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ORIGINAL RESEARCH

Influence of CVD Diamond Tips and Er:YAG Laser Irradiation on Bonding of Different Adhesive Systems to Dentin

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ABSTRACT

Aim: The aim of this study was to compare the microtensile bond strength of three adhesive systems, using different methods of dentin preparation.

Materials and methods: A hundred and eight bovine teeth were used. The dentin from buccal face was exposed and prepared with three different methods, divided in 3 groups: Group 1 (DT)-diamond tip on a high-speed handpiece; Group 2 (CVD)-CVD tip on a ultrasonic handpiece; Group 3 (LA)-Er: YAG laser. The teeth were divided into 3 subgroups, according adhesive systems used: Subgroup 1-Adper Single Bond Plus/3M ESPE (SB) total-etch adhesive; Subgroup 2-Adper Scotchbond SE/3M ESPE (AS) self-etching adhesive; Subgroup 3-Clearfil SE Bond/Kuraray (CS) self-etching adhesive. Blocks of composite (Filtek Z250–3M ESPE) 4 mm high were built up and specimens were stored in deionized water for 24 hours at 37ºC. Serial mesiodistal and buccolingual cuts were made and stick-like specimens were obtained, with transversal section of 1.0 mm^2. The samples were submitted to microtensile test at 1 mm/min and load of 10 kg in a universal testing machine. Data (MPa) were subjected to ANOVA and Tukey's tests ($p < 0.05$).

Results and conclusion: Surface treatment with Diamond or CVD tips associated with Clearfil SE Bond adhesive produced significantly lower bond strength values compared to other groups. Surface treatment with Er: YAG laser associated with Single Bond Plus or Clearfil SE Bond adhesives and surface treatment with CVD tip associated with Adper Scotchbond SE adhesive produced significantly lower bond strength values compared to surface treatment with diamond or CVD tips associated with Single Bond Plus or Adper Scotchbond SE adhesives.

Clinical significance: Interactions between laser and the CVD tip technologies and the different adhesive systems can produce a satisfactory bonding strength result, so that these associations may be beneficial and enhance the clinical outcomes.

Keywords: Dentin bond strength, Adhesive system, Smear layer.

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INTRODUCTION

Dentin bonding depends on a number of factors, both related to substrate and the bonding system.[1] The total-etch adhesive systems are proposed to total removal of the smear layer produced by cutting or abrasion of dentin during the operative procedure through the use of acids.[2] The unfavorable factor in the use of total-etch adhesive systems is the sensitivity of the technique and protocol used with caution, due to the high number of steps and the subjectivity in obtaining or retaining moisture dentin, enabling the occurrence of an incomplete impregnation of the adhesive to dentin.[3-5]

In order to decrease the sensitivity of the technique emerged the self-etching adhesive systems, which dispense etching, washing and drying steps. In these systems, an acid solution is applied on dentin without smear layer removal, which is dissolved or incorporated into the hybrid layer. However, the smear layer has significant buffering capacity, able to change the ionic potential of the acid monomer used in self-etching systems, tending to limit the depth of diffusion in the underlying dentin, depending on its thickness and smoothness.[6] Some studies have shown that the smear layer thickness or the type of instrument used to prepare dentin can change the values of bond strength when using the adhesive systems to dentin.[1,7-16]

With advances in technology, other options besides the traditional cavity preparation with diamond points have emerged; among them are the laser and the CVD tips. The CVDentus® system is obtained by chemical vapor deposition of a diamond film over a molybdenum stem with a high adherence of diamond on the metallic surface. These probes are coupled to an ultrasound machine, causing an oscillatory movement with effective cleaning power, as the ultrasonic action form air bubbles in a liquid, which produces turbulent fluid, releasing energy and moving particles.[3,17] Potential advantages of this preparation method are reduced noise, minimal damage to the gingival tissue, extended bur durability, improved proximal cavity access, reduced risk of hitting the adjacent tooth resulting from the high inclination angles, and minimal risk to the patient of metal contamination.[3] Conservative cavity preparation with well-defined walls and finishing margins can be obtained when CVD tips are used.[18]

Another option for cavity preparation is the Er: YAG laser (Erbium: Yttrium-Aluminum-Granada), used to reduce tooth structure by means of a mechanism for casting of dental tissues. In this laser, the emission wavelength of 2940 nm coincides with the main absorption peak of water, resulting in good absorption in all biologic tissues, including enamel and dentin. The impact of light energy causes an instant vaporization of the water with massive volumetric expansion. This expansion causes surrounding material to ablate.[19] Removal of carious lesions and cavity preparation are possible with Er:YAG laser without thermal damage to the pulp.[20]

These innovative technologies used for dental reduction produce distinct grinding patterns and dentin surfaces with different characteristics, which may influence the bond strength of adhesive systems, thus, the aim of this study was to investigate the effect of different methods of dentin preparation on dentin bond strength of one total-etch and two self-etching adhesive systems. The null hypothesis tested is that the method used to produce the smear layer does not affect the microtensile bond strength of the adhesive systems tested.

MATERIALS AND METHODS

One hundred and thirty-five freshly extracted bovine incisors were cleaned and stored in deionized water inside a freezer at – 18°C until use. The roots were sectioned with steel flexible diamond disk in the handpiece at the cemento-enamel junction. Only the tooth crowns were used. The buccal surfaces were ground with P400 (FEPA) abrasive paper in a polishing machine (DP-10, Panambra, São Paulo, Brazil) under cooling with water, exposing a dentin area of 4 mm diameter. The teeth were embedded in self-cured acrylic resin using a silicone mold.

The specimens were randomly assigned to three groups according to the method of dentin preparation (n = 45):

- *Group 1 (DT):* The conventional diamond tip #3098 (KG Sorensen, Barueri, SP, Brazil) was mounted in a dental turbine (Super Torque, Kavo, Joinvile, SC, Brazil) at high-speed. The dentin was prepared by the same operator, who gently passed the tips 30 times mesio-distally the dentin surface.
- *Group 2 (CVD):* The CVDentus® tip C1 (Clorovale Diamantes, São José dos Campos, SP, Brazil) was operated according to the parameters recommended by manufacturer (30 kHz of frequency, 8 W of electric output power, 120 ml/min rate of water flow; 70% of its maximum power) in an ultrasound device (Profi I AS Ceramic, Dabi Atlante, Ribeirão Preto, SP, Brazil), both under water lubrication. The pattern of wear was performed in the same manner described above for diamond tip, under copious air-water spray.
- *Group 3 (LA):* The Er: YAG laser (Kavo Key Laser III, Kavo Co., Germany) was used to prepare dentin with emission wavelength of 2.94 micron energy per pulse varies between 60 and 500 mJ. The laser was focused to 15 mm of the tooth surface, with the aid of an endodontic file, calibrated with 160 mJ energy per pulse, 10 Hz frequency, resulting in an energy density of 51.3 J/cm^2.[21] The pattern of wear was performed in the same manner described above for diamond under water cooling.

Subsequently, three groups were divided into three subgroups, according to the adhesive systems (n = 15):

- *Subgroup 1 (SB):* Single Bond Plus total-etch adhesive (3M ESPE, St Paul, MN, USA)
- *Subgroup 2 (AS):* Adper Scotchbond SE self-etch adhesive (3M ESPE, St Paul, MN, USA)
- *Subgroup 3 (CS):* Clearfil SE Bond self-etch adhesive (Kuraray Medical Inc, Okayama, Japan).

The adhesive systems were applied according to the manufacturers' instructions (Table 1).

After the adhesive procedures, a block was built up over the treated dentin surfaces with a composite resin (Filtek Z250, 3M ESPE, St Paul, MN, USA), with increments of 2 mm, using a silicon matrix (4 × 4 × 4 mm). Each increment was cured using a halogen photocuring unit CL-K50 (Kondortech, São Carlos, SP, Brazil) with power density of 500 mW/cm^2 for 40 s. To complement the polymerization of the resin, the matrix was removed and the block was additionally cured for 40 s.

After storing the bonded teeth in deionized water at 37ºC for 24 hours, they were longitudinally sectioned in both 'x' and 'y' directions across the bonded interface using a diamond saw in a Labcut machine (Extec Corp, Enfield, CT, USA), under water cooling at 300 rpm to obtain bonded sticks with a cross-sectional area of approximately 1 mm^2. The cross-sectional area of each stick was measured with the digital caliper to the nearest 0.01 mm and recorded for subsequent calculation of the microtensile bond strength (Absolute Digimatic, Mitutoyo, Tokyo, Japan). Each bonded stick was submitted to a microtensile bond strength test using the L2500 specimen holder (Erios, São Paulo, SP, Brazil). The analyses were performed using a universal testing machine (DL 200MF, Emic, São José dos Pinhais, SC, Brazil), at a speed of 1 mm/min.

The samples failed during processing (pretesting failure) were counted as the lowest value measured of the group.

The bond strength values were expressed in Kgf and converted in MPa. The comparison between the groups was done using the mean of each tooth (7 sticks per tooth).

The data were subjected to two-way ANOVA (adhesive and surface treatment factors) and Tukey's test, with a significance level of 5%.

SCANNING ELECTRON MICROSCOPIC (SEM) EVALUATION

For illustrative purposes, one specimen from each type of dentin preparation was prepared for observation under SEM. They were sectioned perpendicular to the long axis using a low speed diamond saw (Labcut 1010, Extec, USA) to obtain dentin disks (1 mm high) surrounded by enamel. The mid-coronal dentin surfaces were prepared in the same manner as used for microtensile test, using a conventional diamond tip on a high-speed turbine, a CVDentus® tip on an ultrasound device and Er: YAG laser.

The specimens were then fixed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 for 12 hours at 4°C. After fixation, the specimens were rinsed with 20 ml of 0.1 M of sodium cacodylate for 1 hour, with three changes, followed by rinsing with deionized water for 1 minute. Subsequently, the specimens were dehydrated in ascending ethanol concentrations (25% for 20 minutes, 50% for 20 minutes, 75% for 20 minutes, 95% for 30 minutes and 100% for 60 minutes). After dehydratation, the specimens were immersed in hexamethyldisilazane solution (Fluka AG, Buchs, Switzerland) for 10 minutes, placed on a filter paper inside a covered glass vial, and dried at room temperature.[21] The specimens were mounted on aluminum stubs, sputter-coated with gold using a sputtering device (Desk II–Denton Vacuum, Moorestown, NJ, USA) for 2 minutes and observed using scanning electron microscopy (JSM 5310–Jeol, Tokyo, Japan).

RESULTS

ANOVA showed a p-value = 0.0001 (F = 15.13), with 4° of freedom, which indicated significant differences between adhesive systems and surface preparation. Tukey's multiple comparison test is presented in Table 2. Surface treatment with Diamond or CVD tips associated with Clearfil SE Bond adhesive produced significantly lower bond strength values compared to other groups. Surface treatment with Er: YAG laser associated with Single Bond or Clearfil SE Bond adhesives and surface treatment with CVD tip associated with Adper Scotchbond SE adhesive produced significantly lower bond strength values compared to surface treatment with Diamond or CVD tips associated with Single Bond Plus or Adper Scotchbond SE adhesives. The number of pretest failures per group is presented in Table 2.

Table 1: Composition of adhesive systems and procedures for bonding according to manufacturers' instructions

Adhesive	*Composition*	*Clinical application procedure*	*pH*
Adper Single Bond Plus Total-etch adhesive (3M ESPE, St Paul, MN, USA)	Bis-GMA, HEMA, dimethacrylate, methacrylate functional copolymer of polyacrylic and polytaconic acid, water, alcohol, photoinitiator	Etch for 15 s, rinse, dry gently; Apply adhesive, scrub for 30 s, air-thin, light cure for 10 s	4.6
Adper Scotchbond SE self-etch adhesive (3M ESPE, St Paul, MN, USA)	*Liquid A:* Water, HEMA, surfactant, pink pigment *Liquid B:* UDMA, TEGDMA, TMPTMA (trimethacrylate hydrophobic), HEMA (phosphate), MHP (phosphate methacrylate), nanoparticles of zirconia, camphorquinone	Apply primer for 20 s; apply bonding agent for 20 s; gently blow air for 10 s, light cure for 10 s	≤1.0
Clearfil SE Bond self-etch adhesive (Kuraray Medical Inc, Okayama, Japan)	Primer: Water, ethanol, MDP HEMA, hydrophilic dimethacrylate, camphorquinone, N, N-diethanol-p-toluidine Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, N, N-diethanol-p-toluidine, silanated colloidal silica	Apply primer for 20 s; gently airblow; apply bonding agent for 20 s; light cure for 10 s.	1.9-2.0 (primer)

Bis-GMA: bisphenol A glycidyl methacrylate; HEMA: bisphenol A glycidyl methacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate

When dentin was prepared with a diamond tip, deep grooves were observed on SEM analysis of surface, with large amount of smear layer and dentin tubules totally or partially obliterated (Figs 1A and B). Figures 2A and B show CVD tip specimens' preparation, with some tubules partially open, and lower amount of smear layer. The dentin surface prepared with Er:YAG laser exhibited tubules completely opened, absence of smear layer and uneven surface (Figs 3A and B).

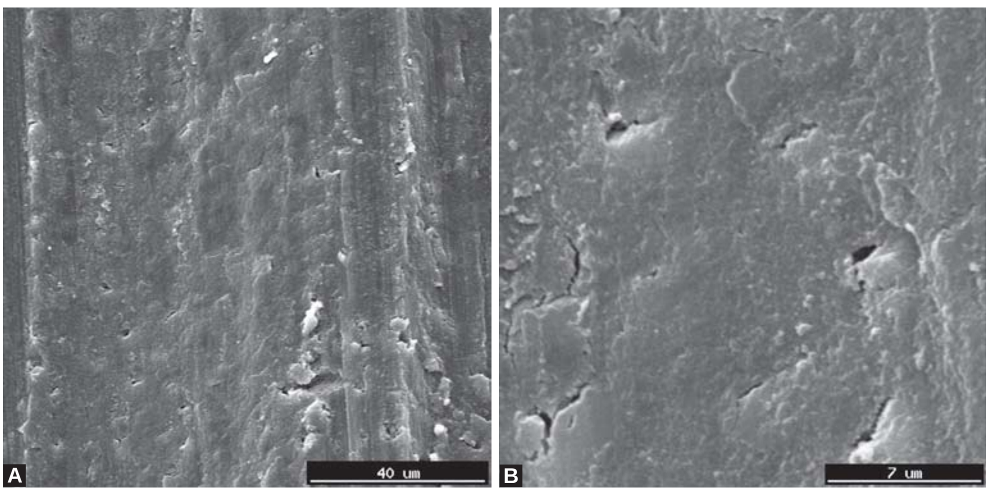
DISCUSSION

In this study, three different methods of dentin preparation were evaluated: The conventional high-speed diamond bur, and the innovative technologies of CVD tip mounted in an

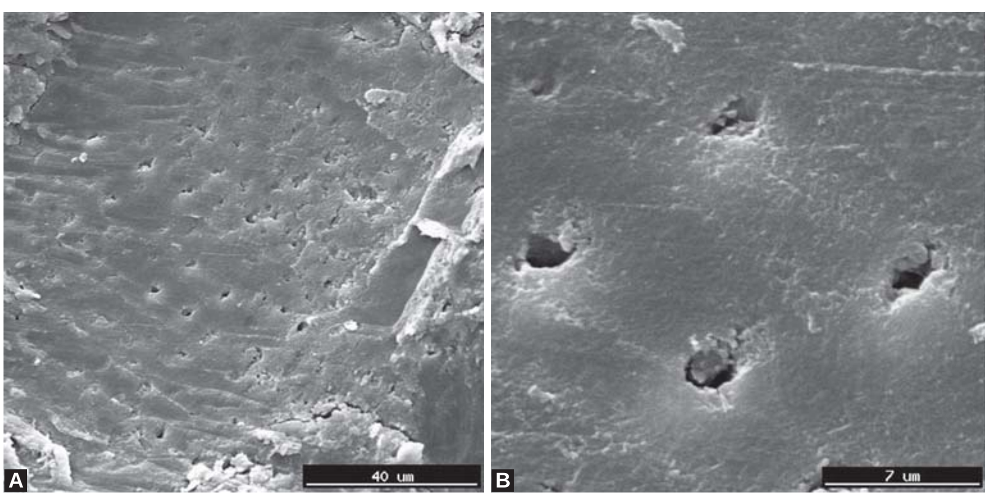
Table 2: Mean (in MPa) and standard-deviation (SD) data and results of Tukey's test for interaction factor. The number of pretest failures per group is presented in parenthesis

Adhesive systems	*Surface preparation*	*Mean ± SD*	*Homogeneous sets*		
Adper Single Bond Plus	Diamond tip	30.33 ± 6.03 (6)	A	—	—
Adper Scotchbond SE	Er:YAG laser	29.81 ± 6.23 (4)	A	—	—
Adper Scotchbond SE	Diamond tip	29.77 ± 6.69 (4)	A	—	—
Adper Single Bond Plus	CVD tip	26.68 ± 5.65 (5)	A	—	—
Adper Single Bond Plus	Er:YAG laser	22.10 ± 5.90 (6)	—	B	—
Adper Scotchbond SE	CVD tip	21.70 ± 5.65 (6)	—	B	—
Clearfil SE Bond	Er:YAG laser	19.20 ± 5.53 (8)	—	B	—
Clearfil SE Bond	Diamond tip	12.41 ± 5.63 (7)	—	—	C
Clearfil SE Bond	CVD tip	10.03 ± 4.34 (8)	—	—	C

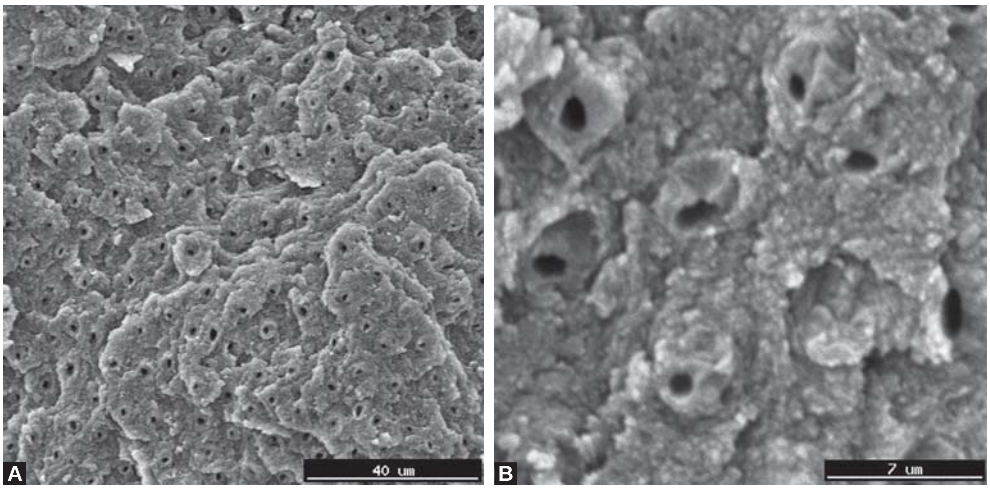
Same superscript letters are not significantly different ($p > 0.05$)



Figs 1A and B: (A) Surface prepared with diamond tip (1000×); (B) 5000×. Dentin tubules totally or partially obliterated and large quantity of smear layer



Figs 2A and B: (A) Surface prepared with CVD tip (1000×); (B) 5000×. Dentin tubules partially opened and small quantity of smear layer



Figs 3A and B: (A) Surface prepared with Er:YAG laser (1000×), (B) 5000×. Presence of opened tubules and absence of smear layer

ultrasonic device and Er: YAG laser. These different devices produced dentin surfaces with distinct grinding patterns and smear layer characteristics. The conventional diamond tip preparation produced a uniform scratched surface, resulting from the abrasion of the diamond particles of the bur with the dentin surface, presenting large amount of smear layer and tubules totally or partially obliterated (Figs 1A and B).

When CVD tip was used, the dentin-prepared surface exhibited grooves and an irregular smear layer. Partially opened dentin tubules result from the lower amount of smear layer (Figs 2A and B). This surface characteristic was previously observed[7,11] and is related to the cleaning ability of the cavitation phenomenon produced by ultrasonic vibrations that forms air bubbles inside a liquid. Some of those bubbles go to the surface, whereas other bubbles grow and implode, liberating a great amount of energy. This energy promotes intense movement of the particles, which cleans the remaining surface, removing the smear layer.[7,17]

The Er:YAG laser is able to promote cavities with no signs of melting or cracking of the surface.[19,22] Dentin irradiated by Er: YAG laser present absence of debris, with evaporation of the smear layer and opened dentin tubules,[12,21,23] caused by the microexplosions that this laser produces due to its thermomechanical ablation.[24] In the present study we also observed these morphological characteristics (Figs 3A and B), with a very irregular prepared surface, showing different levels. Intertubular dentin is selectively ablated more than the peritubular dentin, showing a protrusion of the dentinal tubules, since intertubular dentin contains more water and lower mineral content.[24]

These distinct grinding patterns and smear layer characteristics resulted in different interactions with adhesive systems, affecting bond strength results, thus the null hypothesis was rejected.

In general, the adhesive systems tested produced different bond strength results. Adper Scotchbond SE presented higher bond strength values than the Clearfil SE Bond, and statistical similar means compared to Adper Single Bond Plus. This may be explained due to its high aggressiveness, determined by its low pH value,[25,26] allowing a high reactivity with mineral component and producing a more aggressive effect on dentin.[27]

Moreover, the active application proposed by some adhesive systems can allow a continuous interaction of acidic monomers to dentin, or more monomers are placed indirect contact with dentin, conditioning and promoting continuous penetration throughout the time of application.[16,27] Therefore, as the Adper Scotchbond SE is applied actively and Clearfil SE Bond is applied passively, we can suggest that this variable may also have influenced the results.

Thus, the fact that Clearfil SE Bond is less aggressive and passively applied may have led to lower bond strength observed. When comparing this adhesive system using the three different types of preparation, we observed higher bond strength values when associated to Er:YAG preparation. Maybe, this can be explained by the absence of smear layer produced by this technology.[12,21,23]

Although a thinner and less compact smear layer is produced with CVD tips compared to conventional diamond burs, there were no significant differences when Clearfil SE Bond was applied in both substrates. Previous studies also observed no influence of the thickness of the smear layer when using this mild self-etching adhesive system.[25] Although Clearfil SE Bond is not strong enough to dissolve either thin or thick smear layers, its primer can diffuse through it and produce a hybrid layer within the subsurface intact dentin.[25]

Despite the removal of smear layer and dentin demineralization by acid etching when Adper Single Bond

Plus was used, it was affected by the type of preparation. The teeth prepared with diamond and CVD tips presented significant higher bond strength means than specimens prepared with Er: YAG laser. The changes in dentin surface roughness produced by the different types of preparation tested may modify the interaction of the adhesive to substrate.[12] The lower bond strength values obtained with the association of the total-etch adhesive tested with the laser preparation may also be due to the fact that irradiation of dentin with Er:YAG laser can denature the organic components of subsurface intertubular dentin, and affect the surface morphology and chemistry of dentin, mainly the organic components,[28,29] which may not be favorable for bonding.[21,30] In addition, the remaining denatured collagen fibrils are fused together, thus, the resin diffusion into the interfibrillar collagen spaces is probably prevented, compromising the bonding effectiveness.[24]

The association of Adper Scotchbond SE with CVD tips produced lower means than when dentin was prepared with diamond burs or laser, in spite of the low thickness smear layer formed by this method of dentin preparation and the high aggressiveness of this adhesive system. Cardoso et al.[7] also obtained lower bond strength values when CVD tip preparation was used to prepare dentin compared to conventional diamond tips. They observed the presence of microcracks in the surface layer of prepared dentin, which weakened the surface, resulting from surface tension on the dentin during preparation. Although we observed no microcracks in surface of the prepared dentin, this was observed in that study when cross-sectioned dentin was analyzed, what was not performed in the present study.

The findings of this study confirms the importance of knowing the interactions between these new technologies and the different adhesive systems in order to obtain a satisfactory bonding strength result, so that these associations may be beneficial and enhance the clinical outcomes.

CONCLUSION

According to the limits of this study, it was concluded that:

a. Clearfil SE Bond produced overall lower bond strength values.
b. The surface preparation method altered the bond strength of the adhesive systems tested and the results were material dependent.

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