Bond Strengths of Current Adhesive Systems on Intact and Ground Enamel

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ABSTRACT

Purpose: The goal of this study was to compare the microshear bond strengths of current adhesive systems to intact and ground enamel surfaces.

Materials and Methods: Three commercially adhesive materials were examined: a two-step total-etching self-priming system (Single Bond®, 3M ESPE, St. Paul, MN, USA), a two-step self-etching system (Clearfil SE Bond®, Kuraray Co. Ltd., Osaka, Japan), and an all-in-one system (One-Up Bond® F, Tokuyama Dental, Tokyo, Japan). The labial surfaces of extracted human incisors were used. For intact enamel surfaces, the teeth were polished with pumice. However, for the ground enamel, 0.5 mm deep, flat enamel surfaces at the midlabial aspect were ground with a no. 600 grit silicon carbide paper. Ground and intact enamel surfaces were bonded with one of three bonding systems and a resin composite and were subjected to a microshear bond test.

Results: The two self-etching systems demonstrated lower bond strengths than did the total-etching system on intact enamel (One-Up Bond F, 18.59 MPa; Clearfil SE Bond, 35.71 MPa; Single Bond, 47.20 MPa). No significant difference was found between the total-etching system and the two-step self-etching system on ground enamel (One-Up Bond F, 28.96 MPa; Clearfil SE Bond, 48.51 MPa; Single Bond, 51.07 MPa). Shear bond strengths on ground enamel were significantly higher than those on intact enamel except for the group that was bonded with the total-etching system.

Conclusions: The two-step adhesives showed significantly higher bond strengths than did the one-step adhesive. When applying the total-etching system, no significant differences in bond strength were demonstrated between bonding to intact and to ground enamel.

CLINICAL SIGNIFICANCE

The additional step of removal of the outermost enamel layer is one of the many procedures that can be implemented to increase the bond strength of self-etching systems to intact enamel.

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The concept of altering tooth structures for securely bonded restoration was introduced in 1955 by Buonocore.[1] Subsequent pursuits in the literature indicated that resinous tag-like extensions were established at the interface when materials had contacted a treated enamel surface.[2] The microstructure, tag-like extensions were the penetration of adhesive materials into the microscopic pores that appeared on the acid-treated enamel. However, no tag-like formation was found on enamel surfaces after the application of adhesive resin without the prior application of acid.[2,3] Thus, this micromechanical retention plays an important role in the bonding of most adhesive systems to enamel.[4] Consequently, the acid-etch technique is widely used and has become a standard procedure for enamel surface preparation prior to restoration with resin composites.[5–10]

The acceptability and reliability of bonding performance have increased owing to the introduction of a dentin primer and its benefits to three-step adhesive systems. There are generally three separate steps: etching, priming, and bonding. Because of a multiple-step approach, clinical success of these systems depends on the application techniques and properties of the materials.[11–13] Clinical procedures have been simplified by combining the steps of current adhesive systems into the two-step adhesive systems. These systems can be divided into two groups. One is a total-etching self-priming adhesive system that combines the priming and bonding steps, which are applied after phosphoric acid etching.[12,14] The other group is a self-etching, adhesive system that combines the etching and priming steps together.[12] Most recently one-step self-etching, or all-in-one, adhesive systems have been introduced. This latter system combines the etching, priming, and bonding steps into one application—an etching agent/primer/adhesive.[15]

Most current studies related to adhesive systems deal with cut, flat enamel surfaces,[16–18] whereas there are many differences in the morphologic structures between the intact outer surfaces of enamel and the inner enamel. Surfaces of permanent teeth consist of approximately 70% prismless enamel.[19,20] The amount of prismless enamel might influence the quality of the etch pattern and bond strength. Ideally, to achieve the best etch quality, the enamel surface should be prepared with grinding to remove the prismless enamel. However, for some treatments, such as orthodontic bonded brackets or diastema closure, materials and instruments should be bonded directly onto intact enamel surfaces. Since there are currently many kinds of adhesive systems, their bonding performance should be evaluated on both ground and intact enamel surfaces.

Since Sano and colleagues developed the microtensile test,[21] it has been used to perform many bond strength tests.[21,22] For the microtensile test, however, the trimming step for specimen preparation is important when making the small testing area.[21,22] Because enamel is naturally brittle and fragile, it can crack easily, especially along the enamel prism.[23] The trimming step for the enamel bond strength test should be avoided because it might cause reduction of the bond strength. When conducting the microshear test, the trimming step is not necessary. In addition, since the preparation of small specimens is simple, the use of such a test is quite feasible.

The objective of this study was to compare the microshear bond strength of current adhesive systems to ground and intact enamel surfaces. Moreover, the etching efficiency and resin-enamel interfaces were examined under a scanning electron microscope (SEM). The first hypothesis tested was that there would be no significant difference in the bond strengths of current adhesive systems on intact or ground enamel. The second hypothesis was that there would be no significant difference in bond strengths on each substrate among selected adhesive systems.

MATERIALS AND METHODS

Three commercially available adhesive materials examined were a two-step total-etching self-priming system (Single Bond, 3M ESPE, St. Paul, MN, USA), a two-step self-etching system (Clearfil SE Bond®, Kuraray Co. Ltd., Osaka, Japan), and an all-in-one system (One-Up Bond® F, Tokuyama Dental, Tokyo, Japan).

Bond Strength Measurement
Extracted human incisors that were kept in physiologic saline at 5°C for < 2 months were used in this study. The labial tooth slices were approximately 1.5 mm thick and were prepared by cutting the tooth parallel to the labial surface with a low-speed diamond blade (Isomet®, Buehler Ltd., Lake Bluff, IL, USA) under water coolant. The tooth slices were then divided into two groups, intact and ground enamel. For the intact enamel surfaces, the teeth were polished with pumice and cleaned with an air/water spray from a triple syringe to remove any debris. For the ground enamel, 0.5 mm deep, flat enamel surfaces at the midlabial aspect were ground with no. 600 grit, wet silicon carbide paper. Ground and intact enamel surfaces were randomly assigned to three groups for bonding with one of three bonding systems, according to the manufacturers' instructions (Table 1). Before the adhesive resin was polymerized, a plastic tube (Tygon®, Norton Performance Plastic Co, Cleveland, OH, USA) with an internal diameter and a height of approximately 0.7 and 0.5 mm, respectively, was placed on the bonded area. After the adhesive was light cured, a resin composite (Clearfil AP-X®, Kuraray Co. Ltd., Osaka, Japan) was placed into the tube and polymerized for 40 seconds. In this manner a small cylinder of resin composite was bonded to the surface of enamel. The bonded specimens were stored in tap water at 37°C. Figure 1 demonstrates the specimen preparation for the microshear test.

After storage for 24 hours, the plastic tubes were removed. All resin composite cylinders were checked under a stereomicroscope at ×20 magnification prior to microshear testing. The composite cylinders that presented an apparent interfacial defect were excluded from this study. Ten specimens were tested for each group.

The bonded specimen was attached to the testing device (Bencor-Multi-T®, Danville Engineering Co., San Ramon, CA, USA) with a cyanomethacrylate adhesive (Zapit®, Dental Ventures of America, Corona, CA, USA), and the testing device was placed on a universal testing machine (EZ Test®, Shimadzu Co., Kyoto, Japan).

A thin wire with a diameter of 0.20 mm was looped around the resin cylinder and placed close to the resin-enamel interface for the test. With this orientation, the interface, wire loop, and center of loading cell were aligned as straight as possible to ensure the direction of shear test force. A shear force was applied at a crosshead speed of 1.0 mm/min. The shear bond strengths at failure were recorded and converted into megapascals.

TABLE 1. MATERIALS AND PROCEDURES.

Adhesive	Batch No.	Composition	Procedure
Single Bond	20011204	Etchant: 35% phosphoric acid	Apply 15 s, rinse
		Adhesive: HEMA, dimethacrylate, BIS-GMA, functional copolymer ethanol, water	Apply 2 coats, 10 s light cure
Clearfil SE Bond	011233	Primer: HEMA, MDP, hydrophilic dimethacrylate, water	Apply 20 s, air dry
		Adhesive: MDP, BIS-GMA, HEMA, hydrophilic dimethacrylate, microfiller	Apply and air dry, 10 s light cure
One-Up Bond F	455661C	Bonding agent A: phosphate monomer, MAC-10 monomer; bonding agent B: monomer, water, fluoroaluminosilicate glass	Mix bonding agents, apply for 20 s, 10 s light cure

BIS-GMA = bisphenyl-glycidyl-methacrylate; HEMA = 2-hydroxyethyl methacrylate; MAC-10 = 10-methacryloyloxydecyl dihydrogen phosphate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate.

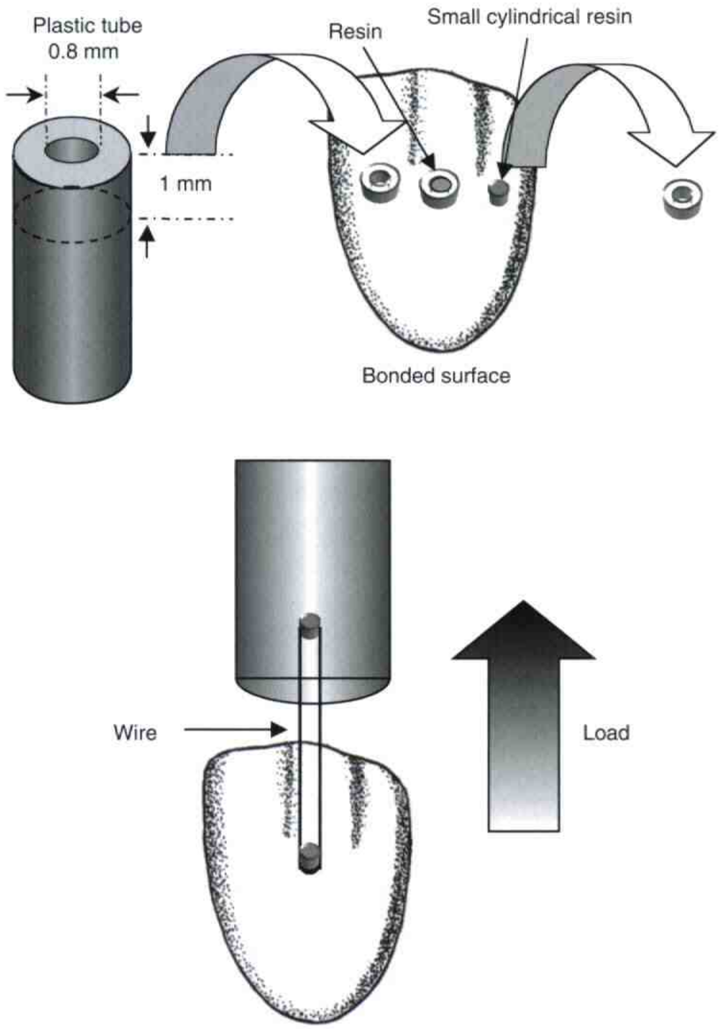

Figure 1. Diagram of specimen preparation for microshear bond strength test.

The debonded specimens were then observed for failure modes under a confocal laser-scanning microscope (1LM21® series, Lasertec Corporation, Tokyo, Japan). The failure modes were calculated as percentages of cohesive failure in enamel, adhesive failure, and cohesive failure in resin.

The means and SDs of microshear bond strengths were calculated.

There were two variables, types of enamel, and types of adhesive systems. The data were analyzed by two-way analysis of variance (ANOVA) and the least significant difference (LSD) multiple comparison test. The difference of failure modes among groups was also analyzed using the nonparametric statistical analysis, the Kruskal-Wallis one-way ANOVA by ranks, at a level of significance of .05.

SEM Evaluation

Etching Efficacy. To observe the effect of acidic conditioning on ground and intact enamel, six additional permanent incisors were prepared and conditioned with each material following the manufacturers' instructions. Phosphoric acid–etched enamel was thoroughly rinsed with water spray for 10 seconds and dried with oil-free air spray. The specimens treated with a self-etching adhesive system were rinsed with acetone for 10 seconds to remove the self-etching prime and dried in a desiccator for 24 hours.[24] The specimens were then coated with gold and observed under the SEM (JSM®-5310LV, JEOL Ltd., Tokyo, Japan).

Enamel-Resin Interface. The observation of the interfaces between the adhesive resins and each group of enamel surfaces, ground and intact enamel, was done under the SEM. Six ground and six intact enamel surfaces prepared from permanent incisors were used and bonded using the same materials and procedures as previously described. Without the placement of plastic tube, the 1 to 2 mm thick resin composite was applied directly on bonded surfaces and polymerized. After 24 hours storage in tap water at 37°C, the bonded specimens were cut perpendicular to the interface with a low-speed diamond disk (Isomet) under water cooling. The cut specimens were embedded in epoxy resin (Epon 815®, Nissin, Tokyo, Japan). The specimens were polished with series of silicon carbide papers (400–1,500 grit) and polished with diamond pastes (from 6 μm down to 0.25 μm). The polished specimens were immersed in 37% phosphoric acid for 5 seconds, washed thoroughly, and dried in the desiccator. After desiccation the specimens were sputter coated with gold and observed under the SEM.

RESULTS

Bond Strength Measurement
Means and SDs in megapascals are shown in Figure 2. The all-in-one adhesive showed significantly lower mean bond strengths than did the two-step adhesive systems. The self-etching systems exhibited significantly lower mean bond strengths than the total-etching adhesive system, Single Bond, on intact enamel (Clearfil SE Bond, $p = .007$; One-Up Bond F, $p < .001$). However, no statistically significant difference was found between the mean bond strength of Clearfil SE Bond and that of Single Bond to ground enamel ($p = .445$). For ground enamel the mean bond strength of One-Up Bond F was significantly lower than those of the other groups ($p < .001$). Mean bond strengths to ground enamel were higher than those to intact enamel (Clearfil SE Bond, $p = .001$; One-Up Bond F, $p = .017$), except for the group that was bonded with Single Bond ($p = .334$).

There was no significant difference for failure modes ($p > .05$). More than 90% of the failures occurred at the resin-enamel interfaces as opposed adhesive failure.

SEM Evaluation
Etching Efficacy. SEM images of ground and intact enamel surfaces, untreated and treated with the adhesives, are shown in Figure 3. Intact enamel surfaces had different surface morphologies depending on whether they were etched with phosphoric acid or self-etching primer. The application of phosphoric acid for 15 seconds results in a uniform removal of enamel apatite crystals. The prism core was preferentially removed, leaving the prism peripheries intact. However, a nonuniform etched pattern was exhibited with the use of self-etching systems. Prismatic structures of enamel were not clearly observed.

The changes of surface morphology on ground enamel demonstrated the same trend as on intact enamel. The acidic primer of Clearfil SE Bond was less effective than phosphoric acid. For the all-in-one system (One-Up Bond F), the smear layer still remained on the etched surfaces, whereas it was not on the ground surfaces of controls, demonstrating that the etching efficacy of the One-Up Bond F primer was weaker than that of the Clearfil SE Bond primer. The nonuniform etched pattern that was found in the Clearfil SE Bond group was rarely defined in the One-Up Bond F group.

Enamel-Resin Interface. In the Single Bond group, in which a total-etching self-priming adhesive system was applied to intact and ground enamel (35% phosphoric acid was used as the etching step), abundant long resin tags were found continuously along the bonding interface. The application of

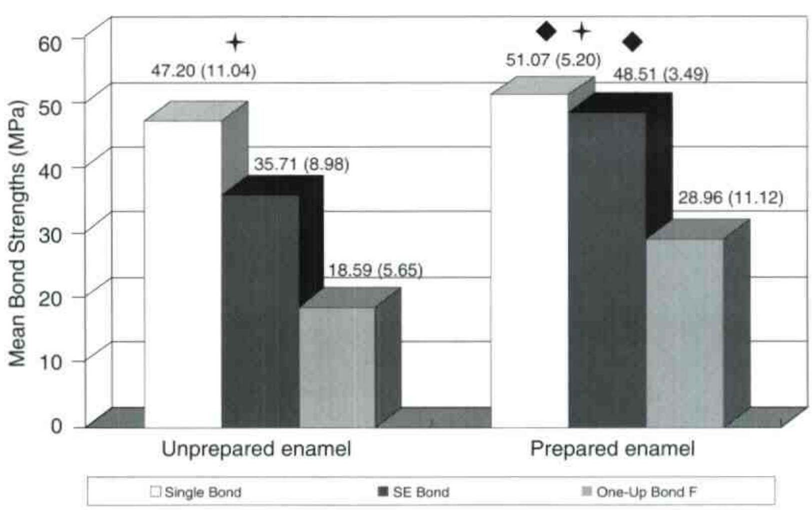

Figure 2. Microshear bond strengths to enamel surfaces. Data with the same symbol demonstrate no statistically significant difference.

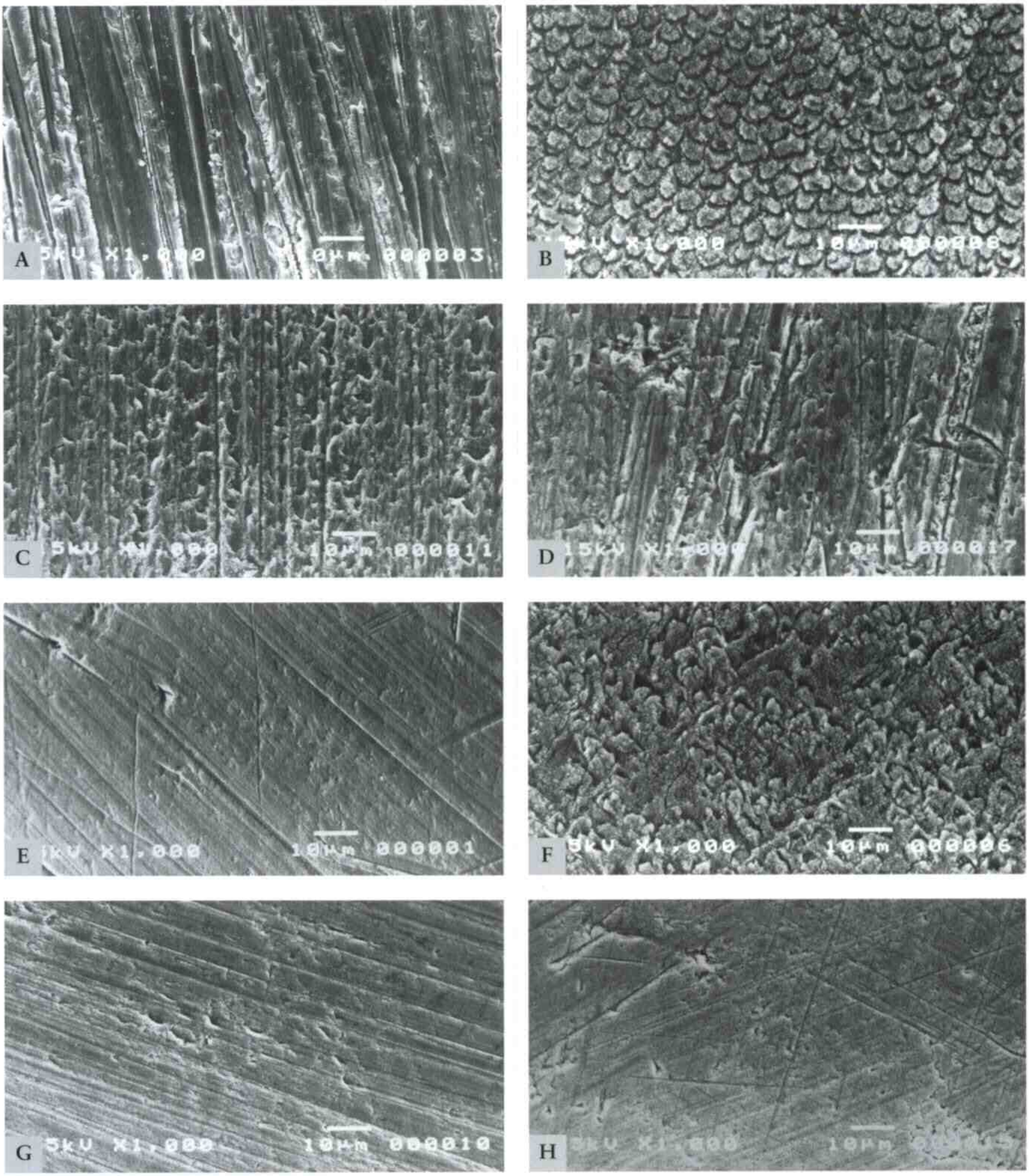

Figure 3. SEM analysis of etched enamel surfaces: A, *ground enamel surface;* B, *ground enamel treated with phosphoric acid (Single Bond);* C, *ground enamel treated with acidic primer (Clearfil SE Bond);* D, *ground enamel treated with acidic primer (One-Up Bond F);* E, *intact enamel surface;* F, *intact enamel treated with phosphoric acid (Single Bond);* G, *intact enamel treated with acidic primer (Clearfil SE Bond);* H, *intact enamel treated with acidic primer (One-Up Bond F).*

Clearfil SE Bond and One-Up Bond F on ground enamel produced short resin tags compared with those of Single Bond. On intact enamel the short resin tags were rarely identified when the self-etching systems were used.

DISCUSSION

The structure of a surface enamel layer has been well documented in the literature as the presence of a thick prismless or aprismatic zone.[19,20,25] Such a surface layer is generally more mineralized than the subsurface enamel and is thought to be resistant to the acid-etch process of bonding systems.[20] Grinding of this layer to expose the prismatic subsurface enamel before acid etching has been recommended.[20] However, some operators prefer to etch and bond materials over the existing enamel without removing any tooth structure when performing a laminated veneer restoration or diastema closure work. In this manner, bonding to intact enamel surface is important and should be an area of concern for clinicians. However, most studies aim to investigate bond strengths of many systems on ground enamel,[16–18] owing to the technical difficulties of measuring bond strength of the convex, intact enamel surface with conventional testing methods. To solve this problem, the testing method that uses relatively small bonding surface areas is preferable.[24]

According to the results of the present study, bond strengths of resin composite to both intact and ground enamel using a total-etching self-priming adhesive system demonstrated the highest result when compared with the two groups that used self-etching systems. High acidity of phosphoric acid produced major changes on enamel by the removal of aprismatic enamel surface and the formation of surface irregularity (see Figure 3B and F). When the adhesive resin is applied on etched enamel surface, it flows into the porosities and forms a micromechanical bond after polymerization.[2–4] This ultrastructure might have contributed to the high bond strength obtained with the total-etching system.

The etching ability of two self-etching systems (Figures 3G and 3H) was demonstrated on intact enamel to be less efficient than the total-etching self-priming adhesive system (see Figure 3F).[17] The outermost intact enamel is hypermineralized, which may prevent the permeation of the self-etching primer, resulting in a deficient penetration of the adhesive resin.[20,25] Clearfil SE Bond produced a mild etching effect and left some areas partially unetched. The poorly defined resin tags on intact enamel may have produced the lower bond strength when compared with that of the total-etching self-priming adhesive system (Single Bond). One-Up Bond F resulted in a rather smooth enamel surface. The lowest bond strength obtained from this all-in-one self-etching adhesive system might be due to the improper penetration of resin into etching patterns that were not well defined. When self-etching adhesive systems composed of acidic monomers are used without rinsing, the calcium and phosphate ions that were dissolved from the demineralization process must be suspended in the watery solution of the primer. When the solvent of the primer evaporates, the solubilized calcium and phosphate within the primer may exceed the solubility product constants for a number of calcium phosphate salts.[26] These high concentrations of calcium and phosphate tend to limit further dissolution of the apatite.[27,28] The binding of calcium ions to phosphate residue contributes to the inactivation of the acidity and thereby inhibits the self-etching effect.[29] These effects might reduce bond strength of self-etching systems on intact enamel.

Clinically, grinding aprismatic enamel surface is an additional step that has been recommended to expose the underlying prismatic enamel. In our study there was no significant difference in the bond strengths to ground enamel treated with Clearfil SE Bond and Single Bond. This result confirmed those of former investigations that reported no significant difference between results with a two-step self-etching adhesive system and other phosphoric acid–etch adhesive systems.[16,24] Clearfil SE Bond even produced less demineralization of enamel than did the phosphoric acid, but it demonstrated a high bond strength to ground enamel.

Thus, One-Up Bond F created less etching pattern and resin penetration on enamel than did Clearfil SE Bond, and the mean bond strength was also low and of significant difference. The difference of the etch pattern among the adhesives might be partially responsible for the difference in bond strengths found in this study. Additionally, the strength of the resin adhesive used might have influenced the strength of the adhesive resin-enamel bond.[30]

Both etching efficacy and strength of resin were thought to be two important contributing factors on the bonding of a self-etching adhesive to enamel.[31] On intact enamel surfaces, both self-etching systems—two step and one step—produced a lower etching effect than did the total-etching system and resulted in a deficient penetration of the resin used. These results are compatible with the decrease in adhesive strengths. However, when the acid-resistant, hypermineralized outermost surface of enamel was ground, the self-etching primer of Clearfil SE Bond tended to have a better etching efficacy for resin penetration and provided adequate bond strength. One-Up Bond F, the least aggressive system compared with Clearfil SE Bond,[32] produced an etching efficacy similar to that of Clearfil SE Bond even though a relatively low bond strength was obtained. It was apparent that the etching efficacy of One-Up Bond F was not weak, but the adhesive bond was influenced by the strength of the resin used.

For self-etching systems, simultaneous etching and priming facilitate the penetration of the adhesive resin into tooth surfaces. This quite fast and simple application procedure provides many advantages when used in dental clinics. The adhesion potential to enamel has resulted in a wide range of bond strength values, especially when applied to intact enamel surfaces.[16–18,22] An additional step or another protocol may be needed to improve the retention of resin used in the self-etching adhesive system. The removal of outermost enamel layer is one of the many procedures to gain higher bond strength of self-etching adhesive systems to enamel. The results from this study were in line with those of Kanemura and colleagues.[22] It was concluded that there were no significant differences in the bond strengths to ground enamel treated with either the phosphoric acid–etching adhesive system or the two-step self-etching adhesive system.

No clinical evidence has been found to prove that the self-etching adhesives produce durable bonding to enamel. Therefore, further studies in the durability of enamel bond strength of current adhesive systems, as well as clinical longevity evaluated by long-term clinical trials, are necessary.

CONCLUSIONS

The most simplified procedural adhesive system, the all-in-one system, resulted in lower bond strengths to both intact and ground enamel than did the two-step adhesive systems. Use of the total-etching self-priming adhesive system produced consistent bond strengths to both intact and ground enamel.

DISCLOSURE

The authors have no financial interest in the companies whose products are mentioned in this article.

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