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RESEARCH AND EDUCATION

Influence of pretreating enamel on the durability of resin bonds

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ABSTRACT

Statement of problem. Minimally invasive restorations adhesively bonded to prepared enamel have been well established clinically. However, studies on the durability of noninvasive restorations are still lacking.

Purpose. The purpose of this laboratory study was to evaluate the tensile bond strength (TBS) of a luting resin and its durability to differently pretreated enamel with 2 different etching procedures.

Material and methods. The proximal surfaces of human third molar teeth were embedded in custom-made holders with an autopolymerizing composite resin. The enamel was pretreated according to the protocol of the respective group (n=16): Minimally invasively prepared (M), unprepared (N), and airborne-particle abraded (O); enamel was either etched with phosphoric acid followed by applying a self-etching primer (PT) or conditioned with a self-etching primer only (T). The bonding surfaces of the specimens were perpendicularly luted to acrylic resin tubes filled with a composite resin (Clearfil F II) with a luting resin (Panavia V5). Each of the 6 groups was further divided into 2 subgroups with either 3–days of water storage at 37 °C or 150 days of water storage with additional thermal cycling (37 500 thermal cycles between 5 °C and 55 °C). TBS was evaluated with a universal testing machine. All specimens underwent failure mode analysis. Statistical analysis was performed with a 3-way ANOVA, followed by 2-way ANOVAs and separate 1-way ANOVAs. For pairwise comparisons of groups, the Tukey post hoc test was performed (α=.05 for all tests).

Results. Statistical analysis revealed that the enamel preparation had no significant effect on TBS after artificial aging when etched with phosphoric acid (*P*=.520). Conditioning with a self-etching primer only showed significantly lower TBS than groups etched with phosphoric acid for the same preparation after 150 days (*P*=.014 for minimal preparation, *P*=.028 for no preparation, and *P*<.001 for airborne-particle abrasion), and artificial aging did not significantly affect TBS (*P*=.763). The mean ±standard deviation values ranged from 9.8 ±8.1 MPa (OT) to 36.4 ±7.4 MPa (MPT) after 150–day storage.

Conclusions. Etching of enamel is essential for optimal resin bonding, but the preparation of enamel or artificial aging had no influence on TBS. (J Prosthet Dent 2025;133:1054.e1-e7)

The combination of the adhesive technique[1] and the properties of contemporary restorative materials enables minimally invasive dental treatment options.[2] Clinical studies of minimally invasive restorations bonded to enamel such as resin-bonded fixed dental prostheses (RBFDPs) and labial or occlusal veneers have shown promising results and durable long-term bonding.[3–9]

Etching enamel with 33% to 40% phosphoric acid for about 30 seconds followed by water spray rinsing has been reported to improve the bond strength of resins to enamel.[10–14] Etching prepared enamel with 35% phosphoric acid for 30 seconds has been reported to result in the removal of about 7 µm of enamel, while longer etching durations result in more removal.[15] Phosphoric acid etching dissolves enamel prisms and interprismatic enamel leading to a microretentive etching pattern and an increase in the surface, its reactivity, and its wettability,[13,16] and high bond strengths of approximately 30 MPa can be obtained.[16] The self-etch systems can involve 2 steps or a single

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Clinical Implications

The findings of this study are promising with regard to less or noninvasive clinical treatment approaches, as artificial aging and different preparation protocols of phosphoric acid-etched enamel, including no preparation, airborne-particle abrasion, and minimally invasive preparation, did not affect the tensile bond strength of the luting resin. Conditioning the enamel with a self-etching primer only appears to decrease the tensile bond strength compared with phosphoric acid-etched enamel and is therefore not recommended unless for bonding interim restorations to enamel.

step, depending on how the acid primer and adhesive resin are provided by the manufacturer.[17] Self-etching systems have been reported to lead to lower enamel bond strength compared with an etch-and-rinse system.[18] However, etching enamel with phosphoric acid before the application of self-etching systems has been reported to improve the bond strength to enamel so that the bond strength values were comparable with those achieved with etch-and rinse systems.[19–21]

Removal of the superficial aprismatic layer of enamel by minimally preparing the bonding surface with a diamond rotary instrument and etching the prepared enamel with phosphoric acid before bonding is a well-established minimally invasive treatment.[22,23] As the preparation is limited to the enamel or defect-oriented, resulting in tooth structure removal of approximately 3% to 30% for ceramic veneers and resin-bonded prosthesis retainers, this procedure represents a minimally invasive treatment approach.[24] However, even less invasive or noninvasive treatment approaches allow maximum preservation of tooth structure and the benefit of complete reversibility should removal and reintervention be indicated. Concerns exist, however, about their predictability and durable long-term bonding to aprismatic enamel, as this superficial, irregular enamel layer is composed of inorganic substances, contains a form of hydroxyapatite not organized into prisms,[25,26] and does not appear to be evenly or easily etchable.[27,28] Noninvasive treatments such as bonding to aprismatic unprepared enamel for fissure sealing or for attaching orthodontic brackets have been widely applied.[29–32] Furthermore, veneers without preparation have been advocated,[33,34] for which the unprepared enamel is typically airborne-particle abraded (APA) before bonding.[35–38] The combination of APA followed by phosphoric acid etching has been reported to lead to even higher bond strength on aprismatic enamel than enamel preparation with a diamond rotary instrument.[39] However, the effect of the enamel preparation and the etching protocol on the bond strength still appears controversial, with studies showing lower bond strengths for intact enamel than for prepared enamel when using a self-etching primer[40] and studies reporting no differences between unprepared and prepared enamel, irrespective of the etching system.[41–43] Laboratory studies evaluating the bond strength to enamel should aim to simulate clinical conditions by conducting artificial aging,[44–47] but most studies reported bond strength values only 24 hours or 3 days after bonding.

This laboratory study, therefore, was designed to evaluate the durability of the bond strength of a luting resin to unprepared, minimally invasively prepared, and airborne-particle abraded enamel with 2 etching procedures, a self-etching primer or phosphoric acid followed by a self-etching primer and to analyze the failure mode. The null hypotheses were that enamel preparation, the etching procedure, and artificial aging would not significantly influence TBS.

MATERIAL AND METHODS

Fifty-eight extracted human third molars without caries or macroscopically visible fractures were collected after patients had provided their written consent. After the removal of soft tissue remnants, the teeth were stored in 0.1% thymol solution (Thymol; Carl Roth GmbH) and, 2 weeks before testing, in distilled water at room temperature. All teeth were fixed in a custom-made apparatus to remove the roots and to bisect the crowns with a precision sectioning machine (IsoMet 1000; Buehler) at 8000 rpm under water cooling. A total of 96 proximal surfaces of tooth crowns with a minimum thickness of 3 mm were collected for TBS testing after the exclusion of teeth with deviating curvatures or fractures after bisection. The flattest surface was defined as the bonding area. Each specimen was then aligned horizontally on a glass plate with prewarmed modeling plate wax (Pluline; Dentalbauer). A custom-made brass cylindrical holder (13 mm in inner diameter and 7 mm in height) provided with a circular groove for retention was placed over each specimen and filled with an autopolymerizing composite resin (Luxatemp; DMG). The specimens were divided into 3 groups (n=32): minimal invasive preparation, no preparation, and APA, each in combination with 2 etching methods with or without 35% phosphoric acid. A total of 96 acrylic resin tubes (17 mm in length and 3.2 mm in inner diameter) were filled with an autopolymerizing composite resin (Clearfil F II; Kuraray Noritake Dental Inc), which was allowed to autopolymerize for 7 minutes. During the polymerization, the specimens were pretreated according to the protocol of the respective group (Table 1). For the MPT and MT groups with the minimally invasive enamel preparation, the bonding

Table 1. Groups listed with differently pretreated enamel

Group	Treatment Protocol
MPT	Minimally invasive preparation; cleaning with sodium bicarbonate powder; etching with 35% phosphoric acid; applying self-etching Tooth Primer
MT	Minimally invasive preparation; cleaning with sodium bicarbonate powder; applying self-etching Tooth Primer
NPT	No preparation; cleaning with sodium bicarbonate powder; etching with 35% phosphoric acid; applying self-etching Tooth Primer
NT	No preparation; cleaning with sodium bicarbonate powder; applying Tooth Primer
OPT	Airborne-particle abrasion with Orthoprophy SA–85; etching with 35% phosphoric acid; applying self-etching Tooth Primer
OT	Airborne-particle abrasion with Orthoprophy SA–85; applying self-etching Tooth Primer

surfaces of the specimens were color-marked (Schneider Maxx 240; Schneider) and prepared with a diamond rotary instrument (8878K.014; Komet Dental, Gebr. Brasseler GmbH) with a grit size of 46 µm and a rotation speed of 30 000 rpm under water cooling with a handpiece (Expertmatic; KaVo Dental) connected to a portable dental unit (Dentalone; NSK). For each group, a new diamond rotary instrument was used. The preparation straightened the tooth surface curvature slightly until the color had been completely removed. The specimens were cleaned with a prophylaxis powder-jet device (Airflow Handy 3.0; E.M.S.) with sodium bicarbonate powder (KaVo Prophyflex Powder; KaVo), rinsed with water spray for 30 seconds, and dried with oil- and water-free air. In the groups with no diamond rotary instrument preparation (NPT, NT), the specimens were also cleaned with sodium bicarbonate powder spray as in groups MPT and MT. In groups OPT and OT, the specimens were airborne-particle abraded for 20 seconds with an intraoral device (Micoetcher IIa; Danville Materials, Zest Dental Solutions) with 85–µm rounded alumina particles (Orthoprophy SA-85; Danville Materials). A custom-made spacer ensured a standardized distance of 3 mm between the device tip and the bonding surface. The APA was performed with a horizontal and then vertical movement (chess-board like movement) with a pressure of 0.25 MPa followed by water spray rinsing for 30 seconds and drying with water- and oil-free air. After the respective preparation protocol, each group was divided into 2 groups (n=16), and the bonding surface of the specimens were either etched with 35% phosphoric acid (K-Etchant Syringe; Kuraray Noritake Dental Inc) for 30 seconds, rinsed with water spray for 30 seconds, and dried with oil- and water-free air followed by the application of a self-etching primer (Tooth Primer; Kuraray Noritake Dental Inc) for 20 seconds (MPT, NPT, OPT), or they were conditioned with the Tooth Primer only (MT, NT, OT). The acrylic resin tubes were luted perpendicularly to the bonding surfaces with a dual-polymerizing luting resin (Panavia V5 opaque; Kuraray Noritake Dental Inc) in a bonding apparatus with a load of 7.4 N (Fig. 1). The luting resin

Figure 1. Bonding apparatus used for luting acrylic resin tubes to bonding surface of specimens.

was light polymerized (radii-cal; SDI) for 5 seconds from each side. The excess was removed with an explorer (XP23/12; Hu-Friedy Mfg. Co LCC) along the edge of the acrylic resin tube, followed by light polymerization (radii-cal) from all sides for 20 seconds, and allowed to autopolymerize for 5 minutes before storage in water. The bonded specimens were stored in water in an incubator (INCU-Line; VWR International LCC) at 37 °C. Each group was then further divided into 2 subgroups (n=8) and was either stored in water for 3 days at 37 °C or for 150 days and then thermocycled (THE-1100; SD Mechatronik) with 37 500 thermal cycles (7500 cycles per month) between 5 °C and 55 °C and a dwell time of 30 seconds[48,49] After the storage period, the bond strength of the luting composite resin to the differently pretreated enamel surfaces was evaluated with a tensile bond strength test (TBS) in a universal testing machine with a chain loop alignment (Zwick Z010; ZwickRoell) at a cross-head speed of 2 mm/minute (Fig. 2).

The fractured interfaces of all debonded specimens were examined visually with a light microscope (Wild M420;Wild Heerbrugg AG) at ×25 magnification. The failure modes of the groups with 150–day storage time were classified as adhesive failure, cohesive failure in the luting or tube filling resin, or enamel fracture. The type of enamel failure was further classified into no enamel fracture (0%), enamel fracture less than half or half of the debonded interface (1% to 50%), enamel fracture over half of the debonded interface (51% to 99%), and complete enamel fracture at the debonded interface (100%). Representative images of failure modes were made with a scanning electron microscope (SEM) (XL 30CP; Philips) after sputtering a 10–nm gold alloy layer (Leica EM QSG 100; Leica Microsystems GmbH) on replicas (Stycast 1266 PTB; Henke).

The data were evaluated with a software program (IBM SPSS Statistics, v20.0 for Windows; IBM Corp). The Shapiro-Wilk test was performed, which revealed

Figure 2. Bonded specimen in universal testing machine.

that data were normally distributed with the exception of OT after 150 days (*P*=.002) and OPT after 150 days (*P*=.018). The homogeneity of the variances was tested with the Levene test, which was not significant (*P*=.800). Since the deviation from normal distribution affected only 2 subgroups, and, considering that the study design was well balanced, statistical analysis was performed with a 3-way ANOVA and with separate 2-way ANOVAs followed by separate 1-way ANOVAs. For the pairwise comparisons of groups, the Tukey post hoc test was performed (α=.05 for all tests).

RESULTS

The 3-way ANOVA test revealed a significant interaction (*P*=.046) between the 2 variables enamel preparation and etching. The other interactions were not statistically significant (enamel preparation×aging: *P*=.480, etching×aging: *P*=.173, preparation×aging×etching: *P*=.776).

The mean ±standard deviation TBS values ranged from 16.4 ±5.5 MPa (OT) to 32 ±8.8 MPa (MPT) after 3 days and from 9.8 ±8.1 MPa (OT) to 36.4 ±7.4 MPa (MPT) after 150–day storage (Table 2).

The enamel preparation significantly affected the resulting TBS, with group OT showing significantly lower TBS values in comparison with groups MT and NT after 150 days (*P*<.05). The preparation protocol did not affect the TBS for groups that were etched with phosphoric acid (MPT, NPT, and OPT). Etching enamel with phosphoric acid significantly increased TBS compared with the application of the self-etching primer only, independent of the enamel preparation protocol for subgroups with 150–day storage time (*P*<.05). However, artificial aging had no significant influence on the TBS values (*P*>.05).

Table 2. Tensile bond strength (TBS) with means ±standard deviation (SD) for all test groups

Group	TBS (MPa)	
	3 Days	150 Days
	Mean ±SD	Mean ±SD
MPT	32.0 ±8.8$^{\alpha Aa}$	36.4 ±7.4$^{\alpha Aa}$
MT	27.4 ±5.6$^{\alpha Aa}$	25.1 ±8.6$^{\alpha Ba}$
NPT	30.7 ±11.3$^{\alpha Aa}$	32.2 ±8.1$^{\alpha Aa}$
NT	22.7 ±8.9$^{\alpha\beta Aa}$	23.0 ±7.0$^{\alpha Ba}$
OPT	30.7 ±10.6$^{\alpha Aa}$	30.3 ±9.1$^{\alpha Aa}$
OT	16.4 ±5.5$^{\beta Ba}$	9.8 ±8.1$^{\beta Ba}$

Different superscript uppercase letters indicate significant differences within each column for same preparation protocol (*P*≤.05).

Different Greek letters indicate significant differences within each column for same etching procedure (*P*≤.05).

Same superscript lowercase letters indicate no significant differences within each row (*P*>.05).

The prevalence of failure modes for all groups after 150 days is shown in Figure 3. The failure mode was predominantly cohesive for groups MPT, MT, NPT, and NT. A dominant adhesive failure mode was observed for group OT. An adhesive mode of failure was mainly observed in groups where the enamel was treated with the self-etching primer only. Enamel fractures were observed in all groups, except for group OT. Higher percentages of enamel fracture were observed in groups that were etched with phosphoric acid. The proportion of enamel fractures of the debonded surfaces is shown in

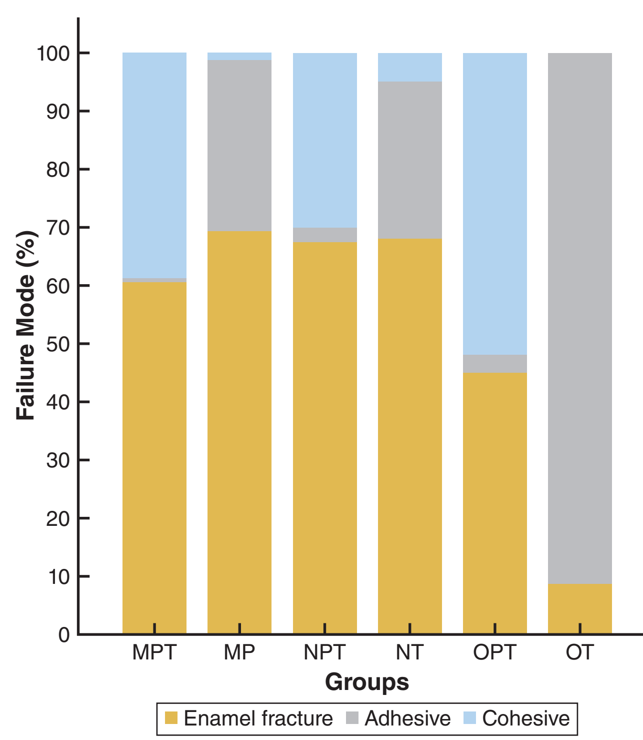

Figure 3. Bar chart of prevalence of failure modes (in percent) of groups with 150-day storage after tensile bond strength testing.

Table 3. Proportion of enamel fractures of debonded interfaces for all groups after 150–day storage with number of specimens and percentage

Group	Proportion of Enamel Fractures of Debonded Interface			
	0%	1% to 50%	51% to 99%	100%
MPT	4 (50.0%)	1 (12.5%)	1 (12.5%)	2 (25.0%)
MT	7 (87.5%)	1 (12.5%)	-	-
NPT	5 (62.5%)	1 (12.5%)	1 (12.5%)	1 (12.5%)
NT	7 (87.5%)	1 (12.5%)	-	-
OPT	3 (37.5%)	-	5 (62.5%)	-
OT	8 (100.0%)	-	-	-

Table 3. Representative SEM images of adhesive and cohesive failure modes are shown in Figure 4.

DISCUSSION

The experimental design and procedure were mainly based on studies which also investigated the TBS and the effect of artificial aging.[48,49] The proximal surfaces of third molars were used following the methodology of other studies evaluating bond strengths to unprepared enamel.[42,43] The luting resin Panavia V5 was tested as it has been shown to have clinically promising results for nonretentive minimally invasive restorations bonded to enamel.[4,50] A custom-made bonding apparatus was used for the luting procedure to ensure that the tubes were perpendicular to the bonding surfaces.[51] The specimens were stored in water and thermocycled as artificial aging to evaluate the long-term durability of the adhesive bonds.[48] A TBS test in a universal testing machine was conducted to evaluate the bond strength of composite resin.[52] To enable a moment-free axial application of the tensile force, a chain loop alignment was used during the TBS test.[48]

The null hypothesis that the enamel preparation would have no significant effect on the TBS was not rejected for the groups that were etched with phosphoric acid, as they did not show significant differences (P>.05). However, this null hypothesis was rejected for the groups that were conditioned with the self-etching primer only, as there were statistical differences with significantly lower TBS for groups OT and NT compared with group MT (P<.05). These findings were consistent with those of a study[40] that reported no statistically significantly differences in bond strength between prepared and unprepared enamel when etching with phosphoric acid but significantly lower bond strengths to unprepared enamel when conditioning with self-etching primer compared with prepared enamel after 24-hour storage. The lower bond strength to enamel that was airborne-particle abraded with Orthoprophy SA-85 (85 µm), group OT, could be explained by the absence of cleaning with sodium bicarbonate powder (60 to 70 µm) in those groups. Orthoprophy SA-85 might not remove contaminants adequately or remnants of powder might have affected the bond strength. Another explanation for the differences in the bond strength values might be the differences in surface roughness. Surface roughness was not investigated in the present study, but it has been reported that a 20-second APA with Orthoprophy SA-85 on bovine enamel resulted in a surface roughness with a mean value of R_a=0.46 µm and an enamel volume loss of 0.01 mm^3.[53] However, a surface roughness with a mean value of R_a=1.4 µm was reported for human enamel when cleaning with sodium bicarbonate powder for 10 seconds (with 0.4 MPa at 5–mm distance to the enamel surface).[54] Despite different methods, sodium bicarbonate appears to lead to a higher surface roughness than Orthoprophy SA-85. The results, however, were not consistent with the finding of another study[39] that reported significantly higher shear bond strength of 42 MPa for the etch-and-rinse method and 34 MPa for a self-etching system for airborne-particle abraded enamel after 24 hours compared with enamel which received no pretreatment, sodium bicarbonate or glycine air polishing, or a minimally invasive preparation. This difference could have been caused by the different pressure, distance to the bonding surface, type of powder, or shorter storage interval before testing. Regarding the powder, APA with 50–µm sharp-edged

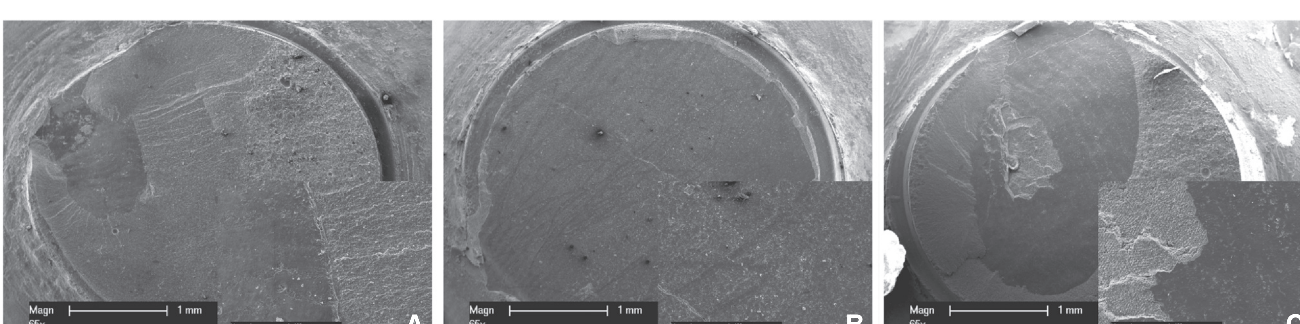

Figure 4. Representative scanning electron microscope images. A, Predominantly cohesive failure mode. B, Adhesive failure mode. C, Debonded interface with 1% to 50% enamel fracture. Original magnification ×65 and ×500.

alumina particles has been shown to be considerably more abrasive to enamel than 85–µm round-shaped alumina particles, as used in the current study.[53]

The null hypothesis that the etching method would have no significant influence on the TBS was rejected for all groups after 150 days of artificial aging, with significantly lower TBS values for groups treated with a self-etching primer only (*P*<.05). When treating enamel with a self-etching primer, the etching pattern of the self-etching primer might not be deep enough to obtain sufficient penetration for bonding resin, which might explain the lower TBS for those groups. This effect has been observed in field-emission scanning electron microscopy.[40,55] Irrespective of the enamel pretreatment, another study[39] also reported significantly lower shear bond strength values in groups conditioned with a self-etching system than in groups etched with phosphoric acid, being consistent with the results of the 150–day storage groups of the present study. In contrast, some studies[41,43] reported no significant differences between differently pretreated enamel for different adhesives systems tested after 24 hours, which, in turn, is comparable with the results of the present study regarding the short-term storage of the groups with minimally invasive preparation and no preparation. However, because of different study designs and methods, the comparison is limited to those studies.

The null hypothesis that artificial aging would not significantly influence TBS was not rejected, as artificial aging with thermocycling did not significantly affect the durability of TBS for the enamel pretreatments and etching procedures tested (*P*>.05). The luting resin tested seems to have sufficient resistance against hydrolysis and thermocycling during the tested time interval. Also, in other studies[49,56] no significant bond strength differences were reported with the same luting resin. However, bond strength to dentin was investigated[49] or the thermocycling method differed from the present study.[56] However, thermocycling with prolonged water storage is a common method of simulating thermal stress and water absorption at the bonding interface to simulate clinical conditions. Adequate artificial aging should ensure complete water saturation of the bonding interfaces for months.[44–47]

The type of failure observed in this study was predominantly cohesive within the luting composite resin for unprepared and minimally invasively prepared enamel. This contradicts the results of another study[39] where the failure mode was predominantly adhesive, irrespective of the enamel treatments and adhesives. However, a shear bond strength test was applied rather than a TBS in the aforementioned study, which might explain the contradictory findings. A cohesive failure mode is to be considered as positive from a clinical perspective, as this type of failure keeps the teeth and hard tissues intact, preventing fractures, and allows rebonding of the debonded restoration after removal of luting composite resins. The results of this study were consistent with those of another study[40] that reported predominantly cohesive failure modes after TBS testing, whereas adhesive failures were found for unprepared enamel that was not etched with phosphoric acid; this most closely corresponds to group OT of the current study. However, enamel fractures, considered the most catastrophic type of failure, were also observed, particularly in groups etched with phosphoric acid.

Limitations of this study included that the specimens could not be standardized because of the natural tooth curvature, the relatively low numbers of specimens, the absence of other oral environmental conditions such as masticatory stress or low-pH challenge and that mechanical fatigue was not tested. Future research on testing different powders and etching times and analyzing the enamel surface and morphology after different enamel treatments and etching procedures is recommended. Further research is also needed on restorations that are to be bonded to enamel with less invasive or noninvasive techniques, as the clinical performance and longevity of those treatment approaches are still unclear.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The preparation protocol for phosphoric acid-etched enamel did not affect the TBS of the luting resin.
2. The use of phosphoric acid on enamel increased the TBS compared with the application of a self-etching primer only.
3. Artificial aging did not affect the TBS of the luting resin.

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