Influence of Activated Bleaching on Various Adhesive Restorative Systems

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ABSTRACT

Statement of the Problem: When adhesive restorations are used in combination with bleaching in clinical applications, it is generally recommended to wait for a period of time between the two procedures. However, it is not clear if a time interval is necessary between applying the bleaching treatments and the silorane-based restorative system.

Purpose: The purpose of this in vitro study was to determine if activated bleaching affects the bond strength of silorane-based restorative, a newly developed restorative that uses a polymerization system that has been reported to be insensitive to oxygen.

Methods: A gel consisting of 38% hydrogen peroxide was applied to ground labial enamel surfaces and activated using a diode laser to bleach the teeth. Then, without waiting for any period of time, four different restorative materials were applied to the bleached enamel surfaces, and these sets were compared with nonbleached control samples. The shear bond strength of the restorative systems to enamel was tested, and data was evaluated using two-way analysis of variance and Tukey HSD tests.

Results: Significant differences (p < 0.05) in shear bond strengths were found among bleached and nonbleached enamel surfaces.

Conclusion: It may be more beneficial to allow a time interval of 2 to 3 weeks between activated hydrogen peroxide bleaching and applying silorane-based composite restoratives or methacrylate-based composites than just applying restorative agents immediately after bleaching. However, further studies are needed to examine the structural effects of activated hydrogen peroxide on enamel.

CLINICAL SIGNIFICANCE

A time interval should be allowed between the application of silorane-based or methacrylate-based restorations and activated hydrogen peroxide bleaching systems.

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INTRODUCTION

Bleaching may be indicated before restorative treatments in order to harmonize the shade of the restorative material with that of the natural teeth.¹ In fact, combining enamel bleaching and composite resin restoration procedures has been reported to achieve a more successful outcome.² Several studies have reported on bonding after bleaching.^{3–9} Most of these studies^{5–9} recommend waiting for a period of time between the bleaching procedures and the application of composite resins. The authors of one study⁹

*Assistant Professor, Department of Operative Dentistry, Faculty of Dentistry, Near East University, Mersin 10, Turkey [†]Consultant, Department of Dentistry, Hospital of Girne, Mersin 10, Turkey recommend waiting at least 1 week between applying enamel bleaching with 38% hydrogen peroxide (H_2O_2) and placing adhesive bonding agents for composite resin restoration.

Light-activated bleaching is a method of tooth whitening.¹⁰ Tooth whitening can be achieved by external bleaching utilizing highly concentrated, 30 to 35% H₂O₂-releasing agents. To enhance or accelerate the whitening process, heat or light, including lasers, can be used in a procedure known as activated bleaching.¹¹ Numerous studies report using diode lasers in activated bleaching^{12–19}; however, while one recent study showed that using light activation with in-office bleaching seems to increase the efficacy of treatment for only a short period of time,²⁰ another study mentioned that as bleaching with diode laser resulted in less tooth and gingival sensitivity, it might be preferred among in-office bleaching systems.²¹

Resin composites can be classified according to various characteristics of the filler used (i.e., type, distribution, average particle size, and physical and mechanical properties).²² In recent years, the development of adhesive technology has proceeded rapidly, and several materials are now available that contain very fine inorganic particles produced using advanced technology that are known as "nanofill" or "nanohybrid" composites. The resin matrix also significantly influences the properties of composite materials.²³ Although all commercially available composites are methacrylate-based resins that undergo a process of radical polymerization, new silorane-based composite materials are based on siloxanes and oxiranes that polymerize through a cationic ring-opening process.^{24,25} Initial evaluations of this new category of composite material have reported acceptable mechanical and physical properties.²⁶ Even for these low-shrinking composites, however, a layering technique is still recommended.27

Reportedly, oxidants like bleaching agents affect the polymerization and bond strength of dental adhesives if applied to the substrate immediately prior to the bonding procedure.²⁸ Whereas oxygen has been shown to inhibit the radical polymerization of monomer

systems used in dentistry,²⁹ the cationic ring-opening polymerization associated with the oxiranes found in silorane have been reported to be insensitive to oxygen,^{29–31} which may offer an advantage for bonding to bleached tooth substrates. However, the adhesive of the silorane-based system is a phosphate-methacrylate based, two-step self-etch system consisting of a hydrophilic primer and a hydrophobic adhesive resin, and in contrast to all other two-step, self-etch adhesive systems, the self-etching primer agent of the silorane adhesive requires polymerization.^{28,32} According to recent literature, silorane adhesive is different than methacrylate-based adhesives.^{33,34}

In an effort to reduce technique sensitivity, application difficulties and chair time, dentin-enamel bonding adhesives have been simplified to include self-etching all-in-one and one-bottle etch-and-rinse systems, in which all ingredients are combined in a single bottle. Choosing the appropriate material is complicated by the great variety of bonding agents available on the market,³⁵ and the efficacy of simplified adhesives, in terms of bond strength and longevity, has been questioned.³⁶

The aims of this study were to determine if the prior application of bleaching treatments would affect the shear bond strength of a silorane-based adhesive restorative system and to compare the shear bond strengths of various silorane- and methacrylate-based composite resin restorative systems applied immediately after activated bleaching. The null hypothesis was that there would be no difference in the shear bond strength results of both bleached and nonbleached samples for silorane-based restorative system and it would exhibit high shear bond strength values to activated H_2O_2 -treated enamel and could be used immediately after bleaching procedures.

MATERIALS AND METHODS

Preparation of Enamel Surfaces

A total of 80 caries-free permanent premolars, which were extracted for orthodontic reasons, were used in

this study. Written informed consent was obtained from all patients. Individual tooth surfaces were hand scaled to remove any remaining soft tissue. All teeth were disinfected in 0.5% chloramine solution for up to 1 week and then stored in distilled water at -20°C for up to 1 month. Crowns were separated from the roots 2 to 3 mm apical to the cementoenamel junction using a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water irrigation at a low speed, and pulp tissue was removed using a dental explorer. With their labial surfaces exposed, the crowns were then embedded in self-curing acrylic resin (Meliodent, Heraeus Kulzer, Dormagen, Germany) into Teflon molds. Specimens were wet ground using 300- and 600-grit silicon carbide paper to achieve evenly flat enamel surfaces, and then cleaned in distilled water to remove any remaining silicon carbide particles. All enamel surfaces were examined under a stereomicroscope (Leica, MZ 12, Leica AG, CH-9435 Heerbrugg, Switzerland), and any specimens with cracks or hypoplastic defects were excluded. Specimens were randomly divided into eight groups, four of which underwent bleaching treatment followed by one of four restorative adhesive treatments, and the other four groups served as control samples that received no bleaching prior to adhesive treatments.

Bleaching Treatment

Bleaching was performed with Opalescence Xtra Boost (Ultradent Products Inc., South Jordan, UT, USA), a 38% H₂O₂ power bleaching gel with a neutral pH (7.0) designed for in-office, chairside accelerated whitening. No etchant was applied to the enamel surfaces prior to bleaching. Enamel surfaces were cleaned with water spray for 5 seconds and dried with oil and water-free compressed air for 3 seconds. The contents of the Opalescence Xtra Boost syringe were raised to room temperature and the plunger was pressed rapidly 20 times to mix the activator and bleaching agent. Next, a layer of the mixed bleaching agent, 0.5 to 1.0 mm thick, was applied evenly over the enamel surfaces while stirring the gel every 5 minutes for optimum effectiveness. The bleaching gel was then immediately activated with a galium-aluminium-arsenide diode laser (Laser Smile Biolase Technology, San Clemente, CA, USA) in the continuous mode. As per the manufacturer's instructions, specimen surfaces were irradiated with 815 nm applied with the hand piece close to the gel in an even sweeping, scanning motion. The operator wore protective eyewear designed specifically to protect against the diode laser wavelength during the treatments. Each sample required two 30-minute bleaching sessions performed at 3-day intervals, for a total of 60 minutes per sample. One bleaching session consisted of three separate applications of bleaching gel for 10 minutes each, including four laser irradiations of 15 seconds each (total irradiation: 1 minute), followed by thoroughly wiping and rinsing the enamel surface between applications.

During the hiatus periods, all specimens were stored at 37°C in artificial saliva prepared from 1.5 mmol/L calcium chloride, 8.2 mmol/L sodium bicarbonate, 4.8 mmol/L sodium chloride, 137 mmol/L potassium chloride, 4 mmol/L potassium dehydrogen phosphate, and 100 mL deionized water. The artificial saliva, changed daily, contained an electrolyte composition similar to that of human saliva and a pH of 7.0.⁵

Application of Bonding Agents and Preparation of Composite Resin Rods

Details of bonding adhesives and composites are provided in Tables 1 and 2, respectively. Before the applications, an adhesive tape with a central orifice of 3 mm in diameter was applied to the enamel surfaces to demarcate the area to be treated. Bonding agents were then cured using a quartztungsten-halogen (QTH) light curing unit (Hilux Ultra Plus, Benlioglu Dental, Istanbul, Turkey) in standard mode. The light intensity of the unit was monitored with a radiometer (Curing Radiometer, Model 100, Demetron/Kerr Corp., Danbury, CT, USA) throughout the experiment and did not drop below 550 mW/cm².

Resin composite materials were applied to the prepared enamel surfaces perpendicularly by attaching a Teflon

TABLE I.	Information	regarding	adhesive	systems
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Group no.	Material, manufacturer, classification	Main components	Lot no.	Applications
1,2	Clearfil SE Bond Kuraray, Okayama, Japan Two-step self-etching adhesive system	Primer: 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), 2- hydroxyethyl methacrylate (HEMA), hydrophilic dimethacrylate, dl-camphorquinone, N,N-diethanol-p- toluidine and water Bond: MDP, Bisphenol A diglycidylmethacrylate (Bis-GMA), HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N,N-diethanol- p-toluidine and silanated colloidal silica	Primer: 00752A Bond: 01083A	Primer was applied with a disposable brush tip and left in its place for 20 seconds. Entire surface was sufficiently dried with a mild oil-free air stream in order to evaporate the volatile ingredients. Bond was applied, gently air dried and light-cured for 10 seconds.
3,4	Silorane System Adhesive 3M ESPE, Seefeld, Germany Two-step self-etching adhesive system	Silane treated silica filler, initiators, stabilizers Primer: HEMA, glycerophosphate-dimethacrylate (GPDM), mono(2-methacryloyloxy) ethylphthalate (MMEP), ethanol, water, dl-Camphorquinone Bond: hydrophobic methacrylates, triethylene glycol dimethacrylates (TEGMA), silane treated silica filler initiators, stabilizers, dl- Camphorquinone	Primer: 8BB Bond: 8AY	Primer was applied onto enamel surfaces and brushed for 15 seconds. After exposing to a gentle air stream, primer was light-cured for 10 seconds. The bottle was agitated and bond was applied onto enamel surfaces. After exposing to a gentle air stream, bond was light-cured for 10 seconds.
5,6	Clearfil tri-S Bond Kuraray, Okayama, Japan A single component, all-in-one self-etch adhesive system	MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, ethyl alcohol, water, silanated colloidal silica	009CA	Bonding agent was applied with a disposable brush tip and left in its place for 20 seconds. Entire adherent surface was sufficiently dried by blowing high-pressure air for more than 5 seconds while spreading the bond layer thinly. Bond was light-cured for 10 seconds.
7,8	Adper Single Bond 2 3M ESPE, St. Paul, MN, USA One-step total-etch adhesive system	Etchant: 35% phosphoric acid Bond: ethyl alcohol, Bis-GMA, silica nanoparticles treated, HEMA, glycerol I,3 dimethacrylate, acrylic acid copolymer and itaconic acid, diurethane dimethacrylate, water	7MU	Etchant was applied onto enamel surface for 15 seconds and rinsed for 10 seconds. After blotting, two consecutive coats of bond were applied to etched enamel for 15 seconds with gentle agitation and gently air thinned for 5 seconds to evaporate solvents. Bond was light-cured for 10 seconds.

tube (3 mm wide and 2 mm high) to the prepared enamel surfaces with impression putty. Composite materials were light-cured 40 seconds from each perpendicular direction using the same QTH light curing unit for a total of 160 seconds. Following curing, the Teflon tube was carefully removed, the composite rod was checked, and any specimens with visible air bubbles or gaps at the interface were discarded.

Shear Bond Strength Testing

The shear bond strength of resin bonding systems was tested according to International Organization for Standardization (ISO) procedures (ISO/TS 11405).³⁷ Prepared specimens were stored in distilled water at 37°C for 24 hours prior to shear bond strength testing, and specimens were debonded using a universal testing machine (Lloyd LRX Universal, Lloyd Instruments,

Group no.	Material, manufacturer, classification, lot no.	Main components
1,2,5,6	Clearfil Majesty Posterior Kuraray, Okayama, Japan Nanohybrid 00001C	Resin matrix: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, dl-camphorquinone Filler: nano and micro inorganic filler: Silanated glass ceramics, surface treated alumina micro filler: 82 vol%–92 wt%
3,4	Filtek Silorane 3M ESPE, St. Paul, MN, USA Microhybrid 8CN	Resin matrix: 3,4-Epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethyl phenylmethylsilane Filler: silanized quartz; yttriumfluoride 55 vol%–76 wt%. 0.1–2 μm
7,8	Filtek Z250 3M ESPE, St. Paul, MN, USA Microhybrid 7XE	Resin matrix: Bis-GMA, UDMA, Bis-EMA Filler: zirconia/silica 0.01–3.5 μm. 60 vol%–84 wt%

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Fareham, Hants, England, UK) at a crosshead speed of 1.0 mm/minute at room temperature ($23 \pm 2^{\circ}$ C). Specimens were secured in a mounting jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA, USA) with the shearing rod against and parallel to the flat prepared bonding sites. The distance from the probe to the enamel surface was monitored using a spacer of two celluloid matrices. Shear bond strengths were calculated by dividing the highest fracture force (N) by the bonded area (3 mm diameter) and recorded in megapascals (MPa). Debonded specimens were examined under a stereomicroscope (Leica, MZ 12) at $25 \times$ magnification, and failure modes were recorded based on the percentage of substrate-free material as either adhesive failure, cohesive failure in composite/adhesive resin with partial adhesive failure (mixed mode), or cohesive failure in enamel.⁵

Statistical Analysis

Data was analyzed using two-way analysis of variance and Tukey HSD tests, with a significance level of 0.05.

RESULTS

Minimum, maximum, and mean shear bond strength values and standard deviations (SDs) and failure modes of the groups are shown in Table 3. Applying bleach was found to have a significant effect (p < 0.05) on the

shear bond strengths to the enamel of all of the samples, including the silorane-based restorative system (Figure 1). Composites applied immediately after bleaching had significantly lower bond strengths than the control groups that were not bleached. However, no significant differences (p > 0.05) in bond strengths were observed among the bleached enamel samples of different composite materials.

Adhesive failures occurred less frequently among the control groups than the groups that were pretreated with bleaching. Among the control groups, the one-step total-etch adhesive had significantly (p < 0.05) higher bond strength than the other systems tested, and the all-in-one self-etch adhesive system showed significantly (p < 0.05) lower bond strength than the other systems tested.

DISCUSSION

The bond strength of dental adhesives to enamel has been shown to be affected by prior treatment of individual tooth surfaces.³⁸ According to the present study, the null hypothesis, which suggests that silorane-based restorative systems could be used immediately after bleaching and would demonstrate higher shear bond strength values to activated H₂O₂treated enamel than other composite systems, must be rejected. **TABLE 3.** The mean, standard deviation, the range of the shear bond strength measurements expressed in MPA, and counts and percentages of failure modes in enamel

Group no.	Groups	Mean ± SD	Range	Statistical category	Adhesive failure mode (%)	Cohesive failure mode (%)	Mixed failure mode (%)
I	Clearfil SE Bond/Clearfil Majesty Posterior Nonbleached Enamel	17.78 ± 3.23	3.22–24.34	a,c,d	2 (20)	—	8 (80)
2	Clearfil SE Bond/Clearfil Majesty Posterior Bleached Enamel	10.03 ± 2.11	3.10-12.63	b	10 (100)	_	_
3	Silorane System Adhesive/Filtek Silorane Nonbleached Enamel	17.43 ± 1.86	15.35–21.63	a,c,d	2 (20)	_	8 (80)
4	Silorane System Adhesive/Filtek Silorane Bleached Enamel	8.50 ± 1.36	4.55–11.43	b	10 (100)	_	_
5	Clearfil tri-S Bond/Clearfil Majesty Posterior Nonbleached Enamel	4.47 ± .98	.43– 7.74	С	8 (80)	_	2 (20)
6	Clearfil tri-S Bond/Clearfil Majesty Posterior Bleached Enamel	8.19 ± 2.69	3.17-10.19	b	10 (100)	_	_
7	Adper Single Bond/Filtek Z250 Nonbleached Enamel	21.52 ± 5.38	10.44–28.92	d	(0)	—	9 (90)
8	Adper Single Bond/Filtek Z250 Bleached Enamel	10.71 ± 2.14	5.29-13.09	b	10 (100)		

Statistical category: identical letters indicate that values are not statistically different (p > 0.05) with Tukey HSD comparison.





Buchalla and Attin¹¹ state that optimal bleaching results cannot normally be achieved with a single application of in-office bleaching, and Joiner³⁹ stresses the importance of saliva usage in bleaching studies. Accordingly, this study utilized a multi-appointment procedure, artificial saliva containing an electrolyte composition similar to that of human saliva, and a bleaching agent with a neutral pH. Although a shear bond strength test has some shortcomings, its usage is still being reported in literature⁴⁰ and it does have its own advantages. While shear bond strength testing can only yield comparative data, specimen preparation and testing is relatively straightforward in comparison to tensile test methods, hence its use in this work.³⁰ Other studies on bleaching used posterior teeth, as did this present study.^{3,41–43}

In addition to specific characteristics of adhesives and composite resins on bond strength, prior management of the tooth surface is another factor that should be considered in restorative treatments. Previous studies indicate that H₂O₂ has chemical or morphological effects on enamel^{5,8,44–46} and that in order to achieve the sufficient shear bond strength of composites, a waiting period of 1 to 3 weeks is necessary before applying adhesives to ensure that residual oxygen on the tooth surface is eliminated.^{5–9} Moreover, according to Luk and colleagues,¹⁰ although the light application can significantly improve the whitening efficacy of some bleaching materials, it also causes significant temperature increases in the outer and inner tooth surfaces. A recent study examining the shear bond strength of a two-step, self-etching adhesive to activated H_2O_2 -treated enamel concluded that activation of H_2O_2 may further reduce the shear bond strength of composite resin restorations to enamel and that a longer time period may be needed prior to bonding following activated bleaching when compared to "nonactivated" bleaching.⁴⁷

In order to determine the effects of activated bleaching on various adhesive restorative systems, this study included control groups of nonbleached samples for every restorative system tested. In comparison to the bleached enamel samples, the nonbleached control groups were found to have significantly higher shear bond strengths, thus demonstrating the importance of the time interval between the application of H_2O_2 bleaching treatment and adhesive restoratives. Despite the fact that silorane is reportedly not affected by oxygen,²⁹⁻³¹ the silorane-based restorative system tested in this study showed significantly lower bond strength to bleached enamel in comparison to nonbleached enamel. This finding may be related to an oxygen reaction induced by the camphorquinone content of the silorane-based restorative system.³⁰

While the shear bond strength of all of the restorative systems applied to bleached surfaces without a time interval demonstrated lower shear bond strengths than those of nonbleached controls, there were no differences among the bleached samples. However, differences in bond strengths were found among the nonbleached control samples, with the highest bond-strength values to enamel observed with the one-step total-etch adhesive system tested. This finding is in line with Gurgan and colleagues³ who suggested that following bleaching, etch-and-rinse adhesive systems could provide higher bond strengths than self-etching adhesives to enamel. Although it is not completely clear if heat activation of the bleaching agents will induce or increase micromorphological changes in bleached enamel, it is likely that enamel dehydration is enhanced by exposure to additional heat during bleaching.¹¹ This water loss may dramatically reduce the wettability of enamel, and result in altered

substrates that interfere with bonding to hydrophilic primer coats.⁴⁸ In line with this suggestion, it is possible that the rinsing step performed with total-etch systems could improve bond strength by contributing to the elimination of residual oxygen from the substrate.²⁸

It has been estimated that bond strengths of 17 to 20 MPa are required to sufficiently resist contraction forces and produce gap-free restoration margins.⁴⁹ In this study, the mean bond strength of a silorane-based restorative applied without bleaching was found to be 17.43 MPa, which is considered sufficient for most clinical adhesive needs. However, further research is needed to investigate the mechanical properties of the hybrid layer created by silorane adhesives and their long-term stability.³² In a recent study, Grégoire and colleagues³⁴ mentioned that the complex chemistry involved explains the stable hybrid layer found when this adhesive is applied to dentin.

Adhesive failures occurred less frequently among the control groups than with the groups that were pretreated by bleaching. Barbosa and colleagues⁵⁰ also used shear bond strength test in a study to investigate the influence of in situ post-bleaching times on resin composite shear bond strength to enamel; the results report a predominance of adhesive failures for the groups in enamel. Gurgan and colleagues³ mentioned that the analysis of bonding sites after shear bond strength test revealed that most of the failures were adhesive in nature at the resin composite/enamel surface interfaces. The adhesive failure showed the weak link between the resin composite and the enamel surface due to the adverse affect of bleaching treatments on the bond strength of the adhesive materials. The least frequency of failures related to mixed failures were found on the specimens treated with etch-and-rinse adhesive systems. Perdigao and colleagues⁵¹ similarly observed and reported that cohesive types of fractures are beginning to be seen when the shear bond strength values exceeded 17.40 MPa.

Researchers have suggested that acid etching can increase the bond strength of single-step, self-etch

adhesive systems,^{40,52} and the manufacturers of single-component all-in-one self-etch adhesive systems suggest that uncut enamel should be treated with a phosphoric acid gel, rinsed with water, and then gently dried before using the bond. However, because this study was conducted with ground enamel surfaces, this adhesive was applied directly to enamel surfaces without any pretreatment. This group demonstrated lower shear bond strength results when compared with two-step self-etch and one-step total-etch applied control groups. A previous study suggested that polymerization of simplified adhesives may be compromised by their high hydrophilic monomer content and the presence of water, subsequently leading to suboptimal curing of the associated resin films.³² In addition, polymerization of all-in-one adhesives may be inhibited by the presence of residual adhesive solvent following air drying, thereby reducing bond strength.⁵³ Nevertheless, as an earlier study points out, it is long-term clinical performance, not bond strength, that is the true criterion of adhesive quality.⁵⁴

The present findings should be interpreted with caution, as the results were obtained under laboratory conditions, and it remains to be seen whether similar findings will occur in vivo.⁵⁵ Moreover, in view of the great variety of bonding materials, resin composites, and compomers available on the market, further studies are required to identify differences in the results between products.⁵⁶ For example, further in vivo studies with total-etch filled adhesive systems may identify if the higher viscosity of filled bonding systems will result in lower bond strengths to enamel than one-bottle adhesive systems without filler particles.⁵⁷ Moreover, the concentrations and characteristics of individual monomers in adhesives and their interactions may also affect the extent of infiltration, ionization, and cross linking obtained on polymerization and thus, the mechanical properties of the adhesive.⁵³ In line with a previous study,⁵⁸ the present study measured the bond strengths of samples in which bonding agents were combined with appropriate composite restoratives, and as a result, the findings cannot offer conclusive data on the adhesive level alone. Thus, it would be useful for further studies to separately examine the effects of monomer concentration, pH, solvent type, composition

and filler content of adhesives, and resin composites with regard to bond strength to bleached enamel.

Within the parameters of this in vitro study, it may be suggested that the shear bond strength values of composite resin restoratives to enamel following activated bleaching may be improved by a 2 to 3 week waiting period between the application of the bleaching treatment and the restorative treatment, and that this waiting period may be recommended for silorane-based restorative systems as well as methacrylate-based composite restorative systems. Further in vivo studies examining the structural changes of activated H₂O₂-treated enamel and its adhesion characteristics to adhesive restorative materials are required.

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