Operative Dentistry, 2007, **32-1**, 84-93

Influence of Previous Provisional Cementation on the Bond Strength Between Two Definitive Resin-based Luting and Dentin Bonding Agents and Human Dentin

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Clinical Relevance

Based on *in vitro* results in extracted teeth, the dual bonding method used was effective in restoring the shear bond strength values that decreased after applying provisional luting agent regardless of the composition of the provisional luting agent (eugenol-based or eugenol-free) used.

SUMMARY

This study evaluated the effect of two different types of provisional luting agents (RelyX Temp E, eugenol-based; RelyX Temp NE, eugenol-free) on the shear bond strengths between human dentin and two different resin-based luting systems (RelyX ARC-Single Bond and Duo Link-One Step) after cementation with two different techniques (dual bonding and conventional technique). One hundred human molars were trimmed parallel to the original long axis, to expose flat dentin surfaces, and were divided into three groups. After related surface treatments for each specimen, the resin-based luting agent was applied in a silicone cylindrical mold (3.5 x 4 mm), placed on the bonding-agent-treated dentin surfaces and polymerized. In the control group (n=20), the specimens were further divided into two groups (n=10), and two different resin-based luting systems were immediately applied following the manufacturer's protocols: RelyX ARC-Single Bond (Group I C) and Duo Link-One Step (Group II C). In the provisionalization group (n=40), the specimens were further divided into four subgroups of 10 specimens each (Group I N, I E and Group II N, II E). In Groups I N and II N, eugenol-free (RelyX NE), and in groups I E and II E, eugenol-based (RelyX E) provisional luting

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DOI: 10.2341/06-27

agents (PLA), were applied on the dentin surface. The dentin surfaces were cleaned with a flour-free pumice, and the resin-based luting systems RelyX ARC (Group I N and E) and Duo Link (Group II N and E) were applied. In the Dual bonding groups (n=40), the specimens were divided into four subgroups of 10 specimens each (Group I ND, ED and Group II ND, ED). The specimens were treated with Single Bond (Groups I ND and ED) or One Step (Groups II ND and ED). After the dentin bonding agent treatment, RelyX Temp NE was applied to Groups I ND and II ND, and RelyX Temp E was applied to Groups I ED and II ED. The dentin surfaces were then cleaned as described in the provisionalization group, and the resin-based luting systems were applied: RelyX ARC-Single Bond (Group I ND and ED) and Duo Link-One Step (Group II ND and ED). After 1,000 thermal cycles between 5°C and 55°C, shear bond testing was conducted at a crosshead speed of 0.5 mm/minutes. One-way ANOVA, followed by a post hoc Tukey test (α=0.05) was done. The dentin-resin-based luting system interfaces were evaluated under a scanning electron microscope. There was a significant reduction in the mean shear bond strength values of groups subjected to the provisionalization compared to the control and dual bonding technique groups (p<0.05). The composition of provisional luting did not create a significant difference with regard to reducing shear bond strength values (p>0.05). With regard to resin based luting systems, the shear bond strength values of the double-bond technique groups were not significantly different from the controls (p>0.05).

INTRODUCTION

Modern dentin adhesive systems, in conjunction with resin-based luting agents, possess many advantages for the definitive cementation of restorations. These advantages include high bond strength to tooth structure (Miyazaki & others; Mitchell & others)[1-2], increased mechanical strength (Mitchell, Douglas & Cheng; Braga, Cesar & Gonzaga),[3-4] reduced post-operative sensitivity (Cagidiaco & others)[5] and low solubility in oral fluids (Yoshida, Tanagawa & Atsuta).[6] Acid-etched enamel provides a reliable bond to resin-based restorative materials (Buonocore).[7] This is related to the homogenous structure of enamel and the high inorganic content of this tissue (Anusavice).[8] However, when dentin is exposed, this heterogeneous tissue, with relatively high inorganic content and intrinsic moisture, creates challenges for the bonding of restorations. During recent years, with the introduction of the new dentin bonding agents, it has been possible to bond restorations to dentin (Van Meerbeek).[9] The bifunctional molecular structures of these bonding agents provide bonding interfaces between dental structures and resin-based restorative materials or resin-based luting agents (Anusavice).[8] Although the clinical performance of these newly developed agents are encouraging, meticulous attention is needed during their application. One of the primary requirements for optimum bonding is a contaminant-free substrate surface, as these contaminants may corrupt the bonding process (Watanabe & others).[10]

When clinical procedures are considered, most prosthodontic restorations require a provisionalization phase (Garber & Goldstein; Shillingburg & others).[11-12] During this phase, the provisional or definitive restorations are luted with the provisional luting agent (PLA). Zinc oxide eugenol-based (ZOE) PLA is widely used for provisional cementation. Eugenol is released from ZOE-based luting materials by the hydrolysis of zinc eugenolate (Hume; Abou Hashieh & others; Camps & others).[13-15] Although eugenol has a bactericidal effect (Hume)[16] in deep preparations, its diffusion may cause pulpal irritation or necrosis (Brännström & Nyborg).[17] Even in small amounts, the released eugenol theoretically may inhibit the polymerization process of resin-based luting systems by reacting with the initiators and the growing polymer chains free radicals (Fujisawa & Kadoma).[18] The negative effects of eugenol-containing PLAs on the retention of a restoration luted with resin-based luting agents (Millstein & Nathanson; Paul & Scharer; Bachmann & others; al-Wazzan, al-Harbi & Hammad)[19-22] include softening of the provisional resin surface (Rosenstiel & Gegauff)[23] and deterioration of the marginal adaptation (Hansen & Asmussen).[24] However, according to some authors, eugenol-containing PLAs did not have a negative effect on resin-based restoratives (Hansen & Asmussen; Schwartz, Davis & Hilton; Ganss & Jung; Jung, Ganss & Senger; Peters, Gohring & Lutz; Leirskar & Nordbo).[24-29]

In some PLA formulations, eugenol is replaced by carboxylic acids (Anusavice).[30] However, a considerable reduction in bond strength after adhesive cementation has been identified with eugenol-free formulations (Watanabe & others; Paul & Scharer; Watanabe & others; Aykent & others).[10,20,31-32] This has been related to the occlusion of dentin tubules with PLA residues and the reaction of zinc oxide remnants with the acidic primer of some adhesive system to avoid resin tag formation (Watanabe & others).[10] Therefore, elimination of PLA from the tooth surface is crucial. There have been different attempts to accomplish complete removal of PLA. Residual PLA was evident on dentin surfaces after cleaning with pumice and water (Mojon, Hawbolt & MacEntee; Grasso & others).[33-34] Cleaning the dentinal surfaces with soap and pumice not only had no beneficial effect, but it reduced shear bond strength in some material combinations as well (Bachmann & others).[21] While re-etching has been found to be effective, most of the dentinal tubules examined still contained PLA remnants (Xie, Powers & McGuckin).[35] Some researchers suggested use of acidic conditioners (Watanabe & others)[31] or degreasing agents (Mojon & others)[33] before primer application in order to restore bond strength reduced by PLA remnants.

Dual bonding technique (DB) has been proposed to overcome problems, such as reduction in shear bond strength associated with provisionalization (Bertschinger & others; Paul & Scharer; Magne & Douglas; Dagostin & Ferrari; Ozturk & Aykent).[36-40] Compared to the conventional method, in which the dentin-bonding agent is applied during the final luting procedure, this method requires hybridization of the exposed dentin surface immediately after tooth preparation and hybridization during final luting procedures (Bertschinger & others; Paul & Scharer).[36-37] The primary advantage of this technique is to protect the tooth from the consequences of microleakage by sealing the dentin tubules that are vulnerable to bacterial invasion (Cox & others; Pashley & others; Nagaoka & others; Cox)[38,41-43] immediately after completion of the preparation. Sealing of the dentin tubules also reduces sensitivity (Cagidiaco & others)[5] by preventing hydraulic fluid flow within the dentin tubules, which is associated with post-operative sensitivity (Brännström; Suzuki, Cox & White; Pashley & others).[44-46] It has been shown that cements can be forced into dentin tubules before the luting agent sets (Zaimoglu & Aydin)[47] and microorganisms and their by-products can penetrate into the patent dental tubules post-operatively (Lundy & Stanley; Nagaoka & others).[42,48] Therefore, the early sealing of dentin tubules also may prevent occlusion of dental tubules by PLA remnants and prevent eugenol diffusion across dentin, which may later inhibit polymerization of the bonding agents (Bertschinger & others).[36]

In this study, two null hypotheses were tested. The first null hypothesis was that there was no significant decrease in shear bond strength values after provisionalization. If evidence was found to reject this null hypothesis, an alternative hypothesis was that provisionalization has an effect on shear bond strength values. The second null hypothesis was that the DB technique has no effect on shear bond strength values. If evidence was found to reject this null hypothesis, an alternative hypothesis was that the DB technique has an effect on shear bond strength values. For this purpose, shear bond strength between human dentin and different resin-based luting agents after provisional cementation with different types of PLA were measured. Scanning electron microscopy (SEM) photomicrographs were used to evaluate the interfacial layers in different bonding techniques and the resin-based luting agents.

METHODS AND MATERIALS

One hundred non-carious permanent human molars, extracted due to periodontal problems, were used in this study. The teeth were stored in sterile saline solution at 4°C and used within one month of extraction.

The teeth were sectioned horizontally at their cemento-enamel junction using a slow-speed rotary diamond blade (Micracut; Metkon, Bursa, Turkey) under copious amounts of water, eliminating the roots. Any remaining pulpal tissue was removed and the teeth were placed with their buccal surfaces facing a glass plate. A 5-cm long plastic tube (diameter, 4 cm) cut from a 10 cc disposable plastic syringe (Hayat Co, Istanbul, Turkey) was centered in an upright position around each tooth. Each tooth was then embedded in an auto polymerizing poly methyl-methacrylate (PMMA) resin (Steady-Resin, Scheu-Dental, Iserlohn, Germany) by pouring PMMA in its liquid state onto the open end of the plastic tube. After polymerization, the plastic tubes were removed and, by using a model trimmer (Wehmer Co, Addison, IL, USA), each specimen was trimmed parallel to the original long axis of the tooth until a flat dentin surface was exposed. Abraded surfaces were then polished under water with a 600 grit abrasive (P 600, Kovax Co, Tokyo, Japan). The resin-based luting systems, provisional luting agents (PLA), their application procedures and the chemical compositions used in this investigation are listed in Table 1. All materials were used according to their respective manufacturers' directions.

The tooth specimens were divided into 10 groups of 10 teeth each, receiving treatments as shown in Table 2. The dentin surface of each tooth was conditioned and bonding agent was applied. The dentin surfaces were kept moist for all bonding procedures by wetting them with a cotton pellet saturated with distilled water and air dried for one second with filtered air from a distance of 10 cm, leaving the surfaces visibly moist as described by Kanca.[49] Resin based luting agent was mixed and injected into a silicone tube (Medinorm AG, Quierschied, Germany) 3.5 mm in diameter and 4 mm in height, while the tube was positioned on the conditioned dentin surfaces. A cylindrically-shaped instrument (Goldstein 2, Hu Friedy Inc, Chicago, IL, USA) was applied over the luting agent in order to ensure sufficient wetting of the dentin surface (Jung & others),[27] and the assembly was polymerized for 60 seconds with a halogen light of 500 mW/mm^2 intensity (Hi-Lux Ultra, Benlioglu, Ankara, Turkey) and the silicone tube was then removed.

The procedures for the shear bond strength test are shown in Figure 1. In Group I C and II C (Control groups), related resin based resin-based luting systems were applied immediately after the dentin surfaces were exposed and polymerized. In the provisionalization groups (Group I N, I E, II N and II E), PLA (RelyX Temp NE or RelyX Temp E) was mixed and applied to the dentin surfaces. The specimens were stored in water at room temperature for a week. One week later, a scaler was used to remove PLA from the dentin surfaces

Table 1: *Materials, Composition, Manufacturers and Lot Numbers of the Materials Used in This Study*

Material Description	Material	Chemical Composition	Manufacturer	Lot #
Bonding Agent	Single Bond	Water, Ethanol, HEMA, BisGMA, Dimethacrylates, Photoinitiators, Methacrylate functional copolymer of Polyacrylic and Polyitaconic acids	3M ESPE, St Paul, MN, USA	3JK
Resin-based luting agent	RelyX ARC	TEGDMA, BisGMA Zirconia/silica filler (% 67.5 by weight)	3M ESPE, St Paul, MN, USA	CWEB
Bonding Agent	One Step	Acetone, Bis-GMA, BPDM, HEMA	BISCO Inc, Schaumburg, IL, USA	0400003481
Resin-based luting agent	Duo Link	BPDM, TEGDMA Glass filler (% 60 by weight) UEDMA* *Base only	BISCO Inc, Schaumburg, IL, USA	0300000770
Eugonel-free provisional luting agent	RelyX Temp NE	Zinc Oxide, White Mineral Oil, Petrolatum	3M ESPE, St Paul, MN, USA	185873
Eugenol based provisional luting agent	RelyX Temp E	Hydrogenated Rosin, Eugenol, Modified Rosin, Silane treated silica, Oleic acid, 2.6-Di-Tert-Butyl-Cresol	3M ESPE, St Paul, MN, USA	146656

TEGDMA: Triethylenglycoldimethacryate; BisGMA: bisphenol-A-diglycidylether dimethacrylate; BPDM: Biphenyl dimethacrylate hydroxyethyl methacrylate; UEDMA: Urethane dimethacrylate

Table 2: *Research Outline of the Experimental Groups*

Treatments		Groups									
		Group I C	Group II C	Group I N	Group II N	Group I E	Group II E	Group I ND	Group II ND	Group I ED	Group II ED
Bonding Agent	Single Bond	•						•		•	
	One Step		•						•		•
Luting Resin	Rely X ARC	•						•		•	
	Duo Link		•						•		•
Provisional Luting Agent	Rely X Temp NE			•	•			•	•		
	Rely X Temp E					•	•			•	•
Provisionalization				•	•	•	•	•	•	•	•
Bonding Agent	Single Bond			•		•		•		•	
	One Step				•		•		•		•
Luting Resin	Rely X ARC			•		•		•		•	
	Duo Link				•		•		•		•

Group I C: Single Bond-RelyX ARC; Group II C: One Step-Duo Link; Group IN: RelyX Temp NE-Single Bond-RelyX Temp NE; Group II N: RelyX Temp NE-One Step-Duo Link, Group IE: RelyX Temp E-Single Bond-RelyX ARC; Group II E: RelyX Temp E-One Step-Duo Link; Group I ND: Single Bond-RelyX Temp NE-Single Bond-RelyX ARC; Group II ND: One Step-RelyX Temp NE-One Step-Duo Link; Group I ED: Single Bond-RelyX Temp E-Single Bond-RelyX ARC; Group II ED: One Step-RelyX Temp E-Duo Link

(H6/H7, Hu Friedy). Any visual remnants were cleaned with a prophy cup, (Young Dental, Earth City, MO, USA) and fluoride-free pumice (Isler Dental, Ankara, Turkey) mixed with water. They were then rinsed with water spray for 10 seconds. Resin-based luting systems were applied to the cleaned dentin surfaces and polymerized. In Group I ND, I ED, II ND and II ED (DB groups), a related dentin-bonding agent was applied immediately after the dentin surfaces were exposed. After hybridization, the dentin surfaces were covered with PLA. The teeth were stored in tap water for one week. After PLA was cleaned from the surfaces, dentin and bonding agent was applied for a second time. The resin-based luting agent was applied on hybridized dentin surfaces and polymerized.

All specimens underwent 1,000 thermal cycles between 5°C-55°C (dwell time, 20 seconds). The specimens were stored in tap water for one week at room temperature before being processed for shear strength testing. For shear bond testing, the specimens were mounted in a universal test machine (Lloyd-LRX, Lloyd Instruments, Fareham, UK) at a crosshead speed of 0.5 mm/minute (Figure 2). Failure load was defined as the point when the load versus displacement plot showed a steep decrease in load. The maximum load at fracture for all groups was divided by the cross-sectional area of the silicone tubes, which was determined to be 9.61 mm^2, and the shear bond strength of each specimen was recorded in MPa. The effect of the different bonding techniques and provisional luting agents on the shear bond strength of the resin-based luting agents was assessed statistically using one-way analysis of variance (ANOVA). Tukey's post-hoc test was used to compute multiple pair-wise comparisons of the data to determine significant differences between groups ($p<0.05$).

For SEM evaluation, specimens were sectioned buccolingually using a low-speed saw (Micracut, Metkon) under water cooling as before. The sectioned specimens were placed in distilled water and subjected to ultrasonic cleaning in an ultrasonic unit for 10 minutes (BioSonic UC50, Coltene Whaledent, Cuyahoga Falls, OH, USA). The specimens were then polished with 600-grit silicone abrasive paper (Kovax Co, Tokyo, Japan), acid etched in 10% H_3PO_4 acid solution (Sigma-Aldrich, Deisenhofen, Germany) for 10 seconds and rinsed in distilled water for 60 seconds. The specimens were then placed in a 5% NaOCl solution (Sigma-Aldrich, Deisenhofen, Germany) and rinsed in distilled water. The conditioned specimens were coated with a thin layer of gold using a Polaron SC500 sputter coater (VG Microtech, Tokyo, Japan) and photographed with a SEM microscope (JSM 5600; JOEL, Peabody, MA, USA) at 1500x, 2500x and 3000x magnifications.

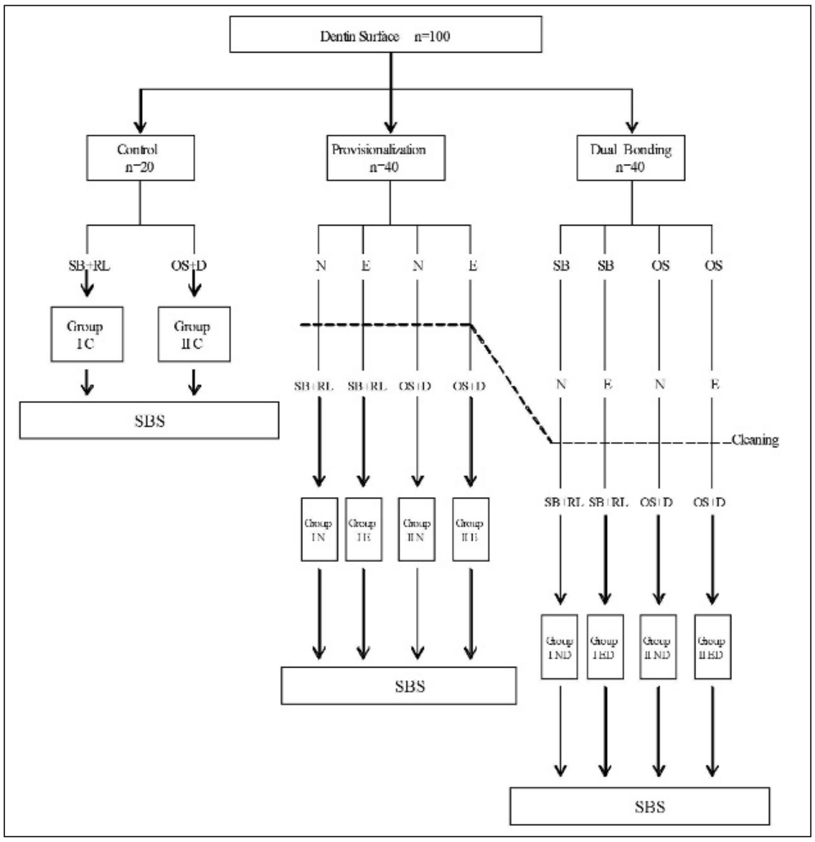

Figure 1: *Procedures for shear bond strength test. SB: Single Bond; RL: RelyX ARC; OS: One Step; D: Duo Link; N: RelyX Temp NE; E: RelyX Temp E; SBS: Shear bond strength test.*

RESULTS

Table 3 presents the descriptive statistical results of the shear bond strength data. Figure 3 shows the graphical demonstration of the shear bond strength values. For each resin-based luting system, the shear bond strength values decreased significantly after PLA application ($p<0.05$). The shear bond strength values of Group I C significantly decreased from 23.485 ± 5.34 MPa to 15.633 ± 4.79 MPa after eugenol-based PLA application (Group I E) and to 12.692 ± 5.47 MPa after eugenol-free PLA application (Group I N). There was no statistical difference between the I E and I N Groups. This was also the case for Group II C, where shear bond strength values decreased from 19.337 ± 5.84 MPa to 11.990 ±2.82 MPa and 12.567 ± 3.26 MPa for Group II N and Group II E, respectively. The highest shear bond strength values were recorded for Group I ND (24.745 ± 4.75 MPa), followed by Group I C (23.485 ± 5.34 MPa) and Group I ED (22.412 ± 3.91 MPa). However, differences between these groups were not statistically significant ($p>0.05$). For the Duo Link-One Step luting system, after PLA application, shear bond strength values decreased significantly from 19.337 ± 5.85 (Group II C) to 12.567 ± 3.26 (Group II E) to 11.990 ± 2.82 (Group II N) ($p<0.05$). Although Group II C had the highest shear bond strength values (19.337 ± 5.85 MPa), the differences between Group II C and Group II ED (17.371 ± 4.55 MPa) and Group II C and Group II ND (17.352 ± 4.96 MPa) were not statistically significant ($p>0.05$).

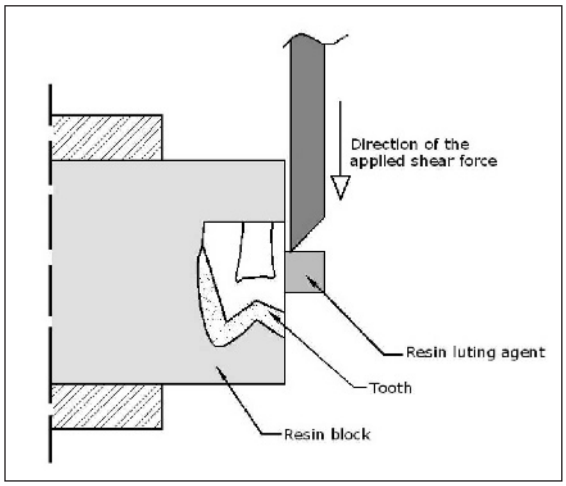

Figure 2. *Schematic drawing of shear bond test apparatus.*

Table 3: *Shear Bond Strengths of Different Bonding Groups (MPa)*

Bonding Groups	N	Shear Bond Strength (MPa)	Standard Deviation	Standard Error	Minimum	Maximum
I C	10	23.485^{c,d}	5.34	1.69	11.00	29.04
I E	10	15.633^{a,b}	4.79	1.52	6.69	21.67
I ED	10	22.412^{c,d}	3.,91	1.24	16.06	28.80
I N	10	12.692^{a,b}	5.47	1.73	5.32	21.85
I ND	10	24.745^{d}	4.75	1.50	17.69	30.56
II C	10	19.337b,^{c,d}	5.85	1.85	11.23	26.25
II E	10	12.567^{a}	3.26	1.03	8.73	17.19
II ED	10	17.371^{a,b,c}	4.55	1.44	10.70	24.90
II N	10	11.990^{a}	2.82	0.89	7.62	15.86
II ND	10	17.352^{a,b,c}	4.96	1.60	9.45	24.10

$^{a,b,c,d:}$ *Groups identified with the same letter are not statistically significant (p>.05)*

Group I C: Single Bond-RelyX ARC; Group II C: One Step-Duo Link; Group IN: RelyX Temp NE-Single Bond-RelyX Temp NE; Group II N: RelyX Temp NE-One Step-Duo Link, Group IE: RelyX Temp E-Single Bond-RelyX ARC; Group II E: RelyX Temp E-One Step-Duo Link; Group I ND: Single Bond-RelyX Temp NE-Single Bond-RelyX ARC; Group II ND: One Step-RelyX Temp NE-One Step-Duo Link; Group I ED: Single Bond-RelyX Temp E-Single Bond-RelyX ARC; Group II ED: One Step-RelyX Temp E-Duo Link

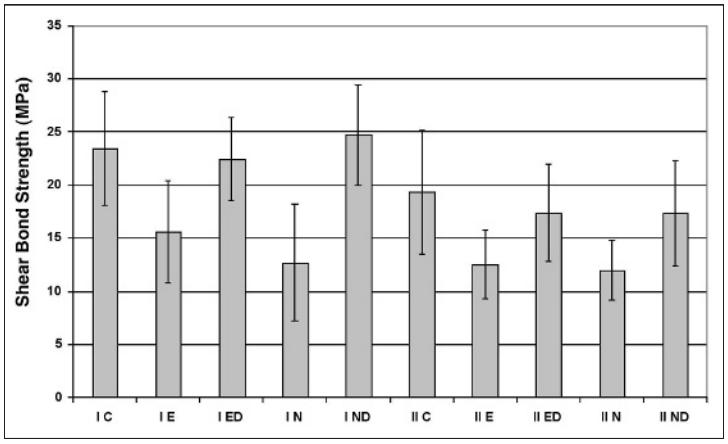

Figure 3. *Mean (± SD) Shear bond strengths of different bonding groups (n=10). Group I C: Single Bond-RelyX ARC; Group II C: One Step-Duo Link; Group IN: RelyX Temp NE-Single Bond-RelyX Temp NE; Group II N: RelyX Temp NE-One Step-Duo Link, Group IE: RelyX Temp E-Single Bond-RelyX ARC; Group II E: RelyX Temp E-One Step-Duo Link; Group I ND: Single Bond-RelyX Temp NE-Single Bond-RelyX ARC; Group II ND: One Step-RelyX Temp NE-One Step-Duo Link; Group I ED: Single Bond-RelyX Temp E-Single Bond-RelyX ARC; Group II ED: One Step-RelyX Temp E-Duo Link.*

SEM analysis of the specimens was performed to evaluate the bonding interfaces. The DB method resulted in more distinct hybrid zones and longer resin tags (Figures 4 and 5) than the conventional bonding method (Figures 6 and 7). The granular material, thought to have originated from the PLA remnants embedded in the bonding agent dentin interface, was evident in the SEM of Group I N (Figure 8).

DISCUSSION

The significant decrease in shear bond strength values of some groups studied after provisionalization leads to a rejection of the first null hypothesis and acceptance of the alternative hypothesis that provisionalization has an effect on shear bond strength values. There was a significant increase in shear bond strength values of the DB groups studied compared to the provisionalization groups. Therefore, the second null hypothesis, that the DB technique had no effect on shear bond strength values, was rejected, and the alternative hypothesis that the DB technique had an effect on shear bond strength values was accepted. The results of this study demonstrate that the decrease in shear bond strength values, which is related to the PLA contamination (Watanabe & others),[10] may be restored with use of the DB technique.

Suzuki and others[45] stated that pulpal recovery from tooth preparation trauma is possible by preventing bacterial leakage and its byproducts. Although vital teeth are more resistant to bacterial invasion into dentinal tubules than are non-vital teeth (Nagaoka & others),[42] and the outward fluid movement retards the inward diffusion of bacterial toxins (Pashley & others),[41] mechanically-exposed dentin requires provisionalization to reduce sensitivity and bacterial microleakage (Cagidiaco & others).[5] It has been stated that tooth sensitivity related to preparation is profound in the first week but decreases with time (Lundy & Stanley).[48] Therefore, the sealing of exposed dentin using dentin bonding agents immediately after tooth preparation (Bertschinger & others; Paul & Scharer)[36-37] and the fabrication of provisional restorations that provide protection for a tooth from the negative effects of thermal stimuli and spatial stabilization are advised (Garber & Goldstein).[11]

Previous studies incorporating various bonding systems, luting agents and application methods have shown that the DB method increased bond strength values compared to the conventional bonding technique (Bertschinger & others; Paul & Scharer; Magne & Douglas; Ozturk & Aykent).[20,36-37,40] This was related to formation of longer resin tags (Magne & Douglas; Ozturk & Aykent)[40,50] and a thicker hybrid zone (Ozturk & Aykent).[40] In a study by Dagostin and Ferrari,[39] in which DB was evaluated, a combination of 4 different bonding agents for initial sealing of the prepared dentin surfaces was used. In their study, the

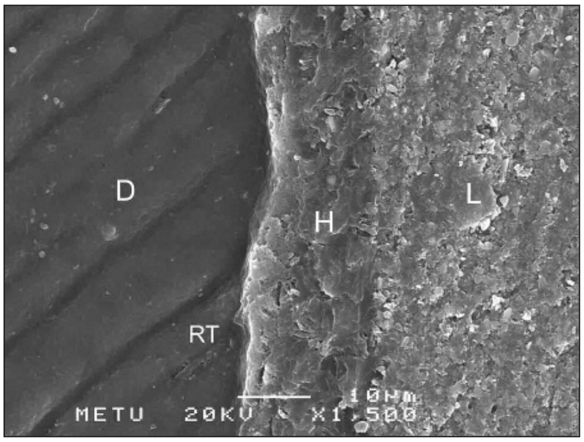

Figure 4. *Scanning electron micrographs of the interfacial layers between dentin and the resin-based luting agent in Group II ND. D, dentin; L, luting agent; H, hybrid zone (original magnification 1500x).*

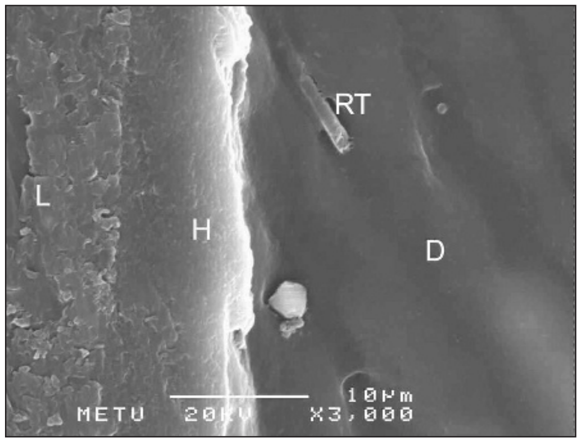

Figure 5. *Scanning electron micrographs of the interfacial layers between dentin and the resin-based luting agent in the Group I ND. D, dentin; L, luting agent; H, hybrid zone.; RT, resin tags (original magnification 1500x).*

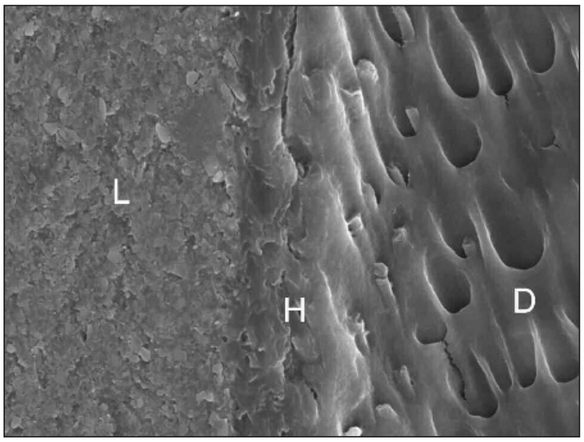

Figure 6. *Scanning electron micrograph of the interfacial layers between dentin and the resin based luting agent in Group II C. D, dentin; L, luting agent; H, hybrid zone; RT, resin tag (original magnification 1500x).*

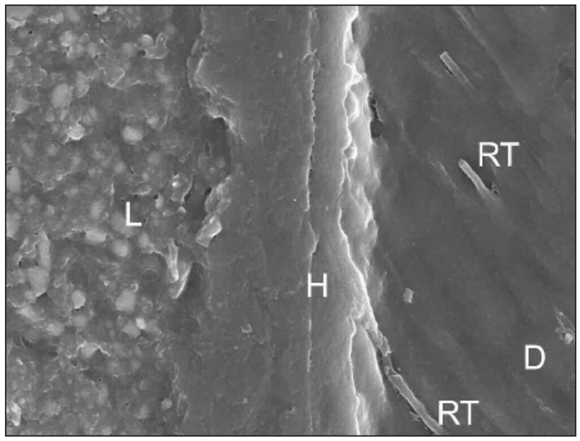

Figure 7. *Scanning electron micrographs of the interfacial layers between dentin and the resin based luting agent in Group I C . D, dentin; L, luting agent; H, hybrid zone; RT, resin tag (original magnification 3000x).*

pared dentin surfaces was used. In their study, the same dentin bonding and luting agents were used before definitive cementation to reduce the varying bonding effects. However, in the current study, two different bonding agents and their respective luting resins were used in combination with two different PLAs. The application of bonding agents with the same brand of luting resins is thought to optimize clinical procedures, as dentin bonding agents and luting resins are dispensed together by the manufacturers and named as "resin-based luting systems." It should be noted that the shear bond strength of composite materials to tooth structure (Miyazaki & others)[1] and their mechanical properties (Anusavice)[8] have been shown to be proportional to their filler content. Although the filler content of luting resins used in the study were relatively similar (RelyX ARC, % 60; Duo Link, % 67.5 by weight) other factors, such as composition of the polymer matrix, may effect the mechanical properties of the luting resin (Braga & others).[4] Therefore, the use of different types of luting resins may increase the bonding variables of adhesive interfaces.

In this study, an increase in the shear bond strength values of the DB method groups (Groups I ND, I ED, II ND, II ED) compared with the control groups (Group I C and II C) was identified; however, this increase was not statistically significant ($p>0.05$). The shear bond strength values of the DB method groups were significantly increased ($p<0.05$) compared to groups in which dentin was contaminated with PLA before luting procedures (Groups: I N, IE, II N and II E). The difference between the findings of this and previous studies (Bertschinger & others; Magne & Douglas; Ozturk & Aykent)[36,40,50] may be related to the different luting systems evaluated and to increased bonding variables.

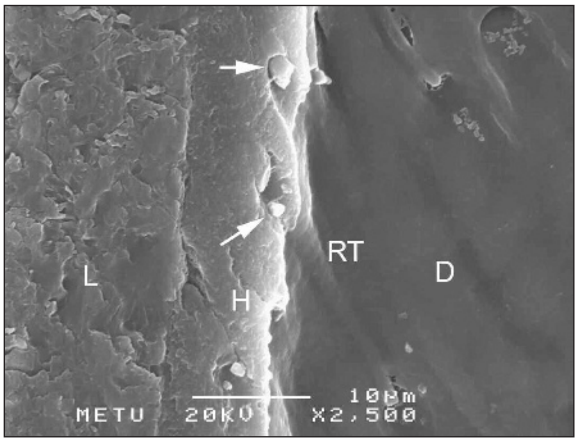

Figure 8. *Scanning electron micrographs of the interfacial layer between dentin and resin based luting agent in the Group I N. Arrows indicate granular substances embedded in bonding agent and dentin interface. D, dentin; L, luting agent; H, hybrid zone; RT, resin tag (original magnification 2500x).*

In this study, dentin surfaces were contaminated with two different PLAs of different compositions (eugenol containing and eugenol-free). In previous studies, it has been stated that acid etching and rinsing procedures are known to be effective in removing contaminants from the dentinal surface (Hansen & Asmussen; Xie & others).[24,35] However, for this study, PLA treatment prior to application of the bonding agents significantly decreased the bond strength values of the resin-based luting systems studied, regardless of the PLA composition (p<0.05).

In SEM examinations, granular substances, thought to originate from PLA remnants and embedded between the dentin bonding agent and dentin the surface, were observed (Figure 8). Watanabe and others[9] also documented granular substances at the dentinal surfaces after etching and indicated that they may have originated from PLA remnants. Contrary to Xie and others,[35] who stated that re-etching after provisionalization restores the original bond strength values, other authors have stated that these substances may be responsible for reducing bond strength values (Watanabe & others).[10]

In this study, a eugenol-based PLA was used (RelyX Temp E, 3M ESPE, St Paul, MN, USA). It has been proposed that dentin provided a major barrier to the diffusion of eugenol inward, and ZOE is an effective barrier against the diffusion of excess unreacted zinc oxide in the set PLA (Hume).[13] The correlation between hydraulic conductance and eugenol diffusion in dentin is not significant for eugenol that is released from zinc oxide eugenol-based cement (Abou Hashieh & others).[14] In previous studies, tooth specimens were treated with Zinc oxide eugenol-based PLAs, and the results indicated that pulpal eugenol concentrations (less than 3.10-3 M) were not cytotoxic to pulpal cells (Hume; Camps & others).[15-16] However, Brännström and Nyborg[44] reported pulpal irritation related to eugenol diffusion when dentin thickness is decreased. Bertschinger and others[36] have stated that the initial application of dentin bonding agents seal dentin against the penetration of eugenol molecules. However, use of the DB method may also reduce the sedative effect of eugenol on dentin, because of the intervening layer of bonding agent.

The effects of the DB method were found to be similar for both luting systems. The DB method was effective in restoring the shear bond strength values that decreased after PLA application *in vitro*. However, it should be noted that this method is more time-consuming and technique-sensitive. While using the DB method, clinicians must be aware of the increased risk of pooling, which would interfere with the marginal fit of indirect restorations while using the DB method.

In this *in vitro* study, only two different resin-based luting systems that use phosphoric acid based conditioners were used, which are limitations of the study. The long-term clinical evaluation of the DB method to validate the results of *in vitro* studies is needed. It would be interesting to evaluate the effects of the DB method and PLA application on new generations of self-etching dentin bonding systems where less aggressive conditioners are used.

CONCLUSIONS

Within the limitations of this study, it was concluded that:

- Regardless of whether or not PLA contained eugenol, the shear bond strength values to dentin significantly decreased compared with the controls (conventional bonding groups).
- When the DB method was used, the PLA application did not reduce the shear bond strength values of two different resin-based luting systems to dentin.
- In SEM examination, the granular substance embedded between the bonding agent and dentin was evident in a provisionalization group.

(Received 14 February 2006)

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