

Influence of Different Primer Application Times On Bond Strength of Self-etching Adhesive Systems to Unground Enamel

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Clinical Relevance

An increase in application time of acid primer does not improve the bonding of self-etching adhesive systems to unground enamel.

SUMMARY

This study evaluated the influence of increasing the application time of acid primer on the bond strength of one- and two-step self-etching systems to unground enamel. Thirty-two human third molars were used in this study. Additionally, four self-etching adhesive systems: Clearfil SE Bond (Kuraray), AdheSE (Ivoclar-Vivadent), Futurabond NR (Voco) and One Up Bond F Plus (J Morita) were used in two conditions according to each manufacturer's recommendations and using double the application time of the primer recommended by the manufacturers. The teeth were randomly separated into groups and sectioned in their central region

in the buccal-lingual direction perpendicular to their long axes, using a double-faced diamond disk. A 6-mm high block was then made with Rok (SDI) resin composite on the mesial and distal faces of each tooth. The samples were then serially sectioned from the resin composite in the occlusal-gingival and buccal-lingual directions at a distance of 1 mm between cuts using a high concentration diamond disk adapted to a precision cutter. The microtensile test was performed in a universal test machine at a speed of 0.5 mm/minute. The fractured specimens were analyzed by scanning electronic microscopy to determine failure modes. The data obtained were submitted to ANOVA and the Tukey Kramer tests. There was no statistically significant difference among the adhesive systems and primer application times. Failure modes varied among the groups and were influenced by the increase in acid primer application time.

INTRODUCTION

The goals of acid etching enamel are to clean dental structure, remove the smear layer, microscopically increase the roughness by removing the prismatic and interprismatic crystals and increase the free surface

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energy to produce sufficient monomer infiltration, seal the surface with adhesive and contribute to the retention of resin composite restorations.¹

It is known that the presence of nanometric-sized spaces at the base of the hybrid layer can be produced by the lack of resin penetration throughout the demineralized layer thickness or by the removal of poorly polymerized resin by oral or dentinal fluids. For this reason, performing acid etching, together with the adhesive system primer, for demineralization and resinous penetration to occur simultaneously, the use of self-etching primers was proposed.²⁻³

The bond strength of adhesive systems to enamel etched with acid attains values of more than 40 MPa,⁴ characterizing a highly reliable bond. However, self-etching adhesives have weaker acids, with a higher pH than that of phosphoric acid, and they are not as effective for demineralizing enamel when compared with conventional adhesives.⁵

Conventional adhesive systems show a deep interprismatic acid etching pattern on enamel, and the pattern for the self-etching adhesive system ranges from absent to moderate.⁶ However, there is no correlation between deep acid attack and bond strength between resin and enamel.⁷ The degree of acid attack of self-etching systems on enamel appears to be minimal, despite the bond strength to enamel being acceptable.⁸⁻⁹

The performance of self-etching systems with regard to bond strength to enamel and clinical marginal degradation is inferior when compared with conventional systems.¹⁰⁻¹² Some authors have suggested techniques that recommend the use of 37% phosphoric acid on enamel before using self-etching adhesives to increase their retentive strength,¹³⁻¹⁶ which could result in self-etching systems being clinically impractical.

Other authors have suggested increasing the time of enamel etching with the acid primer of self-etching adhesives on prepared enamel, which could increase the bond strength and retain the practicality of the self-etching adhesive application by eliminating the clinical steps of washing the acid and drying the teeth.¹⁷⁻¹⁸

To assess the performance of self-etching systems on enamel, the current study evaluated the influence of increasing the application time of the acid primer on the bond strength to unground enamel.

METHODS AND MATERIALS

Thirty-two human third molars were used in this study (n=4). After extraction, the teeth were cleaned with a water slurry of pumice flour in a rubber prophylaxis cup at low speed and stored in distilled water at room temperature to prevent dehydration.

The mesial and distal faces of the specimens were used for bonding to unground enamel. The teeth were randomly separated and sectioned in their central region in the buccal-lingual direction parallel to their long axes using a double-faced diamond disk (Microdont Micro Usinagem de Precisão Ltda, São Paulo, SP, Brazil) at low speed under water/air cooling.

The self-etching adhesive systems were applied on the enamel surface in accordance with each manufacturer's recommendations and also applied at double the length of the primer application time recommended by the manufacturer. The commercial brand name, basic composition, pH, manufacturers, method of use and lot numbers of the adhesive systems used in this study are listed in Table 1.

After application of the adhesive systems, a 6 mm high resin composite block (Rok—Lot #031156/SDI, Melbourne, Victoria, Australia) was prepared. The resin was placed in three increments, each of which was individually light polymerized for 40 seconds with a LED Radii/SDI appliance at 1.500 mW/cm² power and periodically calibrated using the radiometer within the appliance. The samples were stored in distilled water at 37°C ± 1°C for 24 hours.

The samples were then serially sectioned from the resin composite in the occlusal-lingual and buccal-lingual directions at a 1 mm distance between the cuts using a high concentration diamond disk in a precision cutter (Isomet 1000, Buehler, Lake Bluff, IL, USA). The specimens consisted of resin composite bonded to unground enamel (on the mesial and distal faces) in the form of beams. For each tooth, a minimum of four beams was obtained. With the aid of a cyanoacrylate-based adhesive (Super Bonder Gel, Henkel Loctite Adhesives Ltda, Itapevi, São Paulo, Brazil), the ends of the specimens were fixed to the grips of a microtensile device coupled to the universal test machine DL 2000 (EMIC Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, PR, Brazil).

The tensile bond strength was performed at 0.5 mm/minute until the sample ruptured. After the test, the specimen was carefully removed from the device with a scalpel blade and the fracture region area was measured to approximately 0.01 mm with a digital pachymeter (Starret 727-6/150, Itu SP/Brazil) to calculate the final shear force expressed in MPa.

After the microtensile test, the enamel portions were separated and fixed to aluminum stubs (Procind Ltda, Piracicaba, São Paulo, Brazil) with the fractured interfaces facing upward, metalized (SCD 050 Sputter Coater, Baltec) and evaluated by scanning electronic microscopy (JEOL, JSM-5900LV scanning electronic microscope, Tokyo, Japan) to determine the failure modes (adaptation of the model described by Montes and others¹⁹ and Tanumiharja and others): Type 1,

Table 1: Bonding Systems Used			
System Manufacturer Batch #	Composition (main components)	Bonding Steps	pH
Clearfil SE Bond (Kuraray) Primer: 00616A Bond: 00872A	Primer: MDP, HEMA, hydrophilic dimethacrylate, CQ, N,N-Diethanol p-toluidine, water Bond: MDP, BisGMA, HEMA, hydrophobic dimethacrylate, CQ, N, N-Dietanol p-toluidine, Silanate colloidal silica	Apply a layer of primer, wait 20 seconds, dry with a light jet of air, apply the adhesive, remove the excess with a light jet of air and light polymerize for 10 seconds.	2.1
AdheSE (Ivoclar-Vivadent) Primer: J00781 Bond: J03128	Primer: phosphoric acid acrylate, bis-acrylic acid amide, water, initiators, stabilizers Bond: dimethacrylate, hydroxyl ethyl methacrylate, highly dispersed silicon dioxide, initiators, stabilizers	Apply a layer of primer, wait 30 seconds, disperse the excess with a strong jet of air, apply the adhesive, remove the excess with a light jet of air and light polymerize for 10 seconds.	1.7
Futurabond NR (Voco) Liquid A: 620683 Liquid B: 620684	BIS-GMA, hydroxyl ethyl methacrylate, BHT, ethanol, organic acids, fluorides Liquid A–Methacryloyloxyalkyl acid phosphate (phosphoric acid monomer),	Dispense one drop of each agent into the mixing capsule, mix the two agents, apply the mixture to the dental structure for 20 seconds, dry and light polymerize for 10 seconds.	1.4
One Up Bond F Plus (J Morita) Liquid A: 036M Liquid B: 530M	11-Methacryloxy-1,1-undecanedicarboxylic acid (MAC-10), Methyl methacrylate Liquid B–2-Hidroxyethyl methacrylate, L Methyl methacrylate	Dispense one drop of each agent into the mixing capsule, mix the two agents until a homogeneous pink color is obtained, apply the mixture to the dental structure, wait 20 seconds, do not remove the excess and light polymerize for 10 seconds or longer to guarantee color change from pink to colorless.	0.8

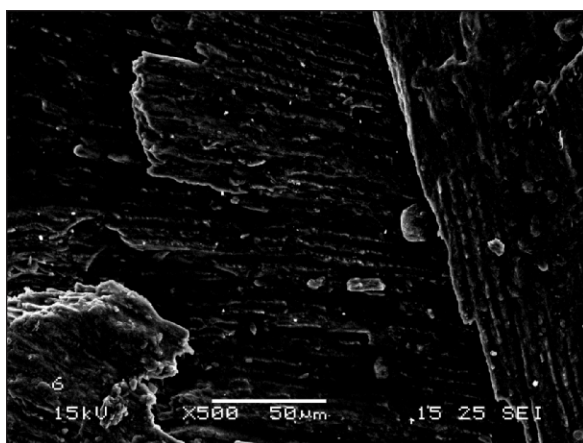


Figure 1. Demonstrates adhesive failure, fracture mode Type 1.

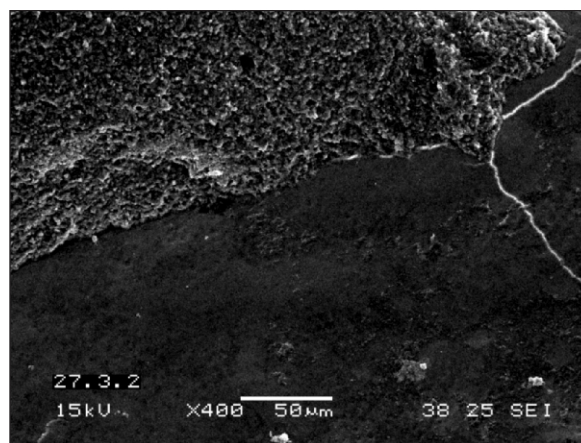


Figure 2. Demonstrates partial adhesive failure and partial cohesive failure in adhesive, Fracture mode Type 2.

adhesive failure between the adhesive and enamel (Figure 1); Type 2, partial adhesive failure between the adhesive system and enamel, and partial cohesive failure in the adhesive (Figure 2); Type 3, completely cohesive in the adhesive system (Figure 3); Type 4, partially cohesive in enamel; Type 5, partially cohesive resin composite.¹⁹⁻²⁰

The data obtained were tabulated and submitted to Analysis of Variance and the Tukey Kramer tests. The

results of the fracture pattern evaluation were submitted to descriptive statistical analysis.

RESULTS

The results are described in Table 2 and Figure 4. The highest mean bond strength value was obtained by the Clearfil SE Bond system when the primer was applied for double the time recommended by the manufacturer. Among all the adhesive systems, only Futurabond NR

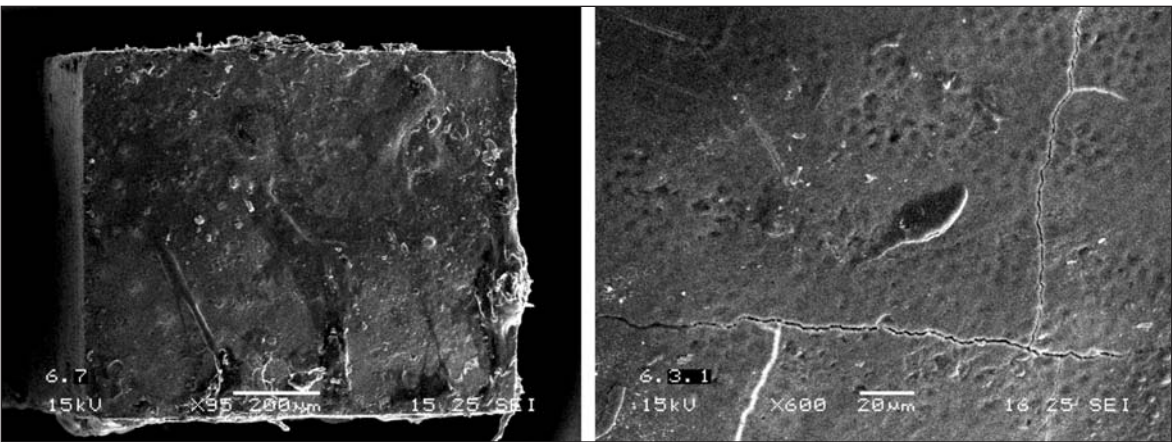


Figure 3. Demonstrates cohesive failure in the adhesive, Fracture mode Type 3.

obtained a lower mean bond strength when the primer was applied for double the time recommended by the manufacturer. There was no statistically significant difference among the groups according to the Analysis of Variance and Tukey Kramer tests ($p=0.3906$, Table 2).

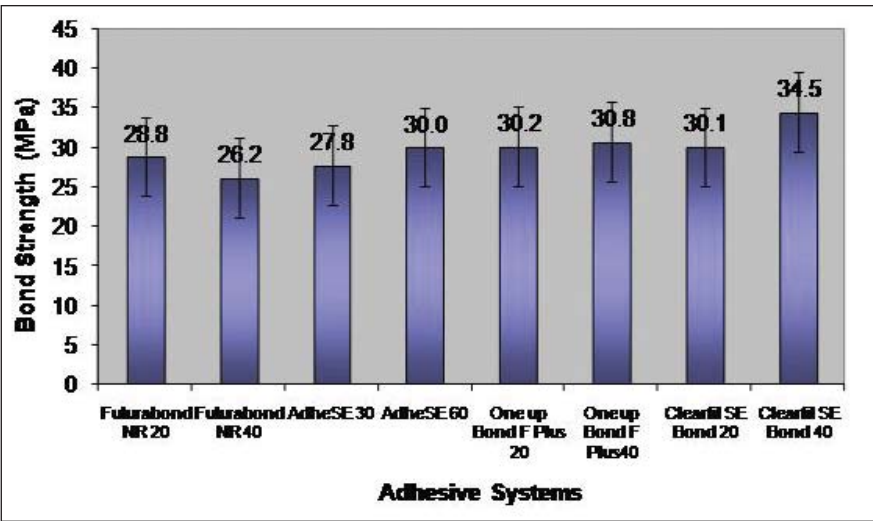


Figure 4. Mean and standard deviation of tensile bond strength as a function of the adhesive systems.

Table 2: Mean and Standard Deviation of Tensile Bond Strength as a Function of the Adhesive System and Primer Application Time			
Adhesives Systems	Primer Application Time	Mean (SD)	Tukey
Futurabond NR	20	28.8 (14.5)	A
	40	26.2 (10.8)	A
AdheSE	30	27.8 (10.5)	A
	60	30.0 (8.6)	A
One Up Bond F	20	30.2 (11.8)	A
	40	30.8 (15.0)	A
Clearfil SE Bond	20	30.1 (16.8)	A
	40	34.5 (16.4)	A

Means followed by the same letter do not differ statistically ($p>0.05$) by the ANOVA and Tukey tests.

However, it was observed that the two-step adhesive systems presented higher mean bond strength values to enamel when they were applied for double the time recommended by the manufacturer.

The data obtained in the fracture pattern

analysis were analyzed by frequencies distribution. The fracture mode analysis of the specimens bonded with one-step application adhesives (Futura Bond and One Up Bond F) demonstrated that, when doubling the primer application time, there was an increase in the percentage of Type 1 fractures, while the percentages of Type 2 fractures remained similar. However, a lower percentage of Type 3 fractures occurred (Table 3 and Figure 5). In the two-step application adhesive systems (Clearfil SE Bond and AdheSE), the percentage of Type 1 fractures diminished with an increase in primer application time, the frequency of Type 2 fractures remained similar and the percentage of Type 3 fractures increased (Table 3 and Figure 5).

DISCUSSION

Enamel debridement by cavity preparation could improve the tissue response to acid etching,²¹ with some studies verifying the bonding of self-etching systems to prepared enamel on the occlusal, lingual and buccal faces.^{10,22-23}

However, it should be taken into account that restorations are commonly extended beyond the margins of the cavity preparations or they are performed without any enamel preparation, such as a number of conser-

Table 3: Distribution and Frequency (%) of the Type of Fracture as a Function of the Group

Group	Type of Fracture			
	1	2	3	4
Futura 20	2 (8.0%)	5 (20.0%)	18 (72.0%)	0 (0.0%)
Futura 40	6 (23.1%)	9 (34.6%)	10 (38.5%)	1 (3.8%)
Adhese 30	5 (27.8%)	10 (55.6%)	2 (11.1%)	1 (5.6%)
Adhese 60	1 (7.7%)	8 (61.5%)	4 (30.8%)	0 (0.0%)
One Up 20	6 (23.1%)	12 (46.2%)	8 (30.8%)	0 (0.0%)
One Up 40	8 (30.8%)	12 (46.2%)	6 (23.1%)	0 (0.0%)
Clearfil 20	5 (22.7%)	9 (40.9%)	7 (31.8%)	1 (4.5%)
Clearfil 40	3 (16.7%)	4 (22.2%)	11 (61.1%)	0 (0.0%)

1-adhesive failure between the adhesive and enamel; 2-partial adhesive failure between the adhesive system and enamel, and partial cohesive in the adhesive; 3-completely cohesive failure in the adhesive system; 4-partially cohesive failure in enamel; 5-partially cohesive resin composite failure.

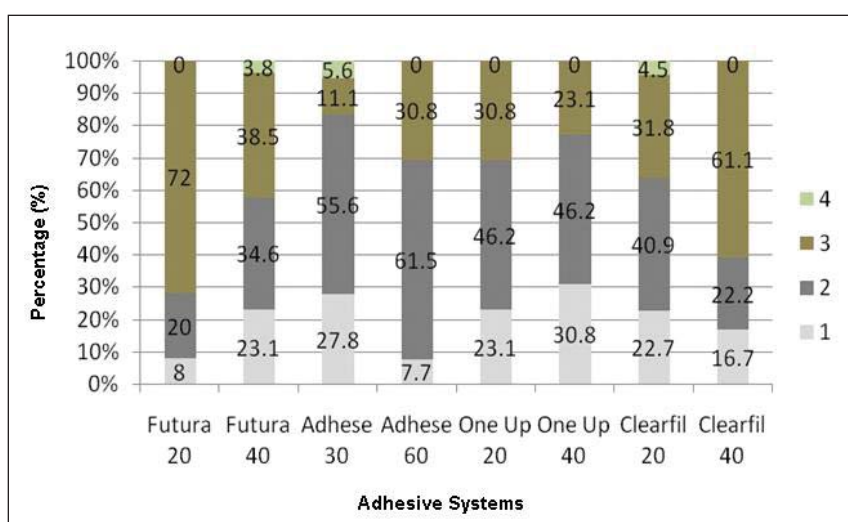


Figure 5. Percentage of fracture modes found in each primer application time.

vative restorative treatments, including diastema closure, tooth recontouring, restoration of fractured teeth, pit and fissure sealing and bonding of orthodontic devices, all performed without tissue instrumentation. Furthermore, important clinical factors, such as the patient's age, environmental factors and the enamel region, could lead to subtle differences in the characteristics of the enamel and influence the ability of acid etching to perform adequate demineralization.²⁴

The current study was conducted using unground enamel on the mesial and distal faces of third molars. The enamel prisms from this dental region are oriented perpendicularly, and it is more difficult for bonding to occur on the sides of enamel prisms (gingival and proximal walls). These factors negatively effect the bonding of conventional total-etch adhesive systems,²⁴⁻²⁵ but this negative effect does not occur with self-etching systems, which are less influenced by orientation of the enamel prismatic structure. Therefore, proximal walls etched with phosphoric acid offer no additional benefit

over a self-etching adhesive.²⁵⁻²⁶

In the current study, one- and two-step adhesives with different pHs were used. The two-step systems consist of an aqueous solution of hydrophilic primer and a hydrophobic adhesive resin applied separately; the one-step systems are complex mixtures

of more acid hydrophilic and hydrophobic components, thus reacting in a different manner to the humid medium.²⁷

With regard to their acidity, adhesive systems can be classified as weak ($\text{pH} \geq 2$), moderate (pH between 1 and 2) and aggressive ($\text{pH} \leq 1$).²⁷⁻²⁸ The more acidic one-step self-etching systems have a more aggressive effect and generate a greater increase in the surface energy of unground enamel; the more hydrophilic resins of these adhesives are capable of penetrating deeper into the etched enamel and producing a well-defined hybrid layer. This well-defined layer does not occur with moderate adhesive systems that produce a hybrid layer with less adhesive penetration into the enamel.²⁹ However, in a one-year clinical study, a two-step adhesive exhibited better retention at the margins of cervical

restorations without preparation, than a one-step adhesive.³⁰ The bond strength produced by less acidic adhesives is not much lower than that of adhesive with previous phosphoric acid application (total-etch adhesives).²⁹ However, all-in-one adhesives seem to be less reliable than two-step self-etching primer adhesives when bonding to enamel.³¹

The difference in the performance of self-etching primers cannot be explained solely by the differences in pH. The demineralization power also depends on various factors: pKa (acid dissociation constant), the structure of the primer components (which may be more or less chelating), the solubility of the salts formed and the application time; therefore, the action does not solely depend on the acid monomers, but also on their concentration.³²

In an attempt to test whether there was improvement in bond strength to enamel with one- and two-step self-etching adhesives with different pHs, the

application of acid primer was performed in accordance with the manufacturers' recommendations and also at double the recommended application time. However, no statistically significant differences occurred among the application times for all of the systems used.

Some authors verified an increase in bond strength in the Clearfil SE Bond and Clearfil Liner Bond II systems after applying the acidic primer for double the time recommended by the manufacturers in *in vivo*³³ and *in vitro* studies.¹⁷⁻¹⁸

In the current study, two-step adhesive systems presented higher mean enamel bond strength values compared with one-step systems when the two-step systems were applied for double the time recommended by the manufacturers. Also, a higher mean bond strength was obtained with the primer of Clearfil SE Bond adhesive when it was applied for double the time recommended by the manufacturer, despite not being statistically different from the other adhesive systems. This is probably due to the combination of stronger water-based acids and hydrophilic monomers in a single solution (one-step self-etching systems), thus making them more unstable and permeable,³⁴ and producing more rapid water absorption through the adhesive layer, compromising their bonding and reducing their clinical life.³⁵

Despite adhering well to enamel before functional and thermal stress, one-step self-etching adhesives are more susceptible to water absorption, making them significantly less effective after these stresses. This reduction results from one-step adhesives becoming permeable membranes after polymerization in the absence of a hydrophobic adhesive agent.³⁶ This could facilitate water absorption between the partially demineralized enamel and the restorative material, and eventually weaken the adhesive interfaces of the enamel.¹¹

The application of a self-etching primer results in a surface etching pattern that could be the result of deficient penetration into the microporosities of the enamel, or it could result from calcium precipitation on the surface, masking the etching pattern and interfering with resin penetration.³⁷⁻³⁹ If the resin does not completely infiltrate into the etched enamel, there may be a region of unprotected enamel prisms, therefore making it more susceptible to hydrolytic degradation.⁴⁰

The results found in the fracture pattern analysis of the specimens bonded with one-step application adhesives (Futura Bond and One Up Bond F) demonstrated that, when doubling the primer application time, there was an increase in the percentage of Type 1 fractures, (adhesive failure between the adhesive and enamel), while the percentages of Type 2 fractures (partial adhesive failure between the adhesive system and enamel and partial cohesive in the adhesive) remained similar for the adhesive One Up Bond F and increased for the

adhesive Futura Bond. However, a lower percentage of Type 3 fractures (completely cohesive in the adhesive system) occurred. It is speculated that, despite their greater acidity, these one-step systems did not form a hybrid layer with high mechanical resistance due to their high hydrophilicity.¹¹ Therefore, the increase in primer application time probably did not lead to a greater occurrence of cohesive fractures in the adhesive (preserving the hybrid layer); instead, it led to a large number of adhesive fractures.

On the other hand, in the two-step application adhesive systems (Clearfil SE Bond and AdheSE), the percentage of adhesive fractures (Type 1) diminished with an increase in primer application time, while the percentage of cohesive fractures in the adhesive (Type 3) increased. Cohesive fractures in the adhesive represent the integrity of the subjacent hybrid layer protecting the dental substrate.⁴¹

One could presume that, by increasing the primer acidity, the manufacturers would solve the problem of bonding to enamel, but this greater acidity would have the effect of retarding the polymerization of light-activated resins by affecting the acid-base reaction of amines generally used in the polymerization initiator systems. This effect also occurs on the tertiary amines of the chemically polymerized resin composites, which are rapidly consumed by the acidity of adhesives. The effect of reduced polymerization results in a significant compromise of bonding at the interface between the resin and adhesive and could cause degradation of the adhesive itself.⁴²⁻⁴⁴

Bittencourt¹² showed that, after 18 months of evaluating self-etching adhesives, a faster marginal degradation was demonstrated, despite being within the acceptable standards of the ADA. Another clinical study showed that some self-etching adhesives were not effective in Class V resin restorations without previous preparation.⁴⁵

Although conventional treatment with phosphoric acid is still considered the safest method for obtaining more durable and more fatigue-resistant bonding to enamel, and despite reservations about the durability of self-etching adhesives bonding to enamel in long-term clinical studies (particularly one-bottle adhesives), the bond strength values of self-etching adhesives are very close to the bond strength values of conventional adhesives. The increase in application time of the acidic primer did not improve the bonding of self-etching adhesive systems to unground enamel.

CONCLUSIONS

The increase in application time of the acidic primer did not significantly influence the bond strengths of one- and two-step self-etching adhesive systems to unground enamel.

The increase in application time of the primer altered the fracture pattern of the specimens.

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