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Effect of Sodium Perborate Treatment on Bond Strength of Two Resin-based Composite Systems to Pulpal Dentine

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ABSTRACT

Objectives: The aim of this study was to assess the effect of treatment with sodium perborate on the shear bond strength of two resin-based composite systems to pulpal dentine.

Materials and Methods: Sixty extracted maxillary anterior teeth were divided into six groups (n=10) according to the type of restorative system: etch and rinse adhesive (Adper Single bond 2) with Filtek Z250 or self-etch adhesive (Silorane system Adhesive) with Filtek P90 and bonding protocol (bonding with no sodium perborate, immediate and late bonding). Bond strength was tested in a universal testing machine, and data was analyzed using two-way analysis of variance with independent samples t-test and one-way analysis of variance with Tukey's HSD post-hoc ($p \leq 0.05$).

Results: Treatment with sodium perborate significantly lowered the shear bond strength between both composite systems and pulpal dentine, ($p < 0.001$). The mean shear bond-strength values were significantly higher in the late bonding compared to the immediate-bonding groups for both composites, ($p < 0.001$).

Conclusions: Treatment of the pulpal dentine with sodium perborate significantly reduced the immediate bond strength between the dentine and the composites. The bond strength can be returned to nearly normal values if the final bonded restoration is delayed for two weeks after treatment.

Keywords: Resin-based composite, Sodium perborate, Bond strength, Pulpal dentine, Intracoronary bleaching.

1. Introduction

Given the rising demand for aesthetic procedures, intracoronary bleaching is considered a cost-effective and conservative method for treating discolored teeth, especially when compared to veneers and crowns. Hydrogen peroxide is the effective element in presently used dental bleaching materials. The low molecular weight of this substance enables it to easily penetrate dentine and release oxygen. The oxygen breaks the double bonds of inorganic and organic compounds within the dentinal tubules (1, 2).

Intracoronary bleaching is frequently succeeded by

the application of bonded restorations. To ensure a proper coronal seal, bonding to the dentine of the pulp chamber must be optimized, as this is crucial for the prognosis of endodontically treated teeth (3). Moreover, effectively bonding the restoration allows for the reinforcement of the weakened tooth structure and the improvement of fracture resistance (4).

Previous studies have yielded mixed results on how various intracoronary materials and their concentrations impact the bond strength of methacrylate-based resin composites to bleached enamel and dentine. One study found no statistically significant differences in the bond-

strength values of methacrylate-based composite to human dentine after intracoronal bleaching with various regimens at different post-bleaching time intervals using a total etch adhesive system. The study concluded that the definitive restoration can be placed immediately after intracoronal bleaching (5). Other studies have found that intracoronal bleaching with sodium perborate can negatively impact the bond strength of methacrylate-based composites to both enamel and dentine (6-8). So, it was suggested that the bleaching agent might have a greater impact on dentine than on enamel, because dentine is a porous material, allowing oxygen to penetrate the dentinal tubules more easily than it does in enamel (6).

The reduction in bond strength to methacrylate-based resin composites has been attributed to the existence of oxygen remaining from hydrogen peroxide liberating products, which may inhibit polymerization of methacrylate-based resin composites (6). However, the reduction in shear bond-strength values was found to be time-dependent, and it was recommended to delay bonding procedures for methacrylate-based resin restorations (7).

The silorane-based resin composite was developed as an alternate to methacrylate-based composites, aiming to address issues related to polymerization shrinkage and the associated stresses (9). The reaction that forms the silorane matrix is cationic ring-opening polymerization of the silorane monomers, which is different from the linear chain reaction of methacrylates that crosslink through radicals. This material class is named after its chemical composition; the silorane molecule features a siloxane core with four oxirane (oxygen-containing) rings that open during polymerization to bond with other monomers (9). Due to the hydrophobic characteristics of the material, exogenous staining and water absorption are reduced (10). Furthermore, the oxirane rings are responsible for the decreased polymerization shrinkage. The opening of the oxirane ring results in volumetric expansion that may counteract to a certain extent for the shrinkage that results from molecular bonding (11).

It has been found that the silorane-based composites showed a significant decrease in shrinkage stresses, lower micro-leakage scores, better marginal adaptation, and decreased cuspal deflection compared to the methacrylate-based composites, which was related to the reduction of the polymerization shrinkage (12-14).

The bond strength of silorane composites has been stated to be close to that of methacrylate-based composites (15,16).

Studies have shown that vital bleaching does not significantly impact the bond strength of enamel-dentine cavities when restored with either silorane- or methacrylate-based composites (17). However, intracoronal bleaching significantly reduced both the immediate and delayed bond strength of bleached dentine to methacrylate-based resin composites when bonded with etch-and-rinse adhesives (18). The silorane resin-based composite adhesive is a two-step self-etch adhesive which is known as silorane system adhesive (3M ESPE, Seefeld, Germany). As a result, it retains the characteristics of conventional methacrylate adhesives, particularly in terms of its bonding approach to tooth tissue (19).

Given the widespread use of bleaching, and the conflicting proof that hydrogen peroxide compounds can impact bonding to enamel and dentine for an average of 7 to 14 days (1), further investigation into how treatment of the pulp chamber dentine with sodium perborate may affect other types of resin-based composites is still necessary. The aim of this study was to assess the effect of treatment with sodium perborate on the shear bond strength of two resin-based composite systems to dentine.

The null hypothesis tested was that the treatment with sodium perborate has no effect on the bond strength of the two resin-based composite systems to pulpal dentine with no difference between immediate and late bonding.

2. Materials and Methods

2.1 Selection and Preparation of Teeth

Sixty extracted human maxillary anterior teeth free of caries and cracks and stored in 0.1% thymol were used in the study. Endodontic access cavity was prepared in each tooth with high speed diamond bur under water coolant. The pulp tissue was removed with # 15 Hedstrom file.

Each tooth was horizontally sectioned 7 mm below the cemento-enamel junction using a double-faced diamond disk (Edenta AG, Hauptstrasse, Switzerland). A 3-mm layer of glass ionomer cement (Chemfil, Dentsply, Konstanz, Germany) was placed 2mm below the cemento-enamel junction to prevent the material from escaping during the bleaching process. Table 1

shows the experimental groups of the study. The sample size was determined according to the literature (16) for

bond strength of composites to dentine (n=10 in each group).

Table 1: The experimental groups

Group	Bleaching and bonding protocol	Restorative System
G1	No bleaching (Control)	Methacrylate-based resin composite (Z250)
G2	No bleaching (Control)	Silorane-based resin composite (P90)
G3	Immediate bonding after bleaching	Methacrylate-based resin composite (Z250)
G4	Immediate bonding after bleaching	Silorane-based resin composite (P90)
G5	Late bonding after bleaching	Silorane-based resin composite (Z250)
G6	Late bonding after bleaching	Methacrylate-based resin composite (P90)

In the control groups (G1 and G2), the access cavities were sealed with temporary filling (Cavit, 3M ESPE, MN, USA), and the teeth were kept in distilled water at 37°C for two weeks before bonding.

2.2 Bleaching Procedure

The intracoronal bleaching material sodium perborate tetrahydrate (Sigma-Aldrich, St. Louis, USA) (2g/1 ml water) was placed into the access cavity and the access was sealed with temporary filling (Cavit, 3M ESPE, MN, USA). The teeth were subsequently kept in distilled water at 37°C. The bleaching agent was replaced after 7 and 14 days. On day 21, the bleaching material was removed and the access cavity was cleaned with water spray for 60 seconds.

Subsequently, the teeth in G3 and G4 were bonded immediately after the bleaching procedure. In G5 and G6, the access cavities were sealed with glass ionomer filling and the teeth were kept in distilled water at 37°C for two weeks before bonding.

2.3 Preparation of Specimens

The teeth were horizontally sectioned at the cemento-enamel junction to separate the crowns from the roots. Subsequently, the crowns were sectioned in a mesial-distal direction with the aid of a double-faced diamond disk (Edenta AG, Hauptstrasse, Switzerland) coupled into a low-speed hand piece to expose (5 x 5 mm²) of dentin. The sections obtained were embedded in stone with the dentine facing up. The specimens were polished using 240 and 600 grit silicon carbide sand paper under copious irrigation to expose a flat surface area of dentine. Next, a 3-mm diameter area was left exposed as a bonding site by positioning a fenestrated PVC film with a 3-mm diameter hole over the flat

dentine surface.

2.4 The Bonding Technique

The manufacturer's instructions were followed for the two resin composite-system bonding protocols. In groups G1, G3 and G5: the dentine surface was etched for 15 seconds with 35% phosphoric acid gel (Scotchbond, 3M ESPE, MN, USA), The bonding agent (Adper Single Bond 2, 3M ESPE, MN, USA) was subsequently applied using a microbrush in two distinct layers. Next, it was air dried for 10 seconds until the surface achieved a uniform glossy appearance, and then light cured for 20 seconds using a light curing source at 1000 mw/cm² intensity (LED.B, Guilin Woodpecker medical instrument, Guangxi, China).

In groups G2, G4 and G6: P90 system adhesive was used following the manufacturer's instructions; P90 self-etch primer (3M ESPE, MN, USA) was applied on the dentine surface. The primer was applied and evenly distributed into a smooth film using a gentle stream of air. After that, the surface was light cured for 10 seconds, followed by P90 bond (3M ESPE, MN, U.S.A) application. After that, the surface was air dried and light curing for 20 seconds using the same light source.

A Teflon mold with a 3-mm diameter and 4-mm height hole was placed over the bonded surface. The mold was incrementally filled with a methacrylate composite (Filtek Z250, 3M ESPE, MN, USA) shade A2 for groups (G1, G3, G5) and with a silorane composite (Filtek P90, 3M ESPE, MN, USA) shade A2 for groups (G2, G4, G6). Each increment was light cured for 40 seconds from above for a total of 80 seconds. The specimens were submerged in distilled water and maintained at 37°C for one week prior to testing.

2.5 Shear Bond Strength Test

The shear bond strength was tested in a universal testing machine (Computer control electromechanical universal testing machine, Jinan testing equipment incorporation, Jinan, PRC); A parallel knife-edge shearing device was positioned over the bonded interface, and the specimen was loaded to failure at a crosshead speed of 0.5 mm/min. Bond-strength values were stated in Megapascals.

The specimens were then evaluated under a stereomicroscope (Leica Microsystems, Wetzlar, Germany) at 40X magnification in order to investigate failure patterns. Failure patterns were classified as adhesive (Type I) if failure occurred at the dentin-experimental material interface, cohesive (Type II) if failure occurred within the experimental material, and mixed failure (Type III) when it involved both the interface and the material. Two representative samples (20%) of the fractured surfaces from each group were air-dried, mounted on aluminum stubs and gold sputtered for evaluation under scanning electron microscopy (SEM) (Eclipse, Nikon, Tokyo, Japan).

2.6 Statistical Analysis

The statistical package for social series software (SPSS Inc., version 26, Chicago, IL, USA) was used for data analysis. The normality was tested using the Shapiro-Wilk and Kolmogorov-Smirnov tests. Means and standard

deviations (SDs) were used as descriptive statistics. The data was subjected to two-way analysis of variance (ANOVA). The main variables were the bleaching protocol and the restorative system. Independent samples t-test was used for differences between the two restorative systems within each bleaching category. One-way ANOVA with Tukey's HSD *post-hoc* was used for differences among the three bleaching categories within each restorative system, ($p \leq 0.05$).

3. Results

Multiple comparison of the data showed that intracoronal bleaching with sodium perborate significantly lowered the shear bond-strength values when comparing control with immediate bonding and immediate bonding with late bonding for both resin-based composite systems ($p < 0.001$), Table 2. The mean shear bond-strength values were significantly higher in the late-bonding groups compared to the immediate-bonding groups for both resin-based composite systems, ($p < 0.001$). The control Z250 group had the highest results in mean shear bond-strength values (8.69 ± 1.49), while the immediate Z250 group displayed the lowest values among all groups (2.40 ± 0.46).

There was no significant difference between the two resin-based composite systems when comparing the two systems in the control ($P = 0.15$), immediate bonding ($P = 0.23$), and late bonding ($P = 0.51$), Table 2.

Table 2: Mean shear bond-strength values in Megapascals (standard deviations) and results of ANOVA and Tukey's test

	P90		Z250		<i>P-value</i> ¹
	Mean	SD	Mean	SD	
Control (No bleaching)	6.50	(.901) ^a	8.69	(4.492) ^a	0.147
Immediate Bonding (Bleaching)	3.06	(.831) ^b	2.40	(1.463) ^b	0.231
Late Bonding (Bleaching)	7.42	(1.824) ^a	7.97	(1.847) ^a	0.508
<i>P-value 2</i>	<0.001		<0.001		

¹ Independent samples t-test.

² One-way ANOVA with Tukey's HSD *post-hoc*.

^{a,b,c} Means within column with different superscripts differ.

3.1 Failure Patterns

The most common failure pattern was the mixed failure (Type III), which accounted for 56.7% (34/60) of the samples when observed under the light microscope level and confirmed with SEM of representative samples

(Fig.1 a & b), followed by adhesive failure (Type I) and cohesive failure (Type II) in (23.3%,14/60 and 20%,12/60) of the samples, respectively. The distribution of failure patterns within each group is presented in Figure 2.

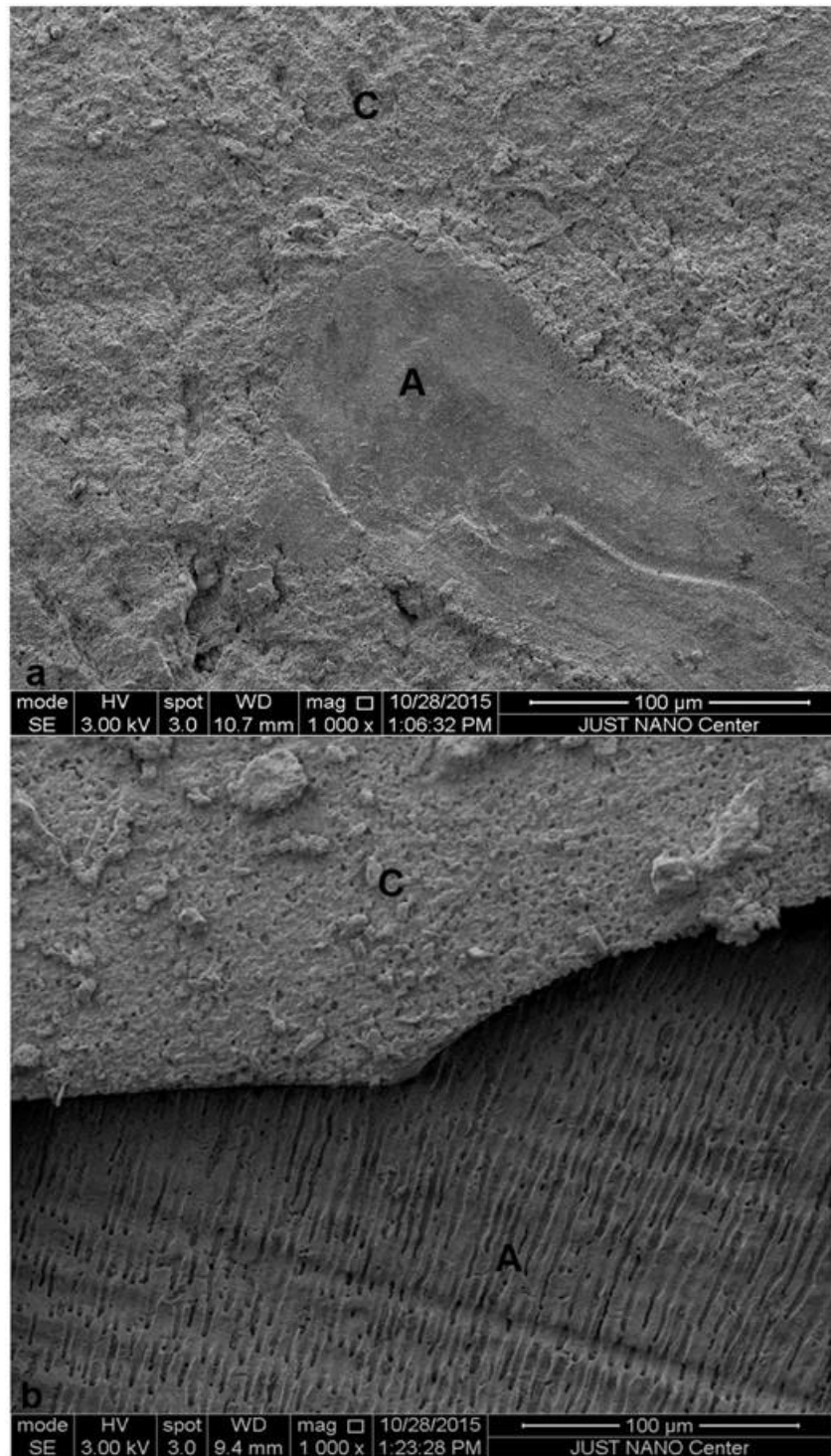


Figure 1 (a & b): Representative scanning electron microscope images at 1000 X magnification

a: Fractured surface from G4 showing areas of cohesive failure within composite (C) and remnant of the adhesive (A)

b: Fractured surface from G3 showing cohesive failure in composite (C) and adhesive failure in dentine (A)

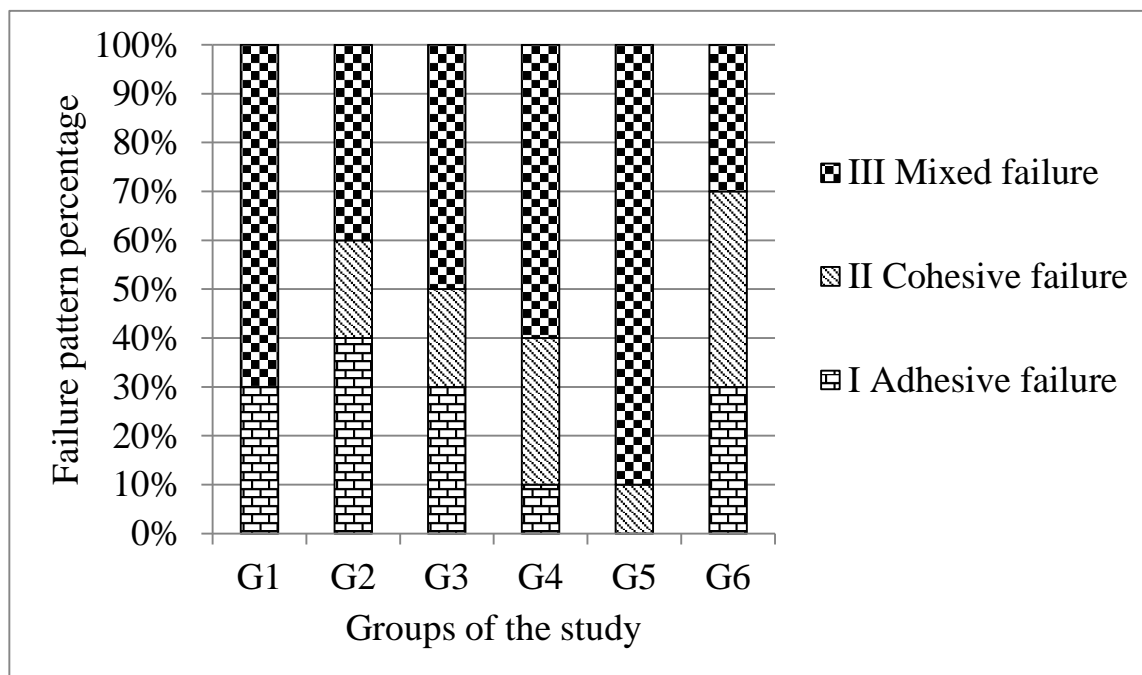


Figure 2: Bar graph of the percentage of failure patterns within each group

4. Discussion

Intracoronary bleaching of teeth using sodium perborate is a widely recognized clinical procedure that yields favorable outcomes. However, multiple studies have indicated that this bleaching process can affect the bonding of subsequent esthetic-restorative treatments to dentine (5-7). According to the results of this study, intracoronary bleaching with sodium perborate significantly decreased the shear bond strength of both composite systems to pulpal dentine, which is consistent with previous findings on methacrylate-based composites (6-8).

One of the assumptions that have been suggested for explaining the decreased bond strength after bleaching is related to the oxygen and water that result from the breakdown of the residual bleaching material in the collagen matrix and dentinal tubules (6). The oxygen interacts with the forming polymeric chain ends, terminating their propagation and decreasing the degree of conversion of both the adhesive systems and composite resins (20). Moreover, it was found that the hybrid-layer thickness was compromised in the dentine that was bleached after endodontic treatment (21).

Multiple studies have found that there is no difference in bond strength to dentine between silorane-based composites and conventional methacrylate-based composites (22,23). These findings were supported by

the findings of the present study, where the differences in the shear bond-strength values were not statistically significant between the two materials apart from the bleaching protocol.

Similar to the methacrylate-based resin composite, the silorane-based resin composite was negatively affected by the intracoronary bleaching, which could be explained by the same mechanism reported in the literature. Given that the silorane-system adhesive retains characteristics of traditional methacrylate adhesives, particularly in its bonding mechanism to tooth structure (19), it can be applied to bond methacrylate-based composite to tooth structure. However, its compatibility with the hydrophobic silorane matrix should be addressed (24).

The present investigation confirmed that immediate bonding after intracoronary bleaching can significantly decrease the shear bond strength of the tested resin composites. However, delaying the bonding of the composite restoration for two weeks increased the bond-strength values to the level observed in the non-bleached control for both resin-based systems. This waiting period might enable the gradual removal of residual oxygen from the bleached surface, therefore eliminating interference with polymerization of the bonded restoration and its detrimental effect on bond strength (7).

The least common pattern of failure was the cohesive failure, which is consistent with the low bond-strength values observed in the study, while the mixed-failure pattern was the most common failure pattern, which is in agreement with previous findings (25). The mixed-failure pattern was also observed in the selected SEM samples. This type of failure is common in the shear bond-strength test, which implies that the dentin/adhesive/composite interface evades stresses, and the shear bond-strength values reflect a complicated mixture of properties related to the bonded substrates rather than revealing the bond strength (26).

The average bond-strength values observed in this study were lower than those stated in the literature (17). Nevertheless, it was found that bonding to dentine that is near the pulp yielded much less bond-strength values compared to bonding to superficial dentine, which may account for variations between studies (27). The present study was conducted on pulp chamber dentin, the innermost type of dentin with the largest tubule diameters and the highest density, resulting in the weakest bond (28, 29).

Another issue that may explain the lower bond-strength values was the variability in teeth according to age range, as it was not standardized. Considering that extracting anterior teeth is very difficult to obtain unless the patient age is old or has periodontal problems; older teeth may exhibit a highly variable history of wear; therefore, a large variation exists in the quantity of secondary and tertiary dentine. Therefore, it is reasonable to predict that dentine adhesion will be less effective on sclerotic dentine compared to unaffected normal dentine (30).

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Even though laboratory tests do not perfectly replicate intraoral conditions, they propose a controlled setting for fabrication and testing specimens, allowing for an equal evaluation of the variables under examination. However, future studies should explore other analytical methods to gain more insights into the impact of intracoronary bleaching on various resin-based composite systems. This study was an *in vitro* investigation; however, the methodology was adopted to mimic the clinical steps by the application of the walking bleach technique and following the manufacturer's direction in the bonding technique.

5. Conclusion

Considering the limitations of this study, it can be concluded that the treatment of the pulpal dentine with sodium perborate reduced the immediate shear bond strength between the dentine and both resin composite systems. However, the shear bond strength may show higher values if the final bonded restoration is delayed two weeks after intracoronary bleaching.

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Conflict of Interests

The authors certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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