Long-term Adhesion Study of Self-etching Systems to Plasma-treated Dentin

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Purpose: To determine the influence of atmospheric pressure plasma (APP) treatment on the microtensile dentin bond strength of two self-etching adhesive systems after one year of water storage as well as observe the contact angle changes of dentin treated with plasma and the micromorphology of resin/dentin interfaces using SEM.

Materials and Methods: For contact angle measurements, 6 human molars were sectioned to remove the occlusal enamel surface, embedded in PMMA resin, and ground to expose a flat dentin surface. Teeth were divided into two groups: 1) argon APP treatment for 30 s, and 2) blown air (control). For the microtensile test, 28 human third molars were used and prepared similarly to contact angle measurements. Teeth were randomly divided into 4 groups (n = 7) according to two self-etching adhesives and APP treatment (with/without). After making the composite resin buildup, teeth were sectioned perpendicular to the bonded interface to obtain beam specimens. The specimens were tested after 24 h and one year of water storage until failure. Bond strength data were analyzed by three-way ANOVA and Tukey's post-hoc test (α = 0.05%). Three beam specimens per group that were not used in the bond strength test were prepared for interfacial SEM analysis.

Results: APP application decreased the contact angle, but increased the bond strength only for one adhesive tested. SEM evaluation found signs of degradation within interfacial structures following 1-year aging in water. APP increased the dentin surface energy, but the effects of APP and 1-year water storage on dentin bond strength were product dependent.

Conclusion: APP increased the dentin surface energy. It also increased the bond strength for Scotchbond Universal, but storage for one year negated the positive effect of APP treatment.

Keywords: plasma, microtensile, dentin adhesion.

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The difficulty of resin infiltration and filling the interfibrillar spaces combined with low monomeric conversion are the main factors that reduce the longevity of dentin-composite bonding. In demineralized dentin not infiltrated by resin monomers, the acidity of etching and acidic primers/adhesives has been implicated in the enzymatic degradation of collagen fibrils, which reduces the durability of restoration.[23,42,43] Thus, depending on the type of adhesive monomer and the bonding technique, the dentin/resin interface may be the weakest part of composite resin restorations.[25]

Contemporary adhesive systems interact with enamel and dentin using two different strategies. One of them, the conventional technique, removes the smear layer by acid etching, while the other – the self-etching technique – incorporates or modifies this smear layer.[40] Self-etching systems are additionally classified based on the number of bottles or steps, since the primer and adhesive resin can be separate or combined in a single bottle. The quality of resin/dentin interfaces formed by different dentin bonding agents is typically evaluated by bond strength testing (especially the microtensile bond strength testing) and microscopy.[30,32]

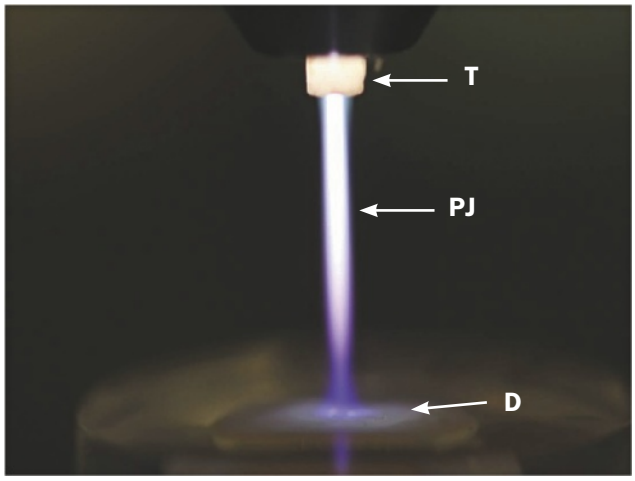

Fig 1 Representative image of dentin specimen being treated with argon plasma (D: dentin specimen; PJ: plasma jet; T: plasma torch tip).

The development of dentin bonding agents and studies on the longevity of composite restorations involve the synthesis of new monomers[39] and the physical/chemical modifications of mineralized dentin surfaces with its collagen fibrils that are directly involved in the bonding process.[35] Other types of dentin teatment have been suggested, such as the application of low-temperature atmospheric-pressure plasma (APP) to modify the dentin surface before bonding.[34,37] The plasma state, also known as the "fourth state of matter", can be generated when a gas becomes ionized. Plasma is formed in nature under different conditions, eg, lightning is a type of dense high-temperature plasma.[29] In dentistry, APP can be indicated to disinfect tooth structure[18] and to enhance the effectiveness of bonding procedures,[33] since APP does not heat the tooth surface.[6,16,29] While promising results have been achieved with plasma application on different types of surfaces, its use in dentin adhesion has not been widely explored. A study by Ritts et al[28] found a significant increase in bond strength in the peripheral dentin area, but no improvement in the central dentin area was reported.

The aim of this study was to evaluate the effect of APP treatment on the dentin bond strength of two self-etching adhesive systems. Additionally, contact angle measurements of plasma-treated dentin and the micromorphology of the resin/dentin interface formed after APP treatment and adhesive application were analyzed. The null hypotheses tested were that neither plasma treatment nor long-term water storage would influence the dentin bond strength.

MATERIALS AND METHODS

APP Device

The APP generator used in this study (KinPen 09, INP; Greifswald, Germany)[13] consisted of a hand-held unit (length 170 mm, diameter 20 mm, weight 170 g) connected to a high-frequency power supply (1.1 MHz, 2 to 6 kV peak-to-peak, 8 W) for the generation of a plasma jet at atmospheric pressure. The hand-held unit has a pin-type electrode (1 mm diameter) surrounded by a 1.6-mm quartz capillary. The operating gas was argon, applied at a flow rate of 5 l/min. The plasma plume emerging at the exit nozzle is about 1.5 mm in diameter and extends into the surrounding air for a distance of up to 15 mm (Fig 1).

Surface Wettability and Surface Energy Assessment

Six intact third molars were used for the surface energy assesment. Region of Interest (ROI) preparation was performed by grinding the enamel surface to a 600-grit finish with SiC papers until a flat dentin area with minimum dimensions of 3 mm width and 6 mm length was obtained. The smear layer was kept intact for the contact angle measurements. All teeth were immersed in water and kept moist until measurements.

The dentin samples were positioned 3 mm away from and perpendicular to the center of the APP hand-held tip. The plasma jet was applied on dried surfaces for 30 s using argon as the gas source at a flow rate of 5 l/min. Dentin ROIs were evaluated for plasma and non-plasma treatments (n = 3). The ROIs were horizontally positioned in a contact angle meter (OCA 30, Data Physics Instruments; Filderstadt, Germany) for measurements. The three liquids employed were purified water, ethylene glycol, and methylene iodide. The different liquids were used as polar and nonpolar solvents in order to calculate free surface energy[15] using Young's equation:

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta,$$

where θ is the measured contact angle and γ_{sv} is the surface energy of the solid-vapor,[8] γ_{sl} the solid-liquid[19] and γ_{lv} the liquid-vapor[4] interface. For surface energy assessment, the Owens-Wendt-Rabel-Kaelble method[24] was used by depositing 0.5-ml droplets of purified water, ethylene glycol, and methylene iodide on the surface of each disk with a micropipette (OCA 30, Data Physics Instruments). Images were captured and analyzed using software (SCA30, version 3.4.6, Samsung; Seoul, South Korea) (Fig 2). The relationship between the contact angle and surface energy was determined. Surface energy was calculated by $\gamma_L = \gamma_{DL} + \gamma_{PL}$, where γ_L is the surface energy, γ_{DL} the dispersion component, and γ_{PL} the polar component. The dispersion component of the surface energy characterizes the interaction between the surface and the dispensed liquid in terms of the nonpolar interactions between molecules.[38]

Microtensile Bond Strength Test

Twenty-eight recently extracted noncarious third molars were obtained under a protocol approved by the New York University College of Medicine Institutional Review Board. The occlusal enamel of each tooth was removed perpendicular to the long axis of the tooth with a diamond saw (model 11-5264, Buehler; Lake Bluff, IL, USA) to expose a flat dentin surface, which

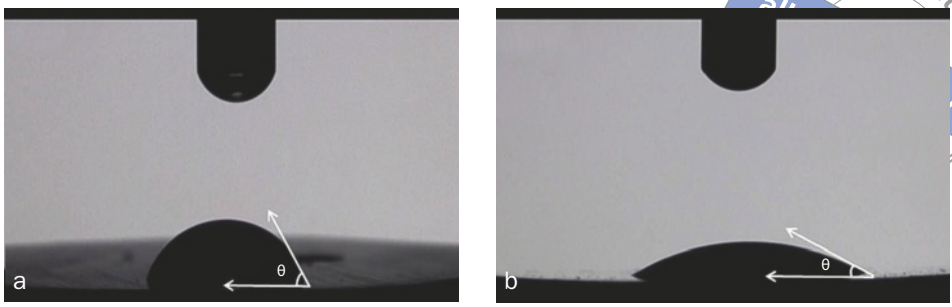

Fig 2 Contact angle measurement. θ is the angle between the surface and the tangent of the water drop placed on the dentin surface. a) Contact angle on untreated dentin; b) contact angle on plasma-treated dentin surface.

Table 1 Composition and batch number of the self-etching adhesives tested in this study

Adhesives	Clearfil SE Bond	Scotchbond Universal
Manufacturer	Kuraray Noritake Dental; Kurashiki, Japan	3M ESPE; St Paul, MN, USA
Composition	Primer: MDP, HEMA, DMA, catalyst, water Adhesive: MDP, HEMA, DMA, bis-GMA, 10% filler, catalyst	MDP, HEMA, dimethacrylate monomers, Vitrebond copolymers, ethanol, filler (5% to 15%), water, silane, initiators
Batch number	062149	488169

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: hydroxyethyl methacrylate, DMA: dimethacrylate.

was later polished using 600-grit SiC papers (Buehler). The specimens were randomly divided into four groups (n = 7), according to the two adhesive systems and APP treatment (with or without). As shown in Table 1, the self-etching adhesive systems selected for this study were Clearfil SE Bond (Kuraray Noritake Dental; Kurashiki, Japan) and Scotchbond Universal (3M ESPE; St Paul, MN, USA). The control group comprised the adhesives applied on untreated dentin, while in the experimental groups, the dentin was treated with APP for 30 s with argon gas. Afterwards, the self-etching systems were used according to the manufacturer's instructions. The hybrid composite resin Amelogen (Ultradent Products; South Jordan, UT, USA) was incrementaly applied up to a thickness of 6 mm to the bonded dentin surface, and teeth were stored in distilled water at 37°C for 24 h.

Teeth were serially sectioned perpendicular to the composite/dentin interface with a low-speed diamond saw (Buehler) under water cooling to form beam specimens (rectangular sticks) with a cross-sectional area of approximately 0.9 mm^2.[25] Twelve beams were obtained from each tooth. There were no pre-test failures. Four specimens were tested immediately after sectioning and another four beams were stored in Eppendorf tubes containing distilled water for 1 year at 37°C before testing. The distilled water was changed monthly. The remaining beams were used to analyze the micromorphology of resin/dentin interfaces. The cross-sectional areas of all specimens were measured individually with a digital caliper (Mitutoyo Sul Americana; São Paulo, SP, Brazil) before testing. Aferwards, each specimen was fixed with cyanoacrylate-based glue (Krazy Glue Gel, Products Advanced Formula, Elmer; Columbus, OH, USA) to a microtensile device attached to a universal testing machine (EZ test, Shimadzu; Tokyo, Japan). The specimens were tested at a crosshead speed of 1.0 mm/min until failure. Bond strength data were expressed in MPa and statistically analyzed by three-way ANOVA (three factors: type of adhesive, application of APP, and evaluation time).

Scanning Electron Microscopy (SEM)

Four bonded beams from each tooth were stored for one year to evaluate the effect of water on the micromorphology of the resin/dentin interfaces. After storage, beams were embedded in epoxy resin (Buehler) and polished with Al_2O_3 papers (800-, 1000- and 1200-grit), followed by diamond pastes (6-, 3-, and 1-µm). Beams were rinsed and placed into an ultrasonic cleaning device for 5 min to remove debris after each polishing step. Afterwards, beams were etched with 50% phosphoric acid for 15 s, washed, and treated with 0.1% with NaOCl for 10 min, followed by dehydration in ascending ethanol concentrations (25%, 50%, 75%, 95%, and 100%), and immersion in hexamethydisilazane for 10 min. After drying overnight at 37°C, embedded specimens were mounted on aluminum stubs, sputter coated with gold (SCD 050, Bal-Tec; Balzers, Liechtenstein), and examined using SEM (JSM 5600, JEOL; Peabody, MA, USA). Representative areas of the adhesive/dentin interfaces were photographed at 1000X.

RESULTS

The surface wettability results are presented in Fig 3. The overall increase in surface energy values for the plasma-treated group was higher compared to untreated surfaces, although the dispersion component was smaller relative to control (untreated).

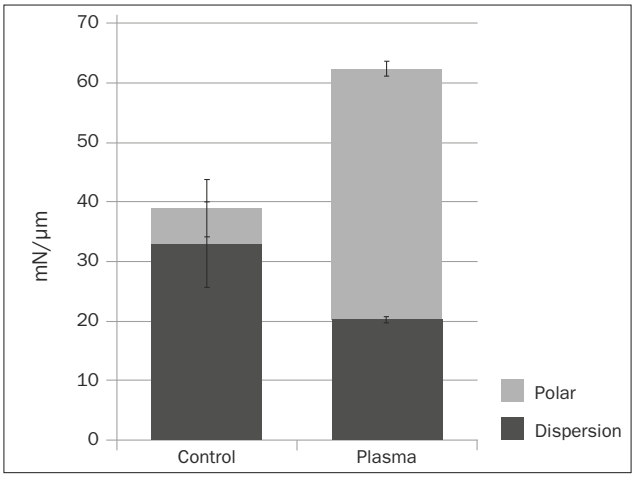

Fig 3 Bar graph of ratio between dispersion and polar components for surface free energy values.

Table 2 Dentin bond strength of self-etching adhesives with or without APP application after 24 h and one year of water storage

Adhesive	APP application	Time	
		24 h	1 year
Scotchbond Universal	no	40.5 (10.0)^{Aa}	32.6 (8.4)^{Aa}
	yes	58.1 (11.3)^{Ab}	33.6 (12.9)^{Ba}
Clearfil SE Bond	no	49.0 (7.0)^{Aa}	44.2 (6.4)^{Aa}
	yes	44.1 (11.2)^{Aa}	36.0 (7.0)^{Aa}

Means followed by different superscript letters (upper case: rows; lower case: columns) are statistically significantly different.

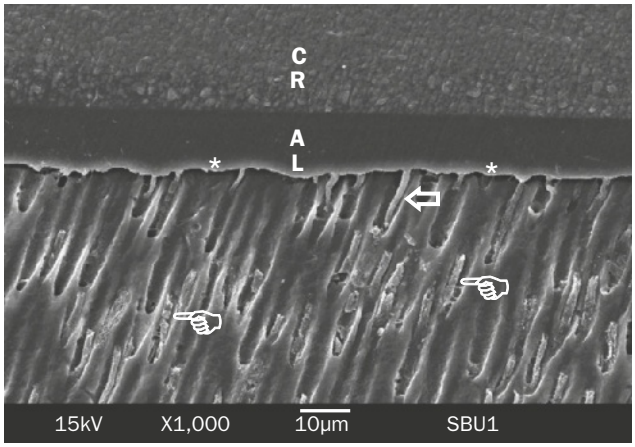

Fig 4 SEM micrograph of the untreated dentin/resin interface bonded with Scotchbond Universal after 1-year water storage. CR: composite resin; AL: adhesive layer; * hybrid layer; arrows: intact resin tag; pointers: porosities and degradation within resin tags.

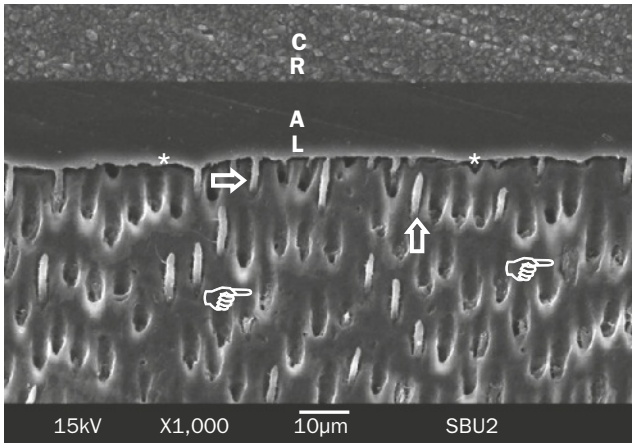

Fig 5 SEM micrograph of the plasma-treated dentin/resin interface bonded with Scotchbond Universal after 1-year water storage. CR: composite resin; AL: adhesive layer; * hybrid layer; arrows: intact resin tag; pointers: porosities and degradation within resin tags.

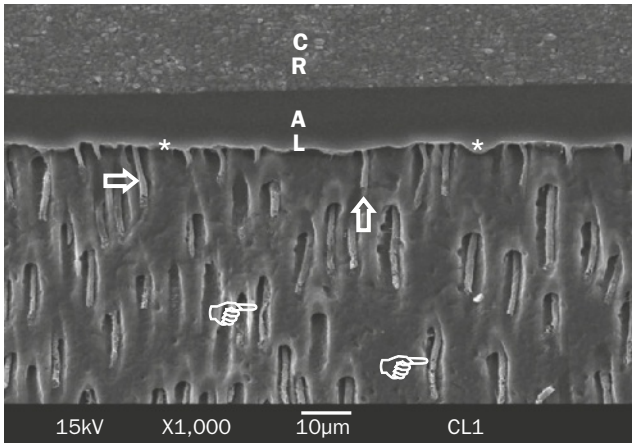

Fig 6 SEM micrograph of the untreated dentin/resin interface bonded with Clearfil SE Bond after 1-year water storage. CR: composite resin; AL: adhesive layer; * hybrid layer; arrows: intact resin tag; pointers: porosities and degradation within resin tags.

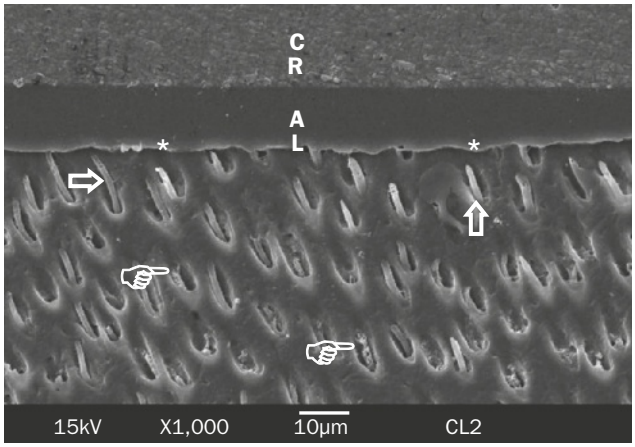

Fig 7 SEM micrograph of the plasma-treated dentin/resin interface bonded with Clearfil SE Bond after 1-year water storage. CR: composite resin; AL: adhesive layer; * hybrid layer; arrows: intact resin tag; pointers: porosities and degradation within resin tags.

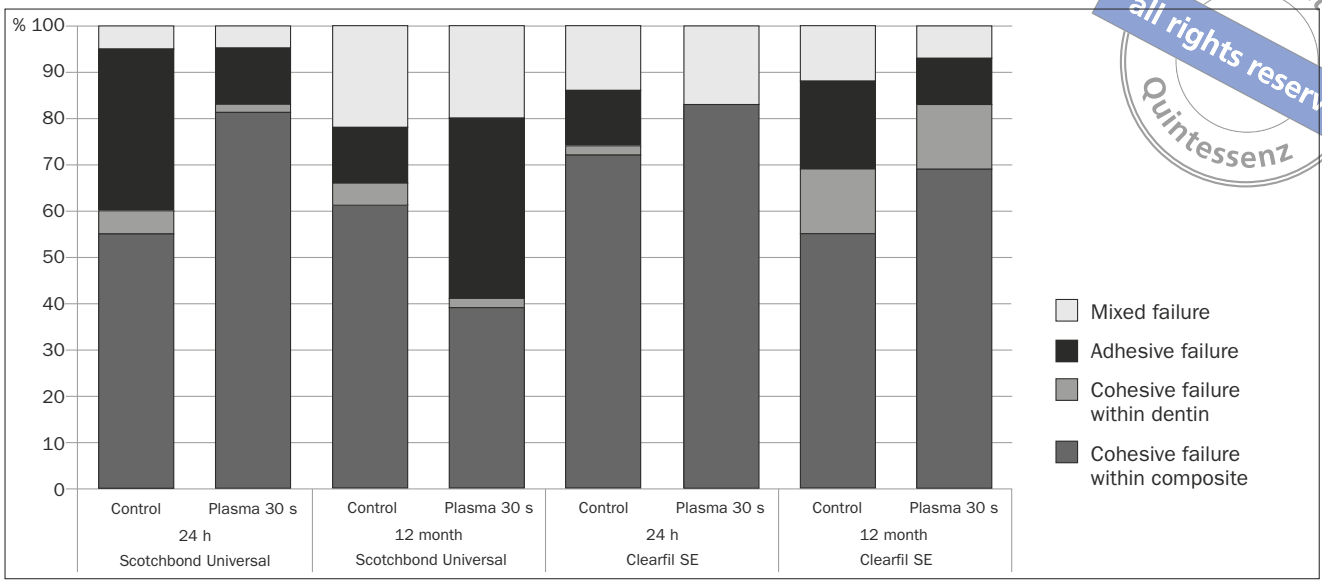

Fig 8 The distribution of failure modes for the experimental groups.

Means of bond strength of self-etching adhesives to dentin are presented in Table 2. Plasma application did not increase the dentin bond strength for Clearfil SE Bond ($p > 0.05$), while for Scotchbond Universal, the bond strength improved following the APP application ($p < 0.05$), but this decreased after one year of water storage ($p < 0.05$). One year of water storage did not change the dentin bond strength of Clearfil SE Bond or Scotchbond Universal without APP application ($p < 0.05$).

All adhesives formed a thin hybrid layer with resin tags in dentinal tubules (Figs 4 to 7). The SEM images of the resin/dentin interfaces stored for one year showed some signs of resin tag degradation. The polymeric structure of most of resin tags presented porosities independent of the type of adhesive and APP application.

The distribution of failure modes for the experimental groups is depicted in Fig 8. Mixed failure was the most predominant failure pattern observed in all groups, followed by adhesive failure along the dentin surface, mixed failure, and cohesive within dentin. For Scotchbond Universal, APP application reduced the amount of adhesive failure; however, after one year, this failure type increased. The use of plasma before applying the two-step self-etching adhesive (Clearfil SE Bond) did not result in adhesive failure at 24 h, but after water storage for one year, similar failure patterns were observed for the control (non-plasma) and plasma groups. In general, the failure patterns of the one-step self-etching adhesive (Scotchbond Universal) indicated that it was more heavily affected by 1-year water storage than was the two-step self-etching system.

DISCUSSION

The null hypotheses stating that neither plasma treatment nor long-term water storage would influence the dentin bond strength were rejected, because the bond strength of Scotchbond Universal increased when applied on plasma-treated dentin and decreased after storage for one year. In this study, two different approaches of self-etching systems were tested: two-step (Clearfil SE Bond) and one-step (Scotchbond Universal). There was no statistical difference between them when used in untreated dentin as well as after 1-year aging.

The application of self-etching adhesive systems involves one or two steps, depending on whether the acidic primer is applied separately or combined with the hydrophobic resin (bonding resin).[40] The self-etching systems can also be classified according to the pH (acidity level), being called “weak”, “moderate”, or “strong”. Systems with low pH (“strong”) remove and demineralize the dentin to allow micromechanical interaction with dentin by the hybridization process, while adhesives presenting a higher pH form a thin hybrid layer and promote chemical interaction with calcium from the hydroxyapatite of dentin. The self-etching systems containing this moderate level of acidity, including both adhesives used in this study, seem to create a more stable interaction zone.[40,41] Clearfil SE Bond and Scotchbond Universal contain MDP monomer with a pH around 2, which partially demineralizes dentin, leaving hydroxyapatite crystals around the collagen fibrils to chemically react with MDP monomer.[21]

No significant difference was found between the adhesives tested in this study. This data did not corroborate with a study that showed lower bond strength for Scotchbond Universal.[21] The authors of that study reported that the presence of polyalkenoic acid copolymer might compete with MDP by binding the calcium.[21] Furthermore, the absolute values obtained by other studies were lower than that those found in the present research.[20,21] After storage for 6 months, Scotchbond Universal and Clearfil SE Bond self-etching adhesives maintained their bond strength values,[20] similar to the results of this study, although here the storage period was one year.

In the current study, only a few samples presented adhesive failures. The standard deviation for bond strength means was approximately 20%; this reflects not only failure at dentin/resin interfaces, but also fractures within surrounding structures, such as dentin and composite resin.[10,31] This fact can be observed in the variability of failure modes obtained in this study.

APP comprises ions, electrons, and neutrons in thermodynamic equilibrium.[3] The microplasma APP used in this study has reached clinical significance in terms of implementation. This technology has been scaled down to smaller dimensions, allowing a portable device for clinical use. The device has sufficient power for increasing surface energy, while presenting safe operating conditions in clinical settings.[17] A recent review emphasized that APPs can improve interactions between materials and biological systems.[9,36,37] It is known that intimate contact, ie, a lower contact angle, is of primary importance for the sucess of adhesive bonds.[2,12] In other studies,[11,28,44] APP treatment durations ranging from 15 s to 1 min changed surfaces by the introduction of new chemical functionalities. However, if the surface undergoes prolonged exposure to APP, the mineralized dentin and collagen fibrils may be overconditioned and denatured, respectively.[11,28,44]

In this study, the surface energy in the plasma-treated group presented significantly higher values vs the control group, mainly by a possible substantial increase in the polar component, despite a decrease in the dispersive component based on changes in chemical composition.[1] APP treatment on enamel improved the surface free energy partially by enhancing surface polarity, thus making the surface more hydrophilic.[5] Three different liquids were used in the present study in order to analyze the polarity changes in dentin surface and at smear layer.[7,15] Smear layer removal and APP application only on dentin may improve the adhesion of all types of self-etching adhesives. The mechanism of APP is related to the alteration of dentin hydrophilicity by increasing the number of carbonyl groups found on the surface and consequently increasing of hydrogen-bonding interactions between collagen fibrils and adhesives.[11,28]

Despite increased wettability of the dentin surface, the bond strength results showed no improvement for Clearfil SE Bond when this adhesive was applied to plasma-treated dentin, but no reduction was observed after one-year water storage. On the other hand, APP application improved the bond strength for Scotchbond Universal at 24 h. However, bond strength decreased to values close control group level after storage for one year. The initial bond strength increase for Scotchbond Universal may be due to the increased reactive surface area of collagen and the composition of this bonding agent. It contains two functional groups, MDP monomer and copolymer, which is similar to that found in resin-modified glass ionomer (Vitrebond, 3M ESPE).[28] The latter adhesive system is able to interact with collagen fibrils and hydroxyapatite.

The bond strength reduction of Scotchbond Universal may be related to the absence of the hydrophobic adhesive applied over the primed dentin, as presented in Clearfil SE Bond. The two-step self-etching adhesive systems possess this hydrophobic adhesive to be applied after the primer, thus forming a layer that decreases the concentration of remaining unreacted acidic primer monomers.[22] The single-step adhesive does not possess this "protective" layer of hydrophobic adhesive resin application, which prevents water absorption and preserves the bonding. As plasma application increases the hydrophilicity of the dentin surface, the accumulation of hydrophylic monomers and water may impair the polymerization reaction of the adhesive monomers, reducing the dentin bond strength over time. Thus, although APP improves adhesion, the polymer degradation of the adhesive system is responsible for the reduction in bond strength as observed in this study.[37]

It has been hypothesized that self-etching adhesives with moderate acidity partially demineralize dentin while monomers simultaneously infiltrate it. As APP application results in an increase of surface wettability,[44] resin tag penetration into dentinal tubules could be facilitated. SEM images of dentin/resin interfaces showed resin tag formation independent of APP application and a thin hybrid layer along the interface.[28] Comparing the two adhesives tested on plasma-treated dentin, Scotchbond Universal presented higher bond strength than did Clearfil SE Bond, but after one year the results did not differ between them. When compared to an etch-and-rinse adhesive, Scotchbond Universal also presented higher bond strength when APP was applied for 30 s as dentin pretreatment.[11]

Both Scotchbond Univeral and Clearfil SE Bond adhesive systems showed signs of resin tags degradation over time, regardless of plasma application (Figs 4 to 7). As the beam specimens were subjected to storage, the direct contact of dentin and resin-based materials with water contributed to the early resin tag degradation at the interface.[14,27] However, the degradation of resin tags did not affect the bond strength of Scotchbond Universal applied without APP or of Clearfil SE Bond, because resin tags contribute little to the bond strength.[26]

CONCLUSION

APP application reduced the contact angle by increasing the dentin surface energy. The immediate effects of APP application on dentin bond strength were product dependent. One-year water storage counteracted the positive effect of APP treatment and reduced the bond strength of Scotchbond Universal.

ACKNOWLEDGEMENTS

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Clinical relevance: In this study, the effect of plasma depended on the type of adhesive system; thus, no bond strength effect may be observed for some commercial bonding agents.