UNIVERSITY OF SIENA
SCHOOL OF DENTAL MEDICINE

PhD PROGRAM
“FACTORS AFFECTING ADHESIVE CEMENTATION OF INDIRECT RESTORATIONS”

PhD THESIS
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TITLE
-Factors affecting adhesive cementation of indirect restorations

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TITLE

Factors affecting adhesive cementation of indirect restorations

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Section 1. Introduction

1.1 Some factors potentially affecting the adhesive-resin cement bonding.

In dental research, a great deal of attention has constantly focused on adhesive procedures that have become routine in the daily dental practice. The coupling of resin-based cements usually requires the adjunctive use of dentin adhesives that are either total-etch or self-etch in nature. The thesis focuses on some factors that can alter this adhesive bond.

For a long time dentists and researchers have assumed that resin cements and composites bond well to dentin adhesives when air inhibition layers are present on the surface of the cured adhesives, while the weak bonds occur between the adhesive and the dentin.¹ ² With the advent of the modern simplified adhesive systems, which are more acidic and hydrophilic in nature, the assumption that bond failures occur between adhesives and dentin and not between adhesives and resin cements may still not be true. There is evidence³ to suggest that simplified self-etch adhesives may not be compatible with chemical polymerization mode of resin cements. The incompatibility between materials is therefore introduced as one of the possible causes of failure in adhesive procedures and in the light of this, the thesis proposes an in vitro protocol based on the application of sustained seating pressure during the luting procedure, in order to verify its effect on the chemical-cured adhesive cementation.

Usually, bonding of resin cement to cured adhesive is achieved in the presence of an oxygen-inhibited layer of unreacted monomers and oligomers,⁴ allowing the materials from both sides to cross the interface and blend together to form an interdiffusion zone, wherein co-polymerization occurs to initiate a chemical bond.⁵ Some clinical procedures, such as long temporary cementation on adhesive sealed dentin can determine this layer absence during the permanent cementation. To date, this oxygen inhibition layer’s role, as far as bond strength is concerned, is not clear.⁶ ⁹ However, even if one considers this aspect, a further objective of the thesis is to investigate the coupling of the resin cement to dentin that had been covered with adhesive before and after temporary cementation.

In order to protect the exposed dentin against bacteria infiltration, the inclusion of antibacterial molecules in adhesive systems has been proposed by manufactures. A
further objective of the thesis was therefore to evaluate possible adverse chemical interactions given by including an added antibacterial monomer in a dual-cured resin cement prototype and to verify its influence on the final bond strength.  In the present thesis, those factors that can influence indirect restoration bond strength are evaluated in the paragraphs that follow.

1.1.1 Incompatibility of the polymerization modes between resin cements and adhesive systems.  
To date, dentin adhesives and resin cements are all cured via a free radical polymerization. Chemical polymerization is traditionally achieved using a binary, redox catalytic system consisting of a peroxide and an aromatic tertiary amine. On the other hand, light-activated polymerization proceeds from the activation of a photoinitiator (i.e. camphoroquinone) to its excited triplet state. This is followed by the reduction of the activated photoinitiator by an amine accelerator to form an intermediate exited complex, releasing free radicals on dissociation.\textsuperscript{10}  
Initially, the incompatibility of simplified adhesives with chemical-cured resin cement or composites was thought to be exclusively caused by interaction of the nucleophilic tertiary amine with the acidic adhesive resin monomers.\textsuperscript{10,11} Charge-transfer complexes that are formed between acidic resin monomers and the tertiary amines impeded free radical generation and resulted in incomplete polymerization of the chemical-cured resin cement or composite.\textsuperscript{2}  
The proposed mechanism has been discussed and assumed as the chemical cause of the above mentioned incompatibility. To circumvent the problem, alternative reducing agents (i.e. acrylic sulphinate salts, ascorbic acid or barbituric acid salts) have been added to simplified adhesives to ensure that optimal polymerization of the resin cements occurs when used in chemical-cured mode.\textsuperscript{12}  
From further studies a research group\textsuperscript{3,13,14} excluded that inherent chemical resin-initiator incompatibility could be the only cause of the adverse reactions with simplified self-etch adhesives. In fact, the authors found similar adverse reactions even when simplified adhesives were used with light-cured composites. In particular, when some time elapsed before composite light-activation.
This issue led the authors to develop an alternative mechanism of the incompatibility in which water is thought to permeate through the adhesive layer and mechanically disrupts the coupling between the adhesive and the composite. In the presence of a slow-setting chemical cured composite, this diffusion process tends to be greater. Thus, at present, the increased permeability in simplified adhesives provides the second major reason for the adverse reactions with chemically cured resin cements.

Researchers came to these conclusions through in vivo and in vitro studies, often combining the results of microtensile tests with the results of microscopy observations. The permeability phenomena has been morphologically described from different kinds of analysis (i.e. SEM and TEM). More precisely, the fluid movement throughout the hybrid layer (dentin-adhesive interface), as well as the fluid movement through the cured adhesive layer (adhesive-composite interface) was found to lead to different, peculiar morphological defects. On the base of their location in the different bonding interfaces and in light of the different materials and methods performed in the experiments, these defects (i.e. voids, resin globules, honeycomb structures) can be assumed as the morphological evidence of adhesive permeability.

1.1.2 In vitro and in vivo evidence of adhesive permeability: total-etch vs. self-etch systems.

Initially, characteristic morphological features were evidenced along the adhesive-dentin interface of some acetone-based adhesives when a wet bonding technique was used. This so called “overwet phenomenon” observed in total-etch (three-step and two-step) adhesives was correlated to transudation of dentinal fluid, from tubules that are opened after acid-conditioning in vital teeth that exhibit a positive pulp-pressure. Then, similar structural defects (resin globules and blisters) were observed along the adhesive-composite interface of self-etch (two-step or one-step) adhesives when used with auto- or dual-cured resin cements, or with chemical cured composite or with delayed light-activated cured composites. For this “new” phenomenon the authors had to consider different causes. In fact, simplified self-etch adhesives do not require the removal of the smear plugs. Moreover, these experiments on hydrated dentin were not performed under dentin perfusion. In addition, the structural defects were never
observed along the dentin-adhesive interface, which instead remained intact. The morphological manifestations were therefore correlated to the osmotic permeability. The latter mentioned studies provided a precise explanation of what presumably occurs. In particular, the fluid movement across the cured adhesive should be caused by a concentration difference between the dentinal tubules (region of low solute concentration) and the adhesive-composite interface (region of high solute concentration), resulting in an osmotic pressure gradient. This fluid movement from the dentin substrate into the unpolymerized interface (i.e. delayed light-activation or slow-chemical-polymerization), produces an immiscible blend of hydrophobic and hydrophilic monomers. All this results in the formation of the characteristic morphological defects observed along the adhesive-composite interface. Emulsion polymerization of the hydrophobic resin in water results in the formation of resin globules and resin polymerization around the osmotic blisters produces the honeycomb resin structures. Finally, the osmotic permeability associated to simplified self-etch adhesives has been unequivocally confirmed in a recent in vivo study, where the fluid movement across the adhesive layer has been found, through resin replicas, even in endodontically treated teeth.

1.1.3 Adhesive systems to protect the exposed dentin after tooth preparation.

Before adhesive permeability knowledge, dentine bonding agents have been recommended as an effective way of sealing dentinal tubules. Many studies have claimed that dentin bonding alone was able to guarantee a tight seal of the tubules. When applied on vital prepared dentin, the adhesive resin material can infiltrate exposed collagen network forming a hybrid layer, resin tags and adhesive lateral branches, thereby decreasing the dentin permeability. 

Indirect restorations often require removal of most of the enamel resulting in an exposed dentine surface. Dentine is a tissue crossed by tubules 0.6-2.0 m in diameter and these tubules provides routes for the passage of bacteria, fluids, chemical substances molecules and ions to and from the dental pulp. The prepared teeth are typically protected by provisional restorations that are retained by temporary cements, that are often subject to problems that include microleakage, dislodgement and even fracture.
These all can affect clinical performance and longevity of the restoration, as well as contribute to potential pulp damage and onset of post-preparation tooth sensitivity, if an appropriate sealing procedure is not employed. Despite the improved sealing performance of available adhesives, to date, it is not clear how effective this adhesive barrier is and how long it lasts. Moreover, if the adhesive system is placed before the temporary cement, it is difficult to predict the overall behavior of this dentin substrate when resin cement is used as the final bond.

In order to “seal” the dentin, self-etch adhesive systems can be used instead of total-etch systems, since the smear layer in the bonding mechanism is incorporated, thus, less post-operative sensitivity is expected to occur. On the other hand, among the multiple bacteria sources that may be implicated in tooth preparation infection, there is also the smear layer. In particular, Brannstrom included: 1) bacterial invasion from the tooth surface via marginal gap formation between tooth and restorative material; 2) bacteria present in the dentinal tubules; 3) bacteria present at the dentino-enamel junction; 4) bacteria recontaminating the surface of tooth prior placing the restoration and 5) bacteria present in the smear layer. Even when the tooth preparation is sealed completely, bacteria existing in the smear layer can multiply and their toxins and degradation products can diffuse into the pulp, resulting in irritation and inflammation.

To reduce the potential risk of pulpal inflammation resulting from bacterial activity, the incorporation of antibacterial monomer in the adhesive system may represent a possible alternative solution. However, there are no benefits if the antibacterial agent decreases the bond strength of the indirect restoration which is bonded with a resin cement by altering its sealing ability to dentin.

In the present research project, after different bonding procedures, the evaluation of the bond quality of the luting cementation system represents the trait de union among all the studies. The experiments were performed in vitro in order to simulate cementation of indirect restorations. The main laboratory examinations performed were microtensile test and scanning electron microscopy observations. One microleakage study was included.
References


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Section 2. The effect of different bonding procedures on problematic bonds between simplified adhesives and chemical-cured resin cementation.

2.1 An in vitro study of the effect of the seating pressure on the adhesive bonding of indirect restorations.
Nicoletta Chieffi, Stefano Chersoni, Michele Vano, Cecilia Goracci, Carel L. Davidson, Franklin R. Tay, Marco Ferrari.

ABSTRACT
Purpose: To evaluate the effect of two different techniques of seating pressure application on the adhesive-dentin bond of indirect restorations. Methods: Eight non-carious human third molars were randomly divided in two treatment groups (four teeth each). Cylindrical composite blocks were luted with a resin cement (Panavia F, Kuraray). In Group 1, the seating pressure was applied for 5 seconds. In Group 2, the resin cylinder was maintained under constant pressure during the entire three minute polymerization period of the resin cement. After storing in distilled water for 24 hours, 0.9 x 0.9 mm sticks were produced from these luted specimens for microtensile bond testing and SEM examination. Results: The Student t-test showed a significant difference (p<0.05) in bond strength between Group 1 and Group 2. SEM revealed the presence of structural defects and resin globules on the seating surface of the composites. These features were exclusively identified from the Group 1 specimens and were probably caused by fluid transudation from the underlying dentin through the simplified self-etch adhesive (ED primer).
Clinical Significance: Pressure application during the entire course of setting of the dual-cured resin cement improves the bond strength and reduces fluid interference from the bonded dentin.
INTRODUCTION

Although posterior composite restorations require minimal intervention and cavity preparation, indirect aesthetic restorations have remained popular among clinicians as they can overcome some of the limitations associated with direct composite placement. When indirect restorations are luted with thin layers of contemporary adhesive cements, polymerization shrinkage stresses are reduced in cavities with high cavity configuration. This results in stronger and more durable bonds to tooth structures and improvements in the fracture resistance of these restorations.

Auto-cured or dual-cured resin cements that utilize peroxide/aromatic tertiary amine catalytic systems are frequently used for bonding of indirect restorations in conjunction with total-etch or self-etch dentin adhesives. Self-etch systems are advantageous in that they are less technique-sensitive and exhibit a lower incidence of postoperative sensitivity. However, when simplified self-etch adhesives were used with auto-cured or dual-cured resin composites, abnormal features were reported along the adhesive-composite interface that were deleterious to the integrity of these. Conversely, the use of the resin-coating technique, which involves the application of an additional less hydrophilic resin layer, was found to improve the bond strength of the resin cements.

Previous studies have examined the effect of the magnitude of seating force on film thickness of luting cements. However, little is known about the effect of seating pressure on the bond strengths of indirect restorations that are coupled to dentin via the use of auto-cured resin cements. As auto-cured resin cements have longer setting times than light-cured resin cements, there is sufficient time for water to diffuse from the underlying bonded dentin across the polymerized hydrophilic adhesive layer via an osmotic gradient.

This study examined the effect of the seating pressure on the coupling of a dual-cured resin cement (Panavia F, Kuraray Medical Inc., Tokyo, Japan) to one-step self-etch adhesive bonded dentin. The null hypothesis tested was that the manner in which the seating pressure was applied during the setting time of the resin cement has no influence on the microtensile bond strength of indirectly composite restorations to dentin.
MATERIALS AND METHODS

Eight non-carious human third molars that were stored in a 0.5% chloramine T solution at 4°C were used within one month following extraction. All the teeth were bonded in their normal hydrated status, as they were retrieved from the storage medium. The composition of Panavia F is shown in Table I. The operating procedures for the experimental and control groups are summarized in Table II.

Table I. Composition of the material employed in this study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Components</th>
<th>Batch #</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Panavia F</td>
<td>Primer a: HEMA, 10-MDP, 5-NMSA, water, accelerator. Primer b: 5-NMSA, accelerator, water, sodium benzene sulfinate.</td>
<td>41170</td>
<td>Kuraray Medical Inc., Tokyo, Japan</td>
</tr>
<tr>
<td></td>
<td>Paste A: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide. Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Abbreviations- HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA: N-methacryloyloxy-5-aminosalicylic acid.

Table II. Procedures employed for the two groups and their microtensile bond strengths.

<table>
<thead>
<tr>
<th>Group designations</th>
<th>Bonding procedures</th>
<th>Pressure applied (MPA)</th>
<th>Laboratory examinations</th>
<th>Microtensile bond strength (MPa)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Control group</td>
<td>Panavia F</td>
<td>1,249 for 5 sec</td>
<td>Microtensile and SEM</td>
<td>9.8 ± 5.7 A</td>
</tr>
<tr>
<td>2 Experimental group</td>
<td></td>
<td>1,249 for 3 min</td>
<td></td>
<td>20.7 ± 7.3 B</td>
</tr>
</tbody>
</table>

*Values are means ± standard deviations in MPa. The different superscripts indicates a statistically significant difference (P< 0.05)
Specimen Preparation

The occlusal enamel was removed using a slow-speed saw with a diamond-impregnated disk (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. The root of each tooth was cut at the same level, in order to obtain tooth samples with a standardized height (5mm). A 180-grit silicon carbide paper was used under running water to create a clinically relevant smear layer on the dentin surface.

Composite cylinders (Tetric-Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein) of 10 mm in diameter and 6 mm in height were prepared using a split aluminum mold, in order to limit and standardize the bonding area. Prior to cementing procedures, the bonding surface of each resin cylinder was sandblasted, etched with phosphoric acid, rinsed with water and dried.

Cementing procedures

The specimens were randomly divided in two treatment groups (N=4). In the Control group, the resin cement Panavia F (Kuraray Medical Inc., Tokyo, Japan) was used according to manufacturer’s instructions. ED Primer (A+B) was mixed and applied with a brush on the dentin surface and left in place for 30 s. After drying the etched surface with gentle air flow, Panavia F resin cement (A+B) was mixed and applied on each of the freshly prepared composite cylinder. The composite block was cemented to the dentin surface using a seating pressure of 1,249 MPa for 5 s. To ensure optimal polymerization of the resin cement along the exposed margins, a layer of Oxyguard was applied after removal of the excess cement with a small scaler and left in place for at least 3 min.

In the Experimental group, the bonding procedures were the same as described in the Control group, except for the application of the cementation pressure. During the entire three-minute period that was necessary for the complete polymerization of the resin cement, the resin cylinder was maintained under a constant seating pressure of 1,249 MPa. The excess cement was removed and the Oxiguard was applied while the specimen was under pressure. For both groups, in order to standardize the applied pressure, a metallic tool (Fig. 1) that delivered 10 Kg was used. This resulted in a seating force of 98,1 N. The pressure [N/m2] was calculated, dividing this force [N] by
the surface area [m2] of the metal weight. The value obtained was finally converted in MPa.

![Image](image.png)

**Fig. 1** The metallic tool used to standardized the seating pressure application. The tooth was positioned on the basal surface of the tool and the composite cylinder was cemented, applying the weight on the upper surface of the tool.

**Bond Strength Evaluation**

After cementation, all the specimens were stored in water for 24 hours at 37°C. Each tooth was then sectioned vertically with the Isomet saw into a series of slabs. The slabs were then sectioned vertically into 0.9 x 0.9 mm sticks, based on the “non-trimming” version of the microtensile bond testing technique. Each stick was measured using a digital caliper to determine the cross-sectional area. The sticks were attached to a testing device with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA). The device was attached to a universal testing machine (Triax digital 50, Controls, Milano, Italy) and loaded in tension at a crosshead speed of 0.5 mm/min until failure.

**Statistical Analysis**

After checking for normal distribution with the Kolmogorov-Smirnov test, the differences in bond strength values between the two groups were tested for statistical significance using the Student t-test. The level of significance was set at $\alpha=0.05$.

**b. SCANNING ELECTRON MICROSCOPY (SEM)**

**Specimens Preparation**
After microtensile bond testing, eight pairs (fractured composite and dentin sides) of specimens from each group were randomly selected for SEM examination. Each specimen was mounted on metallic stubs, sputtered with gold/palladium and observed with a SEM (JSM – 6060LV, JEOL, Tokyo, Japan) operating at 10 or 15 kV.

Images of the two complementary debonded interfaces for each fractured specimen were taken at different standardized magnifications at 100-6,000X. The angle of observation of the samples was 90°. The SEM evaluation was performed by double-blinded operators.

RESULTS

a. MICROTENSILE BOND STRENGTHS

The Control group exhibited significantly lower tensile bond strength than the Experimental group (p<0.05) [Table II], indicating that the application of a sustained seating pressure during the cementation procedure had a positive effect on the final bond strength of the resin cement tested.

b. SEM OBSERVATIONS

The most remarkable finding was the observation of structural defects in fractured specimens derived from the Control group. These defects consisted of compartmentalized structural entities that were nearly identical in almost all of the fractured composite interfaces examined, except for differences in their dimensions (Fig 2a-b; Fig 3). Moreover, the corresponding fractured dentin interfaces in the Control group featured resin globules that represented the separation phase of resin components (Fig 4). These features were completely absent in fractured interfaces of the Experimental group (Fig. 5).
Fig. 2 SEM image of composite side from Group 1, in which the seating pressure was applied for five seconds. (a) A representative medium magnification view (1500x) showing structural defects in the majority of the composite interface (b) A high magnification view (6000x) of a region in (a).
Fig. 3 SEM image (4000x) of composite side from Group I. The micrograph shows the structural design of the faults observed.

Fig. 4 SEM image (6000x) of dentin side of a representative high magnification view of a specimen from Group I. The micrograph shows resin globules.
DISCUSSION

As the microtensile bond strength of the resin cement was significantly higher when the seating pressure was maintained throughout the entire setting period of the cement, we have to reject the null hypothesis tested in this study. Furthermore, the quality of the debonded interfaces was inferior when the seating pressure was only applied for 5 s.

The low microtensile bond strength results observed in the Control group are comparable with those reported in previous studies\textsuperscript{23,24} when the same resin cement was used according to the manufacturer’s instructions. It should be stressed that these low bond strengths were not caused by the incompatibility between acidic simplified dentin adhesives and the basic amine catalysts in auto/dual-curable resin composite. This is due to the inclusion of two different types of sulphinate ternary catalyst\textsuperscript{25} in the ED primer as well as the Panavia F resin cement (Dr. K. Arikawa, Kuraray, personal communication). Should such incompatibility exists, it should be reflected in the bond strength results of both the Control and Experimental groups, and not in the former
only. Thus, it is logical to assert that the low bond strengths and the observation of structural defects and resin globules in the Control group was not caused by acid-base incompatibility of the resin cement system. Indeed, the resin globules seen in ED primer-treated dentin from the Control group (Fig. 4) had also been identified in previous studies that employed single-step self-etch adhesives\textsuperscript{26} or two-step etch-and-rinse adhesives.\textsuperscript{23} These globules represent emulsion polymerization of the hydrophobic components of the resin cement that were in contact with the water that had permeated through the etched and primed dentin.\textsuperscript{12}

Moreover, the structural defects that were identified from the fractured composite side of the specimens in the Control group (Fig. 3) had also been reported previously as “honeycomb structures” or “blisters” in previous studies. Based on SEM and TEM evaluations,\textsuperscript{11,12} these defects were located along the adhesive-composite interface. In those studies, the authors suggested that the blisters were either filled with water that permeated from dentinal tubules, or represented incompletely polymerized regions within the primer layer that resulted from the entrapment of water. When stressed to failure, these blisters may act as stress raisers that expedite crack propagation through the adhesive-resin cement interface.

In order to improve the coupling of resin cements to hydrated dentin bonded with simplified adhesives, the application of an additional coat of a more hydrophobic resin to the primed dentin has been recommended. Although such a technique is useful for direct composite restorations, it has limited use in the bonding of indirect restorations. This is because the addition of an additional hydrophobic resin coating would affect the fit of these indirect restorations. Reduction in the permeability of the primer-cement system to water derived from the underlying sound dentin may account for the higher bond strengths observed in the Experimental group (Group 2). Previous studies\textsuperscript{27,28} that evaluated the long-term durability of resin bonds to dentin confirmed the inverse relationship between water penetration through adhesive systems and bond strength results. The results of this study demonstrated that the detrimental effect of adhesive permeability associated with simplified self-etch adhesives can be eliminated by the application of sustained seating pressure during the luting of indirect restorations. Although, the results of this in vitro study cannot fully predict the clinical behaviour of the same material applied on vital teeth as no fluid diffusion system was applied, such
investigation can contribute to understanding of the adverse interactions\textsuperscript{29} that occur between chemical-cured resin cements and simplified self-etch dentin adhesives. Further studies should be done to test seating pressure effect on auto/dual-curing resin cements when the pulpal pressure is simulated.
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Section 2. The effect of different bonding procedures on problematic bonds between simplified adhesives and chemical-cured resin cementation.

2.2 The effect of application sustained seating pressure on adhesive luting procedure.  
Nicoletta Chieffi, Stefano Chersoni, Federica Papacchini, Michele Vano, Cecilia Goracci, Carel L. Davidson, Franklin R. Tay, Marco Ferrari

ABSTRACT
Objectives: To evaluate the effect of short versus long application seating pressure on the bond strength of resin blocks, luted with a dual-cured resin cement (Panavia F) to pre-coated or non pre-coated dentin with an hydrophobic light-cured adhesive (Clearfil Protect Bond). Methods: Sixteen non-carious human third molars were randomly divided in six Groups (4 teeth each). Cylindrical composite blocks were luted with Panavia F (Group Ia) and with Clearfil Protect Bond with Panavia F (Group IIa) and the seating pressure was applied for 5 seconds. In Groups Ib, IIb, the two bonding procedures were respectively repeated, but the resin cylinder was maintained under constant pressure during the entire three-minute polymerization period of the resin cement. After storing in distilled water for 24 hours, 0.9 x 0.9 mm sticks were produced from these luted specimens for microtensile bond testing and SEM examination. Results: The use of Clearfil Protect Bond with Panavia F produced higher bond strengths than the use of Panavia F (p<0.05). Extending the time of pressure application up to 3 minutes increased the bond strength (p<0.001) and improved the integrity of the interfacial quality.  
Significance: The application of sustained seating pressure during luting procedures and the additional use of a hydrophobic light-cured adhesive both improve the final bond strength of the resin cement.
INTRODUCTION

It has been widely demonstrated that simplified self-etch adhesives can attract fluid from dentin after polymerization.\textsuperscript{1-6} This fluid absorption is due to relatively high concentrations of ionic or acidic monomers in the self-etching primers (in the two-step systems)\textsuperscript{1} or in adhesive (in the single-step systems)\textsuperscript{2-6} essential to enable these systems to diffuse through the smear layer and demineralize the underlying dentine.\textsuperscript{7}

Recently it was reported that incompatibility may be present when auto-cured or dual-cured resin cements were used in conjunction with simplified self-etch dentin adhesives.\textsuperscript{8,9} Adverse chemical interaction and adhesive permeability were identified as the two main factors responsible for the reduction in bond strength, when slow setting resin cements were coupled to bondings on hydrated dentin.\textsuperscript{10} Even when the interaction between acidic simplified dentin adhesives and the basic amine catalysts in dual-curable resin cements may be eliminated by the use of sulphinate-type ternary catalysts in the self-etching primer and the resin cement,\textsuperscript{11} adhesive permeability remains a possible cause of bond strength reduction. This is because auto/dual-cured resin cements have longer setting times than light-cured resin cements and, in time, water can diffuse from the underlying bonded dentin across the polymerized hydrophilic adhesive layer via an osmotic gradient.\textsuperscript{6} A previous study concluded that the additional application of a more hydrophobic resin layer resulted in improved coupling of the dual-cured resin cement to dentin.\textsuperscript{12} Another recent investigation\textsuperscript{13} indicated that pressure application during the entire course of the setting of a dual-cured resin cement improves the bond strength and reduces fluid interference from the underlying, with bonding covered dentin.

The objective of this study was to evaluate the effect of the sustained seating pressure on the integrity of the adhesive bond in resin blocks, in order to simulate indirect restorations, luted with a dual-cured resin cement. The effect of seating pressure was examined by comparing the use of Panavia F as recommended by the manufacturer (i.e., resin cement applied to dentin primed with the one-step self-etch adhesive included in the kit), versus the application of Panavia F to dentin that had first undergone a bonding procedure with the two-step self-etch adhesive Clearfil Protect Bond.
The null hypotheses tested were that: (a) the application of sustained seating pressure during the curing of the resin cement has no influence on the microtensile bond strength of resin blocks to dentin; and (b) the additional application of the two-step self-etch adhesive has no influence on the microtensile bond strength of the dual-cured resin cement.

**MATERIALS AND METHODS**

Sixteen non-carcious human third molars that had been stored in a 0.5% chloramine T solution at 4°C were used within one month following extraction. All the teeth were submitted to bonding in their normal hydrated status, as they were retrieved from the storage medium. The composition of the materials is shown in Table I. The operating procedures for the four experimental and control Groups are summarized in Table II.

**Table I.** Batch number (#), composition and manufacturer of the materials employed in this study.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Components</th>
<th>Manufacturer</th>
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<tbody>
<tr>
<td>Panavia F</td>
<td>Primer a: HEMA, 10-MDP, 5-NMSA, water, accelerator.</td>
<td>Kuraray Medical Inc.,</td>
</tr>
<tr>
<td>(# 41170)</td>
<td>Primer b: 5-NMSA, accelerator, water, sodium benzene sulfinate.</td>
<td>Tokyo, Japan</td>
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<tr>
<td></td>
<td>Paste A: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide. Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass.</td>
<td></td>
</tr>
<tr>
<td>Clearfil Protect Bond</td>
<td>Primer: HEMA, 10-MDP, 12-MDPB*, hydrophilic dimethacrylate, water</td>
<td>Kuraray Medical Inc.,</td>
</tr>
<tr>
<td>(#41112)</td>
<td>Bond: 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, di-Camphoroquinone; N,N-Diethanol-p-toluidine, silanated colloidal silica, surface treated sodium fluoride.</td>
<td>Tokyo, Japan</td>
</tr>
</tbody>
</table>

Abbreviations- HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacryloxyloxydecyl dihydrogen phosphate; 5-NMSA: N-methacryloyl-5-aminosalicylic acid; 12-MDPB: 12-Methacryloyloxydecylypyridinium bromide; Bis-GMA: Bis-Phenol A diglycidylmethacrylate.

*MDPB has been included as for its antibacterial properties.
Table II Procedures employed for the four Groups and their microtensile bond strengths.

<table>
<thead>
<tr>
<th>GROUP DESIGNATION</th>
<th>Microtensile bond strength (MPa)*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>a</td>
</tr>
<tr>
<td></td>
<td>Seating pressure: for 5 sec.</td>
</tr>
<tr>
<td>I (a &amp; b)</td>
<td>Panavia F</td>
</tr>
<tr>
<td>II (a &amp; b)</td>
<td>Clearfil Protect Bond + Panavia F</td>
</tr>
</tbody>
</table>

Values are means (standard deviations) in MPa. Groups with the same superscripts are not significantly different (P>0.05).

a. MICROTENSILE EXAMINATION

Specimen Preparation

The occlusal enamel was removed using a slow-speed saw with a diamond-impregnated disk (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. The root of all teeth were cut at the same level, in order to obtained tooth samples with a standardized height (5mm). A 180-grit silicon carbide paper was used under running water to create a clinically relevant smear layer on the dentin surface.

Composite cylinders (Tetric-Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein) of 10 mm in diameter and 6 mm in height were prepared using a split aluminum mold, in order to limit and standardize the bonding area. Prior to cementing procedures, the bonding surface of each resin cylinder was sandblasted, etched with phosphoric acid, rinsed with water and dried.

Cementing procedures

The specimens were randomly divided in six Groups (N=4). In Group Ia, the resin cement Panavia F (Kuraray Medical Inc., Tokyo, Japan) was used according to manufacturer’s instructions. ED Primer (A+B) was mixed and applied with a brush on the dentin surface and left in place for 30 s. After drying the etched surface with gentle air flow, Panavia F resin cement (A+B) was mixed and applied on each of the freshly prepared composite cylinders.
In Group IIa, the light-curing self-etch adhesive Clearfil Protect Bond (Kuraray Medical Inc., Tokyo, Japan) was used according to manufacturer’s instructions. This material also contains 12-methacryloyloxydodecylpyridinium bromide (MDPB) monomer to include antibacterial properties which may be useful in cases of vital teeth. The self-etching primer was applied with a brush on dentin surface and left in place for 20 sec. After drying the etched surface with mild air flow, the bonding was applied on the etched-primed dentin, gently air flowed and light-cured for 10 sec. Then Panavia F resin cement (A+B) was mixed and applied on each of the freshly prepared composite cylinders.

In these two Groups, the composite block was cemented to the dentin surface using a seating pressure of 1,25 MPa for 5 sec. To ensure optimal polymerization of the resin cement along the exposed margins, a layer of Oxyguard was applied after removal of the excess cement with a probe and left in place for at least 3 min.

In Group Ib and in Group IIb, the bonding procedures were the same as described for Group Ia and Group IIa, except for the application of the cementation pressure. During the entire three-minute period that was necessary for the complete polymerization of the resin cement, the resin cylinder was maintained under a constant seating pressure of 1,25 MPa. The excess cement was removed and the Oxiguard was applied while the specimen was under pressure. In order to standardize the applied pressure, a metallic tool (Fig 1) that delivered 10 Kg was used. This resulted in a seating force of 98,1 N. The pressure [N/m²] was calculated, dividing this force [N] by the surface area [m²] of the metal weight. The value obtained was finally converted in MPa.

**Bond Strength Evaluation**

After cementation, all the specimens were stored in water for 24 hours at 37°C. Each tooth was then sectioned vertically with the Isomet saw into a series of slabs. The slabs were then sectioned vertically into 0.9 x 0.9 mm sticks, based on the “non-trimming” version of the microtensile bond testing technique. Each stick was measured using a digital caliper to determine the cross-sectional area. The sticks were attached to a testing device with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA).
The device was attached to a universal testing machine (Triax digital 50, Controls, Milano, Italy) and loaded in tension at a crosshead speed of 0.5 mm/min until failure.

**Statistical Analysis**

The population of bond strength values was normally distributed according to the Kolmogorov-Smirnov test (p>0.05). The Two-Way ANOVA was applied with bond strength as the dependent variable, material and pressure application time as factors. Tukey’s test was used for post-hoc comparisons.

**b. SCANNING ELECTRON MICROSCOPY (SEM)**

**Specimen Preparation**

After microtensile bond testing, eight pairs (fractured composite and dentin sides) of specimens from each Group were randomly selected for SEM examination. Each specimen was mounted on metallic stubs, sputtered with gold/palladium and observed with a SEM (JSM – 6060LV, JEOL, Tokyo, Japan) operating at 10 or 15 kV. Images of the two complementary detached interfaces for each fractured specimen were taken at different standardized magnifications: 100-6,000X.

**RESULTS**

**a. MICROTENSILE BOND STRENGTHS**

Bond strength results of the four Groups are shown in Table II.

The selection of the luting material was a significant factor with regard to obtained bond strength; with Clearfil Protect Bond + Panavia F measuring significantly higher bond strengths than Panavia F (p<0.05).

Pressure application time was a also significant factor. Extending the time of pressure application up to 3 minutes significantly increased the bond strength (p<0.001).

The interaction between cement and time of pressure application was also significant to obtained highest bond strength. According to the Tukey’s test applied for post-hoc comparisons, Group IIb achieved the highest bond strength and the difference was statistically significant (p<0.05). Group Ib and Group IIa were
comparable ($p>0.05$). The latter was comparable to Group Ia, which had the lowest bond strength.

b. SEM OBSERVATIONS

SEM analysis of the specimens from those Groups where the seating pressure was sustained for a few seconds, revealed the presence of structural defects. These defects consisted of compartmentalized structural entities that were nearly identical in almost all of the fractured composite interfaces examined from Groups Ia (Fig. 2).

![SEM image of fracture composite side of a specimen from Group Ia, where the seating pressure was applied for five seconds. The micrograph (4000x) shows the structural design of the defects observed.](image)

The most significant result in Group IIa (pre-coated light-cured adhesive dentin, with a few seconds of pressure application time), was that separation was between the adhesive and the resin cement (Fig. 3a). Moreover, resin globules that represented the separation phase of resin components were present between the detached adhesive and the resin cement (Fig 3b).
Fig. 3 SEM image of fractured composite sides of a specimen from Group IIa, where the seating pressure was applied for five seconds. (a) A medium magnification view (1000x) of composite side, showing structural defects in most of the adhesive layer, almost completely detached from the resin cement. (b) A high magnification view (4000x) of an area in photo a, showing resin globules.
In contrast to this, such features (structural defects and resin globules) were not found at the fractured interfaces of Group Ib where the seating pressure was sustained for 3 min.

In almost all specimens examined in Group IIb the fracture was located between the resin cement and the composite resin block and a strong adhesion between the adhesive layer and the resin cement was present (Fig. 4).

**DISCUSSION**

As the microtensile bond strength of the resin cement was significantly higher when the seating pressure was maintained throughout the entire setting period of the cement, the first null hypothesis tested in this study has to be rejected.

If the SEM evaluation had not be performed, we could attribute the lower bond strength of the control Group to voids caused by air introduced during hand mixing of the two component pastes of the dual cured cement. However when examining the micrographs another explanation may be found.
As a result of the incompatibility of the absorbed water with the hydrophobic components in the resin cement, material discontinuity (presence of globules)\textsuperscript{15} and irregular features\textsuperscript{12,16} along the adhesive-composite interface were previously reported. The osmotic permeability of the simplified adhesive systems has been described in experiments performed with pulpal pressure simulations\textsuperscript{1,5} and also in those experiments performed without perfusion system\textsuperscript{3,4,17,18}. In these latter studies, as well as in the present investigation, it is reasonable that fluid interferences can be occurred as the samples were retrieved from the storage medium and bonded in their normal hydrates status. Still, transmission of dentin fluid through simplified dentin adhesives was demonstrated to occur in vivo, in endodontically treated dentin\textsuperscript{19}.

It is clear that such structural defects (Fig 2) observed in those Groups where the seating pressure was only applied for a few seconds, were therefore caused by water that had permeated through the etched and primed dentin. These defects were even observed in those samples that had been pre-treated with the hydrophobic light-cured adhesive (Fig.3 a-b). Thus, it is feasible that permeability of the light-cured self-etch adhesive may have compromised the ultimate bond strength result of the resin cement.

Apparently the application of a sustained seating pressure suppresses the absorption of water and the globules formation, resulting in a better quality of the adhesive interface. These results are in agreement with those obtained in a previous investigation\textsuperscript{13}. As a consequence, reduction of water infiltration, coming from the underlying dentin into the primer-cement system may account for the higher bond strength as observed in Groups Ib, IIb.

Since Clearfil Protect Bond together with Panavia F, regardless of pressure application time, had significantly higher bond strengths than Panavia F, the second null hypothesis tested in this study has also to be rejected. This means that the additional application of a hydrophobic light-cured adhesive improves the final bond strength of the resin cement. This latter result confirms those studies\textsuperscript{20,21} showing that the application of an additional hydrophilic resin layer improves resin cement bond strength. Moreover, as the extensive and prolonged antibacterial activity of the adhesive tested in the present investigation has been previously demonstrated\textsuperscript{22,23}, clinical protection against oral bacteria can be expected from this product.

Also the selection of cement in combination with pressure application time was
found to be significant to obtained highest bond strength. In particular, Group IIb (pre-coated light-cured adhesive dentin, with 3 min pressure application time) achieved the highest bond strength among all the other Groups. Thus, it can be speculated that, since the inherent self-etch adhesive system permeability was eliminated by the sustained seating pressure, the application of the hydrophobic light-cured adhesive was more efficient in improving the bond strength of the resin cement, because the incompatibility factors had been reduced. This finding confirms a previous investigation, showing that when simplified adhesives were bonded to dehydrated dentin, no compromised bond strength resulted.

An unexpected outcome of this study was the difference in the kind of fracture observed in Group IIa, located between the adhesive and the resin cement (Fig. 3 a-b) and in Group IIb, between the resin cement and the composite block (Fig. 4). From SEM analysis, it should be stressed that when the short seating pressure was applied (Group IIa), the water that had permeated through the adhesive interfered with the hydrophobic resin cement and resulted in a weaker adhesion. Thus, failure in adhesive/resin-cement bonding interface resulted. On the other hand, with three minutes seating pressure, a stronger adhesion between adhesive and resin cement occurred due to the reduction of the adhesive permeability. Thus, the weakest bonding interface was between the resin cement and the composite restoration. In order to improve the adhesion of the latter bonding interface, the composite block surface could be treated with hydrofluoric acid and bonding.

The results of this study demonstrated that:
To improve coupling of the resin cement to dentin, both the additional self-etch adhesive containing the light-cured hydrophobic bonding layer and the sustained seating pressure during cementation are effective. The latter reduces the incompatibility between simplified-step adhesives and dual-cured resin cements, since it reduces the adhesive permeability associated with simplified self-etch adhesives. However, since pulpal pressure was not simulated in this study, the results of this in vitro study still cannot fully predict the clinical behaviour of these materials applied on vital teeth.
REFERENCES


10. Tay FR, Pashley DH, Yiu CK, Sanares AM, Wei SH. Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured


Section 3. **The final bond of resin cement with adhesive covered dentin before temporary cementation.**

3.1 **The effect of pre-sealing tooth preparations with dentin adhesives on permanent crown cementation: A Pilot Study.**

Nicoletta Chieffi, Cecilia Goracci, Marco Simonetti, Francesca Monticelli, Marco Ferrari

**ABSTRACT**

**Objectives:** 1. to investigate whether the sealing procedures performed before the provisional cementation affect the efficacy of the permanent luting procedures, and 2. to determine the sealing capacity of two different resin-based bonding systems.

**Materials and methods:** Thirty extracted molars were prepared for full-crowns restorations. Samples were randomly divided into three groups (10 teeth each). Dentin desensitizers were applied in two sessions in order to simulate “abutment preparation” and “cementation appointment”. Dentin treatments included: a “one-bottle” self-curing adhesive system (Group 1); a self-etching light-curing two component adhesive system (Group 2); water, that was applied as a control instead of a desensitizing agent (Group 3). Eugenol-free temporary cement was applied on the treated surface and the specimens were stored in distilled water for 1 week. After the removal of cement, the adhesive systems and the resin cement was used to finally cement composite crowns to the abutments. After the luting procedure, specimens were prepared for microleakage test and for a standardized SEM examination. Dye penetration was statistically evaluated using the Kruskal-Wallis Non Parametric Analysis of Variance (p<0.05).

**Results:** Statistical analysis showed no significant difference in the microleakage exhibited by the three groups (p=0.85). SEM observation of Group 1 and Group 2 revealed a perfect bond between the cement and the adhesive and between the adhesive and the dentin. Resin replicas of specimens belong to any of the three groups revealed a gap between the crown and the cement.

**Conclusion:** This pilot study suggests that the application of the two resinous dentin coating, before the temporary cementation, does not effect negatively the seal of luted resin crowns. These findings need to be researched further.
INTRODUCTION

Post-operative sensitivity is frequently encountered in vital teeth that have been prepared for complete-coverage crowns. The prepared teeth are typically protected by provisional restorations that are retained by temporary cements. Frequently, the patients suffer from sensitivity during the temporization phase, when the provisional restorations are removed prior to permanent cementation of the final restorations, following permanent luting or during any combination of these events.\(^{15,26}\)

Various theories have been presented to explain the mechanism of dentin sensitivity.\(^{12}\) Currently, the most widely accepted theory is the one based on hydrodynamics. Displacement of fluids is thought to be responsible for the deformation of nerve fibers located in the dentin tubules.\(^{1}\) Sealing dentin with adhesive resin systems has been proposed as a means to prevent the undesired fluid movement after mechanical dentin exposure.\(^{13}\) Any system that obstructs infiltration of exposed dentin is recommended in preventing dentin sensitivity.\(^{23}\)

In an in vivo study, Cuenin et al\(^4\) reported the direct relationship between dentin hypersensitivity and the accessibility of dentin tubules. Pashley\(^{20}\) showed adhesive resin systems to be effective and reliable in reducing sensitivity. Suzuki\(^{25,26}\) and Lam\(^{14}\) evaluated the pulpal response following sealing of the prepared vital dentin with a dentin bonding system and demonstrated how this procedure not only offered a biologic seal to the vital dentin substrate and tubule complex, but also prevented post-operative sensitivity that often accompanies complete crown preparation.

In case of full coverage-crown preparation, millions of dentinal tubules are exposed.\(^{7,22}\) The provisional restoration and the temporary cement are often subject to problems that include microleakage, dislodgement and even fracture. These all can contribute to the onset of post-preparation tooth sensitivity and potential pulp damage, if an appropriate procedure is not employed. This means that all effort has to be given to prevent hydraulic pressure on the tubule fluids. Such pressure may come along with the cementation itself when the still unset cement penetrates into dentinal tubules, displacing an equal volume of dentinal fluid in the pulp.\(^{19}\)

Moreover, mastication forces can be transferred into hydraulic pressure.\(^{32}\) This mechanism is most likely to be the reason why the non-anaesthetized patient
experiences pain during crown cementation. Therefore, it is most important to maintain dentin protection during the provisional phase of treatment and before the permanent luting of the crown.\textsuperscript{2,7,18}

Some clinical studies\textsuperscript{3,5,7,14,15,26,31} reported on the efficacy of different adhesive systems used for preventing sensitivity on prepared vital abutments. When applied on vital prepared dentin, the adhesive resin material can infiltrate exposed collagen network forming a hybrid layer, resin tags and adhesive lateral branches, thereby decreasing the dentin permeability.\textsuperscript{7}

Several types of bonding systems are available in the market. The so called “one-bottle” and the self-etching priming bond systems were well-accepted by practitioners. Recently a “one-bottle” self-curing adhesive system (Exite DSC, Ivoclar Vivadent, Schaan, Liechtenstein) and a self-etching, light-curing, two component adhesive system (AdheSE, Ivoclar Vivadent) were introduced.

The aims of this study were: 1) to investigate whether sealing tooth preparations with dentin adhesives before provisional cementation affects the efficacy of the permanent luting procedures, and 2) to test the null hypothesis that there is no difference in the ability of the two bonding systems in preventing dye penetration in full-coverage crowns that are sealed with these adhesives and subsequently luted with a resin cement.

**MATERIALS AND METHODS**

*Specimens Preparation*

Thirty molars, extracted for periodontal reasons, were selected for this study. These teeth were free from decay or previous restoration, and were kept in distilled water until the experimental time. Each tooth was prepared, for full-crowns restoration, with a 1.5 mm axial and a 2 mm occlusal reduction. The crown margin was positioned in dentin. A chamfer margin was made on all. The teeth were prepared using diamonds burs in a high-speed handpiece under constant water cooling.

The following treatments simulated the “preparation appointment” and the “cementation appointment”.

*Preparation appointment*- The teeth were randomly divided into three groups of 10 teeth each. The teeth in the first group were treated with a “one-bottle” self-curing, etch-and-rinse adhesive (Excite DSC). The teeth in the second group were treated with a
self-etching, light-curing, two component adhesive system (AdheSE). In the control group, the teeth were treated only with water.

In Group 1, the Excite DSC was used according to the manufacturer’s instructions. The conditioning gel (37% phosphoric acid) was applied for 20 s and the substrate was rinsed with water spray for 15-20 s and then gently dried for 1-2 s. A micro brush, impregnated with the self-curing activator, was used for the application of the light-curing solution of the bonding system. One layer of this bonding system was applied and finally the alcoholic solvent was gently removed with air.

In Group 2, the AdheSE was used, as recommended by the manufacturer. An adequate amount of AdheSE Primer was applied with a brush. Once the dentin surface was thoroughly coated with the primer, the product was brushed into the entire surface for another 15 s. The total reaction time was not shorter than 30 s. The excess AdheSE Primer was dispersed with a strong stream of air until the mobile liquid film disappeared. Then, the AdheSE Bond was applied, dispersed with a very weak stream of air and light-cured for 10 s.

In Group 3, the dentin surface was treated only with water and no adhesive as desensitizer was applied.

Following removal of the oxygen inhibition layer of the adhesives with a wet cotton pellet, impressions were taken of the crown preparations using Permadyne (3M ESPE, Seefeld, Germany) impression material, from which resin crowns (Gradia, GC, Tokyo, Japan) were fabricated in the laboratory.

After impression taking, a layer of an eugenol-free temporary cement (Temp-Bond NE, Kerr, Orange, CA, USA) was applied on the surface of each crown preparation. After setting of the temporary cement, the samples were stored in distilled water for 1 week.

Prior to cementing, the fit of the crowns was observed at 24x magnification under a dissecting microscope. Crowns were accepted only when no discontinuity or gaps were found.8

Cementation appointment- Following the one week storage period, the temporary cement covering the dentin specimens was mechanically removed and the surface was cleaned with polishing brushes.
The dentin surface of each tooth was covered again with the adhesive systems, before the final cementation: Excite DSC was utilized for the Group 1 and Group 3 and AdheSE for the Group 2 following manufacturer’s instructions. The crowns were cemented with a resin cement (Variolink II, Ivoclar Vivadent) without additional pre-treatment of their inner surfaces. Although we understand that the bonding of resin composite crowns to resin cements may be improved by silanization, this procedure was not performed so as the results of the study may be extrapolated to the cementation of metal, ceramometal, and alumina/zirconia-based all-ceramic crowns. After cementation, specimens were prepared for the microleakage test.

**Dye Penetration**

Root surfaces were coated with two layers of nail varnish, up to 1 mm beneath the cervical margins. The specimens were then immersed in an aqueous solution of 2% methylene blue solution at room temperature for 24 h and thoroughly rinsed in tap water. The teeth were sectioned with a low speed diamond disk (Isomet, Buehler, Lake Bluff, IL, USA) under water cooling. Each tooth was sectioned into three vertical slices in a mesiodistal direction.

**Microleakage Evaluation**

The specimens were observed under a stereomicroscope at 25x magnification, and the highest score of each sample was recorded. Dye penetration was ranked by two examiners, and the depth of cervical staining was measured according to the parameters described in a recent study (Fig 1).^10^

![Fig. 1 Schematic illustration of the dye penetration scale, according to the parameters described in the text](image)
Score 0 = no dye penetration; Score 1 = leakage not exceeding the first third of the cervical wall; Score 2 = penetration past the first third of cervical wall; Score 3 = penetration not exceeding the middle of the axial wall; Score 4 = penetration up to the middle of the axial wall. Since data were not normally distributed dye penetration was statistically evaluated using the Kruskal-Wallis non-parametric analysis of variance. The significance level was defined at p<0.05.

**Scanning Electron Microscopy (SEM) Examination**

All slices were evaluated using SEM. The slices were immediately returned to water after evaluation of the dye penetration and prevented from dehydration. Impressions (Provil Novo, Heraeus Kulzer, Hanau, Germany) of these slices were taken and a set of epoxy resin replicas (Alpha Die, Schuetz Dental, Rosbach, Germany) was made for SEM evaluation. The resin replicas were mounted on metallic stubs and sputtered with gold in a Balzers device (Balzers Ltd., London, Great Britain). They were examined using a SEM (Philips 505, Eindhoven, The Netherlands) operating at 10 kV.

All samples were evaluated in double blind for defects/gap at the adhesive interfaces. Defects were recorded at dentin(D)/adhesive(A) interface, at adhesive(A)/resin cement(R) interface and at resin cement(R)/crown(C) interface - as present or not.

**RESULTS**

The results of the statistical analysis showed no significant difference in the microleakage exhibited by the three groups (p=0.85). The dye penetration of control specimens (Group 3) was not significantly different than that of samples treated with Excite DSC (Group I) or AdheSE (Group II). The frequency and median value of the scores for each of the tested groups are listed in Table 1. The interface gaps evaluated using the SEM are reported in Table 2.

| Table 1 Frequency and median value of the scores for each of the tested groups. |
|--------------------------------|-------|-------|-------|-------|-------|
|                              | Score 1 | Score 2 | Score 3 | Score 4 | Median |
| Group 1 Excite               | 6       | 8       | 11      | 5       | 3      |
| Group 2 AdheSE               | 7       | 7       | 6       | 10      | 3      |
| Group 3 Control              | 7       | 8       | 4       | 11      | 2.5    |
Table 2 The table shows the number of samples with gaps in each of the tested groups for any adhesive interface [dentin(D)/adhesive(A) interface; adhesive(A)/resin cement(R) interface; resin cement(R)/crown(C) interface].

<table>
<thead>
<tr>
<th></th>
<th>D/A</th>
<th>A/R</th>
<th>R/C</th>
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<tbody>
<tr>
<td>Group 1 Excite</td>
<td>2</td>
<td>/</td>
<td>30</td>
</tr>
<tr>
<td>Group 2 AdheSE</td>
<td>4</td>
<td>/</td>
<td>30</td>
</tr>
<tr>
<td>Group 3 Control</td>
<td>25</td>
<td>/</td>
<td>30</td>
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</tbody>
</table>

Some important differences between the groups were observed when the specimens were examined using SEM. Resin replicas analysis of Group 1 (Fig. 2) demonstrated the presence of a large gap between the crown and the resin cement in all samples. Spaces were absent along the resin cement-adhesive interface and almost absent along the adhesive-dentin interface. Similar results were obtained from the resin replicas analysis of Group 2 (Fig. 3). Instead, SEM examination of Control group (Fig. 4) revealed gaps between the crown-resin cement interface and also between the adhesive-hybrid layer interface in almost all samples; only the adhesive/resin cement interface was free of gaps.

Fig. 2 SEM image of resin replicas belong to Group 1. A gap (pointer) could be seen between the crown (C) and the cement (R). Absence of spaces between the cement/adhesive (A)/dentin (D) interfaces.
Fig. 3 SEM image of resin replicas belong to Group 2.
A gap (pointer) could be seen between the crown (C) and the cement (R). Absence of spaces between the cement/adhesive (A)/dentin (D) interfaces.

Fig. 4 SEM image of resin replicas belong to Group 3.
A gap (arrow) could be identified between the crown (C) and the cement (R) and another big gap (pointer) between the adhesive (A) and the hybrid layer.
DISCUSSION

While it is desirable to reduce sensitivity, it is also important to evaluate the possible adverse effects of the desensitizing agents on the adhesion of resin to dentin, in terms of bond strength and microleakage. Several studies\(^3,5,11,15,21,23,24,31\) have reported the effects of desensitizing measures on the bond strength of different bonding systems. Few studies\(^9,16,32\) have reported the effect of tubule coating on the eventual microleakage. In fact, bonding agent application is meant to be directly covered with the permanent luting cement, or in the case of direct restorations, with the restorative resin-based composite. Each handling divergent from this procedure should be regarded as a sort of contamination with possible negative effects on the ultimate adhesive qualities (adaptation and bond strength). As most of the dentin substrate consists of intertubular dentin, which is not likely to form on short term a pathway for infiltration towards the pulp, this material might be better untouched during the provisional stage and be “traditionally” conditioned when the permanent luting is being performed. In this view, it might be preferred to employ an unfilled adhesive resin system such as the first generation Gluma, which has shown to be an excellent tubule infiltrating desensitizing agent\(^3,11,24\) without forming a coat on the dentin substrate.

The results of SEM observations of this study demonstrated that using both resin coating materials almost no gaps are formed at dentin/adhesive interface. This observation can support the hypothesis that following the described dentin coating clinical procedure an important reduction of postoperative sensitivity can be expected. Microleakage in this study was assessed using a dye. This method continues to be the most popular of the techniques, which are currently available, but it presents some disadvantages.\(^31\) The main one it is usually associated with the assigning of the numerical scoring that, although carried out by more than one examiner, is somewhat subjective.

Microleakage test results demonstrated no statistically differences in dye penetration between the specimens pre-coated with the adhesives as desensitizers and the specimens uncoated. The two dentin-coating materials (Excite DSC and AdheSe) can not reduce considerably dye penetration of luting resin crown. On the other hand, based on the SEM observations, neither of the two desensitizers, when used at preparation and
cementation appointments, interfered with the adaptation of the resin cement to dentin. In fact, no gaps between adhesive material and resin cement were noted in all groups. According to recent studies, from a clinical point of view, the use of a “one-bottle” system could be indicated because it simplifies the operative protocol and, at the same time, it assures an effective protection. In spite of that, we have to consider that, recently, several studies demonstrated that no perfect seal can be obtained using simplified bonding systems, because of they act as a semi permeable membrane. Although, similar microleakage median values were obtained from the dye penetration analysis of the three groups selected, some important differences among the groups were extrapolated from the SEM examination. In fact, the two groups in which adhesive systems were employed before the temporary cementation showed an almost perfect bond between the resin cement and the adhesive layer and between the adhesive layer and the dentin. This indicates that a good seal was achieved for the dentinal tubules and no interferences of the provisional cement of the dentinal adhesive. Further, after proper debridement, one may achieve a strong bond between the adhesive (utilized as desensitizers) and the luting cement. On the other hand, the control group was characterized by a different SEM characteristic, in which the major gap was observed between the adhesive and the hybrid layer. Clinically this main result in rapid fluid movements and post-operative sensitivity on the one hand, and the ingress of bacterial on the other. Under the experimental conditions of this study, both total-etch and self-etch adhesive systems seem to affect not negatively the dentin sealing, without producing negative consequences during the adhesive luting procedures. However, in order to obtain a good marginal sealing it is important to pre-treat the inner surface of the crowns, before the final cementation, when resin crowns are chosen. The high microleakage median values, registered in any of the groups analyzed, could be related with the absence of the pre-treatment of the inner surface of the resin crowns before the final cementation. In fact, resin replicas of specimens belong to any of the three group revealed the presence of a gap between the crown and the cement. As no statistically difference was observed in preventing dye penetration between the two bonding systems selected, and because of SEM examination showed intact bonding
interfaces (dentin-adhesive-resin cement) for both the adhesives, we cannot reject the null hypothesis.

The results of this preliminary in vitro study cannot fully predict the clinical behaviour of the same materials applied on vital abutments. No artificial aging methods were applied. Moreover, given the limitations in sample size included in the study, it is possible that these results may be due to a low power of the study. However, these findings may be used in future studies which include larger sample sizes to further evaluate this resin system in the effectiveness of decreasing hypersensitivity in the clinical setting.

**CONCLUSIONS**

This pilot study suggests that the application of a resinous dentin coating, before the temporary cementation, does not effect negatively the seal of luted resin crowns. In order to make clinical recommendations it is necessary to conduct other studies, both in vitro and in vivo, using also adequate sample sizes in order to further evaluate this research question.
REFERENCES


Section 3. **The final bond of resin cement with adhesive covered dentin before temporary cementation.**

3.2 **Influence of a resin cement used on control untreated dentin vs. dentin that had been covered with resin sealers and temporary cement.**
Nicoletta Chieffi, Fernanda Sadek, Francesca Monticelli, Cecilia Goracci, Simone Grandini, Carel L. Davidson, Franklin R. Tay, Marco Ferrari

**ABSTRACT**

**Purpose:** To evaluate the effects of dentin adhesives employed as resin sealers and provisional cementation on the bond strengths of a resin cement to dentin. **Methods:** A two-step etch-and-rinse adhesive (Excite DSC – Group I) and two-step self-etch adhesive (AdheSE – Group II) were applied to exposed dentin surfaces prepared from human molars (N=4). Water was used in lieu of a resin sealer in control Groups 3 and 4. A eugenol-free provisional cement (except for Group 4) was applied to the treated surfaces. After storing in distilled water for one week, the provisional cement was removed and cylindrical composite blocks were luted with a resin cement (Variolink II). 0.9 x 0.9 mm sticks were produced from these luted specimens for Microtensile bond testing and SEM examination. **Results:** One-way ANOVA revealed that neither the resin sealer nor the temporary eugenol-free cement had a negative effect on the final bond strength (p>0.05). Mixed failures were predominantly identified from SEM. **Clinical Significance:** The use of dentin adhesives as resin sealers before provisional cementation with a non-eugenol provisional cement does not adversely affect the retentive strength of indirect restorations bonded subsequently with an adhesive and a resin cement.
INTRODUCTION

In crown and bridge restorations, temporization of the prepared teeth is obligatory for the patients' aesthetic and functional needs. Sealing of the freshly-exposed vital dentin during the fabrication of provisional restorations has clinical merits, as external stimuli may trigger rapid fluid shifts within dentinal tubules, and contribute to post-operative sensitivity.¹ Previous studies²⁻⁵ indicated that application of resin sealers immediately after tooth preparation was able to seal the exposed dentin via resin infiltration into the tubules.

Traditionally, zinc-oxide eugenol cement was the first choice for temporary protection of the prepared dentin. However, when the final cementation is planned with resin cements, the eugenol-containing provisional cements should preferably be excluded. Although some studies⁶⁻⁹ reported that eugenol-containing provisional cements did not affect the retention of the final bonded restorations, there were other studies¹⁰⁻¹² that reported the contrary. Similar to other phenolic compounds, eugenol is a radical scavenger that inhibits the polymerization of resin materials.¹³ Residual resin products may also impair hybridization and polymerization of the permanent resin adhesives due to the reduced wettability/reactivity with the dentin substrate.¹⁴ This may result in compromises in bond strength and marginal seal.

Dislodging of the provisional restoration and temporary cement may result in contamination of dentinal tubules with moisture, saliva and blood. As these risk factors may jeopardize bonding to dentin,¹⁵ a double-bonding “resin-coating technique” that consists of sealing dentin with an adhesive before the placement of a eugenol-free provisional cement has been recommended.¹⁶⁻¹⁹ It is not clear whether the subsequent adhesion of indirect restorations will be adversely affected by this double-bonding protocol. This study examined the microtensile bond strengths of a resin cement to dentin that was previously treated with either an etch-and-rinse or a self-etch adhesive in conjunction with a eugenol-free provisional cement. The null hypothesis tested was that the use of adhesives as resin sealers and the eugenol-free provisional cement have no adverse effect on the microtensile bond strengths of indirect composites restorations coupled with an adhesive and a resin cement to dentin.
MATERIALS AND METHODS

Sixteen non-carious human third molars that were stored in a 0.5% chloramine T solution at 4°C were used within one month following extraction. The occlusal enamel was removed using a slow-speed saw with a diamond-impregnated disk (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. A 180-grit silicon carbide paper was used under running water to create a clinically relevant smear layer on the dentin surface. The teeth were randomly divided into four groups (N=4).

Experimental Design

Two different dentin bonding systems were used in the context of resin sealers in the first and the second groups. The eugenol-free temporary cement (Tempofix, Ghimas, Bologna, Italy) was used for the first, second and third groups. The materials tested in this study and their components are summarized in Table I. The operation procedures for the four experimental and control groups are summarized in Table II.

Table I. Compositions of the materials employed in this study.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Components</th>
<th>Batch #</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>AdheSE Primer</td>
<td>Primer: DMA, phosphonic acid acrylate, initiators, stabilizers in a aqueous solution.</td>
<td>F25882</td>
<td>Ivoclar-Vivadent, Schaan, Liechtenstein</td>
</tr>
<tr>
<td>Bond</td>
<td>Bond: HEMA, DMA, silicon dioxide, initiators, stabilizers.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Excite DSC</td>
<td>HEMA, DMA, phosphonic acid acrylate, highly dispersed silicon dioxide, initiators and stabilizers in an alcohol solution. The Excite Brush is coated with initiators.</td>
<td>G03703</td>
<td>Ivoclar-Vivadent</td>
</tr>
<tr>
<td>Resin cement</td>
<td>The monomer matrix is composed of Bis-GMA, urethane DMA, and triethylene glycol DMA. The inorganic fillers are barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide. Additional contents: catalyst, stabilizers, and pigments.</td>
<td>F55945</td>
<td>Ivoclar-Vivadent</td>
</tr>
</tbody>
</table>

Abbreviations: DMA: dimethacrylate; HEMA: 2-hydroxyethyl methacrylate
Table II. Procedures employed for the four experimental and control groups and their microtensile bond strengths.

<table>
<thead>
<tr>
<th>Group designations</th>
<th>Provisional restoration stage</th>
<th>Final cementation stage</th>
<th>Microtensile bond strength (MPa)*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Resin Sealer</td>
<td>Temporary Cement</td>
<td>Dentin Bonding</td>
</tr>
<tr>
<td>1</td>
<td>Excite DSC</td>
<td>Tempofix</td>
<td>Excite DSC</td>
</tr>
<tr>
<td>2</td>
<td>AdheSE</td>
<td>Tempofix</td>
<td>Excite DSC</td>
</tr>
<tr>
<td>3 (control)</td>
<td>Water</td>
<td>Tempofix</td>
<td>Excite DSC</td>
</tr>
<tr>
<td>4 (control)</td>
<td>Water</td>
<td>NIL</td>
<td>Excite DSC</td>
</tr>
</tbody>
</table>

* Values are means ± standard deviations in MPa. Groups with the same superscripts are not statistically significant (P>0.05)

In Group 1, Excite DSC was used according to the manufacturer’s instructions. The conditioning gel (37% phosphoric acid) was applied for 20 s and the substrate was rinsed with water for 15-20 s and then gently dried for 1-2 s. A microbrush, impregnated with the self-curing activator, was used for the application of the light-curing bonding system. One layer of this mixed ‘dual-cured’ solution was applied and finally the alcoholic solvent was gently removed with air.

In Group 2, AdheSE was used as recommended by the manufacturer. An adequate amount of AdheSE Primer was applied with a brush. The dentin surface was thoroughly coated with the primer that was agitated for 15 s. The excess AdheSE Primer was dispersed with a strong stream of air until the mobility of the liquid film disappeared. Then, the AdheSE Bond was applied, dispersed with a very weak stream of air and light-cured for 10 s.

In control Groups 3 and 4, the dentin surfaces were treated only with water, without the use of a dentin adhesive. A layer of the eugenol-free provisional cement was applied on the surfaces of the first three groups of specimens. After setting of the temporary cement, the samples were stored in distilled water for one week at 37°C. In the control Group 4, no provisional cement was applied in order to evaluate the effect of this cement on the final bond strength.

After mechanical removal of the provisional cement, the surface of each specimen in Groups 1-3 was re-etched with phosphoric acid for 15 s. A layer of Excite DSC was applied in the manner described previously. A resin cement (Variolink II, Ivoclar-Vivadent, Schaan, Liechtenstein) was then employed to cement a freshly prepared hybrid resin composite cylinder (Tetric-Ceram, Ivoclar-Vivadent, Schaan,
Liechtenstein) to the bonded dentin. The composite cylinders (10 mm in diameter by 6 mm in height) were prepared using a special metallic tool (Fig. 1), in order to limit and standardize the bond area. In control Group 4, adhesive cementation was made directly on the previously untreated dentin surface.

**Fig.1** The metallic tool used for the composite cylinders preparation.

**Bond Strength Evaluation**

After cementation, all the specimens were stored in water for 24 hours at 37°C. Each tooth was then sectioned vertically with the Isomet saw into a series of slabs. The slabs were then sectioned vertically into 0.9 x 0.9 mm sticks, based on the “non-trimming” version of the microtensile bond testing technique. Each stick was measured using a digital caliper to determine the cross-sectional area. The sticks were attached to a testing device with cyanoacrylate glue (Zap it, DVA, Corona, CA, USA). The device was attached to a universal testing machine (Triax digital 50, Controls, Milano, Italy) and loaded in tension at a crosshead speed of 0.5 mm/min until failure.

**Statistical Analysis**

All the sticks of each group were pooled together for the statistical analysis. The distribution of microtensile bond strength data was first checked for normality with the Kolmogorov-Smirnov test. The data were analyzed with one-way analysis of variance (ANOVA) and Tukey’s multiple comparison tests with $\alpha=0.05$.

**Scanning Electron Microscopy (SEM)**

After microtensile bond testing, several pairs of specimens from each group were randomly selected for SEM examination. Impressions (Provil Novo, Heraeus
Kulzer, Hanau, Germany) of these slices were taken and a set of epoxy resin replicas (Alpha Die, Schuetz Dental, Rosbach, Germany) was made for SEM evaluation. The resin replicas were mounted on metallic stubs, sputtered with gold/palladium and observed with a SEM (JSM – 6060LV, JEOL, Tokyo, Japan) operating at 10 kV.

RESULTS

Bond strength results of the four groups are shown in Table II. No statistically significant differences were observed among the four groups, indicating that neither the resin-coating materials nor the provisional eugenol-free cement had an adverse effect on the final bond strength (p>0.05).

SEM images of the specimens pre-treated with Excite DSC (Fig. 2; A-B) and AdheSE (Fig. 3) as resin sealers demonstrated resin tags formation and mixed failure respectively. SEM analysis of the specimens from Control group 3 revealed the presence of provisional cement remnants that were not completely removed after phosphoric acid etching and rinsing prior to the application of the Excite DSC adhesive (Fig. 4). Images from Control group 4 were similar to Groups 1 and 2 (Fig. 5) in that mixed failures were predominantly observed.
Fig. 2 SEM images (3500x) of a specimen from Group I with the use of Excite DSC as resin sealer. After removal of the provisional cement, Excite DSC and Variolink II were employed for the final cementation. A. The composite side of a fractured stick showing that resin tags were pulled out of the dentinal tubule, along with fracture within the hybrid layer (H). The hybrid layer that was formed around the periphery of the dentinal tubules remained attached to the resin tags (open arrowheads). B. The corresponding dentin side of the fractured stick, showing the patent dentinal tubules that remained after the resin tags were pulled out.

Fig. 3 SEM image (170x) of a specimen from Group 2 with the use of AdheSE as desensitizer). After removal of the provisional cement, Excite DSC and Variolink II were employed for the final cementation. A mixed failure mode was observed the composite side of the fractured stick. C: fractured resin cement; A: fractured adhesive.
Fig. 4  SEM image (3500x) of a specimen from Control group 3. As the specimen was not pre-coated with any adhesive, remnants of the provisional cement (open arrowheads) could be identified from the dentin surface even after subsequent phosphoric acid and rinsing.

Fig. 5  SEM image (85x) of a specimen from Control group 4 with no adhesive applied before provisional cementation, and the use of Excite DSC/Variolink II for the final cementation. A mixed failure mode, involving fractured resin cement (C) and fractured adhesive (A) could be observed from the composite side of the fractured stick.
DISCUSSION

In line with previously published studies\textsuperscript{18,21,22} on the evaluation of microtensile bond strengths to dentin, the microtensile bond assessment was also employed for this study. Our results showed that the application of the resin-coating material and the use of provisional cement by itself did not decrease the bond strength values significantly. Thus, we have to accept the null hypothesis that the use of adhesives as resin sealers and the eugenol-free provisional cement have no adverse effect on the microtensile bond strengths of indirect composites restorations coupled with an adhesive and a resin cement to dentin.

From the SEM analysis, it was evident that those specimens that were not pre-coated with any adhesive before the provisional cement application showed remnants of provisional cement, even after phosphoric acid etching (Fig. 4). A possible explanation is that as the dentin surface was not covered with any adhesive, the simple mechanical removal of the provisional cement was not effective enough to create a perfectly clean dentin surface. On the contrary, when the freshly exposed dentin surface was first sealed with a dentin adhesive prior to the placement of the provisional cement, we speculate that the comparatively smoother surface rendered by the underlying adhesive-layer permitted complete removal of the provisional cement, as the latter was not bonded to the adhesive. In light of these findings, when dentin pre-coating is not performed, a meticulous cleaning of the dentin substrate should be performed to prevent a possible reduction in the adhesion of the final restorations.

In this study, a total-etch system (Excite DSC) and a self-etch system (AdheSE) were selected as resin-coating materials. Dagostin et al.\textsuperscript{17} concluded that total-etch adhesive systems first employed as dentin sealers and consecutively as bonding produce satisfactory final bond strength values. Thus, a similar conclusion may be extrapolated from the microtensile results of the present study with the double application of Excite DSC. Although self-etching primers indeed offer some advantages over total-etch adhesives such as simplification of the chairside procedures, faster manipulation and reduced technique sensitivity, some investigators pointed out that these materials are only able to modify or partially remove the smear layer, as compared with total-etch adhesives and thus might be less effective as bonding.\textsuperscript{23,24} The bond strength of the self-etch adhesive tested in this study was higher when compared with recent studies.
results.\textsuperscript{23-26} This may be due to the additional application of the total etch system (Excite DSC), which is the bonding system recommended by the manufacturer of the Variolink resin cement for the final luting procedure.

A potential problem associated with the resin-coating technique is whether the subsequently applied adhesive and resin cement may couple effectively with the original adhesive surface that is devoid of the oxygen inhibition layer after the placement and the subsequent removal of the non-bonding provisional cement. The oxygen-inhibited layer primarily consists of unreacted monomers and oligomers\textsuperscript{25} that allows the materials from both sides to cross the interface and blend together to form an interdiffusion zone, wherein co-polymerization occurs to initiate a chemical bond. Reports on how the oxygen-inhibited layer affects bond strength have been equivocal. While some reports indicated a positive correlation between the oxygen-inhibited layer increased bond strength,\textsuperscript{28,30} others reported that the presence of oxygen-inhibited layer has no significant influence on bond strength.\textsuperscript{30,31} In addition, others concluded that the presence of an oxygen-inhibited layer was in fact detrimental to bonding additional layers of composite.\textsuperscript{32,33}

Indeed, SEM micrographs of fractured specimens from Groups 1 and 2 revealed that mixed failure along the interfaces was the predominant mode of failure and that pure adhesive failure did not occur (e.g. Fig. 3). These observations provided strong evidence that coupling of the new adhesive to the existing adhesive-covered dentin was not affected by the depletion of the original oxygen inhibition layer. Our results, therefore, were consistent with the recent findings that negate the concept that the oxygen inhibition layer is required for effective coupling of resin composites.\textsuperscript{34} Söderholm and Roberts\textsuperscript{35} showed that during repair of composite materials, the amount of free radicals available in the original resin was reduced to approximately 30%. While this is certainly applies to the coupling of new resin materials to old, existing resin composites, the differences may be accounted for by the half-lives of free-radical decay. It is known that active free radical may remain in polymerized resins for up to 4-5 weeks that slowly decayed with time (Watts DC. personal communication 2004). Clinically, as most permanent restorations are bonded within 1-2 weeks after the resin-coating technique, it is reasonable to speculate that sufficient free-radicals remain within the adhesive-desensitized dentin to ensure optimal coupling between the original
adhesive and the new adhesive. Further studies should be done using electron spin resonance spectroscopy, to quantify the rate of decay of free radicals in polymerized resins that are devoid of the oxygen inhibition layers. This will provide important information on how long clinicians can leave provisional restorations on adhesive-desensitized dentin without adversely affect the coupling strengths of the final restorations.

Although it has been demonstrated that clinically, that hybridizing dentin bondings are able to reduce post-operative sensitivity, recent in vitro and in vivo studies have shown that simplified total-etch and self-etch adhesives act as semi-permeable membranes after polymerization. This means that they permit the continuous transudation of dentinal fluid and do not provide an hermetic seal in vital deep dentin. Moreover, two of these studies further showed that continuous fluid transudation through the adhesive-coated dentin persisted even after a period of temporization with non-bonding provisional cements. Although the relatively slow rate of diffusion of dentinal fluid is unlikely to result in post-operative cold sensitivity, it may interfere with the optimal polymerization of dual-cured or auto-cured composites or resin cements in both direct and indirect restoration. The permeability of contemporary simplified adhesives may influence the clinician’s selection of conventional self-etch and total-etch adhesives with comparatively more hydrophobic, non-solvented coupling resins for the resin-coating technique.
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temporary cement on the bond strength of two resin composite core materials to


Section 4. The effect of additional monomers on a resin cement bond strength.

4.1 The effect of adding an antibacterial monomer on the bond quality of a luting cementation system.
Nicoletta Chieffi, Stefano Chersoni, Federica Papacchini, Michele Vano, Cecilia Goracci, Carel L. Davidson, Franklin R. Tay, Marco Ferrari

ABSTRACT

Purpose: To examine the bond quality of a luting cement after incorporation of an antibacterial molecule into the primer. Methods: Eight non-caries human third molars were randomly divided in two equal groups. Cylindrical composite blocks were luted with Panavia F (Group I) and Panavia Prototype (Group II) and the seating pressure was applied during the entire three-minute polymerization period of the resin cement to minimize fluid interferences from the underlying dentin. After storing in distilled water for 24 hours, 0.9 x 0.9 mm sticks were produced from these luted specimens for microtensile bond testing and SEM examination. Results: No statistically difference in the bond strength between the control Group and the experimental Group was detected (p>0.05). From SEM analysis, between the two groups, no significant differences were found.

Conclusions and clinical significance: Addition of an antibacterial monomer (MDPB) to existing Panavia F luting cementation system does not negatively affect final bond quality and might extend the application of the cement.
INTRODUCTION

Exposure of dentinal tubules during tooth preparation necessitates that they are adequately sealed as soon as possible to prevent microleakage, bacterial invasion,1-3 and minimize postoperative sensitivity.4-7 Treatments available to protect the dentin-pulp system include agents that seal tubules with resins,5,6,8,9 crystal sediments,10-12 or with both materials.13 Over the past years, different results were reported on the contribution of adhesive systems regarding dentin sealing. Some studies14-15 claimed that dentin bonding alone was able to guarantee a tight seal of the tubules. Others16,17 concluded that dentin bonding did not completely seal dentinal tubules and as a result, they could not prevent bacterial penetration. More recently, in vitro18,19 and in vivo20,21 studies have shown that simplified etch-and-rinse and self-etch adhesives behave as permeable membranes after polymerization. These adhesives permit continuous transmission of the dentinal fluid and thus do not provide a hermetic seal when they are applied to vital deep dentin. Additional measures to protect vital teeth are sought in adding antibacterial agents to self-etch adhesives. It has been demonstrated that the recently introduced monomer 12-methacryloyloxydodecylpyridinium bromide (MDPB) provides extensive22,23 and prolonged24 antibacterial activity, before and after polymerization. The new commercially available, self-etch primer Clearfil Protect Bond (Kuraray Medical Inc., Tokyo, Japan) contains this antibacterial resin monomer.25,26 Consequently a prototype of Panavia resin cement (Kuraray Medical Inc., Tokyo, Japan) in which MDPB is included has recently been formulated.

The objective of this study was to investigate whether the quality of bonding of the new antibacterial monomer-containing Panavia prototype is not negatively affected by admixing the agent to the primer of the system. Panavia F was used as control. Micro-tensile bond strength and SEM examination of the interface were used as parameters for bonding quality.

The null hypotheses tested was that the incorporation of an antibacterial resin monomer in a dual-cured resin cement does not alter its quality of bonding to dentin.

MATERIALS AND METHODS

Eight non-carious human third molars that were stored in a 0.5% chloramine T solution at 4°C were used within one month following extraction. All the teeth
underwent bonding in their normal hydrated status, as they were retrieved from the storage medium. The composition of the materials is shown in Table I. The microtensile bond strengths values for the two groups are showed in Table II.

Table I. Batch number (#), composition and manufacturer of the materials employed in this study.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Components</th>
<th>Manufacturer</th>
</tr>
</thead>
</table>
| Panavia F (# 41170)     | *Primer a*: HEMA, 10-MDP, 5-NMSA, water, accelerator.  
                        | *Primer b*: 5-NMSA, accelerator, water, sodium benzene sulfinate. | Kuraray Medical Inc., Tokyo, Japan |
|                          | *Paste A*: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide. |                             |
|                          | *Paste B*: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass. |                             |
| Panavia Prototype (# 41127) | *Primer a*: HEMA, 10-MDP, 12-MDPB, dimethacrylate, water.  
                        | *Primer b*: 5-NMSA, accelerator, water, sodium benzene sulfinate. | Kuraray Medical Inc., Tokyo, Japan |
|                          | *Paste A*: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide. |                             |
|                          | *Paste B*: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass. |                             |

Abbreviations- HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA: N-methacryloyloxy-5-aminosalicylic acid; 12-MDPB: 12-Methacryloyloxy dodecylpyridinium bromide; Bis-GMA: Bis-Phenol A diglycidylmethacrylate
Table II The microtensile bond strengths values for the two investigated groups.

<table>
<thead>
<tr>
<th>GROUP</th>
<th>Microtensile bond strength (MPa)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>I Controll Panavia F</td>
<td>20.71 ± 7.31</td>
</tr>
<tr>
<td>II Experimental Panavia prototype</td>
<td>19.13 ± 6.68</td>
</tr>
</tbody>
</table>

Values are means ± standard deviations in MPa. Groups with the same superscripts differ not statistically significant (P>0.05)

a. MICROTENSILE EXAMINATION

Specimen Preparation

The occlusal enamel was removed using a slow-speed saw with a diamond-impregnated disk (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. All teeth were cut at the same level, in order to obtain tooth samples with a standardized height (5mm). A 180-grit silicon carbide paper was used under running water to create a clinically relevant smear layer on the dentin surface.

Composite cylinders (Tetric-Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein) of 10 mm in diameter and 6 mm in height were prepared using a split aluminum mold, in order to limit and standardize the bonding area. Prior to cementing procedures, the bonding surface of each resin cylinder was sandblasted, etched with phosphoric acid, rinsed with water and dried.

Cementing procedures

The specimens were randomly divided in two groups (N=4). In the Control Group, the resin cement Panavia F (Kuraray Medical Inc., Tokyo, Japan) was used according to manufacturer’s instructions. ED Primer (A+B) was mixed and applied with a brush on the dentin surface and left in place for 30 s. After drying the etched surface with gentle air flow, Panavia F resin cement (A+B) was mixed and applied on each of the freshly prepared composite cylinders.
In the Experimental Group, Panavia prototype was used according to the manufactures instructions and the bonding procedures were the same as described for the Control.

In the two groups, the composite block was cemented to the dentin surface using a constant seating pressure of 1,25 MPa that was maintained during the entire three minutes lasting period required for complete polymerization of the resin cement. While the specimen was still under pressure, a layer of Oxyguard was applied after removal of the excess cement with a probe and left in place for at least 3 min to ensure optimal polymerization of the resin cement along the exposed margins.

In order to standardize the applied pressure, a metallic tool that delivered 10 Kg was used. This resulted in a seating force of 98,1 N. The pressure [N/m²] was calculated, dividing this force [N] by the surface area [m²] of the metal weight. The value obtained was finally converted into MPa.

**Bond Strength Evaluation**

After cementation, all the specimens were stored in water for 24 hours at 37°C. Each tooth was then sectioned vertically with the Isomet saw into a series of slabs. The slabs were then sectioned vertically into 0.9 x 0.9 mm sticks, based on the “non-trimming” version of the microtensile bond testing technique.²⁷ For each group 120 sample sticks were tested. Each stick was measured using a digital caliper to determine the cross-sectional area. The sticks were attached to a testing device with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA). The device was attached to a universal testing machine (Triax digital 50, Controls, Milano, Italy) and loaded in tension at a crosshead speed of 0.5 mm/min until failure.

**Statistical Analysis**

After checking for normal distribution with the Kolmogorov-Smirnov test, the differences in bond strength values between the two groups were tested for statistical significance using the Student t-test. The level of significance was set at α=0.05.
b. SCANNING ELECTRON MICROSCOPY (SEM)

Specimen Preparation

After microtensile bond testing, several pairs (fractured composite and dentin sides) of specimens from the two groups were randomly selected for SEM examination. Each specimen was mounted on metallic stubs, sputtered with gold/palladium and observed with a SEM (JSM – 6060LV, JEOL, Tokyo, Japan) operating at 10 or 15 kV. Images of the two complementary debonded interfaces for each fractured specimen were taken at different standardized magnifications at 100-6,000X.

RESULTS

No statistically difference in the bond strength between the Control group and the Experimental group was detected (p>0.05) [Table II], indicating that the incorporation of the antibacterial resin monomer in the dual-cured resin cement does not alter its bond strength to dentin.

SEM images of the specimens treated with Panavia F (Fig. 1) showed interfaces practically without structural defects. Images from taken from the experimental group (Fig. 2) were similar to the control group, also without structural irregularities.

![Fig. 1 SEM image (1000x) at the composite site of a fractured sample from Group I. The micrograph shows the absence of structural defects.](image-url)
DISCUSSION

Since the microtensile values of the experimental group were comparable to the those of the control group, the null hypothesis tested in this study can be accepted. This means that the monomer (MDPB) incorporated into a dentin bonding system could potentially provide bactericidal activity without causing an adverse effect on bond strength. This antibacterial action is more significant in the case of adhesive systems with a self-etching priming solution since the demineralized smear layer is not removed but only incorporated into the hybrid layer by absence of a rinsing procedure. This implies that residual bacteria may remain at the interface between the tooth and the luting material, constituting a possible cause of secondary caries and damage to the pulp. MDPB is a compound of the antibacterial agent quaternary ammonium and a methacryloyl group, and the antibacterial agent is covalently bound to the polymer matrix by copolymerization of MDPB with other monomers when the material is cured.24 Problems with antibacterial additions may arise when chemical incompatibility with the adhesive monomers occurs as the technical proprieties of the bonding agent
can be impaired by the addition of the antibacterial substances, or the material may become toxic (Schmalz G, personal communication). This latter event can be avoided by applying the material at a safe distance from the pulp, that is when the dentin barrier is equal/thicker than 500 µm no toxicity is detectable with glutaraldehyde-free dentin-bonding agents (Schmalz G, personal communication). Yet, admixing a monomer containing the antibacterial agent remains a potential factor of chemical incompatibility. As such, also simplified self-etch systems exhibit chemical incompatibility with auto or dual curing resin cements. The reduced procedure of the simplified one-step or two-step self-etch adhesive systems is associated with an increase of hydrophilic components in the material in order to facilitate bonding to intrinsically wet dentine surface. These adhesion-promoting monomers have the general structural formula of a hydrophilic group at one end and a monomethacryloyl group at the other, which are connected with a linking group. As a result, the material itself becomes more hydrophilic and capable of attracting dentinal fluid which affects the final bond strength between the adhesive layer and the greater hydrophobic components of the resin cement. When MDPB is incorporated in adhesion-promoting monomers, covalent bonding of both the bactericide and the hydrophilic group of adhesion promoting monomers to polymer matrix occurs after curing. The antibacterial agent dodecylpyridinium bromide remains pendant from the polymer network and, while the immobilized bactericide can act on bacteria that contact the surface, it is not clear how the immobilized agent acts on the affinity with water.

An inverse relationship between water absorption and bond strength of the adhesive resin systems has been demonstrated in literature. Based on our microtensile and SEM results it can be assumed that the antibacterial monomer did not significantly change the water absorption and thus does not interfere with the final bond strength of the resin cement.

Moreover, the presence of an oxygen-inhibited layer in acidic self-etch adhesives was found to be a possible cause of the adverse reaction with self-cured resin cements. Hydrophilic groups are thought to show affinity with water molecules by hydrogen bonding to oxygen. However, alternative reducing agents (i.e. benzoyl peroxide-tertiary aromatic amine and sulphinic acid salts) have been used with dentin adhesive containing acidic resin monomers in order to improve their bonding with
chemical-cured composites. According to these authors the improved bond strength derived from a better affinity between the salts of sulphinic acid and the acid-etched dentin, as well as from a more complete polymerization of the acidic resin monomers. Similarly, the inclusion of two different types of sulphinate ternary catalyst agents in Panavia F system, as well as in the prototype version tested in the present investigation, may have contributed to the results obtained.

Some of the procedures used in this study, such as the application of a sustained seating pressure during the composite block cementation, or the use of only the chemical cured mode of the polymerization in the dual-cured resin cement, differ from the daily practice. Although clinicians during cementation usually apply a seating force that does not envelop the entire polymerization period of the resin cement, in particular, during the auto cured resin cement with a longer setting time, it has to be emphasized that fluid interferences can occur from the underlying dentin. In a recent investigation, the maintained seating pressure protocol was demonstrated to reduce the fluid movement and by consequence an increase in the bond strength, which was related to a lower hydrophilicity of the material used. Even if, as in the present investigation no pulpal pressure was simulated, it may be expected that water interference at the adhesive interface is present since the specimens were bonded in their normal hydrated status, as they were retrieved from the storage medium.

As in the present investigation, the bond strength and the SEM micrographs of Panavia F and Panavia prototype were almost identical, it can be hypothesized that the sustained seating pressure had the same effect on the two materials, notwithstanding their different composition, which evidently has the same effect on the water absorption.

From the results of this in vitro study, it can be concluded that for protection of the dentin and the pulp, resin cements containing the antibacterial molecule (MDPB) can be used without affecting the final bond strength.
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Section 5. Summary, general discussion, conclusions and future research.

5.1 Considering the limits of the currently available adhesive systems and their clinical implications.

Over the years, various clinical applications have been proposed for adhesive systems. The thesis concentrates on when these adhesive materials are used during indirect restorations as materials manufactured exclusively for use with resin cement, as adhesive dentinal “sealers” before temporary cementation and as material with adjunctive antibacterial proprieties.

The research reported in Section 2 confirms that, among factors potentially affecting the coupling of resin cement to dentin, fluid interference from the dentin substrate through the simplified self-etch adhesive system, still represents the major problem in terms of final dual-cured resin cement bond strength.

Simplified bonding strategies proposed by dental manufactures have been well accepted by dentists because they are not time-consuming. Moreover, the different bonding mechanism of self-etch systems has circumvented some of the clinical problems associated with the traditional total-etch bonding procedures. Self-etch systems do not remove the smear plugs, as a result the hydraulic conductance through the dentinal tubules is reduced and less postoperative sensitivity compared with that of total-etch systems occurs.\(^1,2\) In addition, the bonding mechanism of these materials goes back to a dry bonding to smear layers. With self-etch adhesives the technique sensitivity of wet bonding associated with total-etch adhesives is reduced because more hydrophilic and acidic monomers are used, which enable these materials to etch and prime through smear layers and into underlying intact dentin.\(^3,4\)

On the other hand, hydrophilic resin systems attract water because of their higher concentrations of hydrophilic and ionic resin monomers.\(^5\) This is the case above all with one-step self-etch systems. As a result, an impervious seal of the exposed dentin could not be achieved even when bonding is performed in the laboratory on the flat dentin surface and without the additional influence of pulpal pressure, as morphologically evidenced by SEM analysis performed in the studies showed in Section 2, as well as by previous literature data.\(^6,7\) The fluid moves through the self-etch primed dentin and when it comes into contact with the slow-setting resin cement, adverse interactions
occur. These adverse interactions may be responsible for the unexpected premature decoupling of self-cured, core buildup composites that are bonded with single-bottle adhesive systems in vivo, and for the low bond strength value results in vitro, as seen in the present study, as well as in previous research. These issues led to important clinical implications, also found by other researchers. It is suggested to avoid the treatment of dentin surfaces with these acidic adhesives before luting indirect restorations with dual-cure resin composite cements.

On the contrary, the maintained seating pressure during the entire time of dual-cured resin cement setting, improves the bond strength by reducing incompatibility factors (Section 2.1). In light of the encouraging results obtained with the application of a sustained seating pressure, it is important to confirm how effective this is against fluid interference, when the pressure pulp is simulated. In addition, in order to exclude other chemical causes of the incompatibility, the same cementation procedure should be repeated on dehydrated dentin substrate. These further studies could be clinically relevant as a new alternative to circumvent simplified adhesive permeability when indirect restorations require dual-cure resin cements.

In order to overcome, at least in part, the problem of the permeability of simplified one-step systems (in which the resin cement is directly coupled to primed dentin without an additional resin coating) previous studies have proposed the dentin coverage with an additional layer of a greater hydrophobic bonding resin, before the resin cement application. In order to evaluate the significance of the added hydrophobic layer, the second study presented in Section 2 was carried out. This study shows that although fluid flow across bonded dentin cannot be totally eliminated, bond strength of resin cement is substantially improved by the additional application of the greater hydrophobic adhesive layer. The maximum bond strength is obtained with simultaneously applying the maintained seating pressure, which avoids fluid interferences, permitting a superior bond between the adhesive layer and the resin cement, resulting in adhesive-resin cement interfaces completely intact at SEM evaluation.

A potential clinical problem with the use of such a resin coating is the adverse effect on the fit of the indirect restorations. However this can be overcome if the impression is taken after coating the teeth with the additional layer.
When dealing with microleakage prevention after tooth preparation, two papers were included in the thesis (Section 3.1 and Section 3.2). The study presented in Section 3.2 concluded that the use of dentin adhesives as resin sealers before provisional cementation with a non-eugenol provisional cement does not adversely affect the retentive strength of indirect restorations bonded subsequently with an adhesive and a resin cement.

The traditional opinion that a perfect seal could be achieved if dentin tubules and spaces within the demineralized collagen matrix were completely infiltrated by adhesive resin, was based on the assumption that polymerized resins used for bonding are nonporous and impermeable to fluid movement. To date, it is well known that potential leakage pathways within the hybrid layer and the adhesive layer may both contribute to slow fluid flow across a dentin-adhesive joint. However, while water and small ions such as fluoride can certainly move across adhesive-sealed dentin, one wonders if large molecules, such as glucose, bacterial products or hydrolytic enzymes, can permeate from the outside, through the adhesive and dentin, into the pulp. Moreover, the relatively slow diffusion of dentin fluid across the adhesive is unlikely to result in severe postoperative sensitivity. In spite of these logical hypothesis, to date, it appears hard to clinically recommend these materials as dentinal “sealers”. Thus, the conclusions found in Section 3.2 are influenced by the difficulty to achieve an impervious seal of the exposed dentin with adhesive systems.

To circumvent the partial protection offered by adhesives systems, it appears useful to employ therapeutic solutions in which adhesive systems used as resin sealers are helped with the simultaneous use of treatments with crystals, or with agents providing antibacterial activity. Taking this into account, the study in Section 4.1 was carried out and the results obtained suggest that the dual cured resin cement tested, containing the antibacterial molecule (MDPB) in the primer, does not affect the final bond strength, and thus can be safety used in order to protect the exposed dentin and the pulp. Moreover, recently a new technique that uses crystals (oxalate) after acid-etching of dentin, prior to adhesive application, has been developed. Reduction in dentin permeability is thus achieved via subsurface tubular occlusion that does not interfere with subsequent resin infiltration.
In this way of reconsidering the adhesive sealing, the use of adhesive systems to protect the exposed dentin surface still appears to be effective. When considering the potential factors affecting the final bond between adhesive and resin cement, the results of the study presented in Section 3.2 are relevant when referred to the influence that the oxygen inhibition layer may have on the bonding mechanism. Our observations provide strong evidence that coupling of the new adhesive to the existing adhesive-covered dentin is not affected by the depletion of the original oxygen inhibition layer. Further research could be done in this direction, in order to know how long dentists can leave provisional restorations on adhesive-covered dentin without adverse affects on the coupling strength of the final restorations.

The following conclusions can be made:

1. To improve coupling of the resin cement to dentin, both the additional self-etch adhesive containing the light-cured hydrophobic bonding layer and the sustained seating pressure during cementation are effective. The latter reduces the incompatibility between simplified-step adhesives and dual-cured resin cements, since it drastically reduces the adhesive permeability associated with simplified self-etch adhesives.

2. The use of dentin adhesives as resin sealers before provisional cementation with a non-eugenol provisional cement does not adversely affect the retentive strength of indirect restorations bonded subsequently with an adhesive and a resin cement.

3. In order to protect the dentin and the pulp, resin cements containing the antibacterial molecule (MDPB) can be used without affecting the final bond strength.

According to clinical needs, adhesive systems should be able to bond well to dentin substrate which is hydrophilic in nature and to resin composites or cements that are hydrophobic in nature, seal hermetically the dentinal tubules in order to protect exposed dentin against bacteria, prevent post-operative hypersensitivity, provide high bond strength immediately but also in time, finally they should be easy to handle and time saving. Researchers and dentists require that these materials solve a lot of clinical problems. On the base of these stimuli, over the last years, manufactures have tried to improve the features of these materials, but in spite of the collaboration between research, clinical experience, and manufactures, the final ambitious objective of a versatile bond still remains.
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