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Long-term Durability of One-step Adhesive-composite Systems to Enamel and Dentin

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Clinical Relevance

Resin-enamel and resin-dentin bonds formed by one-step adhesive-composite systems from the same manufacturer were not hydrolytically stable after accelerated aging in water.

SUMMARY

This study evaluated the long-term durability of three one-step adhesive-composite systems to ground enamel and dentin. Twenty-seven teeth were randomly divided into three groups of nine. The first group had its crowns sectioned to expose superficial dentin, which was then ground with 600 grit SiC paper. One of three one-step adhesives: a trial bonding agent, OBF-2; i Bond or Adper Prompt L-Pop was applied to the dentin of three teeth and built-up with the corresponding resin composite (Estelite Σ, Venus or Filtek Supreme). The second group of nine teeth had their enamel approximal surfaces ground with wet 600-grit SiC paper, then one of the three one-step adhesives was applied and built-up with resin composite. The bonded specimens were sliced into 0.7 mm-thick slabs. After 24 hours and one year of water storage at 37°C, the slabs were sectioned into beams for the microtensile bond strength test. Failure modes were observed using optical and electron scanning microscopy. The third group of nine teeth had approximal wedge-shaped cavities prepared above the CEJ into

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DOI: 10.2341/07-166

dentin. Two-to-three grains of rhodamine B were added to each of the three adhesives prior to restoring the cavities with resin composite. After 24 hours storage, the teeth were sectioned and their interfaces examined with a laser scanning confocal microscope. The bond strengths of the three adhesive-composite systems to both enamel and dentin significantly lessened after one year of water storage, however, there was no significant difference between the materials.

INTRODUCTION

The continued development of dental adhesives by manufacturers has resulted in the availability of materials that can simultaneously etch, prime and bond to enamel and dentin in one step. A shortage of clinical trials of such materials has resulted in a reliance on *in-vitro* testing in order to evaluate the durability of their adhesion to enamel and dentin, which is important if the marginal integrity of a composite restoration is to be maintained.[1] *In-vitro* durability tests commonly involve storage in water for long periods, thermal cycling and occlusal loading.[1]

Development of the microtensile test has enabled teeth to be bonded and sectioned into slabs prior to testing.[2] Storing slabs less than 1 mm thick in water at 37°C for up to one year will challenge the resin-enamel/dentin bond to hydrolytic degradation and thus provide useful information on the stability of the bond if they are subsequently prepared for bond strength testing.[3] When compared to the storage of whole teeth in water, the narrow diameter of the sectioned slabs can accelerate the speed at which water can enter the resin-enamel/dentin interface. Nanometer-sized channels within the resin/dentin interface allow the ingress of fluid into the hybrid layer.[4] Degradation of the resin-dentin bond is thought to then occur due to either removal of resin components from the hybrid layer or depletion of any incompletely resin-infiltrated collagen fibrils within the demineralized dentin zone.[5]

Single-step adhesives are either supplied as a single bottle or packaged such that the active ingredients are separated until use. One of the active ingredients is a functional monomer, which demineralizes and possibly chemically bonds to the hydroxyapatite of enamel and dentin when hydrolyzed in the presence of water.[6] If the single-step adhesive is supplied as a single bottle, the functional monomer can immediately dissociate into a hydrolytic unstable acidic monomer because of water, which is present at a concentration of 30%-50%.[6] This has led to questions regarding the shelf life of a single-step adhesive, particularly if it is not refrigerated, since hydrolysis of the functional monomers can cause a reduction in their adhesion to dentin.[8] Very little information has been published on the durability of recently introduced one-step adhesives to enamel.

Recent research has shown that selection of a composite material and adhesive from different manufacturers may influence the bond strength of adhesive systems to dentin;[9] however, there has been very little published research on whether this is also true for enamel or after long-term storage periods. On the other hand, it has been recommended that adhesives be tested using a resin-based composite recommended by the same manufacturer to avoid possible adverse chemical interactions.[10]

Therefore, this study evaluated the durability of the bond of three single-step adhesive-composite systems to both enamel and dentin. To avoid possible long-term chemical incompatibility, the adhesives were matched with the manufacturer's recommended resin composite. The null hypothesis was that the durability of the three tested single-step adhesive-composite systems to enamel and dentin would not be affected by long-term water storage.

METHODS AND MATERIALS

Twenty-seven caries-free human third molars, which had been extracted within the previous three days and refrigerated in water following the protocol of the ethics committee of King's College London, were used in this study. These molars were divided into a group of 18 for microtensile bond strength testing and a group of nine for examination of the bonded interface, using laser scanning confocal microscopy.

Microtensile Bond Strength Test

For the group of 18 teeth, each tooth was randomly assigned to one of two groups, for either dentin or enamel bonding. Each group of nine teeth was then randomly subdivided into three groups of three teeth for bonding, with one of three adhesives and the manufacturer's respective composite (Tables 1 and 2).

To obtain superficial dentin, the crowns of the teeth were sectioned using a low-speed diamond saw with water lubrication (Isomet, Buehler, Lake Bluff, IL, USA). Any remaining enamel was removed by polishing with wet SiC paper. An artificial smear layer was obtained by wet grinding with 600-grit SiC paper.

To obtain ground enamel, the approximal surfaces of the teeth were carefully ground with wet 600-grit SiC paper to give a flat surface approximately 3 x 3 mm. One of three adhesives was then applied to the enamel and dentin surfaces according to the manufacturer's instructions (Tables 1 and 2). Each manufacturer's respective resin composite was then applied and incrementally built-up to a height of 5 mm. Light-activated polymerization was performed using a halogen light curing unit (Optilux, Demetron Research Corporation, Danbury, CT, USA) whose power density was checked prior to use.

Table 1: *Chemical Composition of the Three Adhesives*

Material	Manufacturer	Composition
Adper Prompt L-Pop (APL-P)	3M ESPE, St Paul, MN, USA	Methacrylated phosphonic acid ester, water, photoinitator, fluoride complex, phosphonine oxide
i-Bond (i-B)	Heraeus Kulzer, Hanau, Germany	UDMA, 4-MET, acetone, glutaraldehyde, water, photoinitator, stabilizer
OBF-2 (now marketed as One Up Bond F plus)	Tokuyama Dental Company, Tokyo, Japan	Bottle A: MAC-10, phosphonic ester monomer, polyfunctional monomer Bottle B: HEMA, water, photoinitiator, fluoro-alumino silicate glass

All the bonded specimens were then stored in distilled water at 37°C for 24 hours. After storage, each tooth was sectioned into 0.7 mm thick slabs using the water-cooled low-speed diamond saw. Immediately, and after one year of water storage at 37°C, two dentin specimens and three enamel specimens were randomly selected from each sectioned tooth and transversely sectioned into beams with a diameter of less than 1 mm. The widths of each beam were measured with a micrometer (Moore & Wright, Sheffield, England) in order to obtain its cross-sectional area. From the two dentin slabs and three enamel slabs, a maximum of 15 beams could be harvested for testing. Beams that failed during preparation prior to bond strength testing are included in the results as zero bond strengths. The water in which the slabs were stored was changed weekly until testing. In order to carry out the microtensile bond strength test, each beam was carefully glued (Loctite Ltd, Dublin, Ireland) to custom-made metal holders mounted on a SMAC linear electric moving coil actuator (SMAC Inc, Carlsbad, CA, USA) and stressed until failure at a speed of 1 mm/minute.

After carefully removing the broken beams from the testing apparatus, an optical microscope was used to examine all sides of the broken interfaces. From each subgroup, two specimens, which were considered to have failed adhesively, were randomly selected for scanning electron microscopy and their broken ends subsequently gold-coated (SEM, Hitachi S3500 N). The failure mode was subsequently classified as either:

1. Mixed cohesive/adhesive.
2. Cohesive failure in resin.
3. Cohesive in enamel or dentin.

Laser Scanning Confocal Microscope Observation of the Bonded Interface

In the group of nine teeth, approximal wedge-shaped cavities (coronal wall 1.5 mm, pulpal wall 2.5 mm, with a 90° internal angle) were prepared coronal to the cemento-enamel junction using high-speed carbide fissure burs under water-cooling.[11] Two-to-three grains of rhodamine B fluorescent dye were added to each of the adhesives prior to application to the cavity surface. Three teeth were bonded with each adhesive and filled with the corresponding resin composite in two increments. Each increment had a maximum thickness of 2 mm and was polymerized with a halogen light-curing unit (Optilux, Demetron Research Corporation). The roots were removed with a high-speed bur under water-cooling and the pulp removed with a bar broach. The teeth were stored in water for three hours, then sectioned longitudinally using a low-speed diamond saw with water lubrication (Isomet, Buehler) and polished using wet 1200 grit silicon carbide paper.[11] A confocal laser scanning microscope (Ultraview CSU10, Perkin Elmer LSR, Cambridge, UK) was used to obtain images of the bonded enamel and dentin interfaces.

Statistical Analysis

The bond strength data were analyzed using Stata version 9 with significance pre-determined at

Table 2: *Manufacturers' Instructions for Application of the Adhesives and Composite Build-up Material*

Material	Instructions for Application	Composite Build-up Material
Adper Prompt L-Pop (APL-P)	press and fold metal sachet, apply to tooth surface for 15 seconds, gently air dry, apply second coat for 3 seconds, gently air dry, light-cure for 10 seconds	Filtek Supreme (A3)
i-Bond (i-B)	shake capsule and apply to tooth surface 3 times, wait 30 seconds, gentle air drying then thorough air drying, light-cure for 20 seconds	Venus Σ (A3)
OBF 2	Mix one drop of bonding agent A and Bonding agent B, apply to tooth surface, vigorously rub for 10 seconds, apply 2 times, light-cure for 10 seconds	Estelite Σ (A3)

Table 3: *Univariate Summary Statistics of Microtensile Bond Strength Test*

Material	Substrate	1 Day			1 Year		
		n	BS/MPa	Sd/MPa	n	BS/MPa	Sd/MPa
Adper Prompt L-Pop	Dentin	15	36.68	16.83	15	29.47	13.33
	Enamel	15	23.52	13.78	13	18.55	5.85
i-Bond	Dentin	15	38.82	12.53	15	30.48	10.56
	Enamel	15	17.86	8.81	10	20.83	8.27
OBF-2	Dentin	15	43.88	9.39	15	33.41	16.11
	Enamel	15	25.35	9.37	15	15.46	6.86

n Sample size
BS Bond strength
sd standard deviation

Table 4: *Cox's Proportional Hazard Model*

Variable	HR	95 % CI	P>lzl
i-Bond	1.19	0.82 to 1.74	0.357
OBF-2	0.98	0.68 to 1.42	0.924
Enamel	3.96	2.81 to 5.61	0.001
1 year	1.90	1.39 to 2.61	0.001

HR Hazard ratio
95% CI 95% confidence interval about the hazard ratio

α=0.05. The Kaplan-Meier non-parametric analysis was used to estimate the survival function, and the log-rank test was used to compare the survival distributions. Cox's proportional hazard model was used to estimate the effect of the bonding system, substrate and time.

RESULTS

A summary of the descriptive statistics from the results of the microtensile bond strength test are given in Table 3. The data was analyzed for the effect of bonding agent, substrate and storage time using a Cox proportional hazard model following preliminary analysis to establish that the assumption of proportional hazards was valid. The results are shown in Table 4 relative to Adper Prompt L-Pop, dentin and 24-hour storage. There was no significant effect of material, but bonding to enamel and storage for one year caused a significant reduction in bond strength. As a consequence, subsequent analysis was carried out, treating enamel and dentin separately. Kaplan-Meier survival curves are shown in Figures 1 and 2. Within each substrate, the log-rank test, in conjunction with Sidak's adjustment for multiple comparisons, was used to test the equality of the survivor function for each material-storage combination. Of the 30 possible combinations, there were only two significant differences: Dentin, OBF-2 one day—Adper one year; Enamel, OBF-2 one day—OBF-2 one year.

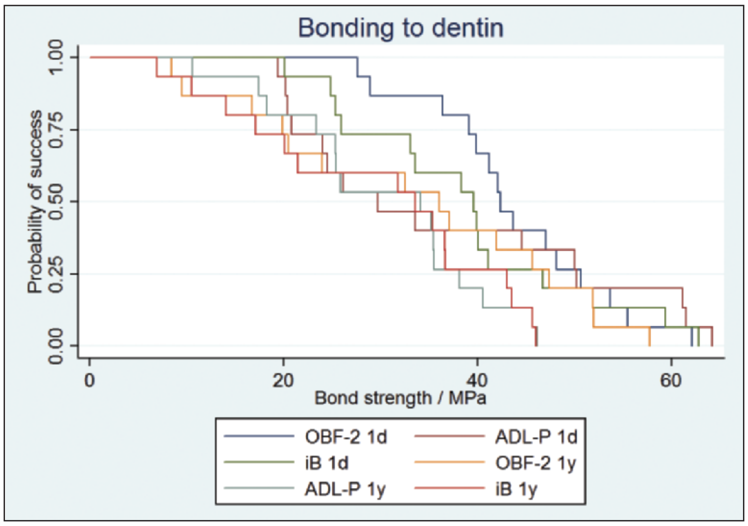

Figure 1. *Kaplan-Meier survival curves for the adhesives bonded to dentin.*

The distribution of the locus of failure for the system-substrate-storage combinations is shown in Figures 3 and 4. The hypothesis of independence between mode of failure and material was tested using χ^2 in a permutation test to allow for the small sample size in some of the cells. For all substrate storage combinations, there were no significant differences.

Scanning electron microscopy revealed that those enamel and dentin specimens that initially appeared to fail adhesively at the bonded interface exhibited a mixed failure mode, which was predominantly cohesive in resin (Figures 5-7). Laser scanning micrographs of the resin-dentin bonded interfaces of the wedge-shaped cavities restored with i-Bond and Venus showed the presence of crack formation at the adhesive interface. In the case of the resin-dentin interface of OBF-2/Estelite Sigma, there is the suggestion of the presence of water droplet formation (Figure 8). All three adhesives appeared to infiltrate both the dentinal tubules and intertubular dentin (Figure 8).

DISCUSSION

In the current study, the long-term durability of three one-step self-etching adhesive-composite systems to enamel and dentin was evaluated. The microtensile bond strengths of all three tested adhesive-composite systems, when bonded to both enamel and dentin, reduced after storage in water for one year. In the case of dentin, these findings are in agreement with the results of previous studies and indicate that the resin-dentin bonded interfaces of all three one-step adhesives were susceptible to hydrolytic degradation.[5,12] Failure patterns were, however, similar for the specimens stored for one day and one year in water. The majority of the enamel and dentin specimens exhibited a mixture of cohesive and adhesive failure at the interface (Figures 5-7). Hydrolytic degradation of the resin-dentin bond is thought to arise due to water penetration through nanoleakage pathways from the dentin margins increasing the number of nanoleakage spaces within the hybrid layer.[13] Laser scanning micrographs of the bonded interfaces of restored wedge-shaped cavities show cracking at the bonded interface (i-Bond) and possibly water droplet formation (OBF-2) (Figure 8). However, the actual mechanism of resin-dentin bond degradation is still unclear. The null hypothesis of the current study must therefore be rejected.

There is very little published research on the stability of the resin-enamel bond formed using recently introduced one-step adhesives after long-term water storage. Previous research has shown that, when enamel was etched with phosphoric acid, the resin-enamel bond did not significantly reduce after one year of water storage.[14] In the current study, the ground enamel was not etched with phosphoric acid to comply with the manufacturers' instructions. The results of the current study are not in agreement with previous research and indicate that the resin-enamel bond formed using a one-step adhesive was not stable in water. The reason for the significant reduction in bond strength is not clear; however, it may have been due to the fact that one-step adhesives can act as semi-permeable membranes.[15] Tay and others found that, when one-step adhesives were bonded to enamel but not covered in composite and then immersed in water, water blisters were observed in the enamel, which resulted in partial delamination of the adhesive.[16] Although the adhesives were covered with composite in the current study, the significant reduction in bond strength after one year of water storage indicates that this phenomenon might have been partly responsible for the reduction.

It has been proposed that the chemical composition of the monomers of the adhesives might affect long-term bond durability.[17] The tested adhesives in the current study differed in the chemical structure of the incorporated monomers. The adhesive iB contains acetone and 4-MET in a single bottle, whereas, OBF-2 contains MAC-10 and water in separate bot-

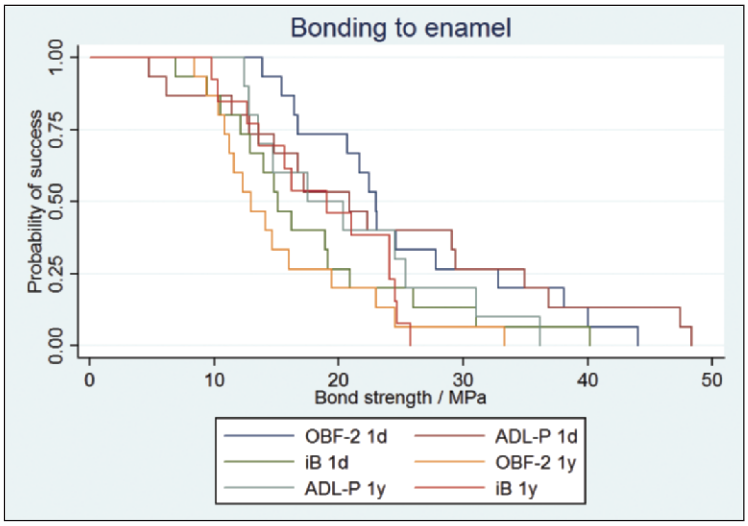

Figure 2. *Kaplan-Meier survival curves for the adhesives bonded to enamel.*

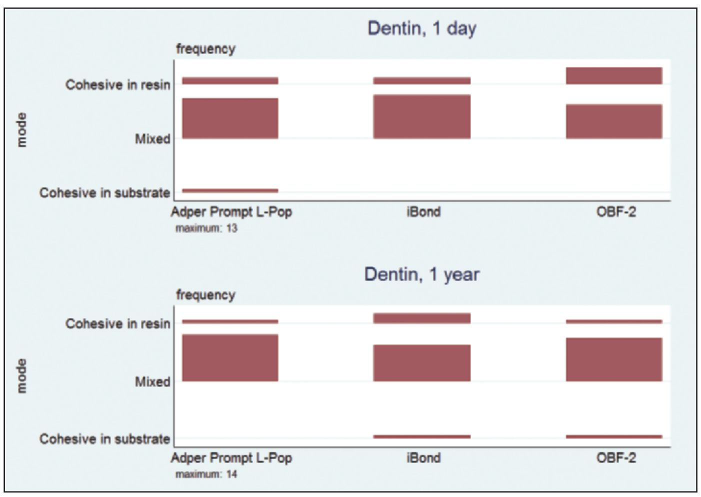

Figure 3. *The distribution of locus of failure of specimens for the adhesive system-dentin-storage period.*

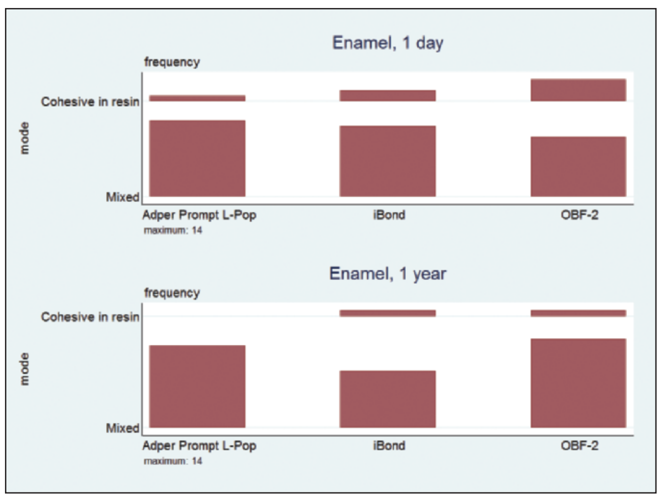

Figure 4. *The distribution of locus of failure of specimens for the adhesive system-enamel-storage period.*

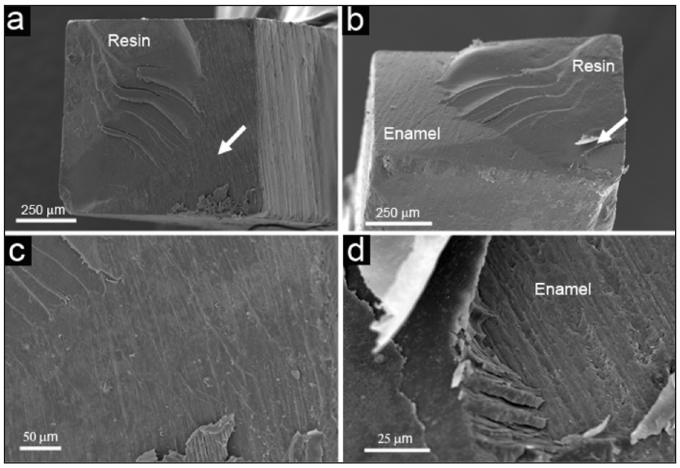

Figure 5. *Scanning electron micrographs of the opposing ends of a fractured beam from the OBF-2 1-Day storage enamel group. A mixed adhesive/cohesive failure mode is evident. a) Resin end of the beam; the arrow indicates the area viewed at higher magnification in 5c, 90x. b) Enamel end of the beam; the arrow indicates the area viewed at higher magnification in 5d, 100x. c) High magnification showing an area of adhesive failure, 300x. d) High magnification showing tearing at the interface of resin and enamel, 1000x.*

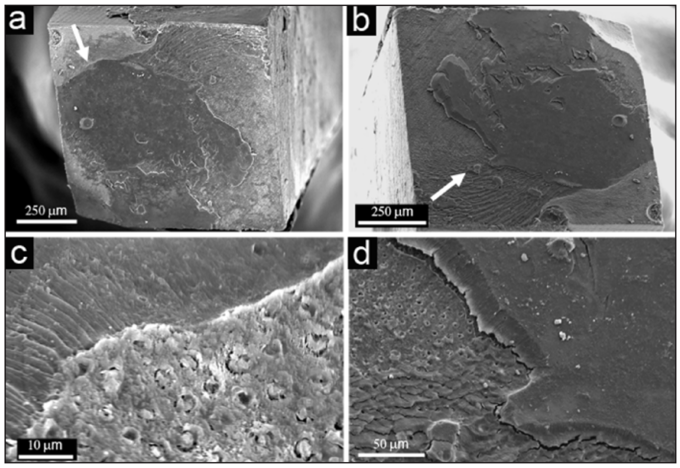

Figure 6. *Scanning electron micrographs of the opposing ends of a fractured beam from the i-Bond 1-Day storage dentin group. A mixed adhesive/cohesive failure mode is evident. a) Resin end of the beam—the arrow indicates the area viewed at higher magnification in 6c, 90x. b) The dentin end—the arrow indicates the area viewed at higher magnification in 6d, 100x. c) High magnification showing cohesive failure of dentin on the resin surface and tearing of resin, 2000x. d) High magnification of the dentin end showing patent tubules, 500x.*

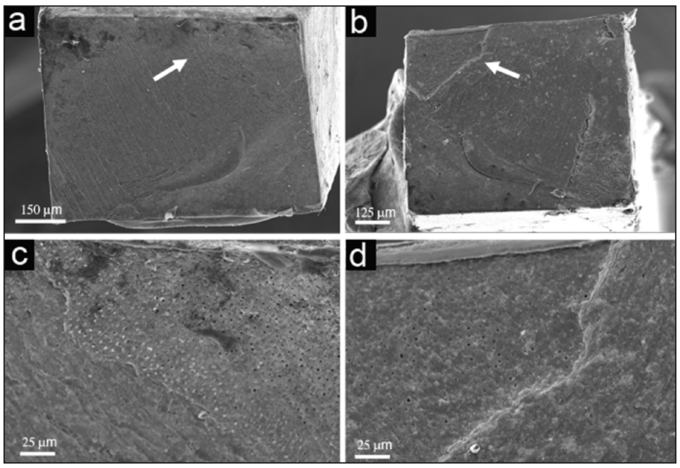

Figure 7. *Scanning electron micrographs of the opposing ends of a fractured beam from the i-Bond 1-Year storage dentin group. A mixed adhesive/cohesive failure mode is evident. a) Resin end of the beam—the arrow indicates the area shown at higher magnification in 7c, 120x. b) The dentin end—the arrow indicates the area viewed at higher magnification in 7d, 100x. c) High magnification showing cohesively fractured dentin on the surface of the resin, 500x. d) High magnification showing adhesive failure at the dentin surface and cohesive failure of resin, 500x.*

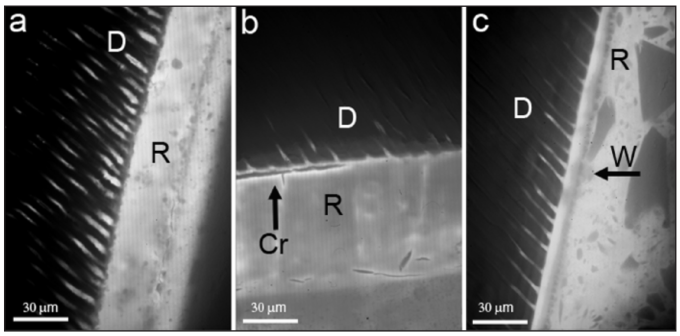

Figure 8. *Fluorescent images of the dentin-resin adhesive interfaces of the three adhesives. a) Adper Prompt L-Pop. b) i-Bond; cracking is visible at the resin-dentin interface (Cr). c) OBF-2; water droplets appear to be visible at the bonded interface of (W). D=dentin, R=resin; CSU10 confocal laser scanning microscope 60x/NA 1.40 oil immersion objective.*

tles. The clinical application of these two adhesives is slightly different after application to the substrate, in that iB must be gently air-dried before polymerization to remove the acetone, whereas for OBF-2, it is not necessary, since the solvent is mainly water. The results of the current study revealed that, for both the enamel and dentin groups, there was no significant difference among the three adhesive-composite systems after one day and one year of water storage. In addition, the mode of specimen failure was similar at each storage interval for all three adhesives. This indicates that the different chemical compositions of the adhesive-composite systems did not have a significant affect on resin-enamel and resin-dentin bond durability and therefore the null-hypothesis must again be rejected.

Previous research has reported that self-etching adhesives, which combine the acidic methacrylate monomers with water in a single bottle, are hydrolytically unstable and require refrigeration to maintain their effectiveness.[8] The current study investigated both single bottle (iB) and two-bottle (OBF-2) self-etching adhesive-composite systems. While adhesives are presented with an expiration date, their date of manufacture is not usually included on their packaging.

Therefore, when the adhesive is first opened, the operator cannot be sure of the time interval since manufacture and also how the material was stored during shipping. In the current study, the three tested adhesives were refrigerated upon delivery and used as soon as possible. For both substrates and storage periods, there was no significant difference between bond strengths of the three systems. This indicates that, if the adhesive is kept refrigerated and used as soon as possible upon delivery, combining the acidic monomers with water in a single bottle did not result in reduced performance compared to an adhesive that presented as two bottles. However, the effect of long-term storage of a single-bottle adhesive at temperatures intermittent between refrigeration and room temperature on the durability of resin-dentin and resin-enamel bond strengths, as occurs in every day clinical dentistry, was not investigated and merits further investigation.

The size of the bonded specimen, when stored in water, has been discussed by researchers, with some preferring to store sectioned beams as opposed to sectioned slabs.[5] This is because, the smaller the cross-sectional area, the greater the potential for an accelerated "aging effect." In the current study, sectioned slabs were stored in water, then sliced into beams after the prescribed period of water storage. Both the enamel and dentin groups significantly reduced in bond strength after one year of water storage, which demonstrates that both the size of the stored specimen and duration of storage were adequate for an effect to be observed.

CONCLUSIONS

1. The resin-dentin and resin-enamel bonds of the three adhesive-composite systems reduced after one year of water storage.
2. There was no significance among the bond strengths of the three adhesive-composite systems after both one day and one year of water storage for each substrate.

(Received 14 December 2007)

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