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Laboratory Research

Influence of Air Abrasion and Long-term Storage on the Bond Strength of Self-etching Adhesives to Dentin

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Clinical Relevance

The adhesiveness of self-etching systems, used with aluminum oxide air abrasion to dentin, decreases over time.

SUMMARY

This study tested the effects of long-term storage and aluminum oxide air abrasion on the bond strength of self-etching adhesive systems. Extracted human third molars were ground flat with 600-grit SiC paper to expose middle coronal dentin. Clearfil SE Bond and One-Up Bond F were applied to dentin surfaces in accordance with manufacturers instructions with or without previous aluminum oxide 50 µm air abrasion. A crown was built up with the resin composite TPH Spectrum and the specimens were stored in water for 24 hours. The bonded assemblies were vertically sectioned into beams for microtensile bond testing. The beams of each tooth were individually immersed in bottles containing water at 37°C for one day, three and six months; the water was changed daily. The specimens were then subjected to microtensile bond testing. The bond strength data were subjected to ANOVA and Tukey Kramer test. Fractured specimens were analyzed in a scanning electron microscope to determine failure modes. Air abrasion improved Clearfil SE Bond bond strength in the three month evaluation. No significant difference was found between the two adhesives systems, but bond strengths gradually decreased over time. Failure modes varied significantly among groups and were influenced by long-term storage and aluminum oxide air abrasion.

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INTRODUCTION

The longevity of esthetic restoration is directly related to the effectiveness of adhesive systems, as the lack of bonding and inadequate marginal sealing may lead to restoration failure.[1-2]

Self-etching adhesive systems use non-rinse acidic monomers that simultaneously condition and prime dentin. The tooth is no longer rinsed, which not only lessens the clinical application time, but also significantly reduces technique-sensitivity.[3] Furthermore, these systems reduce incomplete penetration of the fluid resin into the collagen network and thus might prevent degradation of the hybrid layer in the long term, since these systems simultaneously demineralize and impregnate the dentin with fluid resin.[4] Among the self-etching adhesives, there are two-step systems that require an additional bonding step and all-in-one adhesives, which combine the etching, priming and bonding into a single step application.[5-7]

According to Coli and others,[8] dental pre-treatments that enhance tooth roughness may affect bond strength by improving interfacial contact between dentin and the adhesive surface. Aluminum oxide air abrasion is commonly used in dentistry to increase the adhesion of metal surfaces to resin material[9] and remove caries and faulty restorations for cavity preparations.[10] Moreover, aluminum oxide air abrasion has also been used as a pre-treatment to increase the surface roughness of enamel and dentin surfaces, which, in turn, may positively affect bond strength.[11] Removal of the smear layer by aluminum oxide air abrasion might improve the infiltration of adhesive systems into demineralized dentin, which may result in significantly higher bond strengths.[12]

Durability of the bond between resin and dentin is of critical importance to the longevity of bonded restoration.[13] Adhesive system assessment tests are generally performed 24 hours after specimen fabrication. This time interval is insufficient for demonstrating changes that may occur in the bond strength of the adhesive system over time.[14-15] Thus, it is important to assess the effect of dentin pre-treatment with aluminum oxide air abrasion on the adhesive properties of self-etching adhesive systems over time.

METHODS AND MATERIALS

At this point, 72 human third molars were used in this study (n=72). After extraction, the teeth were cleaned with water slurry of pumice flour in a rubber prophylaxis cup at low speed and stored in distilled water at room temperature to prevent dehydration.

The teeth were longitudinally sectioned at the mid-coronal portion using a double-faced diamond disk (KG Sorensen Ind e Com Ltda, Barueri, São Paulo, Brazil) at low speed under water/air cooling to obtain flat, smooth occlusal dentin of medium depth. A second section was made 5 mm below the cemento-enamel junction, at the root furcation level, using number 2 and 4 spherical burs (SSWhite, Rio de Janeiro, Brazil). The pulp chamber floor and pulp tissue were removed with the aid of dentin curettes (Duflex, SSWhite).

Through access to the pulp chamber, dentin thickness was measured with the aid of a thickness meter, and a 2 mm thickness was uniformly established by means of wear with 600-grit silicon carbide abrasive paper.

In order to facilitate specimen sectioning, pulp chambers were filled with the resin composite TPH Spectrum (Dentsply, Petrópolis, Rio de Janeiro, Brazil) after application of the adhesive system (Clearfil SE Bond or One-Up Bond F). Next, dentin surfaces were finished by manual abrasion using 600-grit silicon carbide abrasive paper (3M, Sumaré, São Paulo, Brazil) for 15 seconds to obtain a uniform smear layer thickness.

The teeth were randomly divided into 12 experimental groups, as described in Table 1 (n=6). Commercial brand name, basic composition, manufacturers method of use and lot of the adhesive systems used in this study are listed in Table 2.

In Groups 1, 2, 3, 7, 8 and 9, the dentinal surface of experimental specimens was air abraded with 50 µm aluminum oxide particles for 10 seconds at 60 psi air pressure by using a Microetcher intraoral appliance (Bioart, São Carlos, São Paulo, Brazil) placed close to the dentin (± 0.5 cm). Figure 1 illustrates an air abrasion treated dentinal surface.

Bonding procedures were carried out in accordance with the manufacturers recommendations. Next, resin composite TPH Spectrum (Dentsply) was inserted in three increments, individually light cured for 40 seconds using an XL 3000 light-curing unit (3M ESPE, Grafenau, Germany), with a constant intensity of 580 mW/cm^2. The light intensity of the light-cure unit was assessed with a radiometer (Demetron Research Corporation, Danbury, CT, USA). A crown was built-up over the adhesive to a final height of 6 mm. Bonded assemblies were stored in distilled water at 37°C for 24 hours.

Table 1: *Experimental Groups*

Group	System	Air Abrasion	Storage Duration
1	Clearfil SE Bond	With	1 day
2	Clearfil SE Bond	With	90 days
3	Clearfil SE Bond	With	180 days
4	Clearfil SE Bond	Without	1 day
5	Clearfil SE Bond	Without	90 days
6	Clearfil SE Bond	Without	180 days
7	One-Up Bond	With	1 day
8	One-Up Bond	With	90 days
9	One-Up Bond	With	180 days
10	One-Up Bond	Without	1 day
11	One-Up Bond	Without	90 days
12	One-Up Bond	Without	180 days

Table 2: *Bonding Systems Used*

System	Composition (main components)	Bonding Steps	Manufacturer and Batch #
Clearfil SE Bond	Primer: MDP, HEMA, hydrophilic dimethacrylate, CQ, N,N-Diethanol p-toluidine, water. Bond: MDP, BisGMA, HEMA, hydrophobic dimethacrylate, CQ, N, N-Dietanol p-toluidine, Silanate colloidal silica.	Gentle air dry, apply primer for 20 seconds, light curing. Apply adhesive, gentle air dry, 10 seconds light curing.	Kuraray Co, Osaka, Japan Batch: 61155
One-up Bond F	Methyl methacrylate; HEMA; MAC 10; F aminosilicate glass, water.	Place one drop of each agent into the mixing receptacle, mix the two agents until a pink, homogenous pink is obtained, apply the mixture to the tooth structure, wait for 20 seconds, do not remove excess, and photo-polymerize for 10 seconds or longer, to guarantee the color change from pink to colorless.	Tokuyama Co, Tokyo, Japan Batch: U4830Z1

Abbreviations: Bis-GMA: bisphenol glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxy methacrylate; CQ: camphorquinone; MAC 10: 11-methacryloyloxy-1, 1-undecandicarboxylic acid.

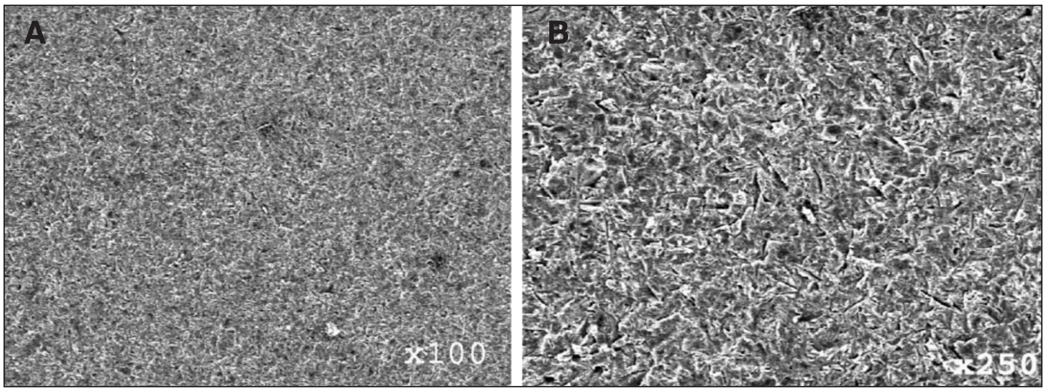

Figure 1. *Figure illustrating aluminum oxide air abraded dentin (magnification 100x and 250x).*

The samples were then serially sectioned vertically from the resin composite, parallel to their long axes in the mesio-distal and lingual vestibular directions at 1 mm intervals using a high concentration diamond impregnated saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). A second section was made perpendicular to the bonded interface to make 1 x 1 mm beams. The top half of each beam was resin composite and the bottom half was dentin. Six or seven beams were obtained from each tooth.

Specimens in Groups 2, 3, 5, 6, 8, 9, 11 and 12 were individually stored at 37°C in vials with distilled water.[16-17] The water was changed daily for 90 (Groups 2, 5, 8 and 11) or 180 days (Groups 3, 6, 9 and 12). The specimens were then prepared for microtensile testing.

The specimens were attached with cyanoacrylate-based glue (Super Bonder Gel, Henkel Loctite Adhesives, Ltda, Itapevi, São Paulo, Brazil) to the flat grips of a microtensile testing device coupled to a universal test machine (Instron Co, Canton, MA, USA) and tested at a crosshead speed of 0. 5 mm/minute until failure. Then, the specimens were carefully removed from the device with a scalpel blade, and the cross-sectional area at the fracture site was measured to the nearest 0.01 mm using a digital pachymeter (Starret 727-6/150, Starret SP/Brazil) to express the results in MPa.

After the microtensile test, the dentinal portions were separated, and the fractured dentin surfaces, facing upwards, were mounted on aluminum stubs (Procind Ltda, Piracicaba, São Paulo, Brazil), metalized (SCD 050 sputter Coater, Baltec), gold sputter-coated and assessed by a scanning electron microscope (JEOL, JSM-5600LV, scanning electron microscope, Tokyo, Japan) to determine the fracture mode. Failure modes were classified as one of five types (adaptation of the model described by Montes and others[18] and Tanumiharja and others[19]): type 1, adhesive failure between adhesive resin and dentin; type 2, partial adhesive failure between adhesive resin and dentin and partial cohesive failure in the adhesive resin; type 3, complete cohesive failure in the adhesive system; type 4, partial cohesive failure in dentin; type 5, cohesive failure in resin composite. The fracture mode results were submitted to non-parametric statistical analysis using the chi-squared and Cochran-Mantel-Haenszel (CMH) tests to study fracture mode distribution.

Table 3: *Comparison of the Means (MPa) of the Air Abrasion Factor Within the Levels of the Time and Adhesive Factors by the Tukey Test*

Storage (days)	Adhesive	Air Abrasion	n	Mean (SD)	Tukey
1	Clearfil	With	36	27.0 (7.2)	A
		Without	41	31.8 (7.5)	A
	One-Up	With	36	28.2 (8.3)	A
		Without	36	29.4 (7.9)	A
90	Clearfil	With	39	26.7 (9.4)	A
		Without	40	19.8 (10.6)	B
	One-Up	With	40	26.9 (8.4)	A
		Without	42	24.2 (8.7)	A
180	Clearfil	With	36	20.7 (7.5)	A
		Without	35	19.9 (6.2)	A
	One-Up	With	38	21.0 (6.0)	A
		Without	37	17.8 (6.8)	A

Means followed by the same letter are not statistically different (p>0.05).

Table 4: *Comparison of the Means (MPa) of the Adhesive Factor Within the Levels of the Time and Air Abrasion Factors by the Tukey Test*

Storage (days)	Airborne Particle Abrasion	Adhesive	n	Mean (SD)	Tukey
1	With	Clearfil	36	27.0 (7.2)	A
		One-Up	36	28.2 (8.3)	A
	Without	Clearfil	41	31.8 (7.5)	A
		One-Up	36	29.4 (7.9)	A
90	With	Clearfil	39	26.7 (9.4)	A
		One-Up	40	26.9 (8.4)	A
	Without	Clearfil	40	19.8 (10.6)	A
		One-Up	42	24.2 (8.7)	A
180	With	Clearfil	36	20.7 (7.5)	A
		One-Up	38	21.0 (6.0)	A
	Without	Clearfil	35	19.9 (6.2)	A
		One-Up	37	17.8 (6.8)	A

Means followed by the same letter are not statistically different (p>0.05).

Table 5: *Comparison of the Means (MPa) of the Time Factor Within the Levels of the Adhesive and Air Abrasion Factors by the Tukey Test*

Adhesive	Air Abrasion	Storage (days)	n	Mean (SD)	Tukey
Clearfil SE Bond	With	1	36	27.0 (7.2)	A
		90	39	26.7 (9.4)	A B
		180	36	20.7 (7.5)	B
	Without	1	41	31.8 (7.5)	A
		90	40	19.8 (10.6)	B
		180	35	19.9 (6.2)	B
One-Up Bond F	With	1	36	28.2 (8.3)	A
		90	40	26.9 (8.4)	A B
		180	38	21.0 (6.0)	B
	Without	1	36	29.4 (7.9)	A
		90	42	24.2 (8.7)	A
		180	37	17.8 (6.8)	B

Means followed by the same letter are not statistically different (p>0.05).

RESULTS

The analysis of variance ANOVA showed that the triple interaction (Time*Adhesive*Air abrasion) was significant; it was necessary to dissociate this interaction for multiple comparisons of means by the Tukey Kramer test ($p<0.05$). Table 3 shows that dentinal air abrasion with aluminum oxide had a significant effect on the Clearfil SE Bond adhesive system after three months of storage ($p<0.05$). There were no significant differences between adhesives in spite of air abrasion or long-term storage ($p>0.05$, Table 4).

The effect of time on bond strength varied depending on the adhesive used and whether aluminum oxide air abrasion was applied ($p<0.05$, Table 5). Clearfil SE Bond with and without air abrasion showed a significantly higher bond strength at day 1 when compared with day 180 (Table 5). Finally, the One-Up Bond F adhesive system showed higher bond strength at day one, irrespective of air abrasion (Table 5).

Differences in frequencies of fracture modes were observed among air abrasion, adhesive systems and storage time (Cochran-Mantel-Haenszel test). Figure 2 illustrates the comparison of fracture mode frequencies at the different levels of air abrasion (with and without). In the group in which aluminum oxide air abrasion was applied, type 3 fractures frequently occurred (46.67%). For the samples that were not submitted to aluminum oxide air abrasion, type 3 fractures were more commonly found (38.53%) than all the other types; however, they were not statistically different from type 2 (34.20%).

Analysis of fracture mode in relation to the adhesive system revealed that, while type 1, 2 and 3 fractures were observed in the Clearfil SE Bond adhesive system with statistically the same frequency, type 3 fractures were more commonly found in the One-Up Bond F adhesive (Figure 2). In contrast, type 2 fractures occurred uniformly between the two adhesives (Figure 3).

Analysis of fracture mode in relation to Time revealed that type 3 fractures were the most common type of failure observed after 1 and 90 days of storage (Figure 4). In contrast, after 180 days storage, type 2 fractures were the most common type of fracture detected, followed by fracture types 3 and 1, and by type 5. No type 4 fractures occurred.

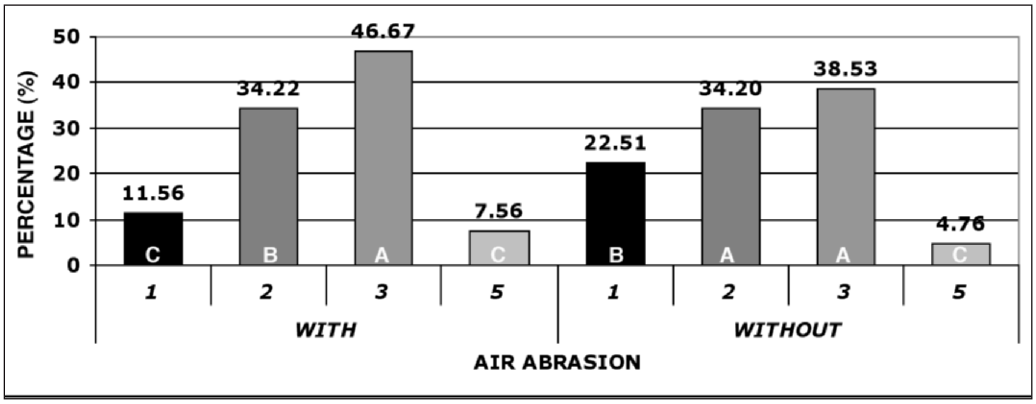

Figure 2. *Comparison of the proportions of the different types of fracture in cases with and without the application of aluminum oxide air abrasion. Bars with the same letters within the same air abrasion level do not differ by the Chi-square test (*p*=0.05).*

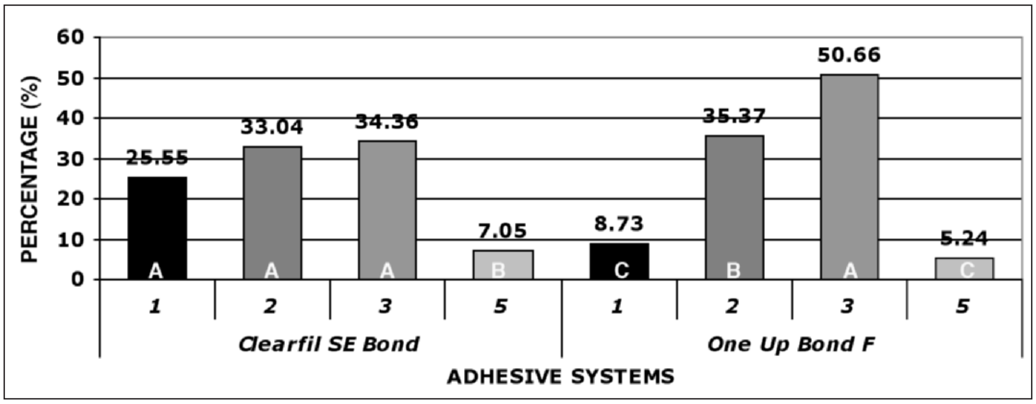

Figure 3. *Comparison of the proportions of the different types of fracture in the two adhesives used. Bars with the same letters within the same adhesive do not differ by the Chi-square test (*p*=0.05).*

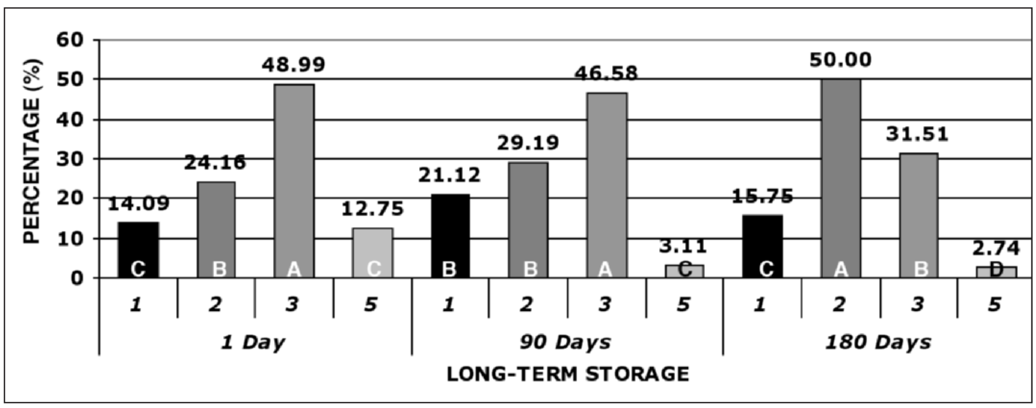

Figure 4. *Comparison between the proportion of samples of time in each type of mode fracture. Bars with the same letters within the same time do not differ by the Chi-square test (*p*=0.05).*

DISCUSSION

The authors demonstrated that Clearfil SE Bond and One-Up Bond F adhesive systems showed similar microtensile bond strength, regardless of aluminum oxide air abrasion treatment or storage time (Table 4).

The Clearfil SE Bond system contains MDP and HEMA monomers; whereas, One-Up Bond F adhesive has MAC 10 and HEMA monomers; the solvent for both systems is water, and they have methacrylate monomers and inorganic loads. The similar composition of the two systems used might partially explain their microtensile bond strength. Consistent with the results of this study, Tanumiharja and others assessed seven different adhesive systems and found no differences among the microtensile bond strengths of self-etching adhesive systems.[19] Nevertheless, some authors[5,20] observed that one-step application systems obtained lower shear bond strength means compared with two-step application self-etching systems.

In contrast, the adhesive systems had a significant influence on fracture modes (Figure 3). Adhesive fractures were three times more frequent in the Clearfil SE Bond system than in the One-Up Bond F system. The latter, in turn, accounted for half of the cohesive fractures in the adhesive. It is possible that the presence of fluoride amino silicate glass in the composition of the One-Up Bond F adhesive system prevented the loss of residual smear layer minerals, which were incorporated into the hybrid layer, resulting in a lower number of adhesive failures. It is known that fluoride has an anticariogenic action and, when incorporated with the dental structure, it increases the dental structures resistance to the acid media.[13] Fluoride can be detected in the hybrid layer and in the subjacent dentin adhering to materials that released fluoride when immersed in water.[21] It is speculated that fluoride prevents degradation of the intrinsic hybrid layer calcium phosphates that tend to solubilize in the long term, resulting in a stable bond to dentin over time.[22] Moreover, adhesive failures in the Clearfil SE Bond system may have been influenced by the slow degradation of the hybrid layer during the storage period.[22]

After three months of storage, dentinal air abrasion with aluminum oxide had a significant effect on the Clearfil SE Bond adhesive system. Although the microtensile bond strength of the two adhesive systems with aluminum oxide air abrasion was higher after three and six months of storage, these differences were not statistically significant (Table 3).

In this study, the purpose of dentinal pre-treatment with aluminum oxide was not to seek an alternative to acid etching, as it was for Roeder and others in 1995,[23] and Rinaudo, Cochran and Moore in 1997,[10] when each concluded that aluminum oxide air abrasion must always be used with acid etching. Instead, it was to seek an association between the mechanical smear layer removal by aluminum oxide air abrasion and chemical removal by the low pH of adhesive systems. Although the results showed that aluminum oxide air abrasion significantly enhanced the bond strength of Clearfil SE Bond after three months of storage, there was no difference at 1 or 180 days (Table 3). Therefore, no immediate or long-term benefits were obtained. Moreover, Chaves, Gianinni and Ambrosano investigated the effect of aluminum oxide air abrasion on self-conditioning adhesive systems and showed that air abrasion did not affect the bond strength to dentin at early time points.[12]

The results of this study demonstrated that air abrasion influenced the fracture mode. Due to the use of air abrasion, 46.67% of the fractures were cohesive in adhesive, a significantly higher percentage than that of the other fracture modes. In the groups without air abrasion, there was no difference between the percentage of cohesive in adhesive fractures and mixed fractures (partially cohesive in adhesive and partially adhesive). Cohesive in adhesive fractures represent integrity in the hybrid layer subjacent to the adhesive, protecting the dentin (Figure 6). In contrast, adhesive failures (Figure 5) denote a rupture at the dentin/resin bond interface, characterized by open dentinal tubules and intertubular dentin with collagen fibers without mineral protection. Los and Barkmeier, in 1994,[9] also found a greater frequency of cohesive failures in aluminum oxide air abraded surfaces after the shear bond strength test and reported an increase in roughness on the surfaces that had been aluminum oxide air abraded, as compared with those that were abraded with 600-grit silicon carbide abrasive paper.

Adhesive restoration longevity studies are important for providing information with regard to materials subject to water diffusion at the interface formed with the dental structure;[24] thus, the durability of adhesive system bonds to tooth structure is crucial to assure that adhesive restorations do not fail with the passage of time.[25] The results showed a reduction in bond strength at the different assessment times, depending on the adhesive system used and whether or not air abrasion was applied (Table 5).

For the two adhesive systems, it was found that the application of air abrasion was found to maintain the bond strength means similarly at day 1 and day 90

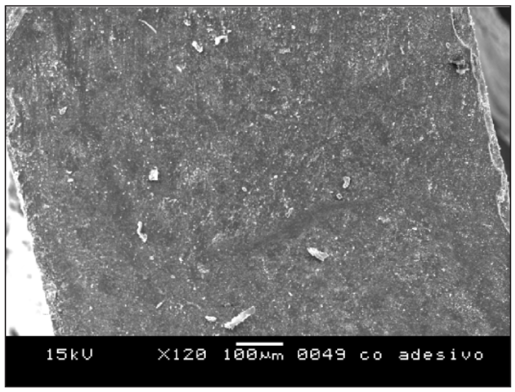

Figure 5. *Illustrates cohesive in adhesive system failure, fracture mode type 3.*

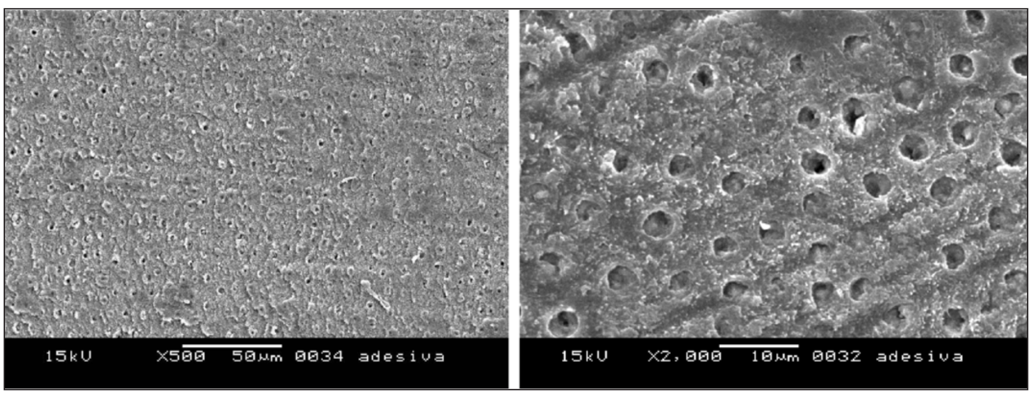

Figure 6. *Illustrates adhesive failure, fracture mode type 1.*

assessments; however, a reduction in bond strength was detected after 180 days of storage for both systems, irrespective of aluminum oxide application.

The cause of interface degradation has been attributed to the hydrolysis of demineralized collagen fibers that were not completely protected by adhesive and also to hydrolysis of the monomers that infiltrated into the dentin by water diffusion through nanoleakage channels that increased in size with the passage of time.[13,17,25-26]

Self-etching systems are composed of acid hydrophilic monomers, water, HEMA and bi-functional dimethacrylates. An increase in the concentration of acid monomers is necessary to dissolve the smear layer and etch the subjacent dentin, and water is used as a means of ionizing these acid resinous components. HEMA is added as a solvent, since some of the acid monomers are not directly soluble in water.[27] Two-step self-etch adhesives consist of a hydrophilic aqueous primer solution and a separate hydrophobic adhesive resin. Nevertheless, one-step self-etch adhesives are complex mixtures of both hydrophilic and hydrophobic components. Compared to two-step self-etch, one step self-etch adhesives consistently achieve lower bond strengths and are less stable bond over time.[28] High concentrations of water may cause harmful effects on polymerization due to incomplete water removal. This also applies to the high concentrations of solvent that may cause incomplete resin polymerization in case of incomplete evaporation.[28] Due to their high hydrophilicity, cured one-step self-etch adhesives have been shown to act as permeable membranes. The presence of these nanometric leakage pores make the hybrid layer of self-etching adhesive systems permeable to water and ion movement.[7,27,29-31] Thus, interface degradation may not necessarily have been caused by discrepancies between the depths of demineralization and resinous monomer infiltration, but by the presence of permeable areas in a polymerized matrix in which water was not completely removed, resulting in incompletely polymerized regions and/or hydrogel formation.

Sample storage also influenced the fracture mode. A decrease in the percentage of cohesive in adhesive fractures and an increase in the percentage of mixed fractures (partially cohesive in adhesive/partially adhesive—Figure 4) were noticed over time. These findings are in agreement with those of Okuda and others, who showed that both Clearfil Liner Bond 2V and Fluorobond adhesive systems demonstrated a trend toward a reduction in cohesive in adhesive failures and an increase in adhesive fractures over time.[13] It is possible that interface degradation influenced the reduction in bond strength means and caused an increase in the percentages of partial adhesive fractures and partial cohesive in adhesive fractures.

It was demonstrated that long-term storage significantly influenced the bond strength of self-etch adhesive systems over time, and both long time storage and dentinal air abrasion with aluminum oxide had a remarkable influence on failure modes. Other variables could also have positive or negative effects on bonding between adhesive systems and tooth structure. Identifying these factors is crucial in achieving stable, long-lasting esthetic restorations.

CONCLUSIONS

- Previous dentinal air abrasion with aluminum oxide did not influence the bond strength means of adhesive systems at different evaluation times, except for the Clearfil SE Bond adhesive system, which, after three months of storage, obtained higher mean bond strength to dentin values when used with aluminum oxide air abrasion.
- There were no statistically significant differences between bond strength means of the two adhesive systems used with and without aluminum oxide air abrasion at the different storage times.
- Sample storage influenced the bond strength means, because bond strength to dentin diminished significantly between 1 and 180 days.
- There were significant differences in the frequency of all types of fracture modes.

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