

# The Influence of Luting Systems on the Microtensile Bond Strength of Dentin to Indirect Resin-based Composite and Ceramic Restorations

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## Clinical Relevance

Compared to self-etch and self-adhesive luting systems, the two etch-and-rinse luting agents evaluated in the current study provided more reliable bonding when used to bond indirect resin-based composite restorations to sound dentin. On the contrary, the self-adhesive luting system showed the highest mean bond strength for the cementation of glass ceramic restorations.

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## SUMMARY

**Microtensile bond strength ( $\mu$ TBS) evaluation and fractographic analysis were used to compare four luting systems in the cementation of resin-based composite (RBC) and ceramic disks to dentin. Forty freshly-extracted molars were transversally sectioned to expose flat, deep dentin surfaces. Forty cylindrical specimens (5-mm diameter and 10-mm height), consisting of 20 RBC disks and 20 leucite-based glass ceramic disks, were produced. The RBC disks were sand-blasted with 50- $\mu$ m  $\text{Al}_2\text{O}_3$ . The ceramic disks were conditioned with 9.5% hydrofluoric acid gel and silane application. All the disks were then bonded to dentin surfaces according to the luting cements to be used: two etch-and-rinse luting agents (XP bond/CoreXFlow; Dentsply [XP]) (Enabond/EnaCem HF; Micerium [ENA]), a self-etch luting system (ED Primer II A+B/Panavia**

**F2.0; Kuraray-Dental [PAN]) and a self-adhesive luting agent (RelyX Unicem; 3M ESPE [UNI]). The adhesive/luting cement systems were applied according to the manufacturers' instructions. The specimens were sectioned perpendicular to the adhesive interface to produce multiple beams, approximately 1 mm<sup>2</sup> in area. All the specimen preparations were performed by the same operator. The beams were tested under tension at a crosshead speed of 0.5 mm/minute until failure. The  $\mu$ TBS data were analyzed by two different one-way-ANOVA and multiple comparison Tukey tests ( $\alpha=0.05$ ). All the fractured beams were observed using a Scanning Electron Microscope (SEM) at 200x magnification for fracture mode determination.**

The mean bond strength in MPa (SD) for the RBC (Co) and ceramic (Ce) groups were: XP-Co = 31.39 (13.51), ENA-Co = 30.93 (10.17), PAN-Co = 18.29 (10.02), UNI-Co = 19.33 (7.91); XP-Ce = 4.83 (1.86), ENA-Ce = 5.15 (1.66), PAN-Ce = 4.36 (1.80), UNI-Ce = 7.16 (2.52). Statistical analysis showed that the bond strengths were significantly affected by the luting agent employed for both the RBC and ceramic groups ( $p<0.001$ ). In particular, the XP-Co group and the ENA-Co group did not differ from each other ( $p>0.05$ ) and showed significantly higher bond strengths than the PAN-Co and UNI-Co groups ( $p<0.05$ ). On the contrary, the UNI-Ce group showed the highest bond strengths compared to the other ceramic experimental groups ( $p<0.05$ ). Regarding failure mode, differences were found between the RBC groups: for the etch-and-rinse luting systems (XP-Co and ENA-Co groups), most failures occurred cohesively in the luting agent, while the self-etch luting system (PAN-Co group) and self-adhesive luting system (UNI-Co group) failed predominantly adhesively at the luting agent-dentin interface. Little differences were found between the ceramic groups, where failure type was primarily adhesive between cement and ceramic.

## INTRODUCTION

Resin-based composites (RBC) and ceramic materials have significantly improved with respect to both mechanical and aesthetic proprieties and they can be used as indirect restorations with predictable results.<sup>1</sup> Indirect adhesive procedures constitute a substantial portion of contemporary aesthetic restorative treatments. Tooth-colored inlays, onlays, veneers and crowns are now routinely bonded to the tooth substrate with adhesive resin cements.<sup>1-3</sup> Bonding to dentin has been referred to as a less reliable technique, especially when compared to enamel bonding, due to the intrinsic characteristics of dentin substrate,

including high organic content, tubular structure variations and the presence of outward fluid movement.<sup>4-5</sup> Therefore, it is important to improve dentin adhesion when placing indirect restorations. However, when bonding RBC or ceramic restorations to tooth structure, two different interfaces need to be considered: the dentin/adhesive-cement interface and the RBC-ceramic/cement interface. The bond strength at both of these interfaces should be optimized, because the lower one will determine the final bond strength of the cemented restoration.<sup>6</sup> Each step of the clinical and laboratory procedures can have an impact on the aesthetic results and longevity of indirect restorations. Cementation is the most critical step and involves the application of both adhesive system and resin luting agent.<sup>7-8</sup> In order to establish a strong, durable bond, which is necessary for the biomechanical aspect of the tooth-restoration system, an appropriate treatment of the fitting surface of the RBC indirect restoration is also of primary importance.<sup>9</sup> Various studies have evaluated different ceramic surface treatments used to optimize bond strength at the ceramic/cement or ceramic/RBC interface.<sup>10-13</sup> The clinical protocol for placing laboratory-processed RBC resins includes the use of dual-polymerizing resin cements; however, the dentin bonding adhesive system can be light-activated, auto-polymerizing or dual-polymerizing.<sup>14-15</sup>

Resin cements are based on the use of an etch-and-rinse or self-etch adhesive, along with a dual resin RBC. Recently, self-adhesive universal resin cements that combine the use of adhesive and cement in a single application that eliminates the need for pre-treatment of tooth have been marketed.

Little information is available in the literature on the bond strength of the complete tooth/indirect restoration complex using different luting systems. The current study investigated the effect of four types of dual-cure luting systems on the bond strength of RBC and ceramic blocks to dentin. The null hypothesis was that there were no differences in bonding effectiveness between etch-and-rinse, self-etch and self-adhesive luting systems.

## METHODS AND MATERIALS

### Tooth Preparation

Forty freshly-extracted human molars, free of cracks, caries and restorations on visual inspection, were selected for the study. Any residual soft tissue and debris was removed from the roots using a scaler. The teeth were then rinsed with water and stored in an aqueous solution of 0.5% chloramines T at 4°C for not longer than three months until the start of the experiment. Each crown was sectioned perpendicular to its longitudinal axis at 3 mm from the cemento-enamel junction using a low-speed diamond saw (Micromet M,

Table 1: Firing Temperatures for Ceramic, Based on Manufacturer's Recommendations

Dentin Firing Cycles				Glazing Cycles		
Drying Time (minute)	Heating Rate (°C/minute)	Firing Temp (°C)	Holding Time (seconds)	Heating Rate (°C/minute)	Firing Temp (°C)	Holding Time (seconds)
2	75	900	120	75	880	60

Remet, Casalecchio di Reno, Bologna, Italy) under copious water spray to expose a flat dentin surface in the middle crown portion.

Each surface was then ground with 180-grit silicon carbide (SiC) paper under running water for 30 seconds to produce and standardize the smear layer thickness on the dentin surface. The bonding surfaces were then examined under a stereomicroscope (Nikon SMZ10, Tokyo, Japan) to ensure that they were free from retained enamel. If any enamel remained, the surface was ground again until all the enamel was removed. The teeth were then randomly divided into eight groups (five for each group).

Twenty RBC specimens were obtained by placing a microhybrid RBC (Enamel-Plus HFO UD3, Micerium, Avegno, Genova, Italy) inside translucent polyethylene cylindrical molds with an inner diameter of 5 mm and height of 10 mm. Each mold was put on a glass surface, then filled with RBC. The RBC was placed in the mold in five increments of about 2 mm. Each layer was individually light cured for 40 seconds (LE Demetron I, Sybron/Kerr, Orange, CA, USA, with a 1200 mW/cm<sup>2</sup> output). RBC cylinders measuring 5 mm in diameter and 10 mm in height were obtained. After having removed the polyethylene molds, the samples were subjected to an additional cycle of polymerization in an RBC oven at 70°C for 10 minutes (Bulb PlusT, Micerium). All RBC surfaces were ground with 600-grit silicon carbide (SiC) paper under running water for 30 seconds to expose filler particles. The RBC surfaces were then submitted to an air-borne particle abrasion with 50-µm Al<sub>2</sub>O<sub>3</sub> (Korox, Bego, Bremen, Germany), using an intraoral air-abrasion device (Micerium, Avegno, Genova, Italy). The tip of the microetcher was kept 5 cm away from the surface of each specimen and applied for 10 seconds at 2.0 bar pressure.<sup>9</sup> All the specimens were then rinsed under running tap water and cleaned in an ultrasonic bath to remove debris.

Twenty silicate glass ceramic cylindrical disks (Reflex, Wieland Dental & Technik GmbH & Co, Pforzheim, Germany) were also prepared. The ceramic was condensed in a cylindrical silicone mold. Dentin powder (shade A3) was mixed with deionized water, vibrated and excess water was removed with absorbent paper to condense the ceramic. All the specimens were fired according to the manufacturer's instructions (Table 1) in a programmable vacuum fur-

nace (Austromat D4, Dekema Dental-Keramiköfen GmbH, Freilassing, Germany). The fired cylinders were polished to standardized dimensions (5 mm ± 0.2 in diameter and 10 mm ± 0.2 thick) prior to glazing by using a variable-speed grinder polisher machine (Ecomet 3, Buehler Ltd, Lake Bluff, IL, USA). The disks were then ground with 400-grit followed by 600-grit wet silicon carbide (SiC) paper under running water until flat surfaces had been attained. All the ceramic disks were rinsed under running water, cleaned in an ultrasonic bath and dried. The bonding ceramic surfaces were then pretreated with 9.5% hydrofluoric (HF) acid for 90 seconds (Porcelain Prep Kit, Pulpdent, Watertown, MA, USA), rinsed with air-water spray for 60 seconds, air dried and silanized (Porcelain Prep Kit, Pulpdent) in accordance with the manufacturer's instructions.

**Group Classification and Bonding Procedures**

Four commercially available luting agents (Tables 2 and 3) were used in the current study in order to form four groups, as follows: *XP group*, *ENA group*, *PAN group* and *UNI group*, in which the cements applied on the dentin surfaces were CoreXFlow (Dentsply DeTrey GmbH, Konstanz, Germany), Enacem HF (Micerium), Panavia F2.0 (Kuraray-Dental, Düsseldorf, Germany) and RelyX Unicem (3M ESPE, Seefeld, Germany), respectively.

**XP Group**

For teeth from this group, the dentin surfaces were first etched for 15 seconds with phosphoric acid gel provided by the manufacturer (Table 2), then thoroughly washed using a water spray for at least 15 seconds. The excess water was blot dried from the dentin surface with a wet cotton pellet, leaving the surface visibly moist. An equal number of drops of the bonding agent and its activator (Table 2) were mixed in a mixing well for two seconds. Generous amounts of mixed adhesive/activator were rubbed onto the moist dentin with a microbrush (Microbrush X, Microbrush Corp, Grafton, WI, USA) for 10 seconds and air-thinned with two-to-three short, moderate blasts of air.

Equal amounts of a recently developed dual-cure self-activating system (Table 2) paste base and catalyst were mixed and applied to the flat dentin surfaces.

**ENA Group**

Teeth from this group were prepared similar to the XP-C group, except for the materials employed (Table 2).

Table 2: Summary of the Materials Used				
Groups	Etching Agent	Bonding Agent	Luting Agent	Manufacturer
XP	Conditioner 36 (Batch #0704001714)	XP Bond (Batch #0605001399) + SelfCure Activator (Batch #0510061)	CoreXFlow (Batch #080403)	Dentsply DeTrey GmbH, Konstanz, Germany
ENA	EnaEtch (Batch #2006104947)	EnaBond Light curing (Batch #2007006817) + EnaBond Catalyst (Batch #2007005180)	Enacem HF (Batch #2007006613)	Micerium, Avegno, Genova, Italy
PAN	/	Panavia F2.0 ED Primer II A + B (Batch #41212)	Panavia F2.0 Paste A + B (Batch #41212)	Kuraray-Dental, Düsseldorf, Germany
UNI	/	/	Relyx Unicem Aplicab (Batch #294071)	3M ESPE, Seefeld, Germany

Table 3: Composition of the Materials Used	
Material	Composition
Enamel-Plus HFO	Tetra methyl methacrylate, trimethyl dioxil diedilmethacrylate, isopropil (hydroxide fenoxyl propilmethacrylate)
Reflex	Silica dioxide (60-70%), alumina oxide (10-15%), sodium oxide (4-8%), potassium oxide (11-16%)*
XP Bond	Carboxylic acid modified dimethacrylate, phosphoric acid modified acrylate resin, urethane dimethacrylate, triethyleneglycol dimethacrylate, 2-hydroxyethyl methacrylate, butylated benzenediol, ethyl-4-dimethylaminobenzoate, camphor quinone, functionalized amorphous silica, t-butanol
SelfCure Activator	Urethane dimethacrylate, 2-hydroxyethyl methacrilate, catalysts, photoinitiators, stabilizers, acetone, water
CoreXFlow	Silanated barium aluminoborosilicate glass, treated hydrophobic fumed silica, aluminum oxide, urethane dimethacrylate resin, urethane modified bis-GMA dimethacrylate resin, polymerizable dimethacrylate resin, polymerizable trimethacrylate resin, benzoyl peroxide
EnaBond Light Curing	Modified acrylate acid, polyacrylate acid, methacrylate, ethyl alcohol, catalysts, stabilizers
EnaBond Catalyst	Ethanol solution**
Enacem HF	Diurethane dimethacrylate, butandiol dimethacrylate, silica bioxide
Panavia F2.0 ED Primer II A + B	10-Methacryloyloxydecyl dihydrogen phosphate, N-Methacryloyl-5-aminosalicylic acid, water, accelerators, N-Methacryloyl-5-aminosalicylic acid, initiators
Panavia F2.0 Paste A + B	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass, filler, initiators, accelerators, pigments, sodium fluoride; 10-Methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-Camphorquinone, initiators
Relyx Unicem Aplicab	Methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiators, glass powder, silica, substitute pyrimidine, calcium hydroxide, peroxy compound, pigment
*The ceramic components are listed as oxides, but in the material, they are presented as either glass (homogeneous) or crystals (leucite).	
**The exact composition is the property of the manufacturer.	

### PAN Group

On teeth from this group, no acid etching was performed, according to the manufacturer's instructions. An equal number of drops of ED Primer II liquids A and B (Table 2) were mixed in a mixing well for two seconds. Generous amounts of the mixed adhesive were rubbed onto the dentin surface with a disposable brush tip, left in place for 30 seconds, then air thinned with two-to-three short moderate blasts of air. The subsequent procedures were the same as for the XP-C and ENA-C group. The materials employed are summarized in Table 2.

### UNI Group

Neither acid-etching or bonding agent application was performed on teeth from this group. The prepared tooth surfaces were rinsed with water spray and the excess water was dried with cotton pellets. The self-adhesive luting agent (Table 2) was mixed for 10 seconds (Rotomix, 3M ESPE) and applied onto the dentin surfaces.

After placing the adhesive/luting cement systems, tooth specimens from each group were randomly divided in two subgroups (Co and Ce). The RBC cylinders and ceramic disks were respectively placed on flat dentin surfaces. The bonded assemblies were held centrally between the two measuring arms of the vertical-



ly positioned digital micrometer. A load pressure of about 5 N was applied on the cylindrical RBC or ceramic specimens in order to standardize and simulate clinical conditions of inlay cementation. This pressure was repeated three times for five seconds each at intervals of 15 seconds. The luting cement thickness was kept at approximately 100  $\mu\text{m}$ . The micrometer arms were slowly adjusted to produce a reading that was 100  $\mu\text{m}$  (mean) thicker than that initially recorded for the respective dentin specimen and RBC/ceramic inlay. During the dwell time and before the resin-luting agent completely polymerized, every excess was carefully removed with a thin instrument used to place RBC fillings and an axial load of 5 Kg was applied for 60 seconds. After the initial self-polymerization, the luting agent was further light-cured using an LED light-curing unit (LE Demetron I, Sybron/Kerr) under a load of 5 N from five directions for 40 seconds, for a total exposure time of 200 seconds.

After 24 hours of storage in distilled water at 37°C, all the specimens underwent 5,000 thermal cycles in deionized water from 5°C to 55°C, with a 30-second dwell time and a five-second transfer between temperature baths. The specimens were successively sectioned perpendicular to the adhesive interface with a diamond saw (Micromet M, Remet) under water cooling/lubrication to produce beams with an adhesive area approximately 1 mm<sup>2</sup>. Four beams from the central part of each specimen were obtained per each tooth. All the bonding procedures and specimen preparations were performed by the same expert operator.

### Microtensile Bond Strength Test

A total of 20 beams for each subgroup were obtained and submitted to a  $\mu\text{TBS}$  test. The beams were attached to the flat grips of a microtensile testing device using a cyanoacrylate cement and stressed in a Universal Testing Machine (LR30K, Lloyd Instruments Ltd, Fareham, UK) managed by software (Nexygen-ondio Version 4.0, Lloyd Instruments Ltd) with a crosshead speed of 0.5 mm/minute until failure. A chain of two links was interposed between the device and the upper clamp of the testing machine. After testing, the specimens were removed from the testing devices and the cross-sectional area of the fracture sites were measured with a digital caliper (series 500 Caliper, Mitutoyo America Corp, Aurora, IL, USA) to calculate the ultimate tensile bond strength expressed in MPa.

### Mode of Failure

After the  $\mu\text{TBS}$  test, both the dentin and RBC/ceramic sides of fractured beams were mounted on aluminum stubs, gold-sputter coated and observed by scanning electron microscope (SEM) (EVO MA 15; Carl Zeiss NTS GmbH, Oberkochen, Germany) at 190x or higher magnification for fracture mode determination. The

failure modes were classified to be among six different types:<sup>16</sup>

- *Type 1*: Cohesive failure in dentin
- *Type 2*: Adhesive failure at the luting-dentin interface
- *Type 3*: Mixed adhesive failure and cohesive failure in dentin
- *Type 4*: Cohesive failure in the luting agent
- *Type 5*: Mixed adhesive failure and cohesive failure in RBC (or ceramic)
- *Type 6*: Adhesive failure at the luting-RBC (or ceramic) interface

### Statistical Analysis

After microtensile testing, statistical analysis was performed using SPSS Advanced Statistical 11.5 software for Windows (SPSS, Chicago, IL, USA).

Two different one-way-ANOVAs were performed, in order to analyze the effect of the factor “luting cement” on the microtensile bond strength of both the RBC and ceramic experimental groups. Beams were used as the statistical unit ( $n=20$ , per group). Post-hoc multiple comparisons were achieved using the Tukey honestly significant difference (HSD) test.  $P$ -values lower than 0.05 were considered to be statistically significant in all tests.

## RESULTS

The mean bond strengths obtained for the RBC and ceramic groups are shown, respectively, in Tables 4 and 5. One-way ANOVA showed that the luting agent that was employed significantly affected the bond strength for both the RBC and ceramic groups ( $p<0.001$ ).

The microtensile bond strengths of acid-etched specimens from the XP-Co and ENA-Co groups did not differ from each other ( $p=0.999$ ) and were significantly higher than those of the PAN-Co ( $p=0.001$  and  $p=0.002$ , respectively) and UNI-Co ( $p=0.003$  and  $p=0.005$ , respectively) groups. The lowest values for the RBC groups were obtained for the specimens from the UNI-Co group, although no statistically significant differences were observed if compared with specimens from the PAN-Co group ( $p=0.990$ ) (Table 4).

On the contrary, the UNI-Ce group showed the highest bond strengths compared to the other ceramic experimental groups. The actual  $p$ -values of the Tukey test were:  $p=0.012$  (UNI-Ce vs ENA-Ce);  $p=0.003$  (UNI-Ce vs XP-Ce);  $p<0.001$  (UNI-Ce vs PAN-Ce). The other ceramic groups did not differ from each other ( $p=0.594$  for ENA-Ce vs PAN-Ce;  $p=0.957$  for ENA-Ce vs XP-Ce;  $p=0.878$  for XP-Ce vs PAN-Ce) (Table 5).

The distribution of failure modes for each RBC experimental group is summarized in Figure 1. The percent-

Table 4: Mean Microtensile Bond Strengths (MPa) and Standard Deviations (SD) for Resin-based Composite Experimental Groups

	XP-Co	ENA-Co	PAN-Co	UNI-Co
$\mu$ -TBS MPa (SD)	31.39 <sup>a</sup> (13.51)	30.93 <sup>a</sup> (10.17)	18.29 <sup>b</sup> (10.02)	19.33 <sup>b</sup> (7.91)

The same superscripted letters indicate no significant differences ( $p>0.05$ ).

Table 5: Mean Microtensile Bond Strengths (MPa) and Standard Deviations (SD) for Ceramic Experimental Groups

	XP-Ce	ENA-Ce	PAN-Ce	UNI-Ce
$\mu$ -TBS MPa (SD)	4.83 <sup>b</sup> (1.86)	5.15 <sup>b</sup> (1.66)	4.36 <sup>b</sup> (1.80)	7.16 <sup>a</sup> (2.52)

The same superscripted letters indicate no significant differences ( $p>0.05$ ).

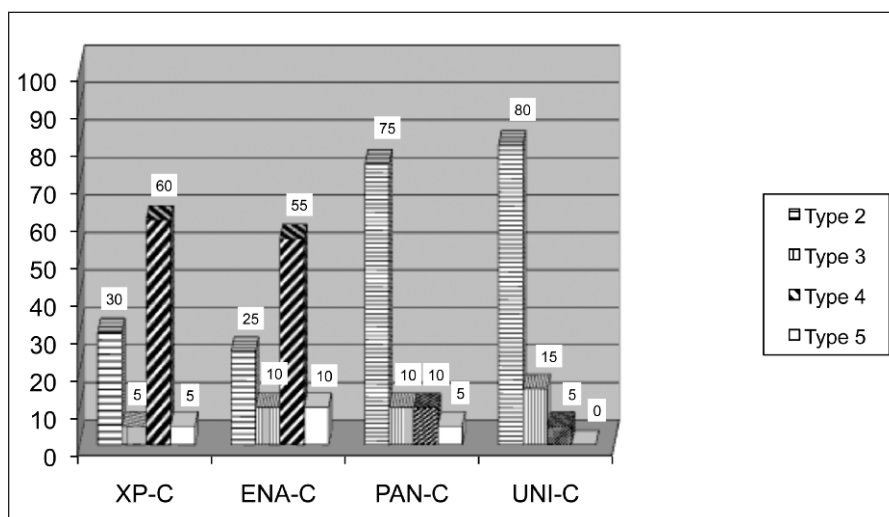


Figure 1: Graphical presentation showing percentages of fracture modes for resin-based composite experimental groups. Type 1 and Type 6 fractures were not observed.

Type 2: Adhesive failure at the luting-dentin interface; Type 3: Mixed adhesive failure and cohesive failure in dentin; Type 4: Cohesive failure in the luting agent; Type 5: Mixed adhesive failure and cohesive failure in RBC.

age of each fracture pattern was calculated for each group. PAN-Co and UNI-Co specimens failed predominantly adhesively between dentin substrate and luting agent (80% and 75%, respectively), while the XP-Co and ENA-Co groups showed a 50% and 60% (respectively) cohesive failure in the luting agent. There were no cohesive failures in dentin and no adhesive failures between the luting agent and RBC.

Small differences were found between the ceramic groups, where failure type was mostly adhesive between cement and ceramic. Some Type 4 failures were also recorded in each ceramic group (Figure 2).

## DISCUSSION

The current study revealed that luting systems significantly affect the bond strengths of indirect RBC and

ceramic restorations to dentin. The null hypothesis of the study, that there were no significant differences in bonding effectiveness between etch-and-rinse, self-etch and self-adhesive luting systems, was rejected. The luting systems involving the etch-and-rinse procedure resulted in significantly higher bond strength than all the other resin cements when used to bond indirect RBC restorations to sound dentin. On the contrary, the self-adhesive luting system showed the highest mean bond strength for the cementation of indirect leucite-based ceramic restorations to dentin.

The luting technique for the RBC or ceramic inlays is important, and the properties of the luting agent are crucial for the longevity of the restorations. Clinical success with ceramic inlays/onlays has been assisted by the ability to develop a reliable bond of resin cement to dental tissues.<sup>17</sup> Because of its high organic content, dentin is a less favorable substrate for bonding than enamel. Therefore, it is important to improve dentin adhesion when placing ceramic restorations.<sup>18</sup> Several *in vitro* studies reported the bond strength of different adhesive systems used in combination with a luting composite to both enamel and dentin.<sup>6,19-21</sup>

In the current study, the etch-and-rinse technique has been shown to improve the dentin bonding of dual-polymerizing resin cement when compared with self-etch (Panavia F2.0) and self-adhesive (RelyX Unicem) luting agents in the cementation of indirect RBC restorations. These findings differ from those recently reported,<sup>16</sup> in which the etch-and-rinse, self-etch and self-adhesive luting agents demonstrated an equal bond

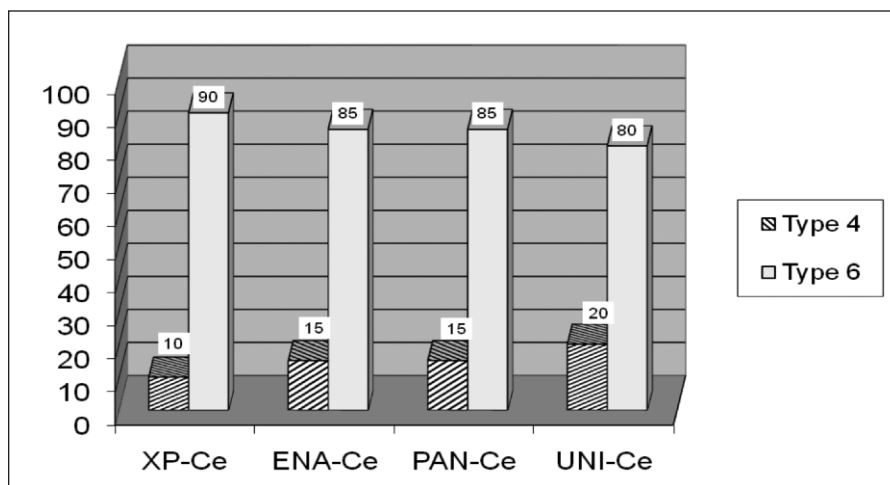


Figure 2: Graphical presentation showing percentages of fracture modes for ceramic experimental groups. Type 1, 2, 3 and 5 fractures were not observed.

Type 4: Cohesive failure in the luting agent; Type 6: Adhesive failure at the luting-ceramic interface.

strength to dentin. That study affirmed that self-etching resin cements offer an alternative to effectively bonding to dentin in terms of bond strength values. Several reports have shown that self-etching adhesive systems can be used for RBC restorations due to their ability to provide high bond strength to dentin and efficient marginal sealing.<sup>22-24</sup> Additionally, it was previously reported that a multi-step application technique (etch-and-rinse system) is time-consuming and rather technique-sensitive and, consequently, may compromise bonding effectiveness.<sup>8</sup> However, several studies demonstrated that the simplification may ease handling for the general practitioner but may not be able to improve adhesive effectiveness.<sup>5,25-27</sup> Furthermore, it was clearly shown that all-in-one adhesives suffer a certain permeability, which was verified both *in vitro* and *in vivo*.<sup>28-32</sup>

Recently, a self-adhesive universal resin cement without surface pretreatment has been introduced (RelyX Unicem, 3M ESPE). This self-adhesive resin cement is based on a new monomer, filler and initiation technology. The manufacturer purports that the organic matrix consists of newly developed multifunctional phosphoric acid methacrylates. The phosphoric acid methacrylates can react with basic fillers in the luting cement and hydroxyapatite of the hard tooth tissue.<sup>16</sup> The bonding mechanism of this cement differs from that obtained with self-etch adhesives, since TEM morphological interface examination did not show distinct demineralization and hybridization.<sup>33</sup>

Among the resin cements investigated, RelyX Unicem was the only one that did not require pretreatment of the dentin. It achieved the lowest mean bond strength value between the RBC experimental groups, although no statistically significant difference was observed if

compared with Panavia F2.0. This latter resin cement system utilizes a self-etching primer (ED Primer II) for simultaneous etching and priming of dentin. It is also dual-curable and contains polysiloxane-coated sodium fluoride fillers for additional fluoride release.<sup>8</sup>

On the contrary, the self-adhesive luting system showed the highest mean bond strength compared to the etch-and-rinse and self-etch luting systems, which did not differ from each other when used to bond glass ceramic restorations to dentin. These findings differ from those reported in previous studies,<sup>34-36</sup> in which Relyx Unicem achieved lower bond strength and demonstrated a worse luting effectiveness when compared to different etch-and-rinse and self-etch luting agents.

Analyses of failure mode showed that, for the RBC groups, etch-and-rinse luting systems (XP-Co and ENA-Co groups) primarily failed cohesively in the luting agent, while the self-etch luting system (PAN-Co group) and self-adhesive luting system (UNI-Co group) predominantly failed adhesively at the luting agent-dentin interface. This lower adhesive failure rate at the luting-dentin interface registered for etch-and-rinse luting systems compared to self-etch (Panavia F2.0) and self-adhesive (RelyX Unicem) luting agents is a consequence of the higher bond strength of the luting cement to dentin. In the current study, most failures in the PAN-Co and UNI-Co groups were adhesive at the luting-dentin interface in accordance with a recent investigation<sup>16</sup> in which most of the self-etch adhesive luting agent specimens showed an adhesive failure at the luting-dentin interface. The PAN-Co group showed a high percentage (75%) of adhesive failure between the luting cement and dentin with similar value (65.9%) to that obtained by Mak and others,<sup>8</sup> who valued the  $\mu$ TBS of resin cement to dentin and indirect RBC. In the ceramic groups, the main failure pattern was adhesive between the luting cement and ceramic, indicating that this is the most critical interface.

As discussed by Shortall and others,<sup>37</sup> the bonding of an indirect composite restoration using resin luting cements relies on both mechanical retention and chemical bonding thanks to residual-free carbon bonds. Even though secondary curing reduces the amount of residual-free carbon bonds, the air-borne particle abrasion of composite leads to a more effective bonding by removing some of the matrix resin and exposing surface filler particles. On the other hand, the bond between the ceramic surface and resin composite can be created via



(i) micro-mechanical bonding by grit-blasting or acid etching with HF and (ii) silane coupling agent.<sup>38</sup> With an acidic catalyst in an alcohol–water solution, the labile alkoxy groups of the silane coupling agent -O-R react with water to form reactive, hydrophilic, acidic silanol groups,  $\equiv\text{Si-OH}$ . The silanols are adsorbed, deposited and polymerized on the substrate surface. When deposited on an inorganic surface, silanol oligomers react with each other, forming branched hydrophobic siloxane bonds, -Si-O-Si-. With a surface of silica containing typically reversibly attached hydroxyl groups, -OH, they form siloxane bonds. As a result of this silanization process, a hydrophobic and branched three-dimensional siloxane film, which allows resin composite bonding, will be formed (20-100 nm).<sup>38</sup>

From the failure data presented in the current study, it seems clear that bond to restorative material is inferior to bond to dentin when testing ceramic material and vice versa when testing RBC. Further studies that simplify the tested systems by luting ceramic to ceramic could be required in order to evaluate the bonding to ceramic materials of those adhesive luting systems that demonstrated reliable bond to enamel and dentin.

This *in vitro* study evaluated the  $\mu\text{TBS}$  of dual-polymerizing resin cements and adhesive systems in laboratory conditions. Therefore, factors, such as dentinal fluid movement and internal stress related to the cavity configuration for indirect restorations, were not simulated. RBC and ceramic cylinders 10-mm thick were used in this study as a standard indirect RBC restoration. They were bonded to a flat dentin surface, which represents a situation of maximum compliance, as there is unrestricted shrinkage strain (free-curing contraction) of the resin cement.

According to the results of the current study, when dual-polymerizing resin cements are used, selection of the dental adhesives is important to avoid early bonding failure. Moreover, it may be concluded that etch-and-rinse luting systems can be suggested for the cementation of indirect RBC restorations. A self-adhesive luting system can be preferred for the cementation of glass ceramic restorations.

## CONCLUSIONS

Within the limitations of the current *in vitro* study, the  $\mu\text{TBS}$  of the tested luting systems to dentin was material-dependent and indicated that resin cements involving the etch-and-rinse technique provided more reliable bonding compared to self-etch luting agents and self-adhesive luting agents when used to bond indirect RBC restorations to sound dentin. On the contrary, the self-adhesive luting system showed the highest mean bond strength for the cementation of indirect glass ceramic restorations.

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