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Effect of Water Storage on Bond Strength of Self-etching Adhesives to Dentin

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Abstract

Aim: The objective of this study was to evaluate the bond strength of self-etching adhesive systems one week and one year after storage in water.

Methods and Materials: Fragments from the buccal surfaces of 45 bovine teeth were prepared (12 mm in length X 5 mm in width X 1.0 mm in thickness). Dentin surfaces were wet-abraded with 600-grit SiC paper to create a standardized smear layer. Samples were randomly assigned to 18 experimental groups (n=5), according to nine adhesive systems tested (Single Bond; Adper Prompt L-Pop; iBond; One-Up Bond F; Xeno III; Clearfil SE Bond; Optibond Solo Plus SE; Tyrian SPE/One-Step Plus; and UniFil Bond) and two water-storage times (one week and one year). Adhesives were applied according to the manufacturers' instructions. Z250 composite was applied into the molds to fill up the internal diameter volume of a Tygon tubing mold (1.0 mm high / 0.7 mm internal diameter). Micro-shear bond strengths were determined using an apparatus attached to an Instron Universal Testing Machine (0.5 mm/min). Data were statistically analyzed using a two-way analysis of variance (ANOVA) and the Tukey's test (5%).

Results: One year after water storage the dentin bond strength of all adhesive systems reduced significantly, except for One-Up Bond F.

Conclusion: Water-storage time decreased the bond strength for most dentin bonding agents tested.

Keywords: Dentin, adhesive systems, bond strength, water storage, composite resin, bond durability

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Introduction

Contemporary self-etching systems have been described since 1994. Phenyl-P was the first acidic monomer responsible for preparing enamel and dentin for bonding of resin-based restorative materials.[1,2] Although self-etching adhesive systems are a relatively new category of dental adhesives, the earlier generations of dentin bonding agents could also be considered as self-etching primers since halophosporous ester-based primers of Bis-GMA or HEMA were applied to unconditioned dentin.[3-5]

Self-etching primers and adhesives are composed of aqueous solutions of acidic functional monomers with a pH relatively higher than phosphoric acid etchants. The water is necessary to provide the medium for ionization and action of these acidic resin monomers. HEMA monomer is added because most acidic monomers have a low solubility in water, while bi- or multi-functional monomers are important to provide strength to the cross-linking at the formed polymer matrix.[6]

The bonding mechanism of acidic monomers is promoted by carboxylic or phosphate acid groups. Self-etching bonding agents can be classified as strong, moderate, and mild, depending on their etching aggressiveness or acid dissociation constants (pKa values). According to the number of clinical steps required for use, they are divided into two-step self-etching primers and single-step "all-in-one" adhesives.[7-9]

The self-etching adhesives offer some advantages over conventional etch and rinse systems such as reduction of postoperative sensitivity, less sensitive technique, and simplification of bonding procedures because they do not require a separate acid conditioning step and moist post-rinse control. However, the most important advantage of self-etching systems is the infiltration of the adhesive resin occurring simultaneously with the self-etching process.[7,10]

A category of bonding agents designed to simplify bonding procedures includes one-bottle self-priming etch and rinse and single-step "all-in-one" self-etching adhesive systems.[7] However, the simplification has resulted in loss of bonding effectiveness because single step self-etching adhesives are more hydrophilic systems and form a hybrid layer more permeable to water.[11] The increase in acidic monomer concentration in self-etching systems is required for etching through the smear layer into the underlying intact dentin.[12] The ionization promoted by water reduces the pH of the adhesive creating an aggressive adhesive solution depending on the concentration of hydrophilic acidic monomer.[11,13] Thus, the premature degradation of resin-dentin bonds has been aggravated by the increase in concentration of hydrophilic acidic monomer in single-step self-etching systems.[14,15]

The bonding mechanism of self-etching adhesives has been intensely investigated and described.[10,12,16,17] However, the durability of composite restorations bonded with self-etching adhesives still remains questionable. The long-term effects of incorporating dissolved hydroxyapatite crystals and residual smear layer remnants within the bond are still unknown. Thus, the purpose of this study was to evaluate the micro-shear bond strength of self-etching systems after one week and one year of water storage compared with the bond strength of an etch and rinse single bottle adhesive to dentin. The null hypothesis tested was bond strength is not influenced by the water storage time.

Methods and Materials

Forty-five freshly extracted bovine incisors stored at 6ºC were used in this study. Buccal and lingual surfaces were wet-abraded with 200-grit SiC paper (Carborundum, Vinhedo, SP, Brazil) to remove the enamel and obtain flat dentin surfaces. The roots were removed using a diamond disk (Isomet, Buehler, Evanstone, IL, USA). The crowns were sectioned longitudinally to create two dentin slices of similar size (12 mm in length X 5 mm in width X 1.0 mm in thickness) resulting in two specimens from each tooth. The flat dentin from the buccal surfaces were wet-abraded with 600-grit SiC paper (Carborundum) to create a standardized smear layer, and the dentin fragments were randomly divided into 18 experimental groups (nine adhesive systems and two storage times). The specimens from the same tooth were not used in the same group.

The materials tested, the manufacturers, and their compositions are described in Table 1. Nine adhesive systems were evaluated: four two-step self-priming systems (Clearfil™ SE Bond; OptiBond® Solo Plus™; Tyrian™ SPE/One-Step® Plus; and UniFil Bond™), four one-step self-etching adhesives (Adper Prompt L-Pop™; iBond™; One-Up Bond F™; and Xeno III™), and one two-step etch and rinse single bottle adhesive (Single Bond™). Adhesive systems and Z250™ composite (3M ESPE) resin were used according to the manufacturer's instructions.

The methodology developed by Shimada et al.[18] was used to prepare specimens for the micro-shear test (Figure 1). Three cylindrical translucent molds (Tygon tubing, TYG-030, Saint-Gobain Performance Plastic, Maime Lakes, FL, USA) were positioned over the bonded dentin of each dental fragment and filled with Z250™ composite resin using a composite instrument (#1/2, Duflex-SS White, Rio de Janeiro, RJ, Brazil). The tube molds were then removed to expose the resin composite cylinders (0.7 mm diameter by 1.0 mm high/0.38 mm^2) bonded to the dentin surfaces. This resulted in three bonded small resin cylinders being obtained for each dental fragment. Composite cylinders were checked under an optical microscope (30X). The cylinders had no interfacial defects, bubble inclusion, and no leakage of composite. The restored dental fragments were stored in distilled water at 37ºC for one week and one year.

Each dental fragment was attached to the testing device with cyanoacrylate glue (Super Bonder, Loctite, Itapevi, SP, Brazil), and each composite cylinder was tested in a 4411 Universal Testing Machine (Instron Corp., Canton, MA, USA). A shear load was applied to the base of the composite cylinder with a thin wire (0.2 mm diameter) at a crosshead speed of 0.5 mm/min until failure.

The micro-shear bond strengths were calculated and expressed in MPa. The mean values for each experimental group were calculated from the bond strength mean of each dental fragments (n=5) which were obtained from the three composite cylinders bonded to dentin. The results were analyzed statistically using a two-way analysis of variance (ANOVA) and the Tukey test at the 5% level of significance. The statistical analysis was done using a personal computer program (SAEG, version 9.0, Federal University of Viçosa, Viçosa, MG, Brazil).

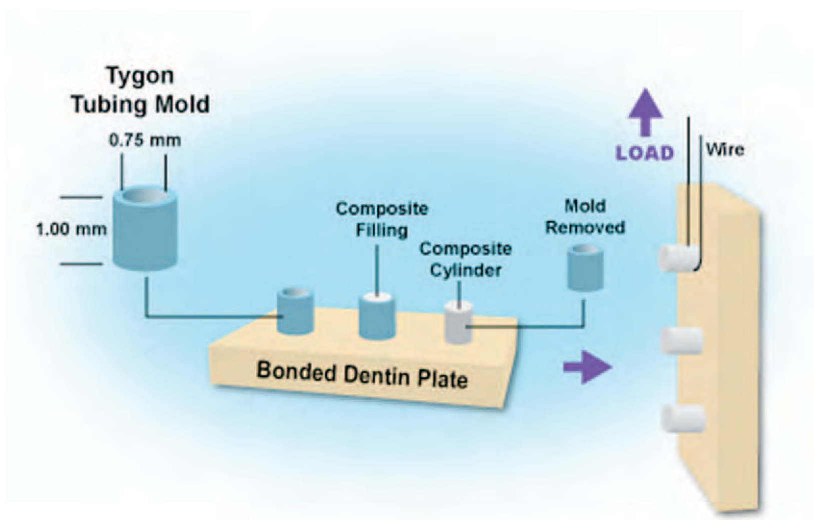

Figure 1. Schematic representation of specimen preparation.

Table 1. Composition of adhesive systems used in this study.

Adhesive Systems	Composition	PH primer	Manufacturer
Single Bond (etch and rinse)	Etchant: 35% phosphoric acid Primer and adhesive resin: Bis-GMA, HEMA, ethanol, water, UDMA, Bisphenol A glycerolate, polyalkenoic acid copolymer, dimethacrylate, camphorquinone	4.3	3M ESPE, St. Paul, MN, USA
Clearfil SE Bond (self-primer)	Primer: Water, ethanol, MDP, HEMA, dimethacrylate hydrophilic, camphorquinone, N, N-diethanol p-toluidine Adhesive resin: MDP, Bis-GMA, HEMA, dimethacrylate hydrophobic, camphorquinone, N, N-diethanol p-toluidine, silanated colloidal silica	2.0	Kuraray Medical Inc., Kurashiki, Okayama, Japan
OptiBond Solo Plus SE (self-primer)	Primer: Ethyl alcohol, water, Alkyl dimethacrylate resins, stabilizers, activators Adhesive: uncured methacrylate ester monomers, photo initiators, stabilizers	1.5	Kerr, Orange, CA, USA
Tyrian SPE/One-Step Plus (self-primer)	Primer: 2-Acrylamido-2-methyl propanesulfonic acid, Bis (2-(methacryloyloxy) ethyl) phosphate, ethanol Adhesive: Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, glass frit	0.5	Bisco Inc., Schuamburg, IL, USA
UniFil Bond (self-primer)	Primer: HEMA, 4-MET, ethanol, water Adhesive resin: UDMA, HEMA, TEGDMA, silanated colloidal silica	2.2	GC Corp., Itabashi-ku, Tokyo, Japan
Adper Prompt L-Pop (self-etching adhesive)	Liquid 1 (red blister): Methacrylated phosphoric esters, Bis-GMA, initiators based on camphorquinone, stabilizers Liquid 2 (yellow blister): Water, HEMA, polyalkenoic acid copolymer, stabilizers	0.8	3M ESPE, St. Paul, MN, USA
iBond (self-etching adhesive)	UDMA, 4-MET, glutaraldehyde, acetone, water, photo-initiator, stabilizer	1.6	Heraeus Kulzer, Hanau, Germany
One-Up Bond F (self-etching adhesive)	Bonding A: Water, MMA, HEMA, coumarin dye, metacryloyloxyalkyl acid phosphate, MAC-10 Bonding B: multifuntional methacrylic monomer, fluoraluminosilicate glass, photoinitiator (arylborate catalyst)	2.6	Tokuyama Dental Corp, Taitou-ku, Tokyo, Japan
Xeno III (self-etching adhesive)	Liquid A: HEMA, purified water, ethanol, butylated hydroxy toluene, highly dispersed silicon dioxide Liquid B: phosphoric acid functionalised polymethacrylate resins, di- and polyfunctionalised methacrylate resins, butylated hidroxy toluene, camphorquinone, 4-dimethylamino-ethyl-benzoate	1.4	Dentsply DeTrey, Konstanz, Germany

Bis-GMA=bisphenol-glycidyl-methacrylate; HEMA=2-hydroxyethylmethacrylate; PAA=polyalkenoic acid copolymer; MMA, methyl methacrylate; MAC-10=methacryloxyundecane dicarboxylic acid; TEGDMA=triethylene glycol dimethacrylate; MMA=methyl methacrylate; MDP=10-methacryloyloxydecyl dihydrogen phosphate; 4-MET=4-methacryloxyethyl trimellitate anhydride; UDMA=urethane dimethacrylate.

After testing, the debonded dentin samples were dehydrated in ascending ethanol concentrations (25%, 50%, 75%, 95%, and 100%), immersed in hexamethydisilazane (HMDS) for 10 minutes, mounted on aluminum stubs after drying, gold/palladium sputter coated (SCD 050, Baltec, Vaduz, Liechtenstein), and then observed under high vacuum of a scanning electron microscope (SEM) (VP-435, Leo, Cambridge, United Kingdom). Photomicrographs of representative areas of the fractured surfaces were taken at 100X, 500X, and 1000x magnification for fracture pattern evaluation.

Results

Mean shear bond strength values are presented in Table 2. The two-way ANOVA revealed there were statistically significant differences for the factor “adhesive system” ($p<0.0001$) and for the factor “storage time” ($p<0.0001$). In addition, it identified a significant interaction between the two factors ($p<0.0001$). The Tukey test showed significant differences among adhesives and storage time ($p<0.05$). One-year water storage produced significant decrease on bond strength for all adhesive systems tested ($p<0.05$), except for One-Up Bond F™. The lowest bond strength was observed for iBond adhesive system in both storage times.

The SEM pictures included were only representative of each fracture pattern according to storage time. Specimens tested after seven days showed fractures between the adhesive resin and composite, cohesive failure within the dentin, or partial cohesive in the adhesive layer (Figures 2 to 4).

The most predominant failure patterns on groups stored for one year were adhesive in nature (between the adhesive resin and composite) (Figure 5).

Discussion

Studies have indicated bond strength and sealing quality produced by bonding agents to dentin substrate decrease with time *in vitro* and *in vivo*.[18-22] In this current investigation

Table 2. Mean values (MPa) and standard deviation of the tensile bond strength of the adhesive systems tested after two storage times (n=5).

Adhesive Systems	Storage Time	
	7 days	1 year
OptiBond SOLO Plus SE	58.9 (8.6) a A	29.9 (7.6) a B
Single Bond	53.4 (6.5) a A	27.2 (4.2) ab B
Clearfil SE Bond	43.1 (7.1) b A	27.6 (5.3) ab B
UniFil Bond	41.5 (7.6) b A	20.7 (7.2) bc B
Adper Prompt L-Pop	29.4 (6.1) c A	16.2 (3.7) cd B
Xeno III	26.5 (7.5) c A	12.5 (4.1) d B
Tyrian SPE+One-Step Plus	25.6 (2.7) c A	12.7 (2.3) d B
One-Up Bond F	22.9 (2.8) c A	22.5 (8.1) ab A
iBond	16.1 (2.0) d A	11.5 (0.4) d B

Means followed by different letters (lower case – column, upper case – row) differ among them by Tukey test at the 0.05 confidence level.

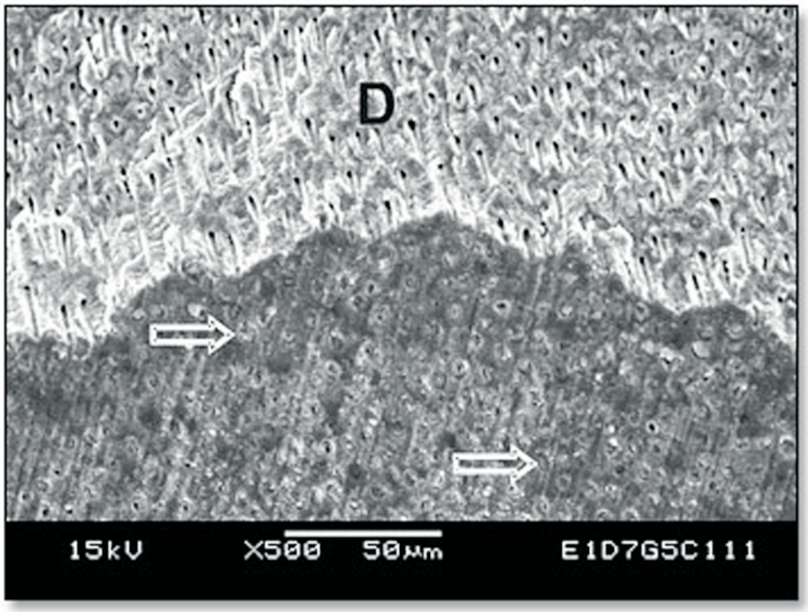

Figure 2. SEM photomicrograph illustrating a mixed fracture for group bonded with One-Up Bond F™ and stored in water for seven days. Fracture within the dentin (D) and failure between dentin and composite with dentinal tubules occluded by resin tags *(arrows)*.

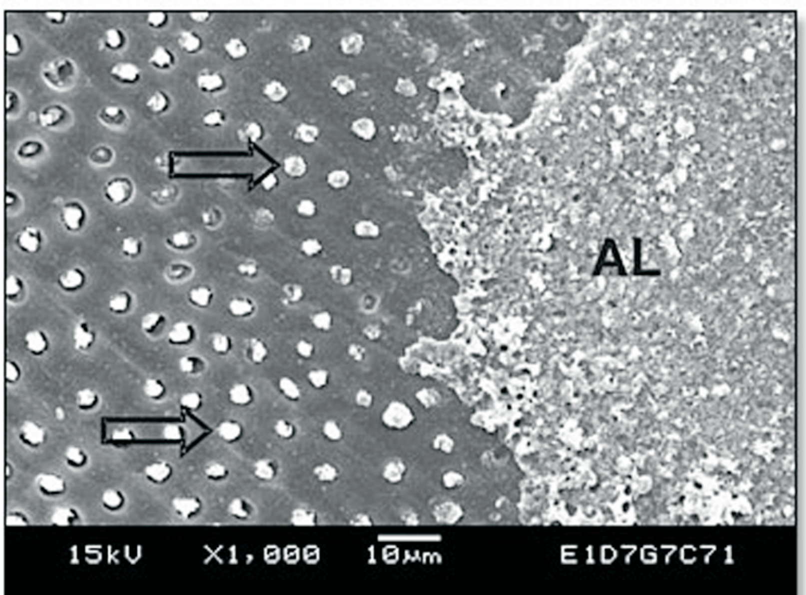

Figure 3. SEM photomicrograph illustrating a mixed fracture for group bonded with Tyrian SPE/One-Step Plus and stored in water for seven days. Cohesive fracture in adhesive layer (AL) and failure between dentin and composite with dentinal tubules occluded by resin tags *(arrows)*.

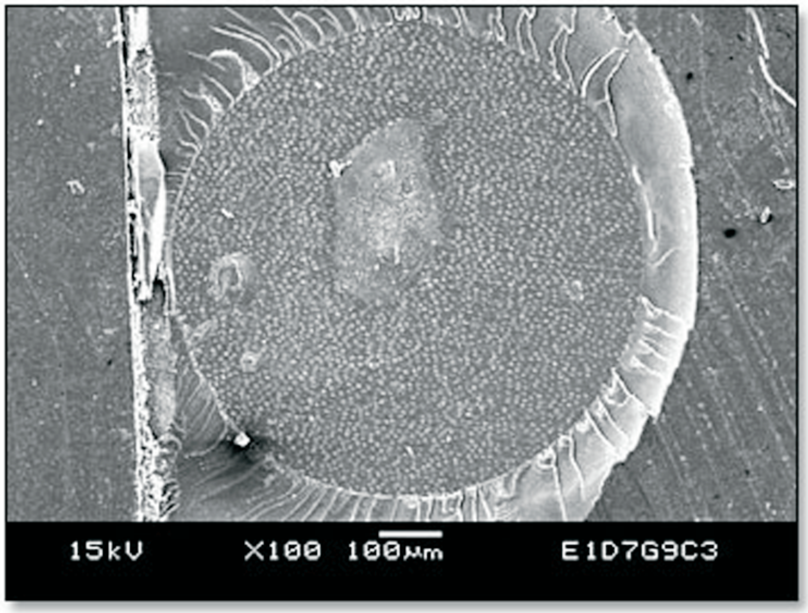

Figure 4. SEM photomicrograph illustrating a fractured surface for group bonded with Xeno III and stored in water for seven days. Adhesive failure exposing dentinal tubules occluded by resin tags.

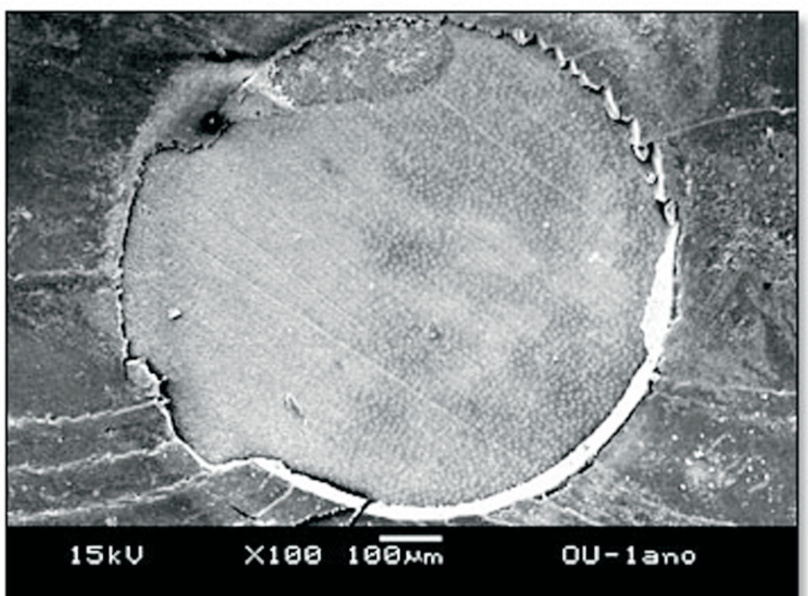

Figure 5. SEM photomicrograph illustrating a fractured surface for group bonded with One-Up Bond F and stored in water for one year. Failure between dentin and composite.

the performance of adhesive systems also indicated a decrease in bond strength for most tested adhesive systems after one year of water storage. The null hypothesis was partially rejected because one of the bonding agents was not influenced by water storage time. The main mechanism contributing to lower bond strength values is related to hydrolytic degradation of the adhesive polymer over time. This degradation of bonds has increased with use of simplified bonding techniques which include the use of more hydrophilic bonding agents.[8,11]

The self-etching primer systems Clearfil™ SE Bond, OptiBond® Solo Plus™ SE, and UniFil Bond™, which comprise the application of an acidic primer and a hydrophilic adhesive resin (two-step), produced the higher bond strengths after one week and one year of water storage. A critical review of the longevity of adhesion to dental tissues[8] examined the degradation process of bonding agents with time and concluded the two-step self-etching adhesives and the three-step etch and rinse conventional adhesive systems are more effective systems in terms of durability. The results of this current bond strength study confirmed the greater effectiveness of two-step self-etch adhesives when compared with simplified dentin bonding systems. The hydrophobic resin applied over primed dentin allows the increase in the degree of conversion of the adhesive resin and reduction of hydrophilic characteristics of the bonding agent.[18]

Because hydrophilicity and hydrophobic components present antagonistic properties, single step self-etching adhesives form a hybrid layer with incomplete adhesive infiltration into the dentin substrate. The formed hybrid layer exhibits microscopic water-filled channels that allow water movement from underlying dentin to the adhesive-composite areas.[10-23] Moreover, the water can diffuse back from the bonded dentin into hydrophilic adhesive resins after drying since hydrophilic resins attract water.[24-26] Thus, the increase in the amount of hydrophilic resin monomers in one-step self-etching adhesive compositions could jeopardize the durability of resin-dentin bonds.[27,28]

The Tyrian™ SPE/One-Step® Plus system is considered a two-step self-etching primer adhesive; however, its bond strength was similar to one-step self-etching adhesives. The SPE primer is considered a strong self-etching adhesive with a very low pH (0.5). Moreover, the hydrophilicity is increased by application of One-Step Plus bonding agent, which contains HEMA, BPDM, and acetone instead of a purely hydrophobic adhesive resin. The adhesive hydrophilicity results in increased water sorption, decreasing the hydrolytic stability.[29,30] The lack of hydrophobic material over primed dentin at resin-dentin interfaces bonded with the Tyrian™ SPE/One-Step® Plus system may be responsible for the low results of bond strength over time.[10,11,14,31]

Studies have shown milder versions of self-etching adhesives offer an appropriate pH for long-term durability of the bonding. Since they are able to demineralize the dentin and infiltrate the adhesive monomer, self-etching bonding agents classified as mild seem appropriate options for clinical use.[7,30] In this study the results indicated one year of water storage did not change the bond strength of One-Up Bond F™ self-etching adhesive. The pH of this material is 2.6, and it is close to the ideal acidity and etching aggressiveness. One-Up Bond F™ is a simplified bonding agent, and the bond strength stability may be related to low etching aggressiveness and low acid dissociation constants.[30] Moreover, each adhesive contains specific functional hydrophilic monomer that can determine its performance and hydrolytic stability over time.

The Single Bond™ adhesive system is a combination of hydrophilic primer (HEMA, polyalkenoic acid copolymer), hydrophobic adhesive resins (Bis-GMA, UDMA, dimethacrylates), and organic solvent (ethanol) in an adhesive solution requiring a previous separate step of phosphoric acid etching.5,32 Although this two-step etch and rinse adhesive showed higher initial bond strength means, such values decreased after one year of water storage. The compromised bonding is related to the difficulty for the simplified two-step adhesives to fully infiltrate the demineralized collagen mesh and to the removal of all residual solvents (ethanol) with lower vapor pressure.[17,22] Single Bond™ adhesive was included in this study for comparison with the performance of self-etching systems. However, the results showed water storage affected the bond strength for all categories tested, except for One-Up Bond

F™. A significant interaction between the two factors (bonding agent and storage time) showed the bond strength for most adhesive systems evaluated in this study can be altered as a function of water storage.

Bovine teeth were used in this study as the histochemical, and the comparative morphological studies have revealed all mammalian teeth are essentially similar.[33] Studies performed with bovine teeth have already generated and will continue to generate important information with regard to adhesion concepts.[6,8,34] Although adhesion studies should ideally be carried out on vital human teeth in the oral environment to evaluate dental adhesives, bovine teeth have been considered as possible substitutes for human teeth in adhesion tests.[33,34]

In summary, for most bonding agents tested, initial bond strength results (seven days) to bovine dentin showed acceptable values. However, this study showed concerns regarding the longevity of resin-dentin bond, especially for the durability of adhesive restorations bonded with some simplified systems.

Conclusions

Regardless of bonding agent brands, the bond strength for most of the tested adhesives one year after water storage showed a reduction of approximately 50%.

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