Original Article

A comparative evaluation of the effect of eugenol exposure time on the bond strength of an etch-and-rinse and a self-etch adhesive to dentin: An *in vitro* study

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Abstract

Aim: The study aimed to comparatively evaluate the effect of eugenol exposure time on the micro-shear bond strength (μ-SBS) of etch-and-rinse and a self-etch adhesive to dentin.

Materials and Methods: One hundred and twelve teeth samples were prepared from bisectioning 56 freshly extracted human mandibular molars and were randomly divided into 14 subgroups of 8 samples each ($n = 8$). Three subgroups containing eugenol and a noneugenol-based restorative material were placed on the dentin surface and left for 24 h, 7 days, and 14 days, respectively, and were compared to a control. Two bonding systems were evaluated: one being etch-and-rinse and the other self-etch adhesive. The μ-SBS were calculated and expressed in MPa.

Statistical Analysis: The data were analyzed using mixed model analysis of variance. The level of statistical significance was set at 5%.

Results: There was a statistically significant reduction in the μ-SBS values when the self-etch adhesive was used, after the removal of eugenol-containing cement placed for 24 h. However, the reduction in the μ-SBS values after 7 days or 14 days was not significant.

Conclusion: Exposure to eugenol containing temporary cement for 24 h significantly reduces the μ-SBS of self-etching adhesives to dentin. However, exposure for 1 week or more has minimal effects.

Keywords: Provisional restorations; resin composite; self-adhesive; shear bond strength; zinc oxide eugenol

INTRODUCTION

Esthetic restorative dentistry has seen a boom in recent times due to its wide spectrum of available treatment options ranging from the routine placement of composite resin restorations, porcelain veneers, tooth whitening, and all-ceramic full and partial coverage restorations.[1-4]

Often, the application of a temporary material is indispensable for sealing the cavity until the next appointment to protect the exposed dentinal surface from sensitivity and plaque build-up. Furthermore, intermediate restoration of multiple carious teeth and the use of pulp capping procedures may warrant using

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temporary restorations. Eugenol-based temporary cement or temporary restorative materials are frequently used to treat teeth that subsequently receive resin composite cores after endodontic therapy.[3,5]

The negative influence of provisional cement, with or without eugenol, on the bond strength of composites to tooth substrates can be attributed to the chemical nature and the amount of the residual cement which jeopardizes the bonding.[6-11]

Bond strength studies have reported no adverse effect of eugenol on the bonding of resin composite to enamel, probably due to the removal of any residual cement as well as superficial enamel by phosphoric acid. Studies of the effect of eugenol on bonding to dentin have yielded contradictory results.[7,8] Despite the numerous studies on the inhibitory effects of eugenol, studies show a lack of consensus pertaining to the inhibitory effects of eugenol.

Several studies have found that eugenol-containing cement did not reduce the bond strength to dentin. However, other studies have reported contradictory results. 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 h to 10 days, depending on the study.[4]

Since the literature provides contradictory information on this topic, the purpose of our study was to evaluate the effects of exposure time of different types of provisional restorative materials (containing or not containing eugenol) on the bond strength of the composite to dentin using two different adhesive strategies.

MATERIALS AND METHODS

The sample size estimation was done using G*Power software (Version 3.1.9.7, University Duesseldorf, Germany). The minimum total sample size required was calculated as 112 ($n = 112$) with 56 samples per group and eight samples per subgroup ($n = 8$), based on an effect size of 0.36, based on the results of a previous study[3] with a power of 80%, and a type I error rate of 0.05. Following approval from the institutional ethics committee, 56 freshly extracted human mandibular molars were procured from the department of oral and maxillofacial surgery of the institution after excluding teeth that were found to be carious, fluorosed, fractured/cracked teeth, and restored.

For the preparation of the samples, the teeth were sectioned below the cementoenamel junction to remove the roots, using a diamond disk. The crowns were sectioned to obtain two halves in the mesiodistal axis, parallel to the long axis of the tooth. Each half was embedded in acrylic resin to facilitate handling, exposing the buccal/lingual surfaces.

The surface of the tooth was prepared with a cylindrical diamond bur until a flat surface on the dentin was obtained. The division of samples was as follows:

- Group I: Etch-and-rinse adhesive system
- Group II: Self-etch adhesive system.

The adhesive systems were further divided into three broad subgroups:

- A: No provisional restorative material (control)
- B: Noneugenol-based provisional restorative material (Cavit, 3M ESPE, Germany)
- C: Eugenol containing provisional restorative material (IRM, Dentsply, Caulk, USA).

Subgroups B and C of each Group were placed on the dentin surface and left for 24 h, 7 days, and 14 days.

The material was mechanically removed with a scaler and the surface was further cleaned with pumice water slurry and rinsed with air-water stream.

Two bonding systems were evaluated: Single Bond (3M ESPE, Germany), which is a two-step etch-and-rinse system, and Adper SE Plus (3M ESPE, Germany), a self-etch system. After the removal of the provisional materials, the adhesive systems were applied on dentin surfaces according to the manufacturer's directions. Upon completion of the adhesive procedures, standardized plastic cylinders were placed onto the dentin surface. The resin composite Filtek Z–350 (3M ESPE, Germany) was inserted into the plastic cylinders and light-polymerized. The plastic cylinder was then removed to expose the resin cylinder.

The embedded specimens were attached to the Hounsfield universal testing machine (Instron, USA), with a shear load application to the base of the resin composite cylinder at a crosshead speed of 0.5 mm/min until failure [Figure 1]. The micro-shear bond strengths (μ-SBSs) were calculated and expressed in MPa.

The collected data were tabulated into a spreadsheet using Microsoft Excel 2019 and then statistical analysis was carried out using GraphPad Prism for Windows, Version 9.5 (GraphPad Software, La Jolla California USA). A mixed-model analysis of variance was used for comparisons between the study groups and for observations collected over time, respectively, after assessing for normality. The $P = 0.05$ was considered as the level of statistical significance.

RESULTS

The mean μ-SBS values of the various subgroups are listed in Table 1 and Figure 2. Analysis of the results demonstrated

Table 1: Mean bond strengths in MPa for both the study groups and their respective subgroups

Groups/ subgroups	Subgroup A (control)	Subgroup B			Subgroup C		
		24 h	7 days	14 days	24 h	7 days	14 days
Group I	14.6±1.01	13±0.794	13.5±0.59^{A}	13.9±0.49	12.9±0.71^{A}	13.5±0.57	13.8±0.5^{A}
Group II	14.2±1.82$^{aa',b'}$	12.3±0.18^{a}	12.4±0.14$^{a',B}$	12.5±0.11	4.05±0.45^{b,B}	11.8±0.32$^{b'}$	12.2±0.38^{B}

Different lowercase letters: Significant differences within subgroups (24 h vs. 7 days vs. 14 days) in each group. Different lowercase apostrophe (') letters: Significant difference between subgroups (A vs. B vs. C) in each group. Different uppercase letters: Significant difference between groups (I vs. II) for each period

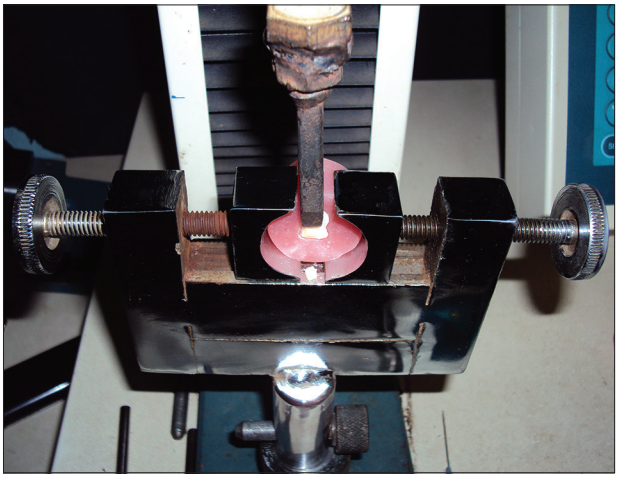

Figure 1: Specimen fitted on the universal testing machine and shear load was applied perpendicular at the base of the resin composite cylinder

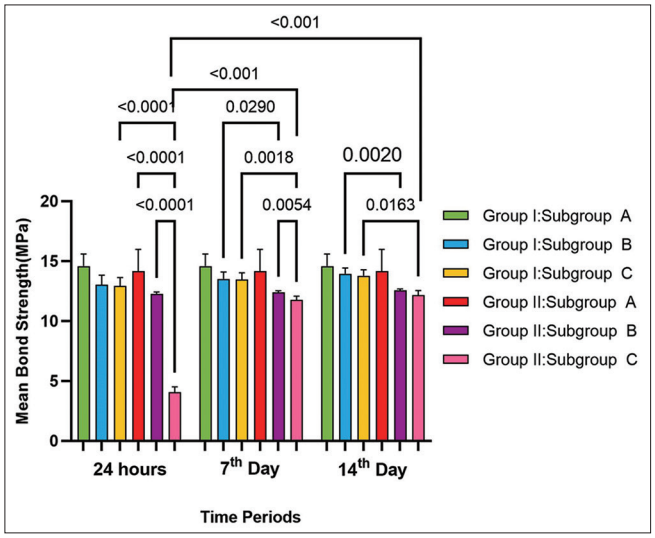

Figure 2: Bar graph showing the mean shear bond strength of the two study groups and their respective subgroups at various periods (24 h, 7 days, and 14 days) and the statistically significant comparisons between them

that there was no significant difference ($P > 0.05$) in the bond strength values when either eugenol-containing or noneugenol-containing cement was used, for either 24 h, 7 days, or 14 days, along with the use of an etch-and-rinse adhesive. There was a statistically significant reduction in the μ-SBS values when the self-etch adhesive was used, after the removal of eugenol-containing cement placed for 24 h ($P < 0.01$). However, the reduction in the μ-SBS values after 7 days or 14 days of use of eugenol-containing cement was not significant ($P > 0.05$). In addition, between-group comparisons inferred that the μ-SBS of the samples in the etch-and-rinse group restored with a eugenol-containing provisional restoration was significantly higher than the self-etched samples at 24 h ($P < 0.001$) and 14th day ($P = 0.02$), respectively. Furthermore, on the 7th day, the self-etched samples displayed a significantly lower bond strength than the etch-and-rinse group when restored with a noneugenol-based provisional restoration ($P = 0.03$).

DISCUSSION

Different factors can affect the bond strength of adhesives to tooth structure such as the capability of the dentinal bonding system to moisten and infiltrate the conditioned dentinal surface and the intensity of the interaction of self-etching adhesive systems with dentin is mostly dependent on the acidity and aggressiveness of the primer used.[12-15]

Mechanical removal of temporary cement is not completely effective, and cement remnants that appeared clean macroscopically were detected under magnification.[6,7] Moreover, eugenol released from zinc oxide eugenol (ZOE) mixtures can also penetrate dentin. Residual cement and eugenol penetration may also impair the polymerization of resin adhesives, leading to a decrease in bond strength and marginal sealing ability.[16,17]

Several studies have found that eugenol-containing cement did not reduce the bond strength to dentin. However, other studies have reported contradictory results. These differences in results can be explained by differences between the adhesive systems and methodologies used. It is worth highlighting that most of the studies have employed earlier generations of bonding systems and etch-and-rinse adhesive systems. The varied exposure times ranging from 24 h to 10 days reported previously, may interfere with the action of eugenol and contribute to an explanation of the different findings described in the literature.[3,5]

Hence, the purpose of our present *in vitro* study was to evaluate the effects of exposure time of different types of provisional restorative materials (containing or not containing eugenol) on the bond strength of the composite to dentin using two different adhesive strategies. Both the

etch-and-rinse adhesive system and the self-etch adhesive system were evaluated and the provisional restorations were used for either 24 h, 7 days, or 14 days.

The results of our present study showed that there was no significant difference in the bond strength values when either eugenol-containing or noneugenol-containing provisional restoration was used, for either 24 h, 7 days, or 14 days, along with the use of an etch-and-rinse adhesive (Single Bond). The possible reason for a lesser reduction in bond strength values after the use of etch-and-rinse adhesive may be that two-step etch-and-rinse adhesives, such as Single Bond, require the pretreatment of dentin with an acid. This acid, usually 30%–40% phosphoric acid, superficially demineralizes dentin to a depth of 9–10 μm and thereby exposes collagen scaffold, which likely reduces the amount of free eugenol and temporary restoration remnants on the dentin surface.[3,7] Any absorbed eugenol previously released from the zinc eugenolate may react immediately with the calcium ions to form calcium eugenolate, which may not have the radical scavenging effect of free eugenol.[18-20] Carvalho *et al.* have reported the usage of orthophosphoric acid in the removal of the smear layer contaminated with eugenol may improve the bond strength,[3] which corroborates with the result of the present study. However, in a recent meta-analysis by Garcia *et al.,* the effect of the adhesive system on the bond strength to dentin was found to be noncontributing.[21]

According to the results of our present study, there was a statistically significant reduction in the μ-SBS values when self-etch adhesive (Adper SE Plus) was used after the removal of eugenol-containing provisional restoration (IRM) placed for 24 h. However, the reduction in the μ-SBS values after 7 days or 14 days of use of eugenol-containing provisional restoration (IRM) was not statistically significant. In the self-etch approach, the infiltration of resin into dentin occurs concurrently with the etching process as in these systems, and the adhesive resin is expected to infiltrate beyond the smear layer and etch the intact underlying dentin to form a true hybrid layer.[22-25] Therefore, it is fair to presume that when eugenol containing temporary restoration was placed over the smear layer and left for 24 h, eugenol probably leached into and through the smear layer to the dentin tubules, contaminating the dentin surface.[3,5]

It has been found that the concentration of eugenol in the aqueous phase is in the order of 10^{-2} M just beneath the ZOE cement and 10^{-4} M adjacent to the pulp, implying that the concentration of eugenol is higher at the dentin surface with a gradual decrease toward the pulp. While, the self-etch systems are applied directly over the contaminated smear layer dentin, elucidating why the reduction in bond strength was more marked in the latter after pretreatment with eugenol-containing temporary restoration for 24 h.[3,5]

The diffusion rate of eugenol released from ZOE restorations is said to increase gradually reaching a peak at 0.3 nmol/min for the first 24 h, following which a decreasing trend has been observed. Furthermore, it has been demonstrated that the concentration of eugenol decreases as it moves toward the pulp chamber. Studies have shown that the inhibitory effects of eugenol on the polymerization of resin material were concentration dependent. Based on the results of the present study, it is expected that eugenol concentration in dentin after 1 week will not significantly affect bond strength. Furthermore, 7 days is also the approximate period, a temporary restoration would be used under clinical conditions.[4,14]

CONCLUSION

Within the limitations of this study, it can be concluded that exposure to eugenol containing temporary cement for 24 h significantly reduces the μ-SBS of self-etching adhesives to dentin. However, exposure for 1 week or more has minimal effects. Therefore, it seems logical to use eugenol-based provisional restorations for a week or longer before placing adhesive resin restorations.

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Conflicts of interest
There are no conflicts of interest.

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