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Two-year Water Degradation of Self-etching Adhesives Bonded to Bur Ground Enamel

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Clinical Relevance

All the adhesives tested showed no difference in bond strength after 24-hours of water storage. After two years of water storage, the bonds produced by some self-etch adhesives were significantly reduced.

SUMMARY

To evaluate the effect of water storage on the microshear bond strength to ground enamel of three "all-in-one" self-etch adhesives: Futurabond DC, Clearfil S Tri Bond and Hybrid bond; a self-etching primer; Clearfil SE Bond and an etch-and-rinse adhesive system, Admira Bond. Sixty human molars were used. The root of each tooth was removed and the crown was sectioned into two halves. The convex enamel surfaces were reduced by polishing on silicon paper to prepare a flat surface that was roughened with a parallel-sided diamond bur with abundant water for five seconds. The bonding systems were applied on this surface. Prior to adhesive curing, a hollow cylinder (2.0 mm in height/0.75 mm in internal diameter) was placed on the treated surfaces and cured. A resin composite was then inserted into the tube and cured. For each adhesive, two procedures were carried out: A—the specimens were kept in water for 24 hours, then the tube was removed and the microshear bond strength was determined in a universal testing machine at a crosshead speed of 0.5 mm/minute; B—the specimens were stored in water for two-years before microshear testing. The fractured surface of the bonded specimens after each test procedure was examined by SEM. For the 24-hour control, there was no significant difference in bond strength between the tested adhesives. After two years of water storage, the bond strength of Admira Bond, Clearfil SE Bond and Futurabond DC decreased, but the reduction was not significantly different from that of 24 hours. For Clearfil S Tri Bond and Hybrid Bond, the bond strengths were significantly reduced compared to their 24-hour results.

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INTRODUCTION

From the perspective of ease of handling, adhesives based on self-etching primers are preferred above total-etch systems. However, for the bond to enamel, the bonding quality in terms of bond strength values is not as consistent as that reported for the same products to dentin. Some authors reported that the bond strength of self-etching primers is inferior to that obtained with adhesive systems utilizing phosphoric acid as a surface conditioner.[1-5] Conversely, other studies that tested composite-to-enamel bond strength with self-etching adhesive systems have reported values as high as 20 to 30 MPa,[6-8] being in the same range as that reported on phosphoric acid-etched enamel. Regarding *in-vitro* microleakage experiments, self-etching adhesive systems were found to be comparable to those with separate etchants with respect to dentin marginal leakage but less effective at the enamel site.[9-11] The reduced effectiveness in adhesion to enamel of some self-etching products has been attributed to the relative mild acidity of the primers, while strong self-etchants are said to produce a more effective enamel etch than mild agents.[12]

The self-etching primer can simultaneously cause both acid-conditioning and priming in one step. These adhesive systems eliminate the separate steps of acid etching and water rinsing, thus simplifying bonding procedures. Such simplification may reduce the technical errors that often follow the use of total-etch adhesives, such as over-etching, over-wetting or over-drying of the prepared tooth surface.

Whether these newer products produce the same superior margins along enamel walls as their etch and rinse predecessors is of critical importance. Representative clinical studies have not demonstrated significant differences in the marginal discoloration of non-retentive Class V restorations between self-etching primers, self-etching adhesives and etch-and-rinse adhesives.[13-15]

Recently, the microshear bond test was introduced as an alternative to the microtensile bond test.[16] The microshear bond test involves the application of a loading force by means of a blade from a universal testing machine to a resin composite cylinder bonded to a substrate disc. Advantages of the microshear bond test include less demanding specimen collection and easier control of the bond test area by means of microbore tubes. Shimida and others modified the microshear bond test by employing a looped orthodontic wire rather than a blade.[17]

In addition, different artificial aging techniques were used in order to predict the clinical performance of tested materials. The most commonly used artificial aging technique is water storage. In this technique, the bonded specimens are stored in fluid at 37°C for a specific period. This period may vary from a few months[18] up to four-to-five years[19-21] or longer. Most of these studies report significant decreases in bond strengths, even after relatively short storage periods.[22-28] The decrease in bonding effectiveness after water storage was supposed to be caused by degradation of interface components by hydrolysis (mainly resin and/or collagen). Also, water can infiltrate and decrease the mechanical properties of the polymer matrix by swelling and reducing the frictional forces between the polymer chains. This causes plasticization of the resin, which makes it weaker.[29-30]

The current study was designed to evaluate the influence of water storage on the microshear bond strength of one total-etch adhesive and four self-etching adhesives to ground enamel. The null hypothesis was that there was no difference between the self-etching materials and the total-etch adhesive in their bond strengths to ground enamel when stored in water.

METHODS AND MATERIALS

Four commercial self-etching bond systems (Table 1) were used in the current study: Futurabond DC (Voco, Cuxhaven, Germany), Clearfil S Tri Bond (Kuraray Medical, Inc, Tokyo, Japan), Hybrid Bond (Sun Medical, Moriyama, Japan) and Clearfil SE Bond (Kuraray Medical, Inc). The first three are "all-in-one" adhesives, while the fourth is a self-etching primer to which a layer of adhesive is applied. The etch-and-rinse system Admira Bond (Voco) was used as a control.

Microshear Bond Strength

Sixty caries-free human molars (gathered following informed consent approved by the Commission for Medical Ethics of the University) were used for the bonding tests. The teeth were washed under running water immediately after extraction and stored in 0.02% thymol solution until the experiment time, which was scheduled within one month after extraction. At that time, the root of each tooth was removed and the crown was sectioned into two halves by cutting parallel to the longitudinal axis and facial surface using a low speed Isomet saw (Buehler Ltd, Lake Bluff, IL, USA) under running water. The convex enamel surfaces on the outermost buccal or palatal slices were reduced up to 0.5 mm by gently polishing on 600 grit silicon carbide paper under running water to prepare a flat enamel surface. This surface was roughened with a parallel-sided medium-grit (100 µm) diamond bur (842, Komet, Lemgo, Germany) for five seconds with abundant water. The inner surface of the crown segment was also polished to create a flat surface on the dentin for ease of fixation during microshear testing. All the tooth-halves were randomly assigned; however, for each adhesive, the buccal and palatal sections were equally distributed. The bonding systems were then applied on the pre-

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Table 1: *Chemical Composition of Tested Materials*

Material/Manufacturer	General Composition	Manufacturers' Instructions for Use
Admira Bond (Voco, Cuxhaven, Germany) Lot #45309	**Acid:** 36% Phosphoric acid. **Bond**: Acetone, bonding ormocer, Dimethacrylate, acid modified methacrylates, initiators, stabilizer.	• Enamel was etched for 30 seconds with 37% phosphoric acid, rinsed with water spray for 20 seconds. • Excess water was removed with air blast for 3 seconds. • Admira Bond was applied with disposable brush, thinned with mild air for 2-3 seconds and light cured for 20 seconds.
Clearfil S Tri Bond (Kuraray Medical Inc, Tokyo, Japan) Lot #00001A	**Adhesive:** MDP, BIS-GMA, HEMA, hydrophilic DMA, microfiller.	• Dispense adhesive in the well. • Apply adhesive with a rubbing motion for 20 seconds. • Gently air-dry for 5 seconds. • Light cure for 20 seconds.
Clearfil SE Bond (Kuraray Medical Inc, Tokyo, Japan) Lot #61739	**Primer:** Water, MDP, HEMA, CQ, DET and hydrophilic DMA. **Bond:** MDP, bis-GMA, HEMA, hydrophobic DMA, CQ, DET, silanated colloidal silica.	• Dispense primer in the well. • Apply primer with a brush and leave for 20 seconds. • Air-blow for 5-10 seconds. • Dispense bond in the well. • Apply bond with a brush and air-thin. • Light cure for 10 seconds.
Futurabond DC (Voco, Cuxhaven, Germany) Lot #010075	**Liquid A:** Water, ethanol, silicium dioxide. **Liquid B:** Acid modified methacrylate (methacrylate ester), HEMA, camphorquinone.	• Dispense one drop of Liquid A and one drop of liquid B into the well and mix for 5 seconds. • Apply adhesive with rubbing motion for 15 seconds. • Gently air-dry for 5 seconds. • Light cure for 20 seconds.
Hybrid Bond (Sun Medical, Moriyama, Japan) Lot #010075	**Base:** 4-META, multifunctional acrylate, HEMA, MMA, acetone, water, and PI. **Brush:** p-toluene sulfinate, sodium-salt, and aromatic amine.	• Dispense one drop of Hybrid Base in the well. • Stir the expressed liquid with a Hybrid Brush. • Apply and keep it moist for 20 seconds. • Air-dry for 5-10 seconds. • Light cure for 3-5 seconds.

Abbreviations: Bis-GMA: bis-phenol A diglycidylmethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; DMA: dimethacrylate; DET: N,N-diethanol p-toluidine; CQ: camphorquinone; 4-META:4-methacryloyloxyethyl trimellitate anhydride; MMA: methylmethacylate; PI:photoinitiators.

pared enamel surface of each half following the manufacturer's instructions (Table 1). Prior to irradiation of the bonding resin on each specimen, polyethylene micro-bore tubing 2.0 mm in height (Norton Performance Plastics, Granville, NY, USA) with an internal diameter of 0.75 mm was placed on the treated surfaces. After light irradiation with a halogen light source (Visulux curing unit, Vivadent, Schaan, Liechtenstein) for 20 seconds, a hybrid restorative composite from the same manufacturer was used. The composite was carefully inserted into the tubing lumens and irradiated for 40 seconds according to the manufacturer's instructions. The output of the light-curing unit was regularly checked to insure it was 500 mW/mm^2.

The resin composites used were Grandio (Voco), Clearfil APX (Kuraray Medical, Inc) and Pecalux (Sun-Medical). The specimens were stored in water at 37°C for 24 hours. After removal from water, the tygon tubing around the composite cylinders was removed by gently cutting the tube into two hemi cylinders using a feather-edge blade. This procedure was done under stereomicroscope, and special caution was taken to avoid applying any stress to the bonded composite cylinders.

Two test procedures were carried out for each adhesive.

Procedure A: Specimens tested for microshear bond strength after 24 hours of water storage.

Procedure B: The specimens were kept in water containing 0.5% chloramine to prevent bacterial growth for two years, then the bond strength measurement was carried out. The water was replaced every three months. During replacement, the specimens were allowed to cool slowly to room temperature and the water was replaced.

For microshear testing, each tooth slice was attached to the testing apparatus (modified Ciucchi's jig, Pashley & others[31]) with a cyanoacrylate adhesive and tested in a universal testing machine (Instron, Corp, High Wycombe, UK). A thin steel wire (0.20 mm D) was looped flush between the load cell projection and the resin cylinder, making contact with the lower half-circle of the cylinder and touching the tooth surface. The force was applied at a crosshead speed of 0.5 mm/minute until failure. Care was taken to keep the composite cylinder in line with the center of the load cell and the wire loop, parallel to the load cell movement direction

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and the bonded surface, in order to maintain a shear stress orientation at the bonding interface. The maximum load at the time of failure was recorded. A total of 12 bond strength values was recorded for each adhesive. The types of failure were observed at 50x magnification and categorized as adhesive, cohesive or mixed.

Each adhesive system was applied according to the manufacturer's instructions.

Statistical Analysis: The bond strength was calculated and the data obtained for the five subgroups were statistically analyzed using one-way ANOVA, followed by the Tukey-Kramer Multiple Comparison Test at a 5% confidence level.

Fracture Surfaces Observation

After microshear testing, the fractured surface of the specimen was inspected by a stereomicroscope at 50x magnification (Olympus, Tokyo, Japan) to evaluate the mode of failure. The mode of the failures was classified into cohesive in enamel or cohesive in resin, adhesive at the enamel/resin interface and mixed. In addition, for representative specimens of each test group, impressions of the fractured surfaces were made using a light-body polyvinylsiloxane impression material (Extrude, Kerr GmbH, Karlsruhe, Germany). The impressions were cast in epoxy resin (Epoxy Die), then mounted on aluminum stubs, sputter-coated with gold and observed by using SEM (Philips XL30, Eindhoven, The Netherlands) operating at 15 kV.

Morphological Study Using SEM

In addition, 20 enamel slices were prepared as mentioned, with four slices for each adhesive. After adhesive application, a resin composite was placed 2 mm thick to form a composite core. The slices were stored in water for 24 hours, after which two slices from each adhesive were kept in 0.1 N HCl for 24 hours to completely dissolve the enamel. The resulting composite replicas were examined using SEM, as mentioned for the adhesive-enamel interface. The other two slices for each adhesive were stored in water for two years before acid dissolution and SEM evaluation.

RESULTS

Microshear Bond Strength Test

After 24-hours of water storage, there were no significant differences in bond strength of the tested adhesives (Table 2). The bond strength of the total etch adhesive "Admira Bond" was slightly higher than that of the other materials. However, that difference was not significant ($p>0.05$).

After two years of water storage, the bond strength of all the tested adhesives was reduced. For Admira Bond, Clearfil SE Bond and Futurabond DC, the reduction in bond strength was not significant ($p>0.05$). For Hybrid Bond and Clearfil S Tri Bond, the reduction was significant ($p<0.05$).

Figures 1a and 1b show the morphological interface between a total-etch adhesive, Admira Bond, and enamel. The enamel was completely removed by dissolving in HCL to show the resin penetration into enamel. Two types of resin penetration are evident: infiltration into and between interprismitic (IP) substances that are of different thicknesses and morphology. In addition, intraprismitic infiltration can be seen (IAP) as hollow porosities at all surfaces of the prisms. Figure 2a shows the interface between Futurabond DC and enamel, which is different from that with the etch-and-rinse system. The interprismitic diffusion (IP) was rather shallow and even absent in some locations. However, the intraprismitic diffusion (IAP) was evident in high magnification (Figure 2b). Many areas of nanometer resin diffusion were seen on the top surface of the prism.

Fracture Surface Observation

There were no pretest failures in any group. Overall, failures were predominately adhesive or mixed. The incidence of cohesive failures in composite was more common with increasing bond strength. The complete results are presented in Table 3.

After 24 hours of water storage, the fracture patterns of the bonded specimens were adhesive (61%) at the resin-enamel interface. Also, mixed types of failures were shown in 27% of the specimens, while 12% showed cohesive failure either in enamel or composite.

Table 2: *Microshear Bond Strength of the Tested Adhesives to Enamel (MPa ± SD)*

Materials	24-hours Water Storage	2-year Water Storage	Statistical Difference
Admira Bond	31 ± 2.3	28 ± 1.6	NS
Futurabond DC	29 ± 3.5	26 ± 2.4	NS
Clearfil SE Bond	28 ± 2.9	24 ± 1.7	NS
Clearfil S Tri Bond	27 ± 1.4	15 ± 2.6	($p<0.05$)
Hybrid bond	26 ± 1.9	14 ± 3.2	($p<0.05$)

NS = not significant (p>0.05).

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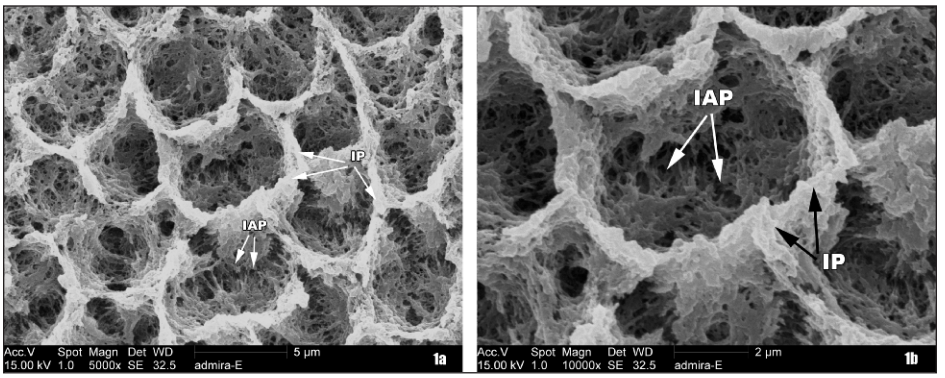

Figure 1a and Figure 1b: *The morphological interface between the total-etch adhesive Admira Bond and enamel. (The enamel was completely removed by dissolving in 0.1 N HCl for two hours to show the resin penetration into enamel). Two types of resin penetration are evident: infiltration into and between interprismitic (IP) substances that are of different thicknesses and morphology. In addition, interaprismitic infiltration can be seen (IAP) (Figure 1b).*

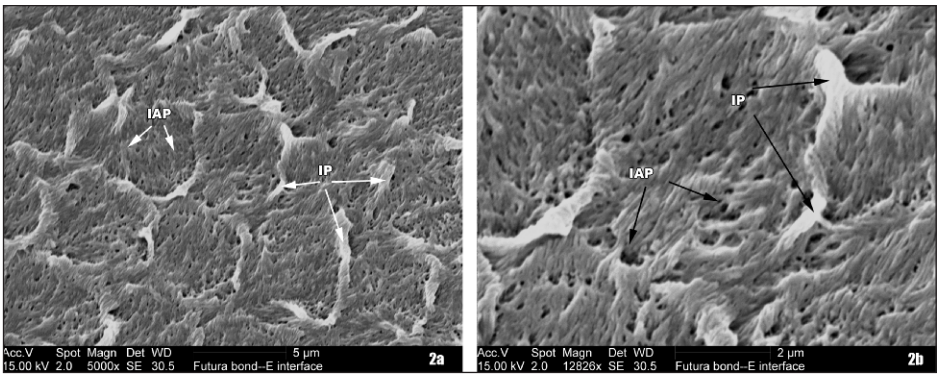

Figure 2a and Figure 2b: *The interface between Futurabond DC and enamel. The interprismitic diffusion (IP) was rather shallow and even absent in some locations. However, the intraprismitic diffusion (IAP) was evident in high magnification (Figure 2b). Many areas of nanometer resin diffusion were seen on the top surface of the prism.*

After two years of wear storage, failures were also mainly adhesive (72%). The mixed type of failure was seen in 27% of the specimens. Cohesive failure in composite was seen in 1% of the specimens. Figure 3 shows the fracture surface of the Admira Bond specimens after two-years of water storage. Resins were still present in the interprismitic substances as well as the top prism itself. Figure 4 shows the fracture surface of Hybrid Bond after two years of water storage. The resins were lost and the interprismitic spaces were visible. Also, the resins were lost from the prism itself.

DISCUSSION

In the current study, the effect of water storage on the bond strength of four self-etching adhesives and one total-etch adhesive to ground enamel was evaluated. Under clinical situations, cycling masticatory function has been reported to fatigue the integrity of the resin enamel bond, thereby permitting micro- or nanoleakage of the peripheral enamel seal.[32-33] In the current study, the bond strength tests were carried out using enamel from the different teeth. Care was taken to apply the adhesive to the enamel surfaces prepared to similar depths to minimize the effects of the enamel prism orientation and variation. The bond strength to transverse sections of enamel prisms has been found to be significantly higher than to longitudinal sections.[34-35]

Table 3: *Failure Patterns of the Tested Adhesives to Ground Enamel After 24-hours and 2-year Water Storage*

Materials	Condition	Adhesive	Cohesive in Enamel	Cohesive in Composite	Mixed
Admira Bond	24-hours 2-year water storage	3 6	2 0	2 1	5 5
Futurabond DC	24-hours 2-year water storage	4 7	1 0	1 0	6 5
Clearfil SE Bond	24-hours 2-year water storage	8 8	1 0	0 0	3 4
Clearfil S Tri Bond	24-hours 2-year water storage	10 10	0 0	0 0	2 2
Hybrid bond	24-hours 2-year water	12 12	0 0	0 0	0 0

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After 24 hours of water storage, the bond strength of tested self-etch adhesives showed a comparable bonding capability to enamel as to the total-etch systems. Other studies, however, suggest that the bond strength of self-etch adhesives to enamel is inferior.[1-5] For the acid-etch systems, the primary mechanism of adhesion to enamel is based on micromechanical retention. When phosphoric acid is applied to enamel, a preferential dissolution of interprismitic enamel permits the creation of 1 µm wide resin tags between the enamel, as well as resin infiltration within the surface of prisms to produce enamel hybrid layers. Resin tags filling the microporous enamel surface have been considered to be of major importance in bonding to etched enamel.[36-37] For the self-etching adhesives, the self-etching primer dissolves the surface area of individual enamel crystallites and widens the intercrystallite spaces. In addition, dissolution along the C-axis of the crystallites was observed.[38] A three-dimensional inter- and intracrystallite nanoretentive etching pattern is thereby created, while simultaneously promoting monomer infiltration. This etching pattern only produced resin infiltration into enamel to a depth of 0.6–0.7 µm.[39] However, even this shallow resin penetration gave good resin–enamel bond strengths. The interprismitic resin tags that are typically observed in phosphoric acid-etched enamel were not seen when bonding with these self-etching primers, which are relatively weak. Apparently, resin infiltration between enamel crystallites creates nanometer-sized resin tags that can contribute to high resin-enamel bond strengths even in the absence of interprismitic resin tags. This resin-infiltrated enamel surface layer might be considered as the resin–enamel interdiffusion zone or hybrid layer.[40-41]

After two years of water storage, the bond strength of all tested adhesives was reduced; however, such a difference was only significant in Clearfil S Tri Bond (Kuraray Medical, Inc) and Hybrid Bond (Kuraray Medical, Inc). Performance of the tested adhesives related to water storage may be dependent on the ability of individual components to resist deterioration by water storage and to the mechanism of achieving bonding to enamel.

Clearfil SE Bond (Kuraray Medical, Inc) is a two-step, mild self-etching primer-adhesive. The primer of Clearfil SE Bond contains 10-MDP as a functional monomer dissolved in HEMA and water to result in a pH of around 2. The primer of this material produced minimal changes in surface topography, resulting in a very superficial resin infiltration. Similar findings were also reported by Perdigão and others.[39] In spite of such low minimal surface changes reported with Clearfil SE Bond, the bond strength was close to that reported with the etch and rise system after 24 hours. Also, the bond was stable after two years of water storage. Again, this was attributed to the inter- and intracrystallite

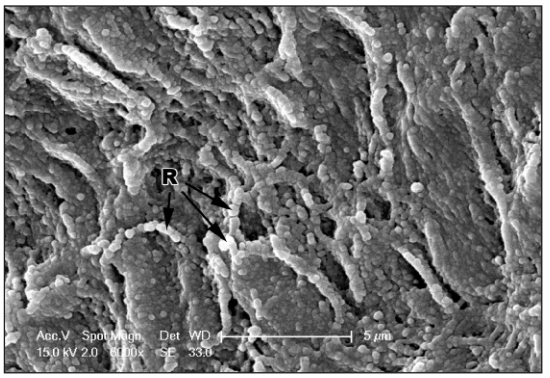

Figure 3: *Fracture surface of Admira Bond specimens after two years of water storage. Resins were still present in the interprismitic substances as well as the top prism itself.*

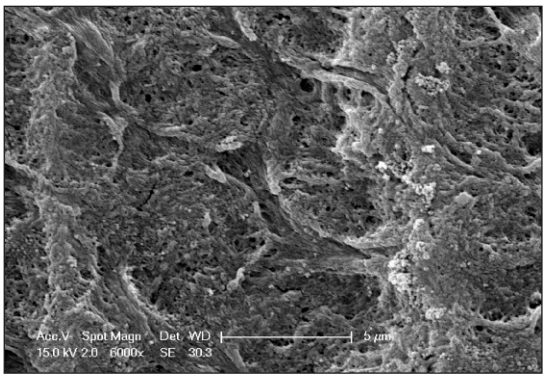

Figure 4: *Fracture surface of Hybrid Bond after two years of water storage. Resins were lost and interprismitic spaces are visible. Also, resins were lost from the prism itself.*

hybridization of the enamel rather than dissolution and resin-tag formation.[40] This was suggested to be further augmented by secondary bonds, which have formed between hydrophilic resins and enamel. Accordingly, enamel adhesion is believed to be a twofold mechanism. One is micro-mechanical interlocking and the other is a chemical interaction between functional monomers of adhesives and enamel.[42] These bonding mechanisms could probably explain the favorable performance of SE Bond (Kuraray Medical, Inc) after two years of water storage.

Futurabond DC (Voco) showed a bond strength value of 29 ± 3.5 MPa after 24 hours of water storage. After two years of water storage, such value was 26 ± 2.4, which is not significantly different from the 24 hour ones. Futura-bond DC contains polyfunctional adhesive monomers (phosphoric acid modified methacrylate esters). These acid esters, when mixed with water, produced a pH value of 1.4.[43] Accordingly, the smear layer was mobilized and the hydroxyl apatite was dissolved (demineralized), creating a retentive pattern on the enamel surface. During this process, the acids in the bonding agent are neutralized by hydroxyapatite in the

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tooth structure; that is, this process only runs until the acid is exhausted. Once the surface has been conditioned, the surface area was increased and bonding resin was penetrated into the etched pattern analogous to conventional etching with phosphoric acid, producing a retentive adhesion.[44] Chemical bonding also takes place in the surface of the tooth structure due to complexation of the calcium by the adhesive.[45]

Hybrid Bond (Sun Medical) is an "all-in-one" adhesive that contains 4-META as an active monomer component. In an aqueous environment, this monomer is converted to the dicarboxylic acid 4-MET, which is the etching component of Hybrid Bond with a pH of around 1.[46] This high acidity resulted in rather aggressive demineralization. Consequently, the underlying mechanism of bonding of Hybrid Bond is primarily diffusion-based, similar to that of the total-etch approach. Furthermore, the hydrophilic end of 4-MET offers the advantages of forming ionic bonds to the calcium in apatite.[46] This bonding mechanism, which seems to encapsulate the apatite crystals with a relatively hydrophobic MMA bonding resin, may explain the excellent performance of this material at 24 hours. However, after two years of water storage, such bond mechanisms were unable to resist deterioration by water exposure. A possible reason for such findings could be the absence of coupling hydrophobic bonding agent. Rapid water sorption can occur via the hydrophilic and permeable adhesive layer.[33]

The chemical composition of Clearfil S Tri Bond (Kuraray Medical, Inc) was quite similar to Clearfil SE Bond (Kuraray Medical, Inc). On enamel, it also produced minimal changes in surface topography, resulting in a very superficial resin infiltration. In contrast to Clearfil SE bond, the material showed lower bond strength value. Again, this could be attributed to the absence of coupling hydrophobic bonding agent, which made such material behave as permeable membranes after polymerization.[47]

In the current study, only adhesives and resin composites from the same manufacturer were used. This eliminated the risk of incompatibility between any of the adhesives and a single resin composite. However, there is some evidence to indicate that different composites with different elastic moduli result in different bond strength outcomes. These should be taken into consideration during interpretation of the outcome of the current study. Future researchers should evaluate the effect of other degradation factors, for example, thermocycling on bond strength to enamel.

CONCLUSIONS

After 24 hours of water storage, the bond strength of self-etching adhesive systems to ground enamel are dependent on the type of adhesive. Some of these adhesives showed bond strength values comparable to that of etch-and-rinse systems. After two-years of water storage, the bond strength of the etch-and-rinse system was stable. For self-etch systems, all adhesives showed a reduction in bond strength that was significant for some materials.

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