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Artículos originales

Effect of different conditions and modes of application on bond strength of adhesives to dentine

Efecto de diferentes condiciones y modos de aplicación sobre la fuerza de unión de los adhesivos a la dentina

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Abstract: Objective: To evaluate the microtensile bond strength in different dentine conditions (etched-E, non-etched-N, dry–D and wet-W) of a multimode adhesive (Scotchbond Universal–SU, 3M/ESPE) and a total etching adhesive (Ambar–AB, FGM) using a sonic device (Smart Sonic Device–SD, FGM). **Material and methods:** In this *in vitro* study, ninety six sound extracted human molars were divided into 12 groups (n=8) according to different dentine conditions and adhesive systems. Enamel was removed and the middle dentine surfaces were polished. Each adhesive system was applied according to the different dentine conditions, and composite resin blocks were incrementally built up and stored for 24 hours. Specimens were sectioned into sticks and bond strength data were analyzed with the Kruskal-Wallis test and Mann-Whitney U tests. **Results:** No effects of sonic application and were observed. In general, AB showed lower results compared to the SU. E and N conditions did not statistically affect the bond strength of SU groups. Dry dentine presented statistically superior bond strength values when compared to wet dentine for SU/E/SD group. **Conclusion:** Adhesion of dry dentine with multimode adhesive system may be superior to wet dentine with sonic application. The modes of application had no influence in bond strength of studied adhesives.

Keywords: Dentin, Dentin-Bonding Agents, Tensile Strength, Ultrasonics.

Resumen: Objetivo: Evaluar la resistencia de la unión microtensil en diferentes condiciones de dentina (grabado-E, sin grabado-N, seco-D y húmedo-W) de un adhesivo multimodo (Scotchbond Universal-SU, 3M/ESPE) y un adhesivo de grabado total (Ambar-AB, FGM) utilizando un dispositivo sónico (Smart Sonic Device-SD, FGM). **Material y Métodos:** En este estudio in vitro, noventa y seis molares humanos extraídos sanos se dividieron en 12 grupos (n=8) de acuerdo con diferentes condiciones de dentina y sistemas adhesivos. Se eliminó el esmalte y se pulieron las superficies centrales de la dentina. Cada sistema adhesivo se aplicó de acuerdo con las diferentes condiciones de dentina, y los bloques de resina compuesta se acumularon de forma incremental y se almacenaron durante 24h. Las muestras se seccionaron en barras y los datos de resistencia de la unión se analizaron con la prueba de Kruskal-Wallis y la prueba de U de Mann-Whitney. **Resultado:** No se observaron efectos de la aplicación sónica. En general, AB mostró resultados más bajos en comparación con el SU. Las condiciones E y N no afectaron estadísticamente la fuerza de unión de los grupos SU. La dentina seca presentó valores de fuerza de adhesión estadísticamente superiores en comparación con la dentina húmeda para el grupo SU/E/SD. **Conclusión:** La adhesión de la dentina seca con un sistema adhesivo multimodo puede ser superior a la dentina húmeda con aplicación sónica. Los modos de aplicación no tuvieron influencia en la resistencia de la unión de los adhesivos estudiados.

Palabras clave: Dentina, Recubrimientos Dentinarios, Resistencia a la Tracción, Ultrasonido .

INTRODUCTION

The interest in aesthetics dentistry led to the development and improvement of concepts, techniques, and materials, essential to restorative therapy [(1)]. In this context, significant improvements occurred in the bond strength and sealing ability with the dentine [(2)]. However, when the mechanisms of adhesion to enamel are compared to dentine it becomes more critical. The predominantly organic composition of dentine formed primarily by heterogeneous collagen fibrils (type I) and dentinal tubules throughout its length, giving this substrate permeability and intrinsic moisture [(3)].

The durability of the adhesion between the adhesive system and tooth substrate is extremely important to the longevity of restorations, [(4)] since the degradation of this interface can weaken adhesion and lead crack formation between the tooth and the restorative material [(5)]. Previous studies have shown that clinically marginal deterioration may be associated with undesirable effects including postoperative sensitivity, marginal discoloration and secondary caries [(6,7)].

The bonding longevity depends on the dentine surface treatment, especially for the multi-mode adhesives [(8)]. Either etch-and-rinse or self-etch techniques may be used; however, when phosphoric acid is applied the action of metalloproteinase may promote degradation of the collagen fibers, causing post-operative sensibility [(8)]. Dry or wet dentine may also can affect the bond strength values. Dry dentine difficult the acid penetration on the collagen fibers and the wet bonding maintains the collagen expanded for the adhesive infiltration but may promote the hydrolytic instability of resin dentine interfaces [(9, 10)].

Since the introduction of adhesive systems various research have been carried out based on the development of different adhesion strategies for clinical practice in order to improve its adhesiveness [(11)]. In this context, ultrasound machines have been proposed to enhance the accommodation and dissemination of restorative materials in the dental substrate, since this device is capable of generating a vibrating sonic energy [(12-14)]. In dentistry, ultrasound devices have been used since 1950 mainly in periodontics and endodontics procedures [(15,16)]. However, there is a lack of information about adhesive application using ultrasonic devices. Thus, it is believed that the adhesive applied with this device could lead to promising results in the bond strength to the dentine [17], since the incomplete penetration of the adhesive in the collagen fibril network in demineralized dentine can be the main local susceptibility of their hydrolytic degradation [6].

The aim of this study was to evaluate the bond strength of a multimode (Scotchbond Universal - 3M ESPE, St. Paul, MN, USA) (SU) and total etching (Ambar - FGM, Joinville, SC, Brazil) adhesive systems in different conditions of dentine substrate using a smart sonic device. The three null hypotheses tested were followed: (i) there would be no difference in the bond strength of a multimode adhesive system when applied to dentine with or without acid etching; (ii) the different dentine conditions (dry or wet) would not affect the dentine bond strength for both adhesive systems and (iii) the use of ultrasonic device to adhesive application would not influence the dentine bond strength.

MATERIAL AND METHODS

Tooth selection and preparation

In this in vitro study, ninety-six freshly extracted, caries and crack-free human molars were used. The use of extracted human teeth was approved by the Local Ethics in Research Committee (protocol no. 53190315.6.0000.5420). The teeth were cleaned, and the occlusal surfaces were transversally sectioned with a low-speed diamond saw (Extec Co., Einfield, CT, USA) under water irrigation to remove the enamel in a metallographic cutter (Buehler, Lake Bluff, IL, USA). The root portion was sectioned 2 mm below the cementoenamel junction. The exposed dentine surfaces were further polished on wet #600-grit SiC paper for 60 s to standardize the smear layer.

The teeth were randomly divided into 12 groups (n=8) according to the dentine surface treatments, dentine conditions, adhesive systems, and application protocols. (Table 1) **Restorative procedures**

The multimode and the total etching adhesive systems were applied in accordance with the manufacturer's instructions. (Table 2) After the bonding procedures, all specimens received a micro hybrid composite restoration (A3D – Opallis, FGM, Joinville, SC, Brazil) in three increments of 2 mm, forming a block of 6 mm thick. Each increment was light polymerized for 40 s using a LED light curing unit set at 1200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Victoria, Australia). The restored teeth were stored in artificial saliva at 37 ºC for 24 h.

Microtensile bond strength test and failure mode

The bonded teeth were vertically sectioned into serial slabs and further into beams with cross sectional areas approximately 0.8 mm^2 obtaining several beams from each group varying from 28 to 32. The specimens were fixed with cyanoacrylate glue (Super Bonder Flex Gel, Henkel Loctite; SP, Brazil) to a base for microtensile test (Microtensile OM 100, Odeme Dental Research, Luzerna, SC, Brazil) and subjected to tensile forces at a crosshead speed of 0.7 mm/min until deboning. Microtensile values (MPa) were calculated by dividing the peak force (N) by the cross-sectional area of the failed interface (mm^2), measured by a digital caliper.

The failure mode of the sticks was classified as cohesive (failure exclusive within dentine or resin), adhesive (failure at resin/dentine interface), or mixed (failure at resin and dentine). The classification was performed under a stereomicroscope at 200x magnification (Stemi SV11, Carl Zeiss, Jena, German). Specimens with premature failures were included.

Statistical analysis

The mean and standard deviations were calculated for each group. The assumptions of equality of variances and normal distribution of data were checked. Since normality was not achieved data were individually analyzed by Kruskal-Wallis test. Multiple comparisons were made using Mann-Whitney U test. All statistical analyses were carried out using BioEstat 2.0 software. Statistical significance was established at $p=0.05$.

RESULTS

The results referents of bond strength are present on Table 3. Related to the total-etch technique, it can be observed that the adhesive AB showed lower results compared to SU ($p>0.05$); except after acid etching and the application of the adhesive multimode with the sonic device in wet dentine, which showed similar values ($p>0.05$). It can be observed that there was no statistically significant difference in dentine bond strength between specimens of the multimode adhesive system groups (SU) when it was applied to dentine with and without acid etching previously, regardless of the dentine condition (dry or wet) ($p> 0.05$).

Related to dentine condition, after acid etching, dry condition showed higher dentine bond strength values compared to wet condition only when the multimode adhesive was applied with SD ($p< 0.05$).

When the types of adhesive application (with SD or conventional) were compared, there was no statistically difference in the same condition of study ($p> 0.05$).

The failure mode distribution of the specimens (Table 4) revealed that more than 60% of failures were adhesive for all bonding systems tested, regardless the dentine condition and the mode of application.

Table 1.
Division of experimental groups

Adhesive System	Phosphoric Acid 37%	Dentin Condition	Application protocol	Groups
Self-etched (Scotchbond Universal)	Yes	Dry	Sonic	SU/E/D/SD
		Wet		SU/E/W/SD
		Dry	Conventional	SU/E/D
		Wet		SU/E/W
	No	Dry	Sonic	SU/N/D/SD
		Wet		SU/N/D/SD
		Dry	Conventional	SU/N/D
		Wet		SU/N/W
Etch-and-rinse (Ambar)	Yes	Dry	Sonic	AB/E/D/SD
		Wet		AB/E/W/SD
		Dry	Conventional	AB/E/D
		Wet		AB/E/W

Note: SU-Single Bond Universal; AB-Ambar; E- etched with phosphoric acid 37%; N-no etched, D- dry dentin; W- wet dentin; SD- sonic device.

Table 2.
Materials, manufacturers, composition and application procedures of the materials used in this study

Material/Manufacturer	Composition	Protocol
Phosphoric acid 37% FGM	Phosphoric acid at 37%	Etch-and-rinse strategy - Apply COND AC 37% FGM on the dentin for 15 s; - Wash the dentin surface with abundant water.
Ambar FGM	UDMA, HEMA, acid methacrylated monomers, hydrophilic methacrylated monomers, ethanol, water, silica nanofiller, photoinitiators, coinitiators, stabilizers	- Apply the adhesive system with the aid of an adhesive applicator (cavibrush or Smart Sonic Device) brushing the adhesive against the dentin surface for 10 s; - Apply a new adhesive layer on the same surface for more 10 s; - Apply a gentle stream of air for 10 s for solvent evaporation and, - Photopolymerization for 10 s.
Scotchbond Universal 3M-ESPE	MDP 10-metacriloyxidecil dihydrogen phosphate, dimethacrylate resins, HEMA 2-hydroxyethyl methacrylate, methacrylate modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	- Apply one layer of the adhesive shaking on the surface for 20 s; - Apply a gentle stream of air for 10 s for solvent evaporation and, - Polymerization for 10 s.
Smart Sonic Device FGM	---	- The adhesive systems were applied according to the manufactures" instructions. - Vibration at 1800rpm.
Radii Plus SDI	---	- Polymerization for 20 s each composite resin increment. - The last increment was photopolymerized for 40 s.,

Table 3.
Dentin bond strength values (Mpa) and standard deviation of different adhesive application protocols

Adhesive system	Adhesive technique		Dry Dentin	Wet Dentin
Self-etch (Scotchbond Universal)	Without acid etching	Sonic	39.5 (11.7) aA	37.3 (12.8) aA
		Conventional	33.5 (11.4) aA	36.7 (12.2) aA
	With acid etching	Sonic	33.2 (11.7) aA	29.0 (8.0) bAB
		Conventional	36.9 (12.5) aA	35.9 (11.7) aA
Etch-and-rinse (Ambar)	With acid etching	Sonic	23.5 (10.1) aB	22.5 (8.9) aB
		Conventional	23.5 (8.0) aB	23.5 (8.7) aB

Note: Different capital letters indicate statistical difference between lines in the same column and different lowercase letters indicate statistical difference between column in the same line, n=8 ($p< 0.05$).

Table 4.
Distribution of failure mode (%) after 24 h within the different groups

Group \ Failure	Adhesive	Cohesive Dentine	Cohesive Resin	Mixed
SU/E/D/SD	84.15	4.88	10.98	0.00
SU/E/W/SD	86.76	2.94	8.82	1.47
SU/E/D	71.79	14.10	12.82	1.28
SU/E/W	76.74	9.30	13.95	0.00
SU/N/D/SD	61.84	15.79	22.37	0.00
SU/N/D/SD	72.94	14.12	12.94	0.00
SU/N/D	61.25	17.50	21.25	0.00
SU/N/W	63.86	10.84	25.30	0.00
AB/E/D/SD	94.44	1.11	4.44	0.00
AB/E/W/SD	86.79	0.00	9.43	3.77
AB/E/D	97.26	0.00	2.74	0.00
AB/E/W	92.54	2.99	4.48	0.00

DISCUSSION

In this study, it was used a total-etch and a multi-mode adhesive to evaluate the different techniques for adhesive application. In most part of results, bond strength values of multimode adhesive showed higher compared to the bond strength of total-etch one. The multi-mode adhesive studied has both a micromechanical interaction (hybrid layer) as a chemical interaction due to the presence of a monomer 10-methacryloyloxydecyl (10-MDP) (18,19). This functional monomer chemically bond to calcium of hidroxiapatite at the same time that it is able to extract calcium from hidroxiapatite resulting in MDP-Ca salt (19,20). Additionally, there is a chemical interaction of copolymer (Vitrebond), present in the SU, with calcium of hydroxyapatite (21,22), allowing the superiority results of this adhesive system. Thus, the lack of chemical interaction of AB adhesive with dentine may be the reason of lower bond strength results compared to the SU adhesive. An *in vitro* study demonstrated the influence that an adhesive with different 10-MDP concentrations have in dentine bond strength (23).

Related to variation of SU technique application it was not observed difference in dentine bond strength values with or without prior etching with phosphoric acid, such as demonstrated in meta-analyses (8,24). However, Rosa et al. (24) observed that the adhesive All-Bond Universal was the only one that showed an improvement in the dentine bond strength with prior phosphoric acid etching. Probably, these results could be due to its ultra-mild acidity (pH 3.1), which was not able to rightly etch and infiltrate the dentine substrate. Unlike, SU has its pH lower compared to the All-Bond Universal, being classified as mild according to its pH (pH 2.7), enabling better conditioning and penetration of the monomer in dentine. (24) Thus, some studies have shown that the effects of phosphoric acid in dentine prior to adhesive system could be material dependent (25).

Regarding the moisture of the dentine, there was no difference between the results of the bond strength to wet or dry dentine, it may be due to the water/ethanol solvents present in adhesives studied. The presence of water may be sufficient to plasticize the collagen fibrils collapsed allowing its re-expansion and reassembly of interfibrillar spaces for resin monomer infiltration (10).

In addition, it is noteworthy that the active application of SU may also have influenced these outcomes as it has been observed a superior adhesion in dry dentine if compared to wet dentine when this adhesive system was actively applied. Other studies have also been observed a superior performance of active application of adhesives (26,27). The mechanical pressure applied in dentine during adhesive application by vigorous rubbing can compress the collapsed-collagen net so that the adhesive can be pulled into the collagen net when the pressure is relieved (28). Moreover, this application can improve both monomer diffusion and solvents evaporation (29).

The durability of bond to dentine depends on the interaction of monomers in the network of collagen fibrils (3,8,24). Insufficient adhesive penetration results in exposed fibers, where the degradation process of the adhesive interface may initiate (5). Degradation may also start breaking the covalent bonds between polymers by water addition, leaving collagen fibrils exposed (5,9). In this context, ultrasonic devices have been proposed to enhance the accommodation and diffusion of adhesive materials in dental substrate, since this device is capable of generating sonic energy vibration (12-14).

It was observed in the present study that the use of ultrasonic device does not influence adhesive bond strength to dentine. These results are consistent with those found by Mena-Serrano et al. (30) In their study it was not observed any improvement on microtensile bond strength to dentine when a self-etch adhesive containing MDP in its composition (All Bond SE) was used; despite of a significant decrease in the dentine permeability when ultrasonic device was used. Finger & Tani (31) also found no difference in shear bond strength values related to the differents methods of adhesive application when different adhesive systems were used in both enamel and dentine.

However, in other studies, it was observed different behaviors through the adhesive application protocol using ultrasonic device. For some self-etch adhesives (One Coat Systems and Clearfil SE) was notice an improvement in bond strength; however, in other self-etch adhesive (Futurabond) any difference in bond strength was observed [14]. In another study, no improvement on adhesion was found when ultrasonic device was used for self-etch adhesives (Clearfil SE and Futurabond) [12]. It was observed that the sonic application of universal adhesives may be an alternative to enamel etching, since various universal adhesives showed an incresed of microshear bond strength values, however for the SU adhesive no difference was found when compared to conventional aplication [32].

Such controversy results in the literature may be due to the different frequencies of ultrasonic device used. In this study, as well as in studies published by Mena-Serrano et al. [14,30] we employed an ultrasonic device that works with a frequency of 170Hz. On the other side, the devices employed by Finger & Tani [31], worked with a frequency of 30Hz and 1MHz, respectively. It is also suggested that for the adhesive systems where the use of ultrasonic device presented no influence during adhesive application, the bond strength can be achieved maximum effect with conventional application and then no improvement was detected when vibrating sonic energy was used. Thus, the benefit of this application protocol may be adhesive dependent.

Some studies have encountered the beneficial action of the ultrasonic vibration in the resinous cements as well as for glass-ionomer cements [13,33]. Additionally, Cuadro-Sanchez et al. [33] observed a high bond strength values of fiber posts to root canal dentine using adhesive systems applied with a ultrasonic device. The adhesive vibrated by the device may be able to reach areas that would hardly be achieved by manual application, such as occurs in roots [14,33]. Kirsch et al. [17] concluded that the sonic application did not improve the post retention in the root canal but may increase the bond strength to coronal dentine.

The more cohesive fractures in SU groups compared with presented in AB groups can be explained by the higher dentine bond strength of SU groups compared to AB. So, the information obtained from microscopic studies of fractured surfaces indicates that SU may, under optimal conditions, interact with the dentine and hence better adhesion enabling cohesive failure before adhesive failure could be reached [34].

Regarding the study limitations, an *in vitro* test cannot reproduce all clinical conditions such as oral temperature changes, occlusal forces, saliva of varying pH, and the acid environment produced by bacteria, which may affect the degradation of the adhesive interface [35]. Thus, future studies should be conducted with the use of different adhesive systems to provide a broad understanding of the ultrasonic devices action and durability of adhesives.

In summary, adhesion of dry dentine with multimode adhesive system may be superior to wet dentine with sonic application; however, the modes of application had no influence in bond strength of studied adhesives.

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Declaración de intereses

* The authors declare that they have no conflict of interest.

Enlace alternativo

https://revistas.upch.edu.pe/index.php/REH/article/view/4430/4974 (pdf)