

THE EFFECT OF ADDING AN ANTIBACTERIAL MONOMER ON THE BOND QUALITY OF A LUTING CEMENTATION SYSTEM

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Abstract

Purpose: To examine the bond quality of a luting cement after the incorporation of an antibacterial molecule into the primer.

Methods: Eight non-carious 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). Seating pressure was applied during the entire three-minute polymerization period of the resin cement to minimize fluid interferences from the underlying dentin. After being stored 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 statistical 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: The addition of an antibacterial monomer (MDPB) to the existing Panavia F luting cementation system does not negatively affect final bond quality and might extend the application of the cement.

Keywords: resin cement; antibacterial activity; bond strength

Introduction

Exposure of dentinal tubules during tooth preparation requires 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 Previously, different results were reported on the contribution of adhesive systems regarding dentin sealing. Some studies claimed that dentin bonding alone was able to guarantee a tight seal of the tubules¹⁴⁻¹⁵. Others concluded that dentin bonding did not completely seal dentinal tubules and, as a result, could not prevent bacterial penetration^{16,17}. Recent *in vitro*^{18,19} and *in vivo*^{20,21} studies have shown that simplified etch-

and-rinse and self-etch adhesives behave as permeable membranes after polymerization. These adhesives allow continuous transmission of the dentinal fluid and thus do not provide a hermetic seal when applied to vital deep dentin. Additional measures to protect vital teeth can be taken by adding antibacterial agents to self-etch adhesives. It has been demonstrated that the recently introduced monomer 12-methacryloyloxydodecylpyridinium bromide (MDPB) provides extensive^{22,23} 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

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not alter its quality of bonding to dentin.

Materials and methods

Eight non-carious human third molars 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 strength values for the two groups are showed in Table II.

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 the 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), 10 mm in diameter and 6 mm in height, were prepared using a split aluminum mould 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 into 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 30s. 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, the Panavia prototype was used according to the manufactures instructions and the bonding procedures were the same as described for the Control Group. In both groups, the composite block was cemented to the

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

Materials	Components	Manufacturer
Panavia F (# 41170)	<i>Primer a:</i> HEMA, 10-MDP, 5-NMSA, water, accelerator. <i>Primer b:</i> 5-NMSA, accelerator, water, sodium benzene sulfinate. <i>Paste A:</i> 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide. <i>Paste B:</i> hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass.	Kuraray Medical Inc., Tokyo, Japan
Panavia Prototype (# 41127)	<i>Primer a:</i> HEMA, 10-MDP, 12-MDPB, dimethacrylate, water. <i>Primer b:</i> 5-NMSA, accelerator, water, sodium benzene sulfinate. <i>Paste A:</i> 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide. <i>Paste B:</i> hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass.	Kuraray Medical Inc., Tokyo, Japan
Abbreviations: HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA: N-methacryloyl-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.

GROUP	Components	Microtensile bond strength (MPa)*
I Control	Panavia F	20,71 (± 7,31) a
II Experimental	Panavia prototype	19,13 (± 6,68) a

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

dentin surface using a constant seating pressure of 1,25 MPa that was maintained during the entire three-minute lasting period required for complete polymerization of the resin cement. While the specimen was still under pressure, a layer of Oxyguard was applied after the 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 delivering 10 Kg was used. This resulted in a seating force of 98,1 N. The pressure [N/m^2] was calculated, dividing this force [N] by the surface area [m^2] 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 into a series of slabs with the Isomet saw. 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 and then 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)

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 statistical 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 composite side from the specimens treated with Panavia F (Fig. 1) showed interfaces practically without structural defects. Composite images taken from the specimens treated with Panavia Prototype (Fig. 2) were similar to that of the Control Group, also without structural irregularities.

In the majority of the specimens examined from both the two Groups, the failure occurred between the dentin and the resin cement (Fig.3).

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

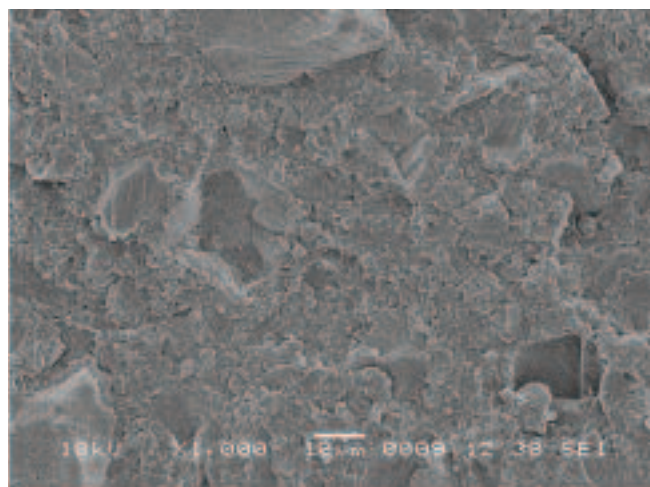


Fig. 1 SEM image at the composite site of a fractured sample from Group I. The micrograph shows a representative medium magnification view (1000x) showing the absence of structural defects.

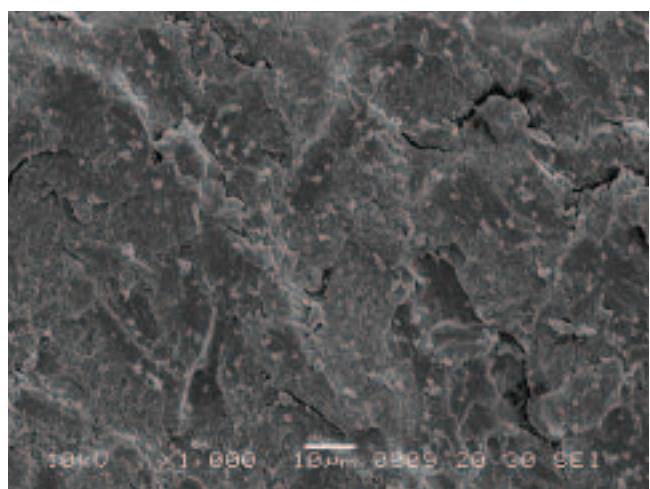


Fig. 2 SEM image (1000x) at the composite site of a fractured sample from Group II. Note the similarity with Fig.1, in its absence of structural defects.

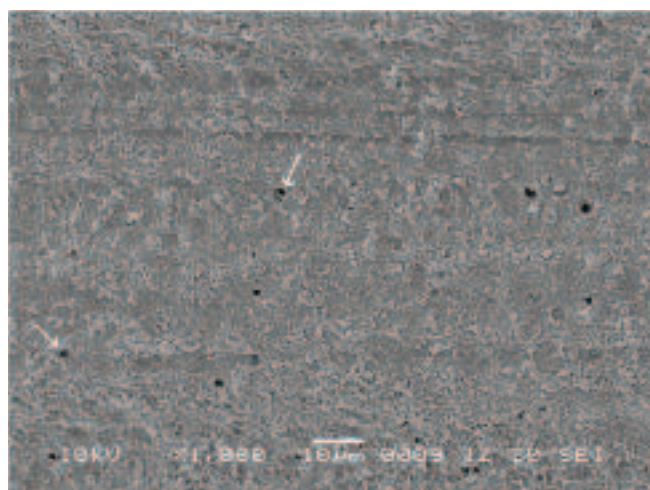


Fig. 3 SEM image at the dentin site of a fractured sample from Group II. The micrograph shows a representative medium magnification view (1000x) in which dentinal tubules can be identified (arrows).

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 incorporated into the hybrid layer by the 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). The latter can be avoided by applying the material at a safe distance from the pulp, for 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.^{28,29,30} The reduced procedure of the simplified 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.^{31,32} 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.³³ As a result, the material itself becomes more hydrophilic and capable of attracting dentinal fluid^{34,35} which affects the final bond strength between the adhesive layer and the greater hydrophobic components of the resin cement.^{36,37} 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 have contact with the surface, it is not clear how the immobilized agent acts on the affinity with water.²³

An inverse relationship between water absorption and bond strength of the adhesive resin systems has been demonstrated in literature.^{38,39} 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.^{28,30} Hydrophilic groups are thought to show affinity with water molecules by hydrogen bonding to oxygen.²³ However, alternative reducing agents (i.e. benzoyl peroxide-tertiary aromatic amine and sulphonic acid salts) have been used with dentin adhesive containing acidic resin monomers in order to improve their bonding with chemical-cured composites.²⁸ According to the authors, improved bond strength is derived from a better affinity between the salts of sulphonic acid and the acid-etched dentin, as well as from a more complete polymerization of the acidic resin monomers.²⁸ Similarly, the inclusion of two different types of sulphinate ternary catalyst agents⁴⁰ in the 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 daily practice. Although clinicians usually apply a seating force during cementation that does not envelop the entire polymerization period of the resin cement - particularly 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.³³ In a recent investigation,³⁴ 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^{19,42,43}.

As in the present investigation, where 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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