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Shear Bond Strength of Self-etch Adhesives to Enamel with Additional Phosphoric Acid Etching

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Clinical Relevance

When using self-etch adhesives to bond composite materials to enamel, there is concern about the ability to achieve bond strengths comparable to approved etch-and-rinse systems. An additional phosphoric acid etching can improve the shear bond strength of self-etch adhesives to enamel.

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SUMMARY

This study evaluated the shear bond strength of self-etch adhesives to enamel and the effect of additional phosphoric acid etching.

Seventy sound human molars were randomly divided into three test groups and one control group. The enamel surfaces of the control group (n=10) were treated with Syntac Classic (SC). Each test group was subdivided into two groups (each n=10). In half of each test group, ground enamel surfaces were coated with the self-etch adhesives AdheSe (ADH), Xeno III (XE) or Futurabond NR (FNR). In the remaining half of each test group, an additional phosphoric acid etching of the enamel surface was performed prior to applying the adhesives. The shear bond strength was measured with a universal testing machine at a crosshead speed of 1 mm/minute after storing the samples in distilled water at 37°C for 24 hours. Fracture modes were determined by SEM examination. For statistical analy-

sis, one-way ANOVA and the two-sided Dunnett Test were used ($p>0.05$).

Additional phosphoric etching significantly increased the shear bond strength of all the examined self-etch adhesives ($p<0.001$). The highest shear bond strength was found for FNR after phosphoric acid etching. Without phosphoric acid etching, only FNR showed no significant differences compared to the control (SC).

SEM evaluations showed mostly adhesive fractures. For all the self-etch adhesives, a slight increase in mixed fractures occurred after conditioning with phosphoric acid.

An additional phosphoric acid etching of enamel should be considered when using self-etch adhesives. More clinical studies are needed to evaluate the long-term success of the examined adhesives.

INTRODUCTION

Patients' demands for esthetic restorations and the search for filling materials that can serve as alternatives to amalgam with equal long-term stability have generated new materials and techniques in adhesive dentistry. Additionally, dentists in private practice have been requesting materials with little technique sensitivity that allow the minimally-invasive treatment of carious defects and the possible repair of defective restorations instead of replacing an entire filling.[1] These requirements need to be met with new materials, such as self-etch adhesives.[1]

The adhesion of hydrophobic resins to hydrophilic substrates, such as enamel and dentin, is mediated by different adhesive systems that generate a microretentive interface and a hybridization of exposed collagen fibers.[2-3]

The desire for simplified self-etch systems has initiated a rapid development and turnover rate of available products, which has also caused some confusion regarding the classification of subsequent generations of materials.[4] A sole chronological classification based on the time when products were introduced is not supported by scientific facts, since various types of adhesives were marketed almost simultaneously. Therefore, adhesives are mainly characterized by their interaction with the smear layer and the mode of application—smear layer modifying, smear layer dissolving and smear layer removing.[5]

The newest types of adhesives are self-etch materials that are subdivided into three categories based upon their pH-value.[6-7] With strong adhesive systems (pH of 1 or below), an enamel etching pattern similar to phosphoric acid etching is achieved.[7-9] Intermediary strong self-etch adhesives have a pH of approximately 1.5 and are more acidic than mild systems, which have a pH of 2.[3,7] Their interaction with enamel is characterized as nanoretentive interlocking with dissolution of peripheral and central parts of the crystallites and an additional inter- and intra-crystallite monomer infiltration.[10]

In terms of bonding to dentin, a bond strength equal to etch-and-rinse systems has been reported.[11] While micromechanical retention and resin infiltration after etching enamel with phosphoric acid was demonstrated to be a reliable clinical procedure,[2-3] the bond strength of self-etch adhesives to enamel is controversially discussed in the literature. In some studies, the most user-friendly one-step self-etch systems produced a lower bond strength than two-step self-etch or etch-and-rinse systems and they are considered to be less reliable when bonding to enamel.[12-15] Also, one-step self-etch systems exhibited fewer gap-free margins when bonded to both enamel and dentin when compared to two-step self-etch and etch-and-rinse systems, especially after thermomechanical loading.[16] Other studies that addressed the enamel bond strength of self-etch systems reported data that are comparable to that observed with etch-and-rinse systems.[17-18] Additionally, there is a consensus that the bond strength of self-etch systems is stronger to ground than unground enamel. Self-etch adhesives also produce a significantly lower bond strength to unground enamel than etch-and-rinse systems, which are dependent on their acidity.[9,19-20]

The simplified bonding procedure, reduction of chairside time, reduced technique sensitivity and the possible prevention of incomplete resin infiltration, in addition to a supposed chemical bonding, are alternative features of self-etch adhesives.[4,7,21-22] However, recent findings have demonstrated that nanoleakage can occur, despite a simultaneous demineralization and resin infiltration of dentin.[23] Thus, one-step self-etch systems could act as permeable membranes.[24]

Therefore, the objectives of this study were to analyze the shear bond strength of a two-step and two one-step self-etch adhesives, as well as compare an etch-and-rinse adhesive to ground enamel with and without additional phosphoric acid etching. The null-hypothesis set forth is that additional phosphoric acid etching of enamel will not increase the shear bond strength of the investigated self-etch adhesives.

METHODS AND MATERIALS

Preparation of Teeth

Seventy caries-free, freshly extracted human molars were used for this study. After removing any debris, the teeth were stored in a chlorhexidine-solution (0.2%) at room temperature until specimen preparation. The teeth were embedded in acrylate (Acryfix, Struers, Willich, Germany) until the cementum-enamel junction was slightly covered (Figure 1). After setting, the excess acrylate was removed to assure a right angle between

the specimen base and tooth axis. The exposed mesial or distal enamel surfaces were ground flat parallel to the tooth surface using a carbide bur (HW 21, Meisinger, Neuss, Germany) in a custom alignment device (F4 basic, DeguDent, Hanau, Germany). All the teeth were positioned in the alignment device in such a way that the carbide bur had maximum contact with the tooth surface. This approach assured the exposure of a 4 mm diameter enamel area by removing only the superficial enamel layer, while avoiding the exposure of dentin. All specimen surfaces were visually examined for dentin exposure after grinding and phosphoric acid etching. The teeth were then randomly divided into one control and three test groups. All the specimens were numbered to avoid mistakes and each test group was subdivided into two groups.

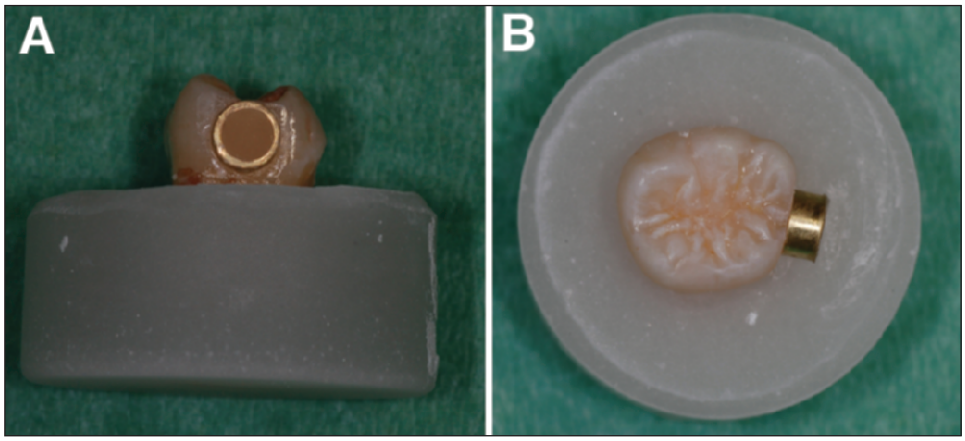

Figure 1. *Lateral (A) and occlusal (B) view of a steel ring attached to the approximal enamel surface.*

Application of Adhesive Systems and Composites

Specifications for all the materials are listed in Table 1.

The enamel surfaces of half the specimens in each test group were etched for 30 seconds with 37% phosphoric acid (DeTrey Conditioner 36, Dentsply, Konstanz, Germany), rinsed with water spray for 30 seconds and air dried until the enamel revealed the typical frosty appearance of acid etching (CPE). The adhesive systems were applied to the etched surfaces according to the manufacturers' instructions. Forced brushing was avoided to preserve the micro-retentive surface.[25] Although the primer applied prior to the adhesive resin was not detrimental to the enamel bond strength, a slight non-significant decrease was detected in previous research.[26] Syntac Classic (SC) is classified as a four-step etch-and-rinse-system. In order to avoid the adverse effects of bond strength caused by application of Primer and Adhesive, only two application steps (phosphoric acid etching and Heliobond) were used for bonding to the enamel surface using SC.

The remaining half of the samples in each test group was treated with adhesive systems without phosphoric acid etching (CSE). The application steps of adhesive systems used within this study are shown in Table 1.

Table 1: *Specifications of the applied materials, manufacturers' instructions for the application of adhesive systems and their classification. In the PE group, additional phosphoric acid etching was performed prior to adhesive application.*

Adhesive System & Charge/LOT	Coding	Application Mode	Classification	Corresponding Composites & Charge Lot	Coding	Manufacturer
Syntac Classic (Heliobond only), H28503	SC	Application of Heliobond, gently air dry, light cure	Smear layer dissolving or smear layer removing/Four-step etch-and-rinse	Tetric Evo Ceram, J02818, H02340, H32750, H31174	TEC	Ivoclar Vivadent, Ellwangen, Germany
Adhese, Primer: G08381, Bond: G06282	ADH	Primer application (30 seconds), gently air dry, bond application, gently air dry, light cure	Smear layer dissolving/ Two-step self-etch	Tetric Evo Ceram, J02818, H02340, H32750, H31174	TEC	Ivoclar Vivadent, Ellwangen, Germany
Xeno III, LOT0511002586	XE	Mixing of components A & B (1:1), application (20 seconds), gently air dry (2 seconds), light cure	Smear layer dissolving/ One-step self-etch	Ceram X mono, 0317	CX	Dentsply, Konstanz, Germany
Futurabond NR, A: LOT590266 B: LOT590267	FNR	Mixing of components (1:1), application (30 seconds), gently air dry (5 seconds), light cure	Smear layer dissolving/One-step self-etch	X-fil, LOT611547	XF	Voco, Cuxhaven, Germany

The adhesive systems were light cured with an LED unit (Bluephase, Ivoclar Vivadent, Ellwangen, Germany) for 10 seconds at 650 mW/cm². The corresponding composites were applied after light curing (Table 1). For composite application, steel rings (inner diameter: 3 mm, outer diameter: 4 mm, height: 2 mm) were filled with composite and placed in the center of the pre-treated enamel surfaces. The composite excess was removed with a thin probe and the mold was covered with a transparent matrix band before light curing the material for 20 seconds at 1100 mW/cm² (Figure 1A and 1B). After each curing cycle, the power output of the LED unit was tested using the integrated testing device to assure identical conditions for all samples.

Table 2: *Shear Bond Strength Values of the Individual Test Groups*

Group	n	Mean (MPa)	SD	p
SC/TEC (Control)	10	29.2	7.3	
FNR/XF (PE)	10	36.1	4.1	<0.05
FNR/XF (SE)	10	24.5	4.9	
ADH/TEC (PE)	8	31.9	4.9	<0.0001
ADH/TEC (SE)	10	18.0	4.1	
XE/CX (PE)	10	29.0	3.9	<0.0001
XE/CX (SE)	10	15.6	4.8	

<0.001

<0.0001

The samples were then stored in distilled water at 37°C for 24 hours until determination of the shear bond strength.

Shear Bond Strength Testing

The specimens were transferred from water storage and mounted in a computer-controlled universal testing machine (Type 20 K, Firma UTS, Ulm, Germany). The specimens were loaded at a crosshead speed of 1 mm/minute until fracture. The shear bond strengths were determined by dividing the maximum load by the covered enamel area (diameter 3 mm).

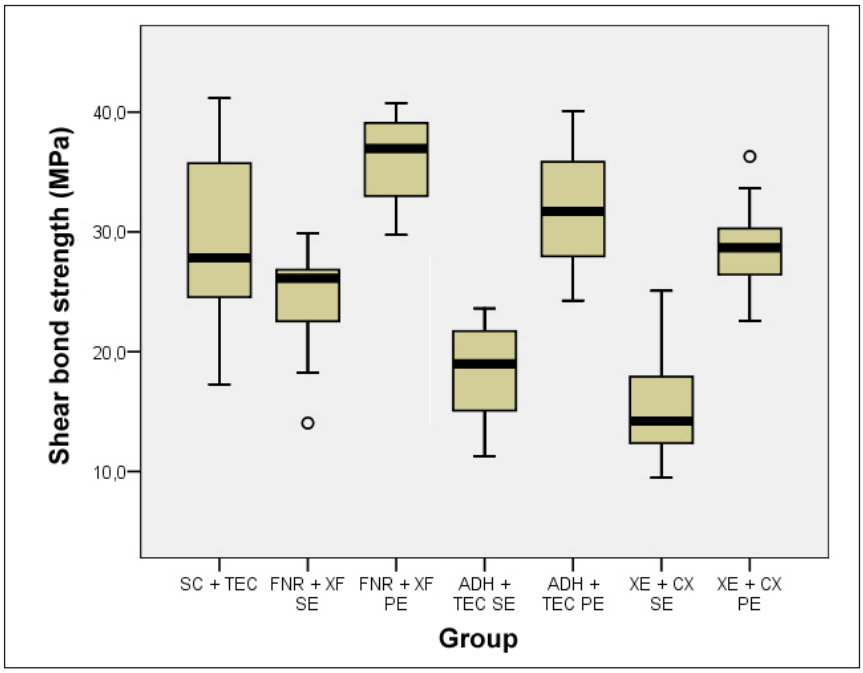

Figure 2. *Box plots showing mean values and standard deviations of the individual experimental groups.*

SEM Analysis

The specimens were mounted on aluminum plates and sputter-coated with Au-Pd (Balzers SCD 004, Oestrich-Winkel, Germany). The fractured surfaces of the specimens were examined using a SEM (S-2300, Hitachi, Tokyo, Japan) at 30x and 400x magnification (voltage: 20 kV).

Statistical Analysis

The mean values for each group were calculated and compared using two-way ANOVA and two-sided Dunnett Test at a significance level of $p<0.05$. The variables were adhesive system and pre-treatment of the enamel surface.

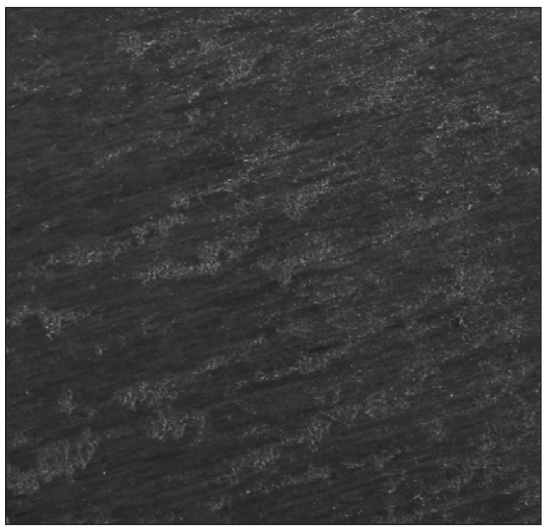

Figure 3. *SEM-Analysis of the enamel surface showing an adhesive fracture (magnification 20x).*

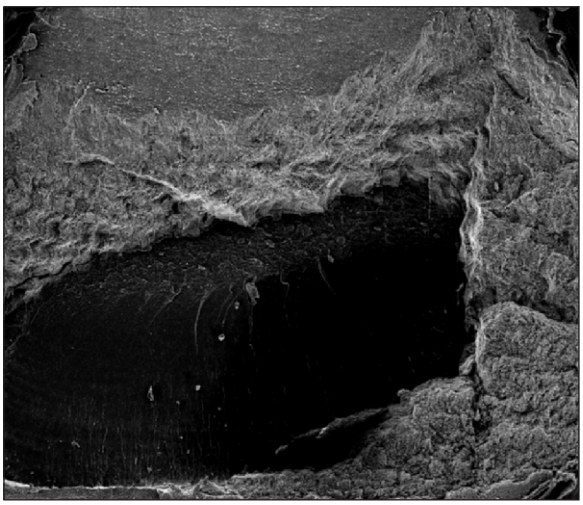

Figure 4. *SEM-Analysis of an adhesive-cohesive fracture with partial enamel disruption at the bottom of the image (magnification 20x).*

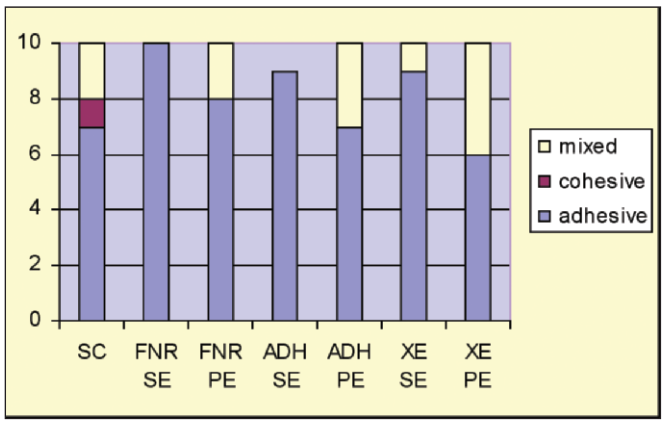

Figure 5. *Distribution of the various fracture types in the individual experimental groups (SE = self-etch adhesive alone; PE = phosphoric acid etching + self-etch adhesive).*

RESULTS

Phosphoric acid etching of the enamel surface prior to application of the adhesive systems led to higher shear bond strengths than self-etch application alone, regardless of the adhesive system used ($p<0.0001$). There were no significant differences between the phosphoric acid etch pre-treatment and the control. However, the application of self-etch adhesives without phosphoric acid etching resulted in shear bond strengths that were lower than the control, which was significant for two of the SE-groups ($p<0.001/0.0001$). Only eight specimens in the ADH/TEC (PE) group were analyzed, as two of the original 10 specimens fractured during mounting to the testing device.

The highest shear bond strength values among all the tested adhesive systems were measured for FNR/XF after additional phosphoric acid etching (36.1 MPa ± 4.1). However, there were no significant differences when compared to the control group SC/TEC (29.2 ± 7.3). All adhesives produced higher shear bond strengths after enamel etching with phosphoric acid when compared to application of the self-etch adhesives alone: FNR/XF 36.1 ± 4.1 vs 24.5 ± 4.9, ADH/TEC 31.9 ± 4.9 vs 18.0 ± 4.1 and XE/CX 29.0 ± 3.9 vs 15.6 ± 4.8. The differences were statistically significant for FNR/XF ($p<0.05$), ADH/TEC ($p<0.0001$) and XE/CX ($p<0.0001$). No statistically significant differences were found for FNR/XF without additional phosphoric acid etching when compared to the control. The lowest shear bond strength was presented by XE/CX used in the self-etching mode when compared to additional phosphoric acid etching and the control ($p<0.0001$). The results are summarized in Table 2 and Figure 2.

When the control was excluded from statistical analysis, the same statistical significance between the groups of phosphoric acid etch pre-treatment and self-etch application alone were found.

SEM evaluation of the fractured surfaces showed mainly adhesive fractures between the enamel surface and the adhesive (Figures 3 and 4). FNR and ADH revealed only adhesive fractures when applied without phosphoric acid etching. After conditioning with phosphoric acid, a slight increase in adhesive-cohesive (mixed) fractures was observed for all self-etch adhesives (Figure 5), which was similar to the control.

DISCUSSION

Self-etch adhesives are a promising development in adhesive dentistry, especially regarding reduction of the necessary application steps and the possibility of a chemical interaction with hydroxy-apatite coated collagen fibers.[21-22] However, bonding to enamel still remains critical and is controversially discussed by various authors.[12-15,17-18]

The mean shear bond strength of the positive control (SC) in the current study was 29.2 MPa. Similar values have been reported when equivalent conditions were applied.[22] Although dentin adhesives should not impair bonding to acid etched enamel, a forced application (rubbing) of adhesives with microbrushes on the etched enamel surface needs to be avoided, because it can cause a reduction in enamel bond strength up to 20% after 24 hours water storage.[25] Additionally, an operator-related technique sensitivity was found, even under standardized experimental conditions.[27] Therefore, the application of adhesives in the current study was performed by one experienced dentist to avoid any operator-related variability. Furthermore, the application of adhesives strictly followed the manufacturers' instructions.

The highest shear bond strength was found with FNR, a one-step self-etch adhesive, although there was no statistically significant difference compared to

the control. One-step self-etch adhesives can produce shear bond strengths to ground enamel that are even higher than those of etch-and-rinse systems. Regarding dentin, total-etch and self-etch-systems performed similarly.[28] Other bond strength studies, however, reported contradictory data. In the contradictory studies, one-step self-etch adhesives usually obtained a lower bond strength than two-step self-etch and three-step etch-and-rinse systems.[6,14] Similar to these studies, the other one-step self-etch system (XE) tested in the current study had significantly lower bond strength values than the positive control (15.6 MPa vs 29.2 MPa). This has to be taken into consideration, because a minimum bond strength of 17 to 20 MPa is required to avoid the formation of gaps at cavity margins and to resist shrinkage forces that occur during polymerization.[29] The specimens were analyzed after 24-hour water storage. Further studies should investigate whether longer periods of water storage would have a similar effect between etch-and-rinse and self-etch adhesives.[30]

When phosphoric acid was applied prior to the self-etch adhesives, a significant increase in shear bond strength values was generated. Various clinical trials also indicated the potential benefit of additional enamel etching with phosphoric acid.[31-36] Enamel margins of Class V cavities showed a significant decrease in marginal leakage after additional enamel etching when using self-etch adhesives when compared to using etch-and-rinse systems.[31] TEM analyses showed that the enamel surface was more micro-retentive, and microtensile bond strengths (μTBS) were significantly increased, for a two-step self-etch adhesive after etching with phosphoric acid.[34] The μTBS of a one-step self-etch adhesive was improved after an additional acid etching step prior to adhesive application to enamel.[35] In dentin, however, the μTBS decreased significantly, and a slight increase in leakage at the dentin margins of Class V cavities due to phosphoric acid etching was found.[31,34-35] Clinical trials demonstrated that, after etching, there were fewer defects and superficial discolorations at the enamel margins in Class V restorations of non-carious lesions after two and three years when a two-step self-etch adhesive was used.[33,36] However, there was no significant influence on retention of the restorations or their clinical performance.[5,33]

The bond strength of self-etch adhesives can be influenced by pre-treatment of the enamel surface before adhesive application.[9,19,37-38] Enamel surfaces in the current study were ground flat with a carbide bur in order to simulate clinical conditions. It should be considered that the μTBS of self-etch adhesives decreased when the enamel surfaces were prepared with diamonds or carbide burs when compared to preparation with silicon carbide paper.[37] This parameter should be considered when comparing bond strength values determined in different studies. On unground enamel, self-etch adhesives showed a lower μTBS when compared to etch-and-rinse systems. Some one-step self-etch systems do not bond to unground enamel.[9,38]

The pH-value of approximately 1.5 of the self-etch systems used in the current study can be classified as "intermediary" strong systems.[15] On ground enamel, a relationship between pH value and the ability to create a microretentive surface has been found.[39] Intermediary-strong adhesives create an irregular, non-homogenous etch-pattern, whereas phosphoric acid removes the enamel smear layer and leads to a honeycomb-structured surface.[19,40] The demineralization depth of the enamel surface was lower for self-etch adhesives compared to the total-etch approach (1.5-3.2 vs 6.9 μm).[10] Because demineralization and resin infiltration occur simultaneously when using self-etch adhesives, dissolved hydroxyapatite crystals and smear layer remnants were incorporated into the polymerized resin layer.[3,5,7] The lower shear bond strength determined for self-etch adhesives without additional phosphoric etching in the current study may be due to lesser demineralization and resin infiltration at the enamel surface, which may have been caused by the inhibition of resin penetration by mineral precipitates.[39]

The failure mode of self-etch adhesives was primarily adhesive when the adhesives were used according to the manufacturers' instructions—without additional phosphoric acid etching.[39]

When phosphoric acid was applied prior to application of self-etch adhesives, an increase in adhesive-cohesive fractures occurred. This could be due to the increased bond strength of self-etch adhesives combined with phosphoric acid etching. At the same time, the incidence of fractures within the superficial enamel layer increased.[32]

Therefore, the null-hypothesis has to be rejected.

CONCLUSIONS

The results of this study show that the enamel shear bond strength of evaluated self-etch adhesives can be significantly increased by additional phosphoric acid etching. Therefore, data from the current study confirms the tested hypothesis.

Within the clinical application, an additional acid etching step could be considered for restorations whose retention primarily depends on a strong bond to the enamel surface, such as large Class IV restorations or restorations with a high C factor.

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