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Effect of Cigarette Smoke on Resin Composite Bond Strength to Enamel and Dentin Using Different Adhesive Systems

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Clinical Relevance

The exposure of dentin to cigarette smoke influences adhesive bonding strength. However, cigarette smoke does not influence the bond strength to enamel.

SUMMARY

Objective: To evaluate the microshear bond strength of composite resin restorations in dental blocks with or without exposure to cigarette smoke.

Method: Eighty bovine dental blocks were divided into eight groups (n=10) according to the type of adhesive (Scotchbond Multi-Purpose, 3M ESPE, St Paul, MN, USA [SBMP]; Single Bond 2, 3M ESPE [SB]; Clearfil SE Bond, Kuraray Medical Inc, Okayama, Japan [CSEB]; Single Bond Universal, 3M ESPE [SBU]) and exposure to smoke (no exposure; exposure for five days/20 cigarettes per day). The adhesive systems were applied to the tooth structure, and the blocks received a composite restoration made using a matrix of perforated pasta. Data were statistically analyzed using analysis of variance and Tukey test ($\alpha < 0.05$).

Results: For enamel, there was no difference between the presence or absence of cigarette smoke (*p*=0.1397); however, there were differences among the adhesive systems (*p*<0.001). CSEB showed higher values and did not differ

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from SBU, but both were statistically different from SB. The SBMP showed intermediate values, while SB demonstrated lower values. For dentin, specimens subjected to cigarette smoke presented bond strength values that were lower when compared with those not exposed to smoke (p<0.001). For the groups without exposure to cigarette smoke, CSEB showed higher values, differing from SBMP. SB and SBU showed intermediary values. For the groups with exposure to cigarette smoke, SBU showed values that were higher and statistically different from SB and CSEB, which presented lower values of bond strength. SBMP demonstrated an intermediate value of bond strength.

Conclusion: The exposure of dentin to cigarette smoke influenced the bonding strength of adhesives, but no differences were noted in enamel.

INTRODUCTION

Of the development of restorative techniques in dentistry, adhesive systems have stood out for their ability to promote a bond between the tooth structure and restorative materials.[1,2] One way of classifying adhesive systems is related to the use of acid etching prior to the application of the adhesive (etch-and-rinse and self-etching). Although the current adhesive systems have been improved significantly after numerous studies, the interface between the substrate and adhesive is the susceptible failure when exposed to the oral environment. The tooth-restoration interface, called the "hybrid layer," is formed by a network of adhesive penetration into dentin tubules and enamel surface irregularities that becomes rigid when polymerized, allowing for the micromechanical retention of a resin restoration. The bonding durability between a restoration and tooth substrate is important for the longevity and clinical success of adhesive restorations.[3] Common clinical findings demonstrate that the exposure of this interface to the oral environment may cause the deterioration of the hybrid layer due to a variety of physical and chemical factors, including hydrolysis and enzymatic degradation of the dentin collagen.[4]

Cigarette smoke is composed of more than 5000 constituents, including carbon monoxide, ammonia, nickel, arsenic, and heavy metals such as lead and cadmium.[5-7] When cigarette smoke comes into contact with the tooth and restorations, it may cause discoloration, surface roughness, and hardness, which are considered important mechanical properties for clinical success of all restorations. Moreover, high temperatures (55°C) can change the properties of adhesive resins, such as sorption and solubility.[5,8] Furthermore, lead and cadmium are accumulated in teeth that are exposed to cigarette smoke, particularly on the enamel surface and within the dentin according to the exposure level,[5] decreasing the bond strength of resin-based restorative materials.[9] Therefore, smoking can affect the chemical and mechanical interaction between the composite resins and dental structures.[9,10]

It is not known to what extent the substances found in cigarette smoke may influence the adhesion of adhesive restorative materials to enamel and dentin and thus interfere with the durability and clinical success of these restorations. There are few studies in the literature that have evaluated the bond strength of different adhesive systems to dentin and the bond strength of resin-based restorative materials in enamel/dentin previously subjected to cigarette smoke. Therefore, the aim of this study is to verify whether cigarette smoke interferes with the adhesion between the dental substrates and various adhesive restorations. The null hypothesis of this study is that cigarette smoke does not affect the adhesion of system adhesives to dental structures.

METHODS AND MATERIALS

For this study, 80 bovine dental blocks were used and divided into eight groups (n=10; Table 1). Prior to the restoration process, half of the samples were exposed to cigarette smoke, as described below.

Specimen Preparation

The bovine incisors were collected, disinfected, and stored in a 0.1% thymol-buffered solution and distilled water. The crown was separated from the root using a double-faced diamond disc (KG Sorensen Ind. Com Ltda, Barueri, SP, Brazil). A metallographic cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) and diamond disc ($4 \times 012 \times 1/2$ inches, Buehler) were used to obtain enamel/dentin blocks with a bonding surface area of 25 mm^2, 3 mm long and having a 1-mm enamel thickness. The enamel surface was ground using No. 600 and No. 1200 grit silicon carbide (SiC) abrasive papers under constant irrigation in a polishing machine (Arotec, Cotia, SP, Brazil). The specimens were then polished with felt disks (Arotec, Cotia, SP, Brazil) and diamond pastes (1, ½, and ¼ μm), with the specific lubricant (Arotec). Samples were placed in an ultrasonic tub (Marconi, Piracicaba, São Paulo, SP, Brazil) for 15 minutes between each application

Table 1: *Experimental Groups for the Adhesion Test in Enamel and Dentin*

Blocks Enamel and Dentin	System Adhesive	Exposure to Cigarette Smoke
SBMP (n=10)	Scotchbond Multi-Purpose (3M ESPE)	No
SB (n=10)	Single Bond 2 (3M ESPE)	No
CSEB (n=10)	Clearfil SE Bond (Kuraray)	No
SBU (n=10)	Single Bond Universal (3M ESPE)	No
SBMP-WS (n=10)	Scotchbond Multi-Purpose (3M ESPE)	Yes
SB-WS (n=10)	Single Bond 2 (3M ESPE)	Yes
CSEB-WS (n=10)	Clearfil SE Bond (Kuraray)	Yes
SBU-WS (n=10)	Single Bond Universal (3M ESPE)	Yes

Abbreviations: SBMP, Scotchbond Multi-Purpose; SB, Single Bond 2; CSEB, Clearfil SE Bond; SBU, Single Bond Universal; WS, with smoke.

of sandpaper and felt and at the end of the polishing procedures. All samples were stored in distilled water at 37°C until use.

Exposure to Cigarette Smoke

A smoke machine developed by the Department of Restorative Dentistry, Piracicaba Dental School UNICAMP, 2011 (registered under No. 01810012043 INPI, National Institute of Industrial Property) was used to expose groups SBMP-WS (Scotchbond Multi-Purpose, 3M ESPE, St Paul, MN, USA), SB-WS (Single Bond 2, 3M ESPE), CSEB-WS (Clearfil SE Bond, Kuraray Medical Inc, Okayama, Japan), and SBU-WS (Single Bond Universal, 3M ESPE; n=10) to cigarette smoke. The cycle was scheduled on a time interval, simulating the smoking behavior usually performed by a smoker, with the smoke remaining in contact with the specimens for three seconds. The machine allows for the ambient air to be inhaled every 10 seconds, thus simulating smoke inhalation and subsequent elimination. The specimens were subjected to smoke from one pack of Marlboro cigarettes (Philip Morris Brazil Ind. e Com, Santa Cruz do Sul, RS, Brazil) per day, for a total of five days.[11] In the interval between one simulation and another, the samples were stored in artificial saliva (1.5 mM Ca, 0.9 mM Pi, 150 mM KCL, 0.05 μg F/mL, 0.1 M Tris buffer [pH=7.0]) at 37°C, and every 24 hours, the samples were washed with distilled water and reimmersed in a fresh solution of artificial saliva to prevent sedimentation.[11,12] Prior to exposure of the samples to cigarette smoke, all samples were isolated with acid-resistant varnish (Colorama, São Paulo, Brazil), with the exception of the polished enamel area. The artificial saliva for all of the groups (exposed and not exposed to smoke) was changed daily.

Microshear Strength Test

Four types of adhesive systems were tested: 1) three-step etch-and-rinse adhesive system (Scotchbond Multi-Purpose [3M ESPE]), 2) two-step etch-and-rinse adhesive system (Single Bond 2 [3M ESPE]), 3) two-step self-etching system (Clearfil SE Bond [Kuraray Medical Inc]), and 4) one-step self-etching system (Single Bond Universal [3M ESPE]). These adhesive systems were applied to the tooth structure according to the manufacturer's recommendations (Table 2), and subsequently, the enamel blocks received a composite resin restoration (Filtek Z350XT Flowable; A3 shade, lot N495761, 3M ESPE) using a matrix of perforated pasta (Furadinho 6, Pastifício Santa Amália, Machado, Minas Gerais, Brazil) that was 1 mm in height and with a 1.15-mm internal diameter. This matrix does not cause tension during its removal after water absorption because the water gelatinizes the starch molecules and consequently reduces its rigidity. All enamel surfaces were etched using phosphoric acid 35% (Ultra Etch, Ultradent Products Inc, South Jordan, UT, USA) for 30 seconds. The photoactivation of the composite was performed using a third-generation LED source (Ultradent) at high-power mode, with a power density of 1400 mW/cm^2 for 20 seconds. The microshear test was carried out using the universal testing machine EZ Test-L (Shimadzu Corporation, Tokyo, Japan) at a speed of 0.5 mm/min. The microshear bond strength results were given in Mega Pascals (MPa) after measuring the bonding area using a digital caliper, according to the following formula:

$$R = \text{Rupture Force}(Kgf) \times 9.8/\text{Area}(\text{mm}^2)$$

where R is the bond strength in MPa.

Table 2: *Instructions for Use of Adhesives According to the Manufacturers*[a]

Adhesive System	Manufacturer's Instructions	Composition
Scotchbond Multi-Purpose (3M ESPE)	Etch enamel and dentin surface with phosphoric acid 35% for 30 and 15 s, respectively. Water rinsing twice as long the etching and dry carefully following the wet-bonding technique. Apply the *primer* to enamel and dentin and dry gently for 5 s (*no waiting*). Surface will appear shiny. Apply the adhesive to enamel and dentin and dry gently for 5 s. Light-curing for 10 s.	PRIMER: Water; 2-hydroxyethyl methacrylate (HEMA); copolymer of acrylic and itaconic acids BOND: Bisphenol a diglycidyl ether dimethacrylate (Bis-GMA) HEMA; triphenylantimony
Single Bond 2 (3M ESPE)	Etch enamel and dentin surface with phosphoric acid 35% for 30 and 15 s, respectively. Rinse for 10 s. Blot excess water using a cotton pellet or mini-sponge. **Do not air dry**. The surface should appear glistening without pooling of water. **Immediately after blotting**, apply two consecutive coats of adhesive for 15 s with gentle agitation using a fully saturated applicator. Gently air thin for 5 s to evaporate solvent. Light-curing for 10 s.	Ethyl alcohol; Bis-GMA; silane-treated silica (nanofiller); HEMA; copolymer of acrylic and itaconic acids; glycerol 1,3-dimethacrylate; water; diurethane dimethacrylate (UDMA); diphenyliodonium hexafluorophosphate; ethyl 4-dimethyl aminobenzoate (EDMAB)
Clearfil SE Bond (Kuraray)	Active application of the *primer for* 20 s. Air dry gently. Apply the *bond*, air dry gently, light-cure for 10 s. Etching enamel surface with phosphoric acid 35% for 30 s, previous application of adhesive.	PRIMER: 10-methacryloyloxydecyl dihydrogen phosphate hydrophilic aliphatic dimethacrylate; dl-camphorquinone (CQ); water BOND: 10-MDP, Bis-GMA, HEMA, CQ, hydrophobic dimethacrylate; N,N-diethanol P-toluidine; colloidal silica
Single Bond Universal (3M ESPE), self-etching method	Apply the adhesive for 20 s. Air dry gently for 5 s and light-cure for 10 s. Etch enamel surface with phosphoric acid 35% for 30 s, previous application of adhesive.	10-MDP; dimethacrylate resins; HEMA; Vitrebond copolymer; filler, ethanol; water; initiators; silane

[a] *As informed by the manufacturers' material safety data sheets.*

After the enamel microshear strength test, the enamel surface was removed to expose the dentin. SiC sandpaper, with a No. 600 granulation, was used to standardize the smear layer. The same protocol was used for the restorative procedures in dentin, and dentin microshear testing was performed. After the microshear test, bond failure mode was classified in percentages, as 1) cohesive in tooth tissue (enamel or dentin), 2) adhesive, 3) cohesive in the composite, and 4) mixed using a stereomicroscope (MZ75, Leica Microsystems, Heerbrugg, Switzerland) under 100×.

Statistical Analysis

The bond strength microshear data were subjected to analysis of variance (ANOVA) and Tukey tests ($\alpha<0.05$).

RESULTS

Table 3 presents the enamel microshear values. ANOVA showed significant differences for the factor adhesive system ($p<0.001$). However, no statistical difference was found for the factor exposure to cigarette smoke ($p=0.1397$) or for the interaction between the factors (cigarette smoke × adhesive

Table 3: *Mean (SD) Enamel Microshear Strength*[a]

Exposure to Cigarette Smoke	Adhesive System			
	Scotchbond Multipurpose (3M ESPE)	Single Bond 2 (3M ESPE)	Clearfil SE Bond (Kuraray)	Single Bond Universal (3M ESPE)
Without	17.55 (2.9)	12.75 (4.56)	19.00 (3.48)	19.09 (3.45)
With	14.30 (2.55)	11.17 (5.0)	19.23 (4.30)	18.72 (2.7)
Pooled data	15.93 (3.13) B	11.97 (4,73) C	19.12 (3.81) A	18.90 (3.01) AB

[a] *No effect of treatment ($p=0.1397$).Different letters indicate a significant difference ($p\leq0.05$) between adhesive systems.*

Table 4: *Mean (SD) Microshear Strength in Dentin*[a]

Exposure to Cigarette Smoke	Adhesive System			
	Scotchbond Multipurpose (3M)	Single Bond 2 (3M)	Clearfil SE Bond (Kuraray)	Single Bond Universal (3M)
Without	14.60 (3.20) aB	15.24 (4.93) aAB	19.94 (4.45) aA	18.15 (4.49) aAB
With smoke	14.17 (5.05) aAB	10.16 (3.80) bB	9.97 (1.90) bB	18.82 (4.40) aA

[a] *Distinct letters (uppercase in the row and lowercase in the column) are statistically different (*$p<0.05$*).*

systems; $p>0.050$). Among these adhesive systems, CSEB showed higher values and did not differ from SBU, but both were statistically different from SB. SBMP showed intermediate values, whereas SB demonstrated the lowest values.

Table 4 presents the dentin microshear values. There was a significant difference between the factors exposure to cigarette smoke ($p<0.001$) and adhesive system ($p<0.001$) and for the interaction between the factors ($p<0.001$). For the groups without exposure to cigarette smoke, CSEB showed higher values that were statistically different from SBMP (lower values). SB and SBU showed intermediary values, without a significant difference between them. For the groups with exposure to cigarette smoke, SBU showed the highest values, which differed statistically from SB and CSEB, which presented the lowest values and did not differ statistically from each other. SBMP presented an intermediate value. When exposed to cigarette smoke, dentin showed lower values for SB and CSEB, which differed statistically from the groups that were not exposed to cigarette smoke. The data obtained in the fracture pattern evaluation were analyzed by frequency distribution (Figure 1). The mixed failure was the predominant pattern in almost all groups in enamel and dentin, with the exception for CSEB with exposure to cigarette smoke, which showed more adhesive failures.

DISCUSSION

In this study, there was no significant difference between the enamel groups with or without exposure to cigarette smoke, but there were differences between the adhesive systems (Table 3). The adhesion mechanism in enamel basically occurs through micromechanical retention from the infiltration of the adhesive system into enamel porosities that result from prior conditioning with phosphoric acid.[13] Based on this finding, it can be supposed that the incorporation of heavy metals did not affect the adhesion to enamel. The application of phosphoric acid for all groups provided demineralization of enamel, and contaminants may be removed from the enamel during this preparation, which could avoid bond degradation.

In relation to the adhesive systems for the enamel surface, CSEB showed the highest bond strength results. This agent contains functional monomers,

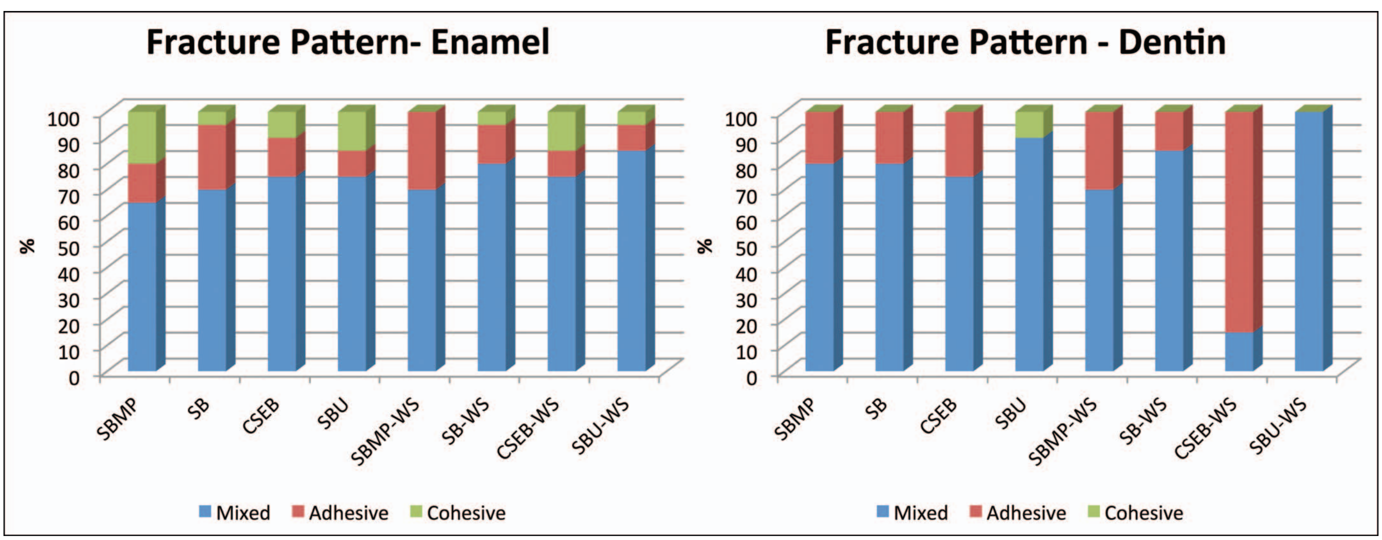

Figure 1. *Percentage of the failures: mixed, adhesive, and cohesive failure.*

which can establish chemical interactions between the adhesive and the hydroxyapatite in the dental substrate.[14] The commonly used functional monomer in this adhesive is the phosphate monomer, 10-methacryloxydecyl dihydrogen phosphate (10-MDP). The AD–Concept can explain the results for CSEB, for which phosphate-based monomers (such as phenyl-P and 10-MDP, which are part of the acidic primer) have a potential for chemical bonding with calcium in hydroxyapatite. Therefore, all of the acids interact with the calcium in the hydroxyapatite, forming ionic bonds that are stable.[14] Moreover, etching of the enamel surface removes the smear layer and increases the reactivity of 10-MDP with the calcium from hydroxyapatite, thus improving the bond strength.[15]

SBU presented intermediate values, without a significant difference from the other groups. SBU is a one-step self-etching adhesive; this adhesive category presents complex mixtures of hydrophilic and hydrophobic components to produce thinner hybrid layers when compared with two-step and etch-and-rinse adhesives.[16-18] SBU also contains functional monomers and a Vitrebond copolymer. Both compounds interact with the calcium from hydroxyapatite.[19] Previous studies[20,21] demonstrated that etching the enamel significantly increased bond strength values for the one-step multimode adhesive, SBU, and the two-step self-etching adhesive, CSE.

SB presented the lowest bonding values to enamel, and SBMP presented intermediate values, without any significant differences from SBU. SB is classified as a two-step etch-and rinse adhesive because the primer (part hydrophilic) and bond (part hydrophobic) are in the same bottle[2,22] and because the solvent is mostly water (enamel contains only 4% water). The infiltration of the two-step etch-and rinse adhesive system is lower when compared with three-step etch-and rinse adhesives. The three-step etch-and rinse adhesives contain hydrophilic functional monomers in the primer, which allow for the monomer to permeate into the demineralized matrix, while the hydrophobic monomers contained in the bonding agent facilitate adhesion of the composite restorative material to the conditioned tooth surface.[23] Moreover, SB showed problematic solvent evaporation,[24] and the presence of the solvent in the adhesive layer decreased the degree of conversion,[24] which consequently provided a lower bond strength.

When considering the dentin surface, there was a statistical difference between some groups with and without exposure to cigarette smoke, with SB-WS and CSEB-WS presenting lower bond strength values when compared with their respective smokeless groups (Table 4). It can be suggested that the heavy metal contaminants interfered with the chemical interaction between the functional monomers 10-MDP and the Ca hydroxyapatite, the main bonding mechanism for CSEB-WS. There were no statistical differences between the groups with or without exposure to cigarette smoke for the SBU group, which may indicate that there are other monomers in addition to 10-MDP, including the Vitrebond copolymer, that might have the chemical property to interact with the tooth structure, since this adhesive demonstrated better results. However, more studies are necessary to verify this interaction, because there are no studies in the current literature.

SB also showed a lower bond strength for the WS groups. This result was expected: the total etching would remove the mostly heavy metals incorporated from cigarette smoke. However, the presence of heavy metals in an adhesive with problematic solvent evaporation could inhibit the degree of conversion and bond strength of this adhesive. This mechanism did not occur with the three-step etch-and-rinse adhesives, since the primer adhesive system (hydrophilic) and bond (hydrophobic) are in different bottles.[2,22]

Only SBU maintained the same bond strength when exposed to cigarette smoke when comparing the adhesive systems that showed higher bond strengths in dentin without smoke (SB, CSEB, and SBU). SBMP presented a lower bond strength in dentin without smoke; however, this system also maintained the bond strength for the group with smoke exposure. Almeida e Silva and others[9] indicated that contamination by cigarette smoke decreased the bond strength between dentin and composite resin because of the reduced diameter of the particles from cigarette smoke, which are capable of being absorbed into the dentin hydroxyapatite based on the exposure level,[5] reducing the contact between the adhesive and dentin and thus reducing the bond strength values.[3,5] After acid etching, it can be inferred that heavy metals may remain in dentin, even after washing the surface, which can damage simplified adhesive systems.

The null hypothesis of this study was partially accepted: cigarette smoke did not affect the adhesion to enamel but reduced the adhesion to dentin for the SB and CSEB adhesive systems.

CONCLUSION

The exposure of cigarette smoke influenced the bonding strength of some adhesives to dentin, but no changes were observed for enamel.

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Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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