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Effect of Eugenol Exposure Time and Post-removal Delay on the Bond Strength of a Self-etching Adhesive to Dentin

JPL Silva • DM Queiroz • LH Azevedo
LC Leal • JL Rodrigues • AF Lima
GM Marchi • M Brito-Júnior • AL Faria-e-Silva

Clinical Relevance

The presence of a provisional restoration containing eugenol for one week or longer does not interfere with the bond strength of self-etching adhesives to dentin. If a eugenol-based provisional restoration remained in place for only 24 hours, a delay of one week re-establishes proper bond strength.

João Paulo Lopes Silva, undergraduate student, Department of Dentistry, State University of Montes Claros, Minas Gerais, Brazil

Déborah Mafra de Queiroz, undergraduate student, Department of Dentistry, State University of Montes Claros, Minas Gerais, Brazil

Luiz Henrique Azevedo, undergraduate student, Department of Dentistry, State University of Montes Claros, Minas Gerais, Brazil

Lidyane Caldeira Leal, DDS, graduate student, Department of Dentistry, State University of Montes Claros, Minas Gerais, Brazil

João Lima Rodrigues, DDS, professor, School of Dentistry, North of Minas United Schools, Montes Claros, MG, Brazil

Adriano Fonseca Lima, DDS, DMD, PhD candidate, Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

Gisele Maria Marchi, DDS, DMD, PhD, professor, Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

Manoel Brito-Júnior, DDS, DMD, professor, Department of Dentistry, State University of Montes Claros, Minas Gerais, Brazil

*André Luis Faria-e-Silva, DDS, DMD, PhD, professor, Department of Dentistry, Federal University of Sergipe, Aracaju, Sergipe, Brazil

*Reprint request: Departamento de Odontologia, Campus da Saúde "Prof. João Cardoso Nascimento Júnior", Rua Cláudio Batista s/n, Bairro Sanatório, Aracaju/ SE, Brazil 49060-100; e-mail: faria_silva@ufs.br

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ABSTRACT

Objectives: This study evaluated the effects of the exposure time of eugenol-based provisional restorative material and the time elapsed between the provisional material removal and the adhesive procedure on the bond strength of the composite to dentin. Materials and Methods: Human third molars were sectioned into two

halves that were enclosed in resin cylinders. The cavities were prepared over the buccal/lingual faces with diamond burs. Zinc oxide and eugenol (ZOE) provisional material was inserted into cavities and left for 24 hours, 7 days or 14 days. The cavities not restored with ZOE were used as controls. After ZOE removal or over fresh dentin (control), self-etching Adper SE Plus was applied immediately, after a 7- or 14-day delay. The cavity was restored with non-eugenol provisional material during this delay period. Cylinders of resin cement were built-up over the hybridized dentin. A shear load was applied to the cylinders at a crosshead speed of 0.5 mm/minute until failure. The data were statistically analyzed using two-way ANOVA and Tukey's tests (α=0.05). Results: Using IRM as a provisional restoration for 24 hours followed by its removal and immediate adhesive application resulted in the lowest values of shear bond strength. There was no significant difference between the other experimental conditions. Conclusions: The use of IRM for 24 hours adversely affected the shear bond strength of a self-etching adhesive to dentin. The recovery of the proper bond strength occurred one week after IRM removal.

INTRODUCTION

The fundamental principle of bonding to dental hard tissues is based on micromechanical interlocking of the adhesive resin with dentin/enamel surfaces.[1] In this approach, proper bonding depends on adhesive penetration between the spaces created by acidic etching and an adequate polymerization of the bonding resin.[2-3] Over the last decade, a number of investigations have reported that several factors might interfere with the bonding ability of adhesive systems to enamel or dentin, including the adhesion strategy,[4] conditioning time,[5] solvent removal method,[6-7] thickness of the adhesive layer,[8] substrate structure[9] and even the provisional restorative material previously used.[10-15]

Among the provisional restorative materials, zinc oxide eugenol (ZOE) is widely used as a temporary material during endodontic treatments and restorative dentistry due to its sedative effect on sensitive teeth, its low cost, ease of removal and proper seal against leakage.[16] However, eugenol (2-methoxi-4-allyphenol) is a radical scavenger and may inhibit the polymerization of resin materials,[17] including the adhesive system that contributes to a reduction in bond strength.[11,13,18-20] The hydroxyl group of the eugenol molecule tends to protonize the free radical formed during polymerization of the adhesive, thereby blocking its reactivity, reducing the degree of conversion and, consequently, bond strength.[21,22]

Contradictory findings exist regarding whether or not the prior use of eugenol-containing temporary restorations affect the bond strength of composites to dentin. Several studies have found that eugenol-containing cements did not reduce the bond strength to dentin.[10,12,15,23-24] However, other studies have reported contradictory results.[11,13,18-21] These differences in results can be explained by differences between the adhesive systems and methodologies used. Furthermore, the exposure time to eugenol-containing provisional material over dentin has varied from 24 hours to 10 days, depending on the study. This factor may interfere with the action of eugenol and also contributes to an explanation of the different findings described in the literature. In addition to the exposure time to eugenol-containing material, the delay period between removal of the provisional material and the adhesive application may also interfere with bond strength.[25]

Thus, the current study evaluated the effects of exposure time of different types of provisional restorative materials (containing or not containing eugenol) and the time elapsed between removal of the provisional material and adhesive procedure on the bond strength of the composite to dentin. The null hypotheses to be tested were that neither the exposure time to eugenol-containing material nor the post-removal delay interferes with bond strength.

METHODS AND MATERIALS

Sixteen non-carious human third molars stored in 0.05% thymol saline solution for up to six months were used. In order to obtain two halves, the teeth were sectioned along the mesio-distal axis, parallel to the long axis of the tooth, using a slow-speed diamond saw (#7020, KG Sorensen, Barueri, SP, Brazil) under water-cooling. Each half was embedded in acrylic resin to facilitate handling, keeping the buccal/lingual surfaces exposed. The cavity preparation was performed with a cylindrical diamond bur (#3097, KG Sorensen) operated with a high-speed handpiece using copious air-water spray. The bur was used parallel to the bucco-lingual surface until a flat surface on the dentin was obtained, with sufficient area to build-up two resin composite cylinders (1 mm in diameter).

The specimens were randomly allocated in accordance with exposure time to the provisional ZOE-based restorative material and the time that elapsed between their removal and the adhesive procedure. Thirty specimens were used as controls; they did not receive the ZOE-based restorative material. IRM (Dentsply, Petrópolis, RJ, Brazil) was used as the ZOE-based restorative material. This material was prepared according to the manufacturer's instructions, inserted into the cavity and left for 24 hours, 7 days or 14 days. The material was then mechanically removed with a scaler until the dentin surface was visually (macroscopically) free of the material. The cavity was

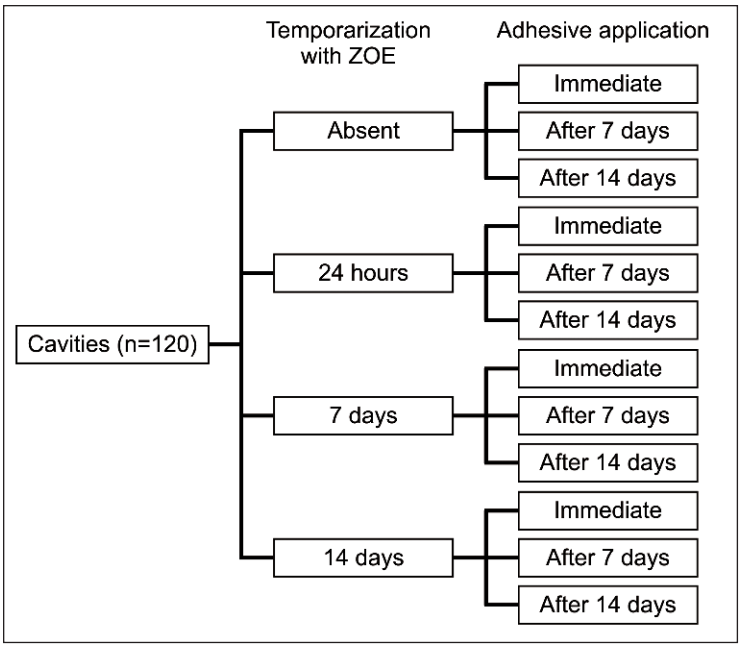

Figure 1. *Experimental design.*

further cleaned with pumice-water slurry in a slow-speed handpiece and rinsed with an air-water stream. After removal of the ZOE provisional material, the cavity immediately received the adhesive application, or a non-eugenol provisional restorative material was inserted during the delay period (7 or 14 days) before the use of the adhesive. The same procedure was performed with the specimens that did not receive the eugenol-containing material. A zinc oxide and zinc sulphate-hydrated provisional restorative material (Coltosol, Coltène AG, Alstätten, Switzerland) was used for this purpose. The non-eugenol material was removed in the same manner as the ZOE material. The specimens were stored in 100% humidity at 35°C during use of the provisional restorations. The experimental design is represented in Figure 1.

After removal of the provisional materials, the self-etching adhesive Adper SE Plus (3M ESPE, St Paul, MN, USA) was applied over the walls of the cavity. The adhesive was applied over the freshly prepared cavity for the control specimens that did not receive provisional restoration. The adhesive was applied according to the manufacturer's instructions. One drop of liquid A was applied on the surfaces of the cavity walls. Liquid B was then applied over liquid A and agitated for 20 seconds. After brief air-drying, liquid B was re-applied and light-polymerized for 10 seconds. Upon completion of the adhesive procedures, polyvinyl tubes with a cylinder-shaped orifice (1 mm inner diameter × 2 mm high) were individually placed onto the dentin surface. The resin composite Filtek Z-350 (3M ESPE) was inserted into the tube and light-polymerized for 20 seconds. Light-polymerization procedures were performed using the LED unit Optilight LD Max (Gnatus, Ribeirão Preto, SP, Brazil) with approximately 500 mW/cm^2 of irradiance. The tubes were then removed to expose the resin cylinders. Two cylinders were made per specimen.

The embedded specimens were attached to the testing device, and each resin composite cylinder was tested using a mechanical testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil). A thin steel wire (0.2 mm in diameter) was looped around each cylinder and a shear load was applied to the base of the cylinder at a crosshead speed of 0.5 mm/minute until failure. The microshear bond strengths were calculated and expressed in MPa. The average value of the two bonded cylinders for each substrate in the same specimen was recorded as the microshear bond strength for that specimen.

The data were subjected to two-way ANOVA and the Tukey's tests at a 95% confidence level. The factors evaluated were "exposure time to ZOE material" and "the time elapsed between ZOE removal and the adhesive procedure."

RESULTS

ANOVA showed a significant effect only for the factor "exposure time to ZOE material" (p=0.02) and interaction between the factors (p=0.003). The factor "time elapsed between ZOE material removal and the adhesive procedure" yielded p=0.06. Comparisons, according to the Tukey's test, are displayed in Table 1. There was no difference between the use and lack of use of ZOE, independent of time of use, when the adhesive procedure was performed for 7 or 14 days after ZOE removal. The use of ZOE interfered with bond strength only when the adhesive procedure was performed immediately. The only experimental condition that yielded different values from the other was the exposure to ZOE for 24 hours and the adhesive application immediately after its removal. This experimental condition showed the lowest shear bond strength.

Table 1: *Results of Bond Strength in MPa*

	Moment of Adhesive Application		
Use of ZOE-based Material	Immediately	After 7 days	After 14 days
Without ZOE	24.3 (8.4) Aa	23.3 (4.5) Aa	26.8 (4.7) Aa
ZOE during 24 hours	13.9 (3.4) Bb	24.8 (6.9) Aa	24.4 (3.2) Aa
ZOE during 7 days	26.0 (3.8) Aa	25.2 (4.6) Aa	24.1 (6.8) Aa
ZOE during 14 days	24.1 (4.2) Aa	23.8 (6.6) Aa	24.5 (2.9) Aa

**Means followed by distinct letters in the same line and small letters in the same column are significantly different at p<0.05.*

DISCUSSION

Several studies have evaluated the effects of provisional restoration with eugenol-based materials on the bond strength to dentin.[10-13,15,18-21,23-24] However, these studies reported conflicting results. Despite other methodological differences, the exposure period to ZOE has not been standardized during these studies, which may have affected the results. The outcomes of the current study show that both the period of ZOE exposure and the post-removal delay time influenced the bond strength of the evaluated self-etching adhesive to dentin. The use of provisional restorations with ZOE for 24 hours, followed by its removal and immediate adhesive application, produced the lowest values for bond strength. Thus, the null hypotheses for the current study were rejected.

The primary aim of provisional restorations is to protect the dentin-pulp complex from physical-chemical stimuli prior to definitive restoration.[26] For this purpose, studies have demonstrated adequate performance of provisional restoration with eugenol-based materials. These materials offer easy handling, provide proper sealing capacity, have a sedative effect on dental sensibility and are easily removed.[16,24,27] However, it has been demonstrated that the eugenol contained in these materials can interfere with the polymerization of adhesive resin and compromise the sealing and retention of adhesive restorations.[28]

The mixture between eugenol and zinc oxide generates a chelation reaction that results in a set mass of unreacted zinc oxide particles in a matrix of zinc eugenolate.[29] If the reaction was complete, it would be expected that the non-eugenol would be available to diffuse into dentin. However, this reaction is reversible, and hydrolysis of the eugenolate in the presence of moisture may again liberate the eugenol.[30] It has been demonstrated that the concentration of eugenol decreases as it moves towards the pulp chamber.[31] Eugenol can demineralize the dentin, releasing calcium, and this reaction reduces its diffusion rate.[15] This means that the polymerization inhibition effect of eugenol is probably higher near the surface. Thus, differences in the adhesive approach may interfere with eugenol's effect on bond strength.

Etch-and-rinse adhesive systems use 30%-40% phosphoric acid and demineralize the dentin to a depth of around 3-5 μm[1]. This mineral removal also seems to remove residual eugenol. On the other hand, self-etching adhesives result in the shortest depths of demineralization.[1] Thus, the effect of eugenol is more pronounced for self-etching adhesive, such as that used in the current study.[11] This reduction in the bond strength of adhesive to dentin by ZOE was observed in the present study. However, this negative effect was not observed when the provisional restoration with the ZOE material was used for one or two weeks. The same negative effect was observed when non-eugenol material was used for similar periods after ZOE removal and prior to the adhesive procedure.

The remnants of provisional materials have also been associated with the reduction of bond strength of adhesive to dental substrate. In the current study, provisional material removal was performed with a scaler, followed by cleaning with pumice-water slurry using a slow-speed handpiece. Both approaches have been proven effective in removing the remaining provisional material and permitting bond strength similar to that achieved without the use of provisional restorations.[24] Thus, it is expected that the combination of the two approaches does not permit the provisional material to interfere with bond strength. This can be confirmed by the results obtained when ZOE was used for longer periods. Furthermore, using two provisional restorations (ZOE followed by non-eugenol material) also does not affect bond strength.

The outcomes of the adverse effects of ZOE on bond strength are probably related to the inhibitory action of eugenol on the polymerization of the adhesive resin. Several studies using ZOE provisional restoration for seven days also found no adverse effect of eugenol on the bond strength of adhesive to dentin.[10,12,15,23-24] In opposition to this, Carvalho and others[11] found lower bond strength of a self-etching adhesive to dentin when ZOE had been previously placed over the substrate for 24 hours. However, all cited studies evaluated the effects of ZOE provisional restorations used for 24 hours or 7 days. Thus, the time-dependent reduction of the effect of eugenol was not evaluated in these studies.

It has been reported that eugenol diffusion from ZOE provisional restorations appears to depend more on the role of the hydrolysis of eugenol from the material than on dentin permeability.[32] The diffusion rate of eugenol released from ZOE restorations increased to a peak of about 0.3 ηmol per minute for the first 24 hours.[25] Following this period, the diffusion rate decreased slowly. Previous studies have proven that the inhibitory effects of eugenol on the polymerization of resin material is concentration-dependent.[28] Based on the results of the current study, it is expected that the eugenol concentration in dentin after one week will not significantly affect bond strength. Furthermore, a delay period of one week after ZOE removal is sufficient to decrease the eugenol concentration to non-inhibitory levels.

The outcomes of the current study showed that exposure to ZOE for one week did not reduce the shear bond strength of a self-etching adhesive to dentin. Even when the ZOE restoration remained in place for only 24 hours, a post-removal delay period of one week re-established the bond strength. Since the same time was necessary for both the exposure to ZOE and the delay after its removal, it seems more logical to use ZOE pro-

visional restorations for a week or longer. Clinically, seven days is a reasonable period for provisional restorations in most situations. However, further studies are necessary to evaluate other periods of exposure between 24 hours and one week. This will allow the shortest period of a ZOE provisional restoration to be determined in order to permit proper adhesive procedures.

CONCLUSIONS

The previous use of provisional restorations containing eugenol for one or two weeks did not interfere with bond strength. In contrast, the exposure to eugenol-based material for only 24 hours reduced the bond strength of a self-etching adhesive to dentin. However, a delay period of one week after ZOE removal was sufficient to re-establish bond strength.

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