomer mixture. This ionization occurs either *in situ* from the water content of the dentin or from that produced during the neutralization reaction of the phosphate monomers with the basic filler of the cement.³⁵ The bonding mechanism is essentially similar to glass ionomers with an intermediate interfacial layer incorporating partially dissolved smear particles and possible regional formation of a nano-hybrid layer.³⁶

Regarding subsection to the different application/storage conditions, VL (etch-and-rinse) resin cement showed a drop in bond strength to ND with P_1T_0 compared with those bonded and stored with P_0T_0 ; whereas, PF and RX resin cements revealed no significant difference from P_1T_0 . The cause of the drop in bonding of VL may be related to the removal of the smear layer through acid etching, which may

have increased the outward flow of dentinal fluid along the dentinal tubules.³⁷ The outward flow could counteract the adhesive monomer penetration, dilute their concentration, and prevent the optimal polymerization.³⁸

On the contrary, in the PF group applied under intrapulpal pressure (P_1), the smear plugs remained in the dentinal tubules so that under simulated intrapulpal pressure, moisture contamination by dentinal fluids transudation would be minimal.²⁸ This helps to provide a barrier against the effect of outward flow of dentinal fluids. The RX self-adhesive resin cement can benefit from the presence of intrapulpal pressure because it can provide additional hydration to the dentin. The presence of water is believed to optimize the chemical reaction of the self-adhesive resin cement with dentin.³⁹ In addition, the protective effect of the smear layer may accentuate the nonsignificant effect of intrapulpal pressure on this resin cement type.³⁹

Thermocycling is considered as an adjunct to assess the effect of thermal stresses and prolonged water exposure on the bond strength to dentin.⁴⁰ The protocol for thermocycling applied in this study for all resin cements, which was 10,000 cycles, was reported to correspond to one year of *in vivo* simulation.⁴¹ The results of this study showed that thermocycling had no significant effect on the bond strength of PF and RX resin cements to ND and CAD. The chemical bond formed between hydroxyapatite and RX and the functional phosphate monomer (MDP) of PF, can account for this dentin bond stability.⁴² This chemical bond was reported not to dissociate in water, according to the adhesion-decalcification concept.⁴³ The VL showed also a decrease in bond strength after thermocycling (T_1) compared with its bond strength without intrapulpal pressure and thermocycling (P_0T_0). The HEMA content in the Adper Scotch Bond MultiPurpose adhesive system used might be the cause of the compromised bond strength obtained. HEMA is likely to absorb large amounts of water within the adhesive and the hybrid layer during thermocycling; hence, water that remains entrapped at the resin-dentin interface jeopardizes the stability of the bond.¹⁵

One of the new findings of the present study was that the combination between bonding under intrapulpal pressure simulation P_1 and thermocycling T_1 dramatically decreased the bond strength of VL and RX resin cement systems. With respect to RX, the chemical bond formed between the phosphoric acid monomer and calcium of hydroxyapatite that resist-

ed the effect of P_1 or T_1 alone was not sustained when the conditions were combined. Both conditions could have synergistic action, causing added degradation of bond strength. The recorded predominant adhesive mode of failure for RX when P_1 and T_1 were combined may justify this explanation.

The results of this study highlight that various structural components and properties of dentin, as well as biological and clinical factors of the oral cavity, could directly affect the adhesive bond. Therefore, much of our understanding of dental bonding has to be tested on normal as well as on clinically relevant caries-affected substrate faced in dental practice while simulating the *in vivo* conditions.

CONCLUSIONS

Etch-and-rinse resin cement is sensitive to dentin substrate and storage conditions. Resin cement with self-etch adhesive containing MDP revealed more reliable bonding to ND and CAD even when combining intrapulpal pressure and thermocycling. Self-adhesive resin cement bonding still cannot compete with other resin cements.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

- Heymann H, Swift EJ, Ritter AV, & Sturdevant CM (2012) *Sturdevant's Art and Science of Operative Dentistry* Elsevier/Mosby St Louis, Mo **6th edition**.
- Hikita K, Van Meerbeek B, De Munck J, Ikeda T, Van Landuyt K, Maida T, Lambrechts P, & Peumans M (2007) Bonding effectiveness of adhesive luting agents to enamel and dentin *Dental Materials* **23(1)** 71-80.
- Yang B, Ludwig K, Adelung R, & Kern M (2006) Micro-tensile bond strength of three luting resins to human regional dentin *Dental Materials* **22(1)** 45-56.
- De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, & Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin *Dental Materials* **20(10)** 963-971.
- Wang Y, Spencer P, & Walker MP (2007) Chemical profile of adhesive/caries-affected dentin interfaces using Raman microspectroscopy *Journal of Biomedical Materials Research Part A* **81(2)** 279-286.
- Haj-Ali R, Walker M, Williams K, Wang Y, & Spencer P (2006) Histomorphologic characterization of noncarious

- and caries-affected dentin/adhesive interfaces *Journal of Prosthodontics* **15**(2) 82-88.
7. Nakajima M, Kunawarote S, Prasansuttiporn T, & Tagami J (2011) Bonding to caries-affected dentin *Japanese Dental Science Review* **47**(2) 102-114.
 8. Mount GJ, Tyas JM, Duke ES, Hume WR, Lasfargues JJ, & Kaleka R (2006) A proposal for a new classification of lesions of exposed tooth surfaces *International Dental Journal* **56**(2) 82-91.
 9. Mobarak EH, El-Badrawy W, Pashley DH, & Jamjoom H (2010) Effect of pretest storage conditions of extracted teeth on their dentin bond strengths *Journal of Prosthetic Dentistry* **104**(2) 92-97.
 10. El-Deeb HA, Al Sherbiney HH, & Mobarak EH (2013) Bond durability of adhesives containing modified-monomer with/without fluoride after aging in artificial saliva and under intrapulpal pressure simulation *Operative Dentistry* **38**(1) 48-56.
 11. Nakajima M, Sano H, Zheng L, Tagami J, & Pashley DH (1999) Effect of moist vs dry bonding to normal vs caries-affected dentin with Scotchbond Multi-Purpose Plus *Journal of Dental Research* **78**(7) 1298-1303.
 12. Erhardt MC, Toledano M, Osorio R, & Pimenta LA (2008) Histomorphologic characterization and bond strength evaluation of caries-affected dentin/resin interfaces: Effects of long-term water exposure *Dental Materials* **24**(6) 786-798.
 13. Mobarak EH, & El-Badrawy WH (2012) Microshear bond strength of self-etching adhesives to caries-affected dentin identified using the dye permeability test *Journal of Adhesive Dentistry* **14**(3) 245-250.
 14. Alleman DS, & Magne P (2012) A systematic approach to deep caries removal end points: The peripheral seal concept in adhesive dentistry *Quintessence International* **43**(3) 197-208.
 15. Sauro S, Mannocci F, Toledano M, Osorio R, Thompson I, & Watson TF (2009) Influence of the hydrostatic pulpal pressure on droplets formation in current etch-and-rinse and self-etch adhesives: A video rate/TSM microscopy and fluid filtration study *Dental Materials* **25**(11) 1392-1402.
 16. Ulker M, Ozcan M, Sengun A, Ozer F, & Belli S (2010) Effect of artificial aging regimens on the performance of self-etching adhesives *Journal of Biomedical Materials Research Part B Applied Biomaterials* **93**(1) 175-184.
 17. Mobarak E, El-Deeb H, & El-Samman M (2013) The difference in microtensile-bond strength jigs influences the test outcome *Journal of Dental Research* **92**(Special Issue B) (IADR Irish Division Annual Scientific Meeting) Retrieved online 15 Nov, 2013 from: <http://www.iadr.ie/wp-content/uploads/2013/11/IADR-Irish-Division-Annual-Scientific-Meeting-2013-No-headers-new.p>
 18. Van Landuyt KL, Mine A, De Munck J, Jaecques S, Peumans M, Lambrechts P, & Van Meerbeek B (2009) Are one-step adhesives easier to use and better performing? Multifactorial assessment of contemporary one-step self-etching adhesives *Journal of Adhesive Dentistry* **11**(3) 175-190.
 19. Ceballos L, Camejo DG, Victoria Fuentes M, Osorio R, Toledano M, Carvalho RM, & Pashley DH (2003) Microtensile bond strength of total-etch and self-etching adhesives to caries-affected dentine *Journal of Dentistry* **31**(7) 469-477.
 20. Omar H, El-Badrawy W, El-Mowafy O, Atta O, & Saleem B (2007) Microtensile bond strength of resin composite bonded to caries-affected dentin with three adhesives *Operative Dentistry* **32**(1) 24-30.
 21. Scholtanus JD, Purwanta K, Dogan N, Kleverlaan CJ, & Feilzer AJ (2010) Microtensile bond strength of three simplified adhesive systems to caries-affected dentin *Journal of Adhesive Dentistry* **12**(4) 273-278.
 22. Yoshiyama M, Urayama A, Kimochi T, Matsuo T, & Pashley DH (2000) Comparison of conventional vs self-etching adhesive bonds to caries-affected dentin *Operative Dentistry* **25**(3) 163-169.
 23. Luhrs AK, Guhr S, Gunay H, & Geurtsen W (2010) Shear bond strength of self-adhesive resins compared to resin cements with etch and rinse adhesives to enamel and dentin *in vitro Clinical Oral Investigations* **14**(2) 193-199.
 24. Pashley DH, Agee KA, Nakajima M, Tay FR, Carvalho RM, Terada RS, Harmon FJ, Lee WK, & Rueggeberg FA (2001) Solvent-induced dimensional changes in EDTA-demineralized dentin matrix *Journal of Biomedical Materials Research* **56**(2) 273-281.
 25. Carvalho RM, Mendonca JS, Santiago SL, Silveira RR, Garcia FC, Tay FR, & Pashley DH (2003) Effects of HEMA/solvent combinations on bond strength to dentin. *Journal of Dental Research* **82**(8) 597-601.
 26. Guimaraes LA, Almeida JC, Wang L, D'Alpino PH, & Garcia FC (2012) Effectiveness of immediate bonding of etch-and-rinse adhesives to simplified ethanol-saturated dentin *Brazilian Oral Research* **26**(2) 177-182.
 27. Mak YF, Lai SC, Cheung GS, Chan AW, Tay FR, & Pashley DH (2002) Micro-tensile bond testing of resin cements to dentin and an indirect resin composite *Dental Materials* **18**(8) 609-621.
 28. Hiraishi N, Yiu CK, King NM, & Tay FR (2009) Effect of pulpal pressure on the microtensile bond strength of luting resin cements to human dentin *Dental Materials* **25**(1) 58-66.
 29. Escribano N, & de la Macorra JC (2006) Microtensile bond strength of self-adhesive luting cements to ceramic *Journal of Adhesive Dentistry* **8**(5) 337-341.
 30. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH, & Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel *Clinical Oral Investigations* **9**(3) 161-167.
 31. Tonial D, Ghiggi PC, Lise AA, Burnett LH Jr, Oshima HM, & Spohr AM (2010) Effect of conditioner on microtensile bond strength of self-adhesive resin cements to dentin. *Stomatologija* **12**(3) 73-79.
 32. Tezvergil-Mutluy A, Lassila LV, & Vallittu PK (2007) Degree of conversion of dual-cure luting resins light-polymerized through various materials *Acta Odontologica Scandinavica* **65**(4) 201-205.

33. Spencer P, & Wang Y (2002) Adhesive phase separation at the dentin interface under wet bonding conditions *Journal of Biomedical Materials Research* **62(3)** 447-456.
34. Fukeygawa D, Hayakawa S, Yoshida Y, Suzuki K, Osaka A, & Van Meerbeek B (2006) Chemical interaction of phosphoric acid ester with hydroxyapatite *Journal of Dental Research* **85(10)** 941-944.
35. Al-Assaf K, Chakmakchi M, Palaghias G, Karanika-Kouma A, & Eliades G (2007) Interfacial characteristics of adhesive luting resins and composites with dentine *Dental Materials* **23(7)** 829-839.
36. Tay FR, Pashley EL, Huang C, Hashimoto M, Sano H, Smales RJ, & Pashley DH (2001) The glass-ionomer phase in resin-based restorative materials *Journal of Dental Research* **80(9)** 1808-1812.
37. Belli S, Unlu N, & Ozer F (2001) Bonding strength to two different surfaces of dentin under simulated pulpal pressure *Journal of Adhesive Dentistry* **3(2)** 145-152.
38. Hashimoto M, Tay FR, Svizero NR, de Gee AJ, Feilzer AJ, Sano H, Kaga M, & Pashley DH (2006) The effects of common errors on sealing ability of total-etch adhesives *Dental Materials* **22(6)** 560-568.
39. Mazzitelli C, Monticelli F, Osorio R, Casucci A, Toledano M, & Ferrari M (2008) Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin *Dental Materials* **24(9)** 1156-1163.
40. Yang B, Adelung R, Ludwig K, Bossmann K, Pashley DH, & Kern M (2005) Effect of structural change of collagen fibrils on the durability of dentin bonding *Biomaterials* **26(24)** 5021-5031.
41. De Munck J, Van Landuyt K, Coutinho E, Poitevin A, Peumans M, Lambrechts P, & Van Meerbeek B (2005) Micro-tensile bond strength of adhesives bonded to Class-I cavity-bottom dentin after thermo-cycling *Dental Materials* **21(11)** 999-1007.
42. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **83(6)** 454-458.
43. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (2011) State of the art of self-etch adhesives *Dental Materials* **27(1)** 17-28.