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Laboratory Research

The Influence of Temperature of Three Adhesive Systems on Bonding to Ground Enamel

RS Alexandre • RH Sundfeld
M Giannini • JR Lovadino

Clinical Relevance

The temperature of adhesive systems must be considered an important factor for the good clinical performance of bonding procedures.

SUMMARY

This study evaluated the microtensile bond strength test (µT), micromorphology of resin-enamel interface (RET) and etching patterns (EP) promoted by the etch-and-rinse adhesive, Prime&Bond NT (PB), and two self-etching adhesives, Clearfil SE Bond (SE) and Adper Prompt L-Pop (APR), to ground bovine enamel surfaces, when applied at temperatures of 5°C (C), 40°C (H) and 20°C (R). *Materials and Methods.* Sixty-three bovine incisors were randomly divided into nine experimental groups (n=7) according to adhesive systems and temperatures. The buccal enamel surfaces were flattened with 600-grit SiC paper and abraded with a diamond bur under water-cooling. The adhesive systems were applied according to the manufacturer's instructions. After the restorative procedures, the specimens were sectioned into five slabs. Four slabs were prepared for µT and one for interface analysis. For etching pattern analysis, the remaining 16 bovine enamel fragments were used (n=2). The adhesives were applied and the surfaces were rinsed with organic solvents after application. The specimens for RET and EP analysis were prepared for SEM analysis. *Results.* No significant differences among the adhesives were found at R temperature. However, at 5°C, PB and APR presented lower bond strength than SE. At H temperature, higher bond strength was observed for PB than for APR and SE. At C and H temperature, formation of the interdiffusion zone was impaired and the treated enamel surfaces presented an undefined EP.

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*Rodrigo Sversut de Alexandre, DDS, MS, ungraduate student, Department of Restorative Dentistry, Piracicaba Dental School, UNICAMP, Piracicaba, Brazil

Renato H Sundfeld, DDS, MS, PhD, associate professor, Department of Restorative Dentistry, Araçatuba Dental School, UNESP, Brazil

Marcelo Giannini, DDS, MS, PhD, associate professor, Department of Restorative Dentistry, Piracicaba Dental School, UNICAMP, Brazil

José Roberto Lovadino, DDS, MS, PhD, associate professor, Department of Restorative Dentistry, Piracicaba Dental School, UNICAMP, Brazil

*Reprint request: Av Limeira 901, Piracicaba, SP-CEP 13414-018, Brazil; e-mail: rodrigos.alexandre@yahoo.com.br and rodrigos.alexandre@ig.com.br

DOI: 10.2341/07-79

***Conclusion:* The variation of temperature of bonding agents affected µT, RET and EP for all materials tested.**

INTRODUCTION

Development of the enamel acid etching technique prior to application of a hydrophobic resin material has had considerable effect on the bonding efficiency of resin materials.[1-3] In order to enable efficient adhesion to enamel and dentin, new materials and techniques have been proposed for dental etching.[4] Thus, two-bottle adhesive systems were developed, followed by single-bottle adhesives,[5-6] which require total etching with phosphoric acid prior to application.[7]

Self-etching adhesives with acidic monomers have been recently introduced; these adhesives etch the dental surface without the use of etchants.[8-9] They are considered user-friendly, because of the number of steps required in the bonding protocol.[9-14] Such adhesives can be classified into two-step (acid primer + bonding agent) and one-step systems, also called all-in-one.[4] It has been postulated that all-in-one self-etching adhesive systems present similar etching patterns on intact and ground enamel as that obtained with phosphoric acid.[4,9] However, some papers have reported that these systems produce lower bond strength values compared with conventional systems that use phosphoric acid as the etching agent.[4,13,15-16]

To achieve an effective bonding procedure, the self-etching adhesive system should meet some aspects, such as dissolution of the smear layer, enamel de-mineralization, penetration and co-polymerization.[4,17] With the purpose of accomplishing all the steps, self-etching formulations became more complex.[18] These complex formulations, or so-called “difficult” formulations, are formed by different components having different functions[17] that have been shown to slow chemical deterioration during storage time at different temperatures.[18-19]

The temperature of adhesive systems should be considered during application, since a low temperature of 5°C can be reached during storage in the refrigerator. Higher temperatures of around 40°C can be reached at places close to ovens, under the direct incidence of sunlight or even in clinical procedures in warmer areas or seasons. These conditions may influence chemical reactions and, consequently, interfere with restorative procedures, compromising the quality of bonding to the dental structure and durability.[19-21] However, the chemical complexity of each system can make it sensitive to external factors, such as temperature.[19-20,22-24] Temperature can influence monomer solutions and change some properties, such as viscosity and degree of conversion, which are important for the bonding procedure.[25]

This study tested the effects of adhesive systems on bond strength applied to enamel at different temperatures, resin-enamel interface and etching pattern micromorphology. The null hypothesis tested was that there is no difference in the effectiveness of adhesive systems when applied to ground enamel at different temperatures.

METHODS AND MATERIALS

Microtensile Bond Strength

This study used the one-bottle conventional adhesive system, Prime&Bond NT (PB) (Dentsply Caulk, Milford, DE, USA) and two self-etching adhesives: Adper Prompt L-Pop (APR) (3M ESPE Dental Products, St Paul, MN, USA) and Clearfil SE Bond (SE) (Kuraray Medical Inc, Kurashiki, Japan). Before the test procedure, the sets of materials were stored at refrigerator temperature (4°C). The adhesives were applied at different temperatures: cold 5°C (C), room temperature 20°C (R) and heated 40°C (H).

Sixty-three extracted, caries-free bovine incisors were used in this study. After removing the roots, the teeth were cleaned and stored in thymol solution at 0.1% for no longer than two months. The labial surface of each tooth was ground using SiC abrasive papers (320, 400 and 600-grit) under water-cooling to create a flat enamel surface. A diamond bur (#4138, KG Sorensen, Barueri, SP, Brazil) was then used to abrade the surface using a high-speed handpiece under water for 10 seconds. Next, the teeth were randomly divided into nine groups (n=7), based on the adhesive systems and temperatures. This n value was obtained after power analysis of 80%, showing that the sample size was adequate.

The adhesive systems were placed in eppendorf tubes for heating or cooling. The tubes were partially immersed in water to increase (40°) or decrease (5°C) the adhesive temperature. The Adper Prompt L-Pop adhesive did not require storage in eppendorf tubes, because of their blister pack presentation. These adhesives were maintained in their own blisters during heating and cooling. The control groups comprised the adhesives used in environmental conditions, with maintenance of 20°C room temperature.

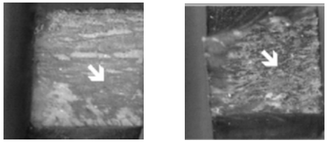
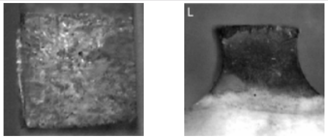
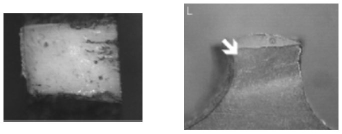
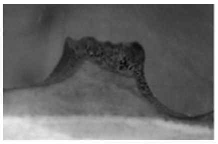
The adhesive systems were applied following the manufacturers’ instructions; their compositions are presented in Table 1. Specimens receiving the conventional adhesive system were conditioned with 35% phosphoric acid (Dental Conditioning Gel, Dentsply Caulk) for 30 seconds. The acid was then rinsed with distilled water for 15 seconds and air-dried.

Immediately after adhesive application on the ground enamel surfaces, a resin composite block (2 mm thick, Esthet X, A_2 shade, Dentsply Caulk) was built-up over bonded enamel, followed by light-curing (Optilux 501,

Table 1: *Adhesive Systems, Composition and Mode of Application According to Manufacturers' Instructions*

Adhesive Systems	Etching Agent	Primer	Adhesive	Application Mode
Prime&Bond NT Dentsply–Caulk	35% phosphoric acid	UDMA, PENTA, R 5-62-1 resin, T resin, D resin, silanated colloidal silica, cetylamine hydroxyfluoride, camphorquinone, stabilizers, acetone.		Acid etching (30 seconds) Rinse (15 seconds) and air-dry Apply two coats of adhesive. Air-dry (20 seconds at 20 cm) Light-cure (10 seconds)
Clearfil SE Bond Kuraray Medical Inc, Japan	*Primer*: MDP; HEMA; Hydrophilic Dimethacrylate; camphorquinone; water.		*Adhesive*: MDP; HEMA; Bis-GMA; Hydrophobic Dimethacrylate; N,N diethanol p-toluidine; camphorquinone bond; silanated colloidal silica.	Air-dry the enamel surface. Apply two coats of primer with slight agitation (20 seconds) Air-dry (20 seconds at 20 cm). Apply one coat of the adhesive with slight agitation (20 seconds seconds) Light-cure (10 seconds)
Adper Prompt L-Pop 3M ESPE	Liquid 1 (red blister): Methacrylated phosphoric esters; Bis-GMA; camphorquinone; stabilizers. Liquid 2 (yellow blister): water; HEMA; polyalkenoic acid; stabilizers.			Mixture of system (red and yellow blisters) Apply two coats with slight agitation (15 seconds) Air-dry (20 seconds at 20 cm) Light-cure (20 seconds)

Table 2: *Classification of Types of Fracture Occurring After Microtensile Testing and Stereomicrograph Showing the Fracture Pattern*

Adhesive	Total or partial enamel exhibition (➘), yet the surface remained planned out.	
Adhesive/Cohesive	Surface totally or partially covered by the adhesive.	
Cohesive on Resin	Surface totally or partially covered by resin. Identification of the interface from the lateral view (➘).	
Cohesive on Enamel	Strongly stained surface and irregularity evidenced by the lateral analysis.	

L—lateral view

Sybron Kerr, Danbury, CT, USA) for 40 seconds (600 mW/cm^2). The specimens were stored in tap water at 37°C for 48 hours.

The bonded specimens were serially sectioned into 1-mm thick slabs using a low-speed diamond saw (Isomet; Buehler Ltd, Lake Bluff, IL, USA). Six slabs were selected from each tooth. Two bonded slabs were used for interface analysis and four were used for the microtensile bond strength test; these slabs were trimmed with a superfine diamond bur (#1093, KG Sorensen) to form hour-glass-shaped bonded specimens approximately 1 mm^2 at cross-sectional areas.

The specimens were attached to the grips of the microtensile device with cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA) and subjected to bond strength testing in a universal testing machine (Instron 4411, Corona, CA, USA) at a crosshead speed of 0.5 mm/minute until failure.

The results were expressed in MPa and the data were submitted to statistical analysis (two-way ANOVA and Tukey test—$p<0.05$). The tested specimens were observed under a stereomicroscope with the aid of a dye for mineralized tissues[1] to evaluate the percentage of fracture types. The fracture patterns for each group are presented in Table 2.

Morphologic Analysis of the Interface

Each specimen was embedded in epoxy resin (Epoxide, Buehler Ltd) and polished with 600-, 800-, 1200- and 2000-grit SiC abrasive papers (Norton, Saint-Gobain Abrasives, Garulhos, SP, Brazil), followed by diamond pastes (6, 3, 1, 0.25 µm, Arotec SA Ind, Cotia, SP, Brazil). After polishing, the specimens were ultrasonicated for 30 minutes.

The ultrasonically cleaned specimens were dehydrated in ascending ethanol concentrations, stored for 24 hours in the oven at 37°C and submitted to conditioning with argon plasma (Plasma Serializes, Anatech Ltd, Hayward, CA, USA) for seven minutes at 100 mTorr of pressure and power of 100 watts. The specimens were then sputter-coated with gold (Desk II, Denton Vacuum Inc, Moorestown, NJ, USA) and observed under a scanning electron microscope (JSM–5600, JEOL USES, Inc, Peabody, MA, USA).

Analysis of Etching Pattern

Sixteen bovine incisors were selected for analysis of the conditioning effects of 35% phosphoric acid and the self-etching systems. The teeth had their roots removed and the crowns were cleaned and stored in 0.1% of thymol solution. Sixteen enamel fragments measuring 5 x 6 mm were obtained from the central area of the crowns. The specimens had their buccal surfaces flattened, following the same preparation employed for the restorative procedures. Two fragments were used for analysis of the smear layer, and others were used for analysis of the etching pattern promoted by phosphoric acid and the self-etching systems, according to different adhesive temperatures.

Specimens with 35% phosphoric acid employed for evaluation of the etching pattern had their surfaces etched for 30 seconds. Then, the surfaces were washed for 15 seconds and air dried. Only SE Bond Primer was used for the Clearfil SE Bond adhesive system; for the Adper Prompt L-Pop system, the adhesive was applied following the manufacturer's instructions.

For dissolution and removal of the resin adhesive solution of the self-etching systems, the specimens were thoroughly rinsed with acetone and ethanol solutions. The specimens were dehydrated, sputter-coated and observed using a scanning electron microscope.

Table 3: *Microtensile Bond Strength Values (MPa) for the Three Adhesive Systems at Three Different Temperatures (mean ± standard deviation)*

Adhesive	**Temperature**		
	20°C (R)	5°C (C)	40°C (H)
Prime&Bond NT	(11.61 ± 1.50) **Aa**	(11.04 ± 3.52) **Ba**	(14.12 ± 3.01) **Aa**
SE Bond	(12.93 ± 1.65) **Aa**	(15.17 ± 5.12) **Aa**	(7.71 ± 2.97) **Bb**
Adper Prompt L-Pop	(10.86 ± 3.12) **Aa**	(8.69 ± 3.12) **Ba**	(9.27 ± 2.66) **Ba**

Means followed by different letters show statistical difference (Two-way analysis of variance—α=0.05)
Capital letters—comparison of adhesives at each temperature (column)
Lower case letters—comparison of temperatures for each adhesive (line)

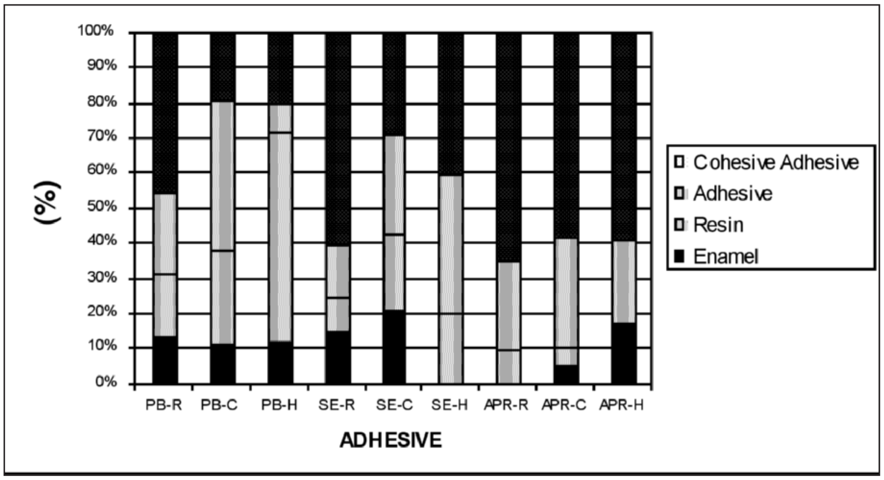


Figure 1. *Graphic illustration of fracture pattern of distribution (%). (PB) Prime & Bond NT; (SE)—Clearfil SE Bond; (APR) Adper Prompt L-Pop; R- Room temperature; H—heated temperature and C—cool temperature.*

RESULTS

Microtensile Bond Strength

The mean bond strength values and standard deviations of the three adhesive systems at the three temperatures are shown in Table 3.

No significant difference in bond strength was observed at room temperature among the three adhesive systems. The temperatures of the same adhesive did not present a reduction in bond strength, except for the adhesive SE-H. However, when compared at the same temperature, there was a statistical difference among the adhesives, whether cold or heated. For the cold adhesives, SE showed the highest bond strength. Among the heated adhesives, PB presented the highest values. Analysis of variance detected the interaction between adhesive factors and temperature.

Figure 1 graphically depicts the percentage of distribution of fracture patterns observed for the adhesives PB, SE and APR when applied at three different temperatures. The adhesive system PB showed a high incidence of adhesive and adhesive/cohesive fractures, except for PB-H, which predominantly presented cohesive fractures in resin. The adhesive SE-R presented a high incidence of adhesive/cohesive fractures and its equivalence in the occurrence of adhesive, cohesive enamel and cohesive resin fractures. SE-C presented the same fracture types; however, when heated, there was a high incidence of adhesive fractures (adhesive/cohesive and only adhesive).

The adhesive APR presented a high incidence of cohesive adhesive fractures at all temperatures. However, the application of APR-H was related to a relevant amount of enamel fracture.

Interface and Etching Pattern Morphology

In specimens not submitted to any etchant, formation of a compact layer of debris was observed on the enamel surface, combined to superficial irregularities left on the surface by the diamond bur (Figure 2).

The morphological aspects of the etched surfaces and bonded interfaces at each temperature of the adhesives are shown in Figures 3, 4 and 5. The etching pattern of the group conditioned with 35% phosphoric acid was characterized by preferential de-mineralization of interprismatic areas of enamel, showing conditioning pattern Type II (Figure 3A). The interface analysis of PB-R shows interprismatic penetration of the adhesive, in addition to penetration of the adhesive into intercrystallite spaces (Figure 3B). The interface corresponding to the PB-H adhesive showed morphological similarity to PB-R (Figure 3C). For the PB-C group, a reduction in adhesive penetration was observed in the interprismatic and intercrystallite spaces, with porosities in the adhesive layer (Figure 3D).

The etching pattern formed by the SE Bond adhesive varied according to the temperature used. For SE-R, the preferential dissolution of interprismatic enamel was observed (Figure 4A). However, this dissolution was less for SE-R than for phosphoric acid etching. For SE-H and SE-C, dissolution of the interprismatic enamel was reduced (Figure 4B,C), mainly in the warmed group, which presented a higher amount of the remaining smear layer (Figure 4D).

Analysis of the bonded interface formed by the SE adhesive demonstrated compatible penetration with the etching pattern. When the adhesive was used at 20°C and 5°C, mild penetration in the interprismatic areas and intercrystallite spaces were observed (Figure 4E,F). For SE-H, there was a presence of pores close to the enamel surface.

Application of APR self-etching adhesive promoted an etching pattern (Figure 5A) and bonded interface morphologically similar to that obtained with phosphoric acid (Figure 5D). A very thin adhesive layer was

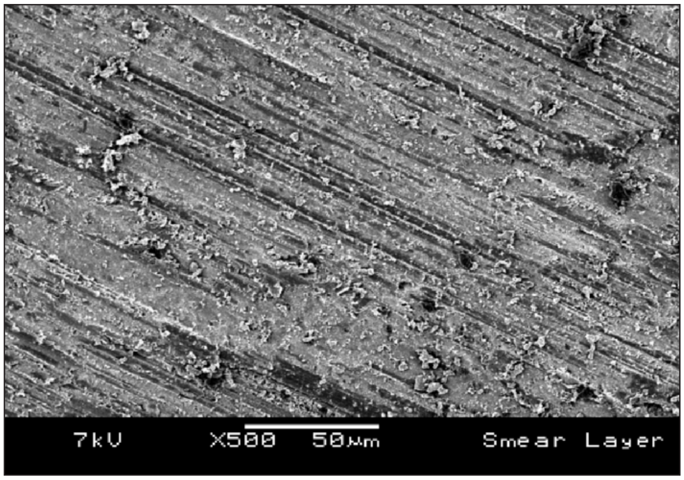


Figure 2. *Scanning electron micrograph of smear layer surface formed after diamond bur abrasion.*

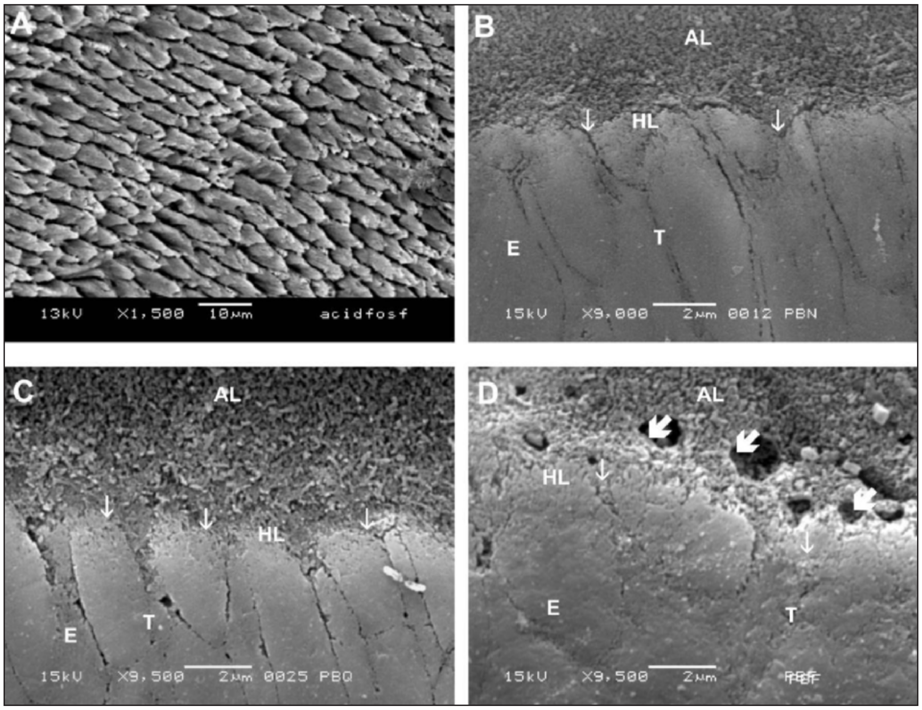


Figure 3. *Scanning electron micrograph of the etching pattern produced by 35% phosphoric acid and interface analysis of Prime&BondNT adhesive systems at three different temperatures A) preferential etching of interprismatic enamel (etching pattern type II). B) Interface analysis of PB-R showed adhesive interprismatic penetration (↓) and in intercrystallite spaces, forming the hybrid layer (HL). C) Interface analysis of PB-H showed similar characteristic as PB-R. D) Interface analysis of PB-C showed presence of pores in the adhesive layer (⬋) discreet interprismatic penetration (↓). AL—Adhesive Layer; T—Tags; HL—Hybrid Layer; E—Enamel.*

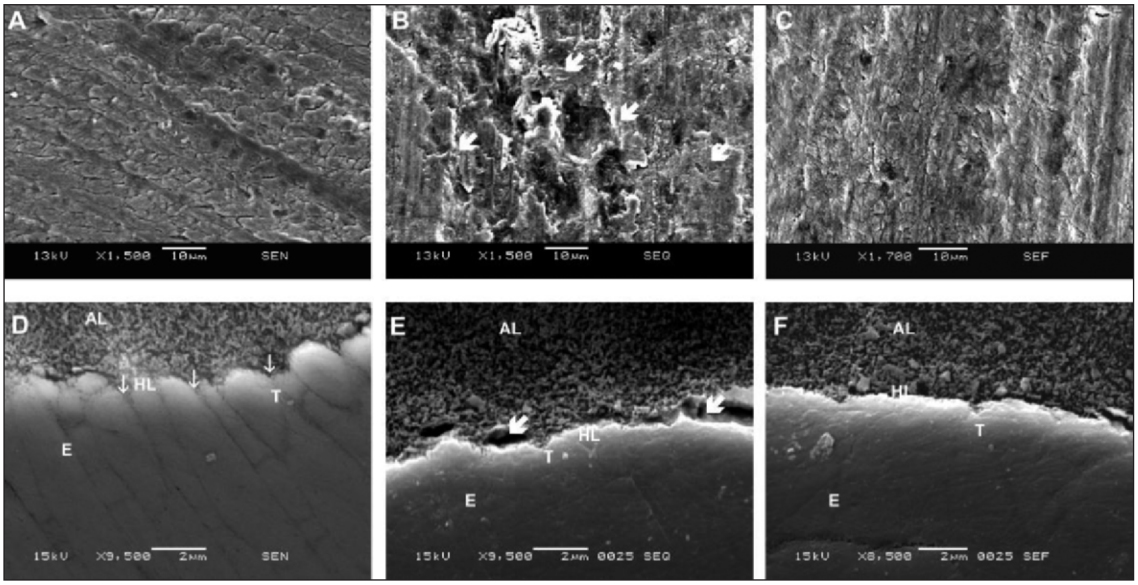


Figure 4. *Scanning electron micrograph of the etching pattern and interface analysis produced by SE Bond adhesive systems applied at three different temperatures. A) Moderate etching pattern produced by SE-R with demineralization of the interprismatic space (Type II). B) Etching pattern produced by SE-H showing presence of the remaining smear layer (⬋). C) Mild etching pattern shown by SE-C with demineralization of the interprismatic space. D) Interface analysis of SE-H showing hybrid layer formation and interprismatic penetration (↓). E) Interface produced by SE-H. Presence of pores on the enamel surface (⬋) and minimal interprismatic penetration (T). F) Interface produced by SE-C was similar to that produced by SE-H, but without the presence of porosities AL—Adhesive Layer; T—Tags; HL—Hybrid Layer; E—Enamel.*

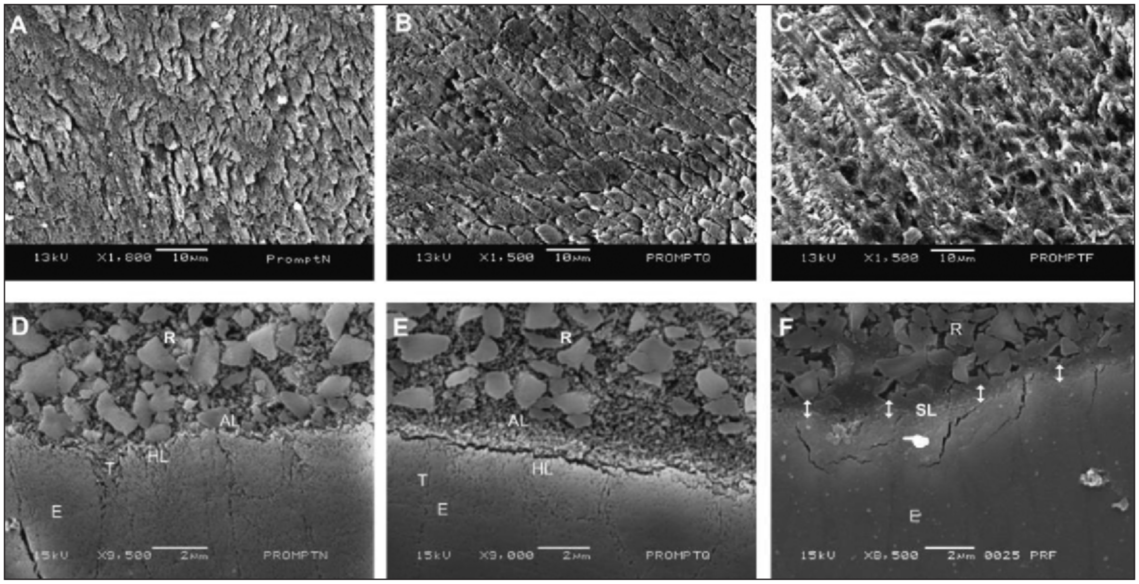


Figure 5. *Scanning electron micrograph of the etching pattern and interface analysis produced by the Adper Prompt-L-Pop adhesive systems applied at three different temperatures: A) Preferential etching of interprismatic enamel (etching pattern type II) similar to phosphoric acid. B) Etching pattern produced by APR-H showing less aggressiveness than APR-R. C) Etching pattern produced by APR-C showing presence of the remaining smear layer. D) Interface analysis of APR-R showing large interprismatic penetration, formation of the hybrid and thin adhesive layers. E) interface analysis of APR-H. F) Interface analysis of APR-C showing the smear layer not dissolved by the adhesive (⇳)and modified enamel structure below the bonded interface (☜).*

observed for this self-etching adhesive. However, the temperature change influenced the morphological characteristics. For the APR-H group, a moderate etching pattern was observed, with preferential dissolution of the external area of prisms (Figure 5B). The interface formed by the adhesive APR-H presented less penetration of adhesive resin than APR-R.

When APR-C was used, a moderate etching pattern was seen, with a small amount of the remaining smear layer (Figure 5C). Investigation of the bonded interface showed the presence of agglomerates of the smear layer close to the enamel surface. Mild penetration in the interprismatic areas and intercrystallite spaces were observed with a modified enamel structure below the bonded interface (Figure 5D-F).

DISCUSSION

Enamel, in its natural form, presents an aprismatic layer that is less susceptible to the action of acid etching.[26-28] However, for standardization of the microtensile test, the surfaces were ground to eliminate this layer. This might have contributed to the good results of the self-etching adhesives.[9-10]

The ground enamel surface exposed to phosphoric acid presented similar morphological alterations to those described in the literature,[29] which is characterized by preferential dissolution of enamel prism edges (Figure 3A). Phosphoric acid produced a defined etching pattern according to the results of previous studies.[9,30]

The quality of etched enamel depends on the acid concentration used, time and type of enamel structure;[29,31] acid aggressiveness is related to its dissociation constant (pKa). Self-etching systems are formed by acidic monomers that are susceptible to polymerization, which contain mono- or di-ester as a functional group.[10,12] Thus, at least one or two hydrogen atoms of phosphoric acid are substituted by a methacrylate group. The dissociation potential of di-ester of phosphoric acid is lower than phosphoric acid and thus is considered a less aggressive acid. Water is essential for acid ionization and is the common component present in the two acidic solutions tested. The Adper Prompt L-Pop system is a solution of mono-ester acid monomer. Its acidity content is higher than that of di-ester and presents pH=0.8,[4] which, at room temperature, was not sufficient to produce selective dissolution on the enamel surface for adhesive resin retention (Figures 5A,D). On the other hand, the SE system is composed only of a mono-ester group of phosphoric acid (MDP) and presents the highest pH. It produced mild de-mineralization on the enamel surface (Figures 4A,D), following some previous reports.[10-12,30,32]

SE Bond de-mineralization effects should be at least the same for Adper Prompt L-Pop adhesive. A possible explanation could be the difference in acid ester concentrations in the systems. The concentration of ester of phosphoric acid in Adper Prompt L-Pop is approximately 80%, compared to 25% to 30% in the SE Bond system.[10] Moreover, in the Adper Prompt L-Pop system, the ester of phosphoric acid and water are distributed in separate compartments at a ratio of 4:1 and are activated immediately before utilization,[14] which can increase its de-mineralization effects.

The differences in morphology produced by different etchants may also be observed on enamel-resin bonded interfaces. Penetration of the adhesive resin into microporosities formed by acid etching produced areas formed by hydroxyapatite crystals involved with resin, which is the denominated hybrid layer.[7] When applied at room temperature, the self-etching system formed a thin hybrid layer with short resin tags (Figure 4 D), when compared to APR-R.[9,30] The APR adhesive showed similar morphological interface as PB, probably due to the aggressiveness of the acidic solution.[9,11,30]

The Adper Prompt L-Pop adhesive applied at room temperature formed a continuous hybrid layer with resin diffusion in the interprismatic and intercrystallite spaces, presenting defined prolongations that involved the center of the enamel prisms (Figures 5A,D).[9,30] Although thinner, APR-R adhesive presented similar morphological interface as the PB-R adhesive. Both presented a continuous hybrid zone with resin diffusion into the interprismatic and intercrystallite spaces, showing more defined and longer resin prolongations involving the center of enamel prisms.[9,11,30] In general, APR presented a very thin adhesive layer (Figures 5D,E,F), due to a lower concentration of hydrophobic monomer and a high amount of water.

Based on their capacity to dissolve the smear layer and demineralize dentin, the degree of acidity of self-etching systems is classified into mild, moderate or aggressive.[10] This classification was used for the intact enamel surface.[9] However, this study showed that this classification can also be accepted for the ground enamel surface. In addition, it showed that the degree of demineralization was important for appropriate penetration of the adhesive resin into the conditioned enamel surface and that this condition was dependent on the acidity of each system.

The adhesives applied at room temperature did not present any statistical difference in bond strength (Table 3). This has also been demonstrated by other authors, who observed similar results for self-etching and conventional adhesives, when applied on ground enamel.[12,30,33-36] These results suggest that, if effective bond to enamel is desired, the number of microporosities created by area unit is more important than aggressive dissolution of enamel.[13,37-38] Additionally, the capacity for chemical bonding between the self-etching system and hydroxyapatite might have contributed to the results that were found.[4,17]

One important characteristic of adhesive systems is their low viscosity, due to the addition of solvents and diluent monomers, which allow for the excellent ability of volatilization and penetration.[3,39-40] However, other factors, such as temperature, can influence properties of the adhesive system.[20-21,23] It has been reported that low temperatures can lead to a reduction in solvent and water evaporation,[41] decreasing bond strength due to the presence of a high amount of residual solvent and water inside the adhesive layer.[42-43] Porosities were observed in group PB-C, suggesting the presence of residual water and solvents. Also, lower bond strength and a higher incidence of adhesives/cohesive fracture for PB-C was found.

Smear layer incorporation by self-etching adhesives can interfere with the bonding mechanism. The reduction of adhesive viscosity, due to low temperature, can impair dissolution of the smear layer and enamel demineralization.[25] The best results for SE-C adhesives can be related to the fact that the SE Primer is composed of monomers with low viscosity and high hydrophilic concentration, compared with other adhesives. Dissolution of the smear layer and the products from de-mineralization of this adhesive were possibly not impaired. The high bond strength presented by SE-C can be related to its two-bottle presentation and application mode. Individual and active application of SE Primer, followed by the waiting time before application of the adhesive, might have facilitated evaporation of the solvent, even at low temperatures. However, for the adhesive APR-C, the formulation with more viscous monomers might have contributed to reducing the action of the acidic monomer. Figures 5C and 5F show that APR-C did not solubilize the smear layer and it was unable to etch the enamel.

In groups using self-etching adhesives, there was a reduction in bond strength, mainly for the adhesive SE-H. The temperature increase elevates the vapor pressure in the solution and facilitates evaporation of the solvent. This evaporation can quickly reduce the concentration of water into the mixture, reducing the de-mineralization capacity of these adhesives by dispersion of the smear layer and de-mineralization products.[44] Moreover, these adhesives presented incomplete diffusion in the smear layer, with a mild etching pattern. The SE-H group exhibited the presence of porosities close to the enamel surface, which were formed by the non-solubilized smear layer, and was probably produced during SEM preparation (Figure 4E). These components can make adhesion more susceptible to degradation.[45] In addition, this precipitation can provide low cohesive force, which might lead to a higher incidence of adhesive/cohesive fractures (Figure 1).

Another important factor that must be considered is the chemical deterioration of these adhesive systems, since some components of these products are unstable at high temperatures, such as HEMA, MDP, BIS-GMA and DMA.[46-47] This deterioration before the application of adhesive systems can result in higher adhesive degradation over time, thus decreasing adhesive durability.[18-19] This may have contributed to lower bond strength and differences in the interface and etching pattern morphology.

The degree of conversion may also have a significant influence on the physical properties of a resin material.[48-49] Polymerization temperature can interfere with the degree of conversion of monomers and consequently affect their properties.[22,50] Radical mobility increases with heating, leading to additional polymerization, which ensures good results for low viscosity systems.[25,49-50] The adhesive PB did not show any statistical differences among the three temperatures. It presented the highest bond strength values compared to the other heated adhesives, without damage to its bonded interface. This can be explained by its better conversion of monomers, in addition to its appropriate solvent evaporation and increased resin flow.[51]

CONCLUSIONS

Although self-etching adhesive systems have been developed to reduce the sensitivity of adhesive application, the complexity of their chemical reactions make them sensitive to external and internal factors, sometimes not dependent on the operator; the absence of standardization of their temperatures can lead to different results. Their application on ground enamel at room temperature (20°C) promoted similar bond strengths as conventional adhesives. The null hypothesis was not accepted, since the authors of this study observed that bond strength to a ground enamel surface was temperature-dependent, suggesting that the temperature of adhesive systems must be considered an important factor for good clinical performance of bonding procedures.

(Received 16 April 2007)

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