

COMPARATIVE STUDY OF THE MICROTENSILE BOND STRENGTH OF THREE DIFFERENT TOTAL ETCH ADHESIVES WITH DIFFERENT SOLVENTS TO WET AND DRY DENTIN. (*In Vitro* Test)

Noé Orellana¹, Robert Ramírez¹, Miguel Roig², Luís Giner², Montse Mercade², Fernando Durán², Gerardo Herrera¹

¹ Dental School, Los Andes University (ULA), Mérida/Venezuela.

² Dental School, International Catalonia University (UIC), Barcelona/Spain.

ABSTRACT

The aim of this study was to compare the microtensile bond strength of three different total etch adhesives: XP Bond (Caulk-Dentsply) versus Excite (Ivoclar/Vivadent) and Prime & Bond NT (Caulk-Dentsply).

Forty two (42) third human molars were cut to expose the dentinal surface. They were divided into three groups of 14 teeth (G1: XP Bond, G2: Excite, G3: Prime & Bond NT) and two groups of seven teeth for each moisture condition: moist dentin (GM) and dry dentin, (GD). The total-etch technique was used with each moisture variation. The adhesives and composites A3 (Ceram Duo G1, G3 and Tetric Ceram G2) were applied according to manufacturer's instructions. Teeth were cut with an ISOMET 1000 (Buehler Ltd.) to obtain 1 mm² x 10 mm bars, which were subject to a traction test at 5 mm/min in a universal testing machine (Adamel Lhomargy DY 36). The collected data were recorded and analyzed using an experimental design

for studying two factors of fixed effects with software Statgraphics version 5.1.

For the variable type of adhesive, we found $p=0.000$; for the variable substrate condition, $p=0.0012$, and for interaction between both factors, $p=0.0457$, which indicates significant statistical differences. The values for microtensile bond strength were $G1M=55.0642$ MPa Standard deviation (SD) 3.09768; $G1D=39.115$ MPa SD 2.86789; $G2M=34.1607$ MPa SD 2.86789; $G2D=32.7373$ MPa SD 2.77065; $G3M=37.3407$ MPa SD 2.86789 and $G3D=31.0593$ MPa SD 2.77065.

XP Bond showed the greatest values of microtensile bond strength under both conditions. Moist substrate increases the values of microtensile bond strength for the adhesives tested; however, Excite shows lower susceptibility to variation of dentinal moisture.

Key words: tensile strength, adhesives, dentin bonding agents, solvents, ethanol, acetone, tert-butyl alcohol

ESTUDIO COMPARATIVO DE LA FUERZA DE ADHESIÓN DE TRES ADHESIVOS DE GRABADO TOTAL CON DIFERENTES SOLVENTES EN CONDICIONES DE DENTINA HÚMEDA Y SECA. (Estudio *in Vitro*)

RESUMEN

El objetivo de esta investigación fue comparar la resistencia adhesiva en micro-tensión del sistema adhesivo de grabado total XP Bond (Caulk- Dentsply) vs. Excite (Ivoclar/ Vivadent) y Prime & Bond NT (Caulk-Dentsply).

Cuarenta y dos (42) terceros molares humanos fueron cortados exponiendo la superficie dentinaria. Se dividieron en 3 grupos de 14 dientes (G1 XP Bond, G2 Excite, G3 Prime & Bond NT) y a su vez en 2 grupos de 7 dientes para cada condición de dentina húmeda (GH) y/o seca, (GS). Se empleó la técnica de grabado total con la respectiva variación de humedad, siguiendo las instrucciones del fabricante se colocaron los adhesivos y las resinas compuestas restauradoras color A3 (Ceram Duo G1, G3 y Tetric Ceram G2). Los dientes fueron seccionados con una sierra ISOMET 1000 (Buehler Ltd.) hasta obtener barras de 1mm² x 10 mm, que fueron sometidas a tracción a una velocidad de 5 mm/min en la máquina de pruebas universales (Adamel Lhomargy DY 36). Los datos recolectados fueron grabados y analizados utilizando un diseño experimental para el estu-

dio de dos factores de efectos fijos utilizando el software Statgraphics versión 5.1.

La variable tipo de adhesivo obtuvo un valor $p=0,000$, para la variable condición del sustrato $p=0,0012$ y las interacciones entre ambos factores $p=0,0457$ lo que indica diferencias estadísticas significativas. Los valores de resistencia a la tracción obtenidos fueron $G1h=55,0642$ MPa Desviación Estándar (DE) 3,09768; $G1s=39,115$ MPa DE 2,86789; $G2h=34,1607$ MPa DE 2,86789; $G2s=32,7373$ MPa DE 2,77065; $G3h=37,3407$ MPa DE 2,86789; $G3s=31,0593$ MPa DE 2,77065.

En las condiciones en que se realizó esta investigación, XP Bond presentó los mayores valores de resistencia adhesiva en ambas condiciones. La condición de sustrato húmedo aumenta los valores de resistencia adhesiva para los adhesivos evaluados; sin embargo el adhesivo Excite presenta una menor susceptibilidad a la variación de la humedad dentinal.

Palabras clave: resistencia a la microtensión, adhesivos de grabado total, adhesión seca versus adhesión húmeda, solventes para adhesivos dentinarios.

INTRODUCTION

Adhesive systems have revolutionized the practice of restorative dentistry thanks to their reliable bonding to enamel and dentin. These developments, which continue today, have produced results that are reflected by materials that are easy to use and have greater bond strength and lower degradation in the oral environment. Nevertheless, it is advisable to pay proper attention to the technique and to have a thorough understanding of the adhesive process. These materials applied thinly serve to bond the restorative material effectively to the tooth structure, thus reducing and avoiding marginal microleakage. Adhesion to enamel has been reliable since it was introduced by Bonuocore in 1955¹ and has provided an ideal morphological surface since then, according to Schwartz². However, the moist, heterogeneous tubular ultra-structure of dentin poses a real challenge to adhesion.

The development of adhesive systems has been directly proportional to the improvement in aesthetic materials. There is a tendency to use adhesives with a simplified application technique, even though it seems to reduce the bond strength to dentin³ and increase its hydrolytic degradation, because adding increasingly hydrophilic monomers accelerates its degradation in the hybrid layer⁴. Three-step, ethanol-based total etch adhesives have been shown to provide greater microtensile bond strength to moist dentin than two-step adhesives⁵. Nevertheless, the number of steps and the difficulty in standardizing drying after washing causes dentists to choose adhesives with fewer steps.

One of the greatest achievements of manufacturers in the development of new adhesive materials has been simplifying the application procedure. The first step was to reduce the conventional three-step etch and wash adhesives to two step adhesives combining the first and second adhesive agents in a single bottle. Faster bonding and easy handling made one-bottle adhesives popular among dentists all over the world. Nowadays nearly all manufacturers produce one-bottle total etch adhesives and have begun to develop new adhesive formulae maintaining the classic one-bottle presentation (primer and adhesive). To increase the affinity of the monomers to partly demineralized dentin, manufacturers use different kinds of solvents (ethanol, acetone and water), which work under different moisture conditions and are susceptible to small variations in moisture.

A one-bottle total etch adhesive system made by Dentsply that uses the compound tert-butyl alcohol as a solvent has recently appeared on the market. According to the manufacturer, tert-butyl alcohol facilitates the performance of the adhesive because

it increases work time, significantly reduces sensitivity to the technique, and provides high bonding values to dentin and enamel.

Except for the results referenced by the manufacturer, to date we have not found any prior research measuring Xp Bond bond strength which we could contrast with other total etch adhesives or fifth-generation adhesives, as they are also known. Therefore we propose to determine and compare the microtensile bond strength of the new Xp Bond (tert-butyl alcohol) one-bottle total etch adhesive system to the conventional adhesives Excite (ethanol) and Prime & Bond NT (acetone) under moist and dry dentin conditions.

To do so, we proposed the following hypotheses:

Alternative hypothesis: The bond strength of XP Bond is superior to the bond strength of other one-bottle total etch adhesive systems when applied to dry dentin and moist dentin.

Null hypothesis: The bond strength of XP Bond is not superior to the bond strength of other one-bottle total etch adhesive systems when applied to dry dentin and moist dentin.

MATERIALS AND METHODS

Sample selection

For this study, 42 human third molars, extracted for different reasons, were selected. They were stored in 0.05% Chloramine T solution at room temperature. Inclusion criteria were that they should have no alteration in their anatomical integrity, no caries and no kind of filling. The molars were cleaned with a brush and pumice stone solution in water.

Sample preparation

Teeth were mounted in acrylic cubes to facilitate handling during sample preparation. The occlusal enamel was eliminated under refrigeration in a model trimmer with a carborundum disc until a flat dentin surface parallel to the occlusal surface was obtained. Specimens were inspected under magnification (2.5x Heine HR binocular microscope, Munich, Germany) to determine whether or not there was any remaining enamel. This exposed dentinal surface was polished with a series of Wetordry THREE-M-ITE 180, 400, 500 and 600 grit sandpaper to provide a homogenous dentinal smear layer.

Adhesive procedure and application of the restorative composite resin

The molars were divided at random into six experimental groups of 7 teeth each (n=7). Three (3) one-bottle total etch adhesive systems were used in this study, following manufacturer's instructions. (See Table 1 for a description of the materials used.)

Table 1: Chart showing the different adhesive systems used in this study

<i>Manufacturer</i>	<i>Adhesive</i>	<i>Classification</i>	<i>Composition</i>	<i>Manufacturer's instructions</i>
Dentsply De Trey	Xp Bond	One-bottle total etch	PENTA, UDMA, TEGMA amorphous functional silica nanofilling HEMA Tert-butyl alcohol.	<ol style="list-style-type: none"> 1. Apply etching acid for 15 seconds. 2. Wash etching acid for 15 seconds. 3. Remove excess water. 4. Apply adhesive and let act for 20 seconds. 5. Apply air gently to evaporate solvent. 6. Light-cure for 10-20 seconds. 7. Apply restorative material.
Dentsply De Trey	Prime & Bond NT	One-bottle total etch	PENTA, UDMA, T-resin cross linking agent. D-resin (small hydrophilic molecule) acetone, silica nanofilling.	<ol style="list-style-type: none"> 1. Apply acid for 15 seconds. 2. Wash etching acid. 3. Apply adhesive and dry to evaporate solvent. 4. Light-cure for 10-20 seconds. 5. Apply restorative material.
Ivoclar Vivadent	Excite	Total etch	HEMA, dimethylacrylates, phosphoric acid acrylate, highly dispersed silicon dioxide, initiators and stabilizers in alcohol solution.	<ol style="list-style-type: none"> 1. Apply the phosphoric acid gel for 15 seconds. 2. Remove the etch gel with water spray. 3. Remove excess water with high volume evacuation tip. 4. Apply adhesive with a micro applicator on all prepared dentinal surfaces for 10 seconds. 5. Evaporate the solvent for 3 seconds. 6. Polymerize for 10-20 seconds. 7. Apply restorative material.

The dentinal surface that had been prepared was etched with 37% Total Etch[®] phosphoric acid (Ivoclar Vivadent, Liechtenstein Switzerland) for 15 seconds. It was immediately sprayed plentifully with air and water for 10 seconds and dried under a jet of air at a distance of 10 cm until the dentin was dry for the dry groups, or with a slightly moist gauze for the moist groups, leaving the dentinal surface visibly wet, as follows:

Group 1

Xp Bond, dry (G1D): 37% orthophosphoric acid (Ivoclar Vivadent) was applied for 15 seconds to the dentinal surface, which was then washed with water and thoroughly air-dried for 10 seconds. The dentin was dried for 10 seconds with an air syringe at a distance of approximately 10 cm from the surface (dry technique). Then the Xp Bond adhesive was applied according to the manufacturer's instructions.

Xp Bond moist (G1M): 37% orthophosphoric acid was applied for 15 seconds to the dentinal surface, which was then washed with water and dried with a small piece of gauze, leaving the surface visibly moist (moist technique). Then the Xp Bond adhesive was applied according to the manufacturer's instructions.

Group 2

Prime & Bond NT, dry (G2D): Teeth were treated in the same way as in group 1, except that Prime & Bond NT was used as an adhesive.

Prime & Bond NT, moist (G2M): Teeth were treated in the same way as in group 2 except that in this case Prime & Bond NT was used as an adhesive.

Group 3

Excite dry (G3D): The samples were treated in the same way as groups 1 and 3, except that we used the adhesive Excite for this group.

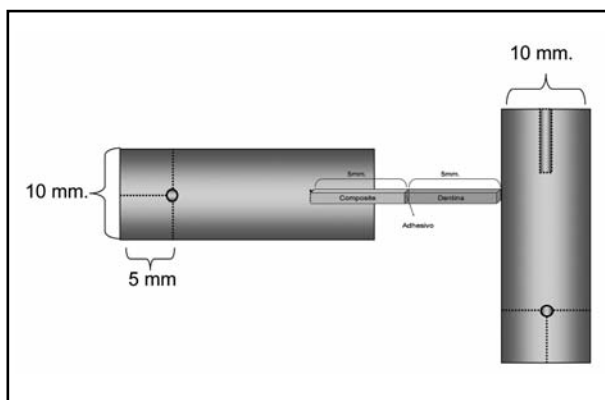


Fig. 1: Schematic drawing of the securing system.

Excite moist (G3M): Teeth were treated in the same way as groups 2 and 4, except that the adhesive system Excite was used for this group.

For each adhesive system we used a composite from the same manufacturer: Ceram duo color dentin A3 for the Dentsply adhesives, and Tetric Ceram A3 for the Excite group. The composite was applied in incremental 1 mm. layers up to a height of 5 mm. In all groups, each layer was polymerized for 20 seconds with a light-curing unit (Blue phase C8, Vivadent Ivoclar) at 800 mW/cm². Forty-two teeth were prepared altogether, 14 for each adhesive, divided into 7 for each condition (moist or dry dentin).

Microtensile bond strength test

Samples were stored in water at room temperature for 24 hours before cutting. The restored teeth were mounted on a precision saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) with a 0.4 mm low-speed diamond disc, with distilled water as a refrigerant. Specimens were cut into slices approximately 1mm thick and 10 mm long, in vestibulo-lingual direction and perpendicular to the adhesive interface. After it was cut into about 8 pieces, the whole tooth was rotated 90 degrees and cut again in mesio-distal direction. As a result of these cuts, rectangular prisms with a 1 mm² cross section were obtained, formed by two arms with an adhesive interface in between. On one side there was a 5 mm arm of composite and on the other a 5 mm arm of dentin obtained from a third cut. A total 15 bars, 10 mm long with 1 mm² cross section were prepared for each experimental group (n=15). They were inspected at 2.5 magnification under Heine HR binocular microscope to make sure they were made up only of dentin, adhesive and composite.

For the traction test, 0.5 mm thick plastic strips (Plakene tropic 111), 2 cm long by 1cm wide, were prepared and then glued with cyanoacrylate (Loc-tite, Spain) to the specimens on the dentin and composite segments respectively on two of their faces, leaving the adhesive interface free. To center the sample on the plastic strips, a 1mm cylinder was drawn in the middle of one end of the strip. The opposite end of the strip was pierced with the tip of a hot explorer at the intersection of a vertical and a horizontal line drawn at 5mm from the edges of the plastic strip. A 0.5 mm 50-100 nylon thread was passed through this hole to form a loop. (See Fig. 1.)

The samples mounted on the strips were fixed to the loading cell on the universal tester machine (Adamel Lhomargy DY 36), which was activated at a speed of 15 mm/min until it reached 10 N, and then continued at a speed of 5 mm/min. The Micro-Tensile Bond Strength (MTBS) data expressed in megapascals (MPa) were recorded. These results were obtained by dividing the force applied at the time of breakage (peak load) by the bonded area (1 mm²). Any specimens that broke during transport and mounting were discarded from the study. For data analysis we used an experimental design for the study of two fixed effect factors using Statgraphics software version 5.1.

RESULTS

Six (6) specimens were discarded from the study; three (3) due to contamination of the adhesive interface with glue when the samples were mounted on the plastic strips (2 G1M + 1 G3M), two (2) that broke when they were dropped while the samples were being mounted on the universal testing machine (1G1M + 1G2M) and one that became detached spontaneously (1G1D).

Variance analysis for bond strength expressed in MPa with a 95% confidence level revealed three p-values lower than 0.05: the bond values obtained with the different adhesives (P-value = 0.0000), type of substrate (P-value = 0.0012) and interaction between the two factors (P-value = 0.0457); see Table 2.

A multiple range comparison was needed to identify which adhesives differed significantly from each other regarding bond strength. The top of Table 3 shows 2 homogeneous groups according to the alignment of the X sign in the column. It shows that there is no statistically significant difference between Excite and Prime Bond NT, but there are significant differences between them and Xp Bond.

Table 2: Variance analysis for bond strength (MPa)

Source of variation	Sum of squares	Degrees freedom	Mean square	F- Coefficient	P-Value
<i>Main effects</i>					
A: Substrate	1298.31	1	1298.31	11.28	0.0012
B: Adhesive	3153.02	2	1576.51	13.69	0.0000
<i>Interactions</i>					
A*B	739.274	2	369.637	3.21	0.0457
Residues	8981.49	78	115.147		
Total (Corrected)	13835.8	83			

Source: Orellana N. (2007)

Table 3: Multiple range comparison for bond strength (MPa) according to adhesive

Adhesive	Count	Mean LS	Sigma LS	Homogeneous groups
Excite	29	33.449	1.99382	X
Prime Bond NT	29	34.2	1.99382	X
Xp Bond	26	47.0896	2.11071	X
<i>Contrast</i>		<i>Difference</i>		<i>+/- Limits</i>
Excite - Prime Bond NT		-0.751		5.61358
Excite- Xp Bond		*-13.6406		5.78047
Prime Bond NT - Xp Bond		*-12.8896		5.78047

* Indicates a significant difference

Source: Orellana J. (2007)

Table 4: Multiple range comparison for bond strength (MPa) according to substrate

Substrate	Count	Mean LS	Sigma LS	Homogeneous groups
Dry	44	34.3039	1.61857	X
Moist	40	42.1885	1.70115	X
<i>Contrast</i>		<i>Difference</i>		<i>+/- Limits</i>
Moist - Dry		*7.88464		4.67476

* Indicates a significant difference

The lower half of Table 3 shows the estimated difference between each pair of means. It can be seen that the bond strength of Xp Bond is higher than those of Excite (-13.6406) and Prime Bond NT (-12.8896) but there is no statistically significant difference between Excite and Prime Bond NT (-0.751). The asterisk beside the two pairs of adhesives indicates statistically significant difference at 95% confidence level.

In order to determine which kind of substrate is better for bonding, we conducted another multiple range comparison. It was found (see Table 4) that

the mean value for the moist substrate (42.19) is higher than the mean value for the dry substrate (34.30) with a statistically significant difference (7.88464).

The bond values of the different adhesives according to the substrate show interaction. This interaction is shown in Table 5, where it can be seen that Excite has low sensitivity

to type of substrate, since the mean values in Mpa for moist substrate (34.16) and dry substrate (32.73) are very similar to each other, but lower when compared to Xp Bond. Prime Bond NT is slightly more sensitive to substrate than in Excite is, and has higher bond values for moist substrate (37.34), and slightly lower values for dry substrate (31.06). For Xp Bond, the bond values were higher than for the rest of the adhesives, both on dry substrate (39.11) and moist substrate (55.06), and it had the greatest sensitivity among all the adhesives tested in this study to variations in moist or dry condition (Table 5).

Table 5: Mean bond strength (MPa) for adhesives according to substrate

<i>Level</i>	<i>Frequency</i>	<i>MEAN</i>	<i>Standard error</i>	<i>Lower limit</i>	<i>Upper limit</i>
<i>ADHESIVE ACCORDING TO SUBSTRATE</i>					
Moist Excite	14	34.1607	2.86789	28.4512	39.8703
Dry Excite	15	32.7373	2.77065	27.2214	38.2533
Moist Prime Bond NT	14	37.3407	2.86789	31.6312	43.0503
Dry Prime Bond NT	15	31.0593	2.77065	25.5434	36.5753
Moist Xp Bond	12	55.0642	3.09768	48.8972	61.2312
Dry Xp Bond	14	39.115	2.86789	33.4055	44.8245

Source: Orellana N. (2007)

DISCUSSION

The evaluation of the tooth-adhesive interface often involves an attempt to determine interfacial bond strength. Although there does not seem to be consensus on the most effective way to measure bond strength, in the past decade there has been an increase in the use of the methodology known as Microtensile Bond Strength (MTBS)⁶. This microtensile methodology is based on the idea that better understanding of the interface bond strength can be obtained from multiple specimens (1mm² in a surface area) obtained from a tooth, whether in the shape of a rectangular prism or an hourglass⁷. This kind of information could lead to a better selection of the technique and restorative material, as *in vitro* results could be extrapolated more easily to daily clinical practice.

In previous decades the most frequently used laboratory procedure to measure dentin bonding was shearing. Flat dentinal surfaces were prepared on human or bovine teeth, onto which the adhesive system and restorative material were applied. A shearing force was applied to the dentin-resin interface, often using a knife or probe and the specimens were evaluated to determine the nature of the breakages (adhesive, cohesive or mixed).

However, this technique has been left aside due to the frequency of cohesive faults in the dentinal substrate, which is directly proportional to the bond strength⁸ and because it lacks the sensitivity needed for discovering subtle differences between bonding systems and procedures, as compared to the traction technique or MTBS⁹.

Therefore we prefer the MTBS system for evaluating bond strength, as it has advantages over the conventional shearing and bonding resistance to traction methodology for the following reasons:

1. Several specimens or compound samples of dental structure, adhesive and composite can be prepared from just one tooth¹⁰.

2. Substrates of clinical significance such as dentin caries, sclerotic dentin, cervical zone and enamel can be evaluated¹¹.
3. Results are more reliable because specimens with a smaller surface area are more homogeneous and have fewer defects, enabling greater bond strength values¹².
4. Regional differences in bond strength within a single tooth can be evaluated¹³.
5. The technique has fewer cohesive faults in dentin compared to other techniques such as shearing¹³.

This study used MTBS on rectangular specimens, (non adjustment method), because for hourglass shaped samples, greater concentrations of stress are generated at the adhesive interface, which may cause premature interface micro-faults and alter the results¹⁴. However, hourglass shaped specimens (adjustment method) with a 1 mm radius curve seem to be better for testing bond interfaces because there are fewer cohesive faults in the specimen¹⁵.

Devices ranging from a universal testing machine to more specific instruments with traction function are used for microtensile tests. Securing the sample to the different devices used for tensile testing requires an additional part to link or connect the specimen to the device. The one most frequently used is the Ciucchi Jig, but depending on the device it is possible to get adaptations of this jig or make a new one. In this study we used plastic strips glued with cyanoacrylate (Loctite) to the dentin and composite segments of the specimens respectively, on two sides, leaving the interface free. A nylon thread was passed through the plastic strips and used to mount the specimens on the testing machine in a manner similar to that described in the study by Kerby et al¹⁶.

The specimens were subject to the traction mode test on a Dy 34 universal testing machine

(Adamel Lhomargy), activated at a speed of 15 mm/minute up to 10 N after which it continued at a speed of 5 mm/min. Although in previous studies^{17,18} the speed for microtensile tests seems to be standardized at 0.5 mm/min, Yamaguchi et al.¹⁹ found that when the machine's speed is set within the range of 0.5-10 mm/min, it does not affect the variation of resistance to traction in micro-tension, because of the small size of the specimens. This is why we used the speed described above and recorded maximum fault load for this research.

During this study, in which we evaluated the microtensile bond strength of adhesive systems applied to dry and moist dentin, we found that adhesion to moist dentin resulted in greater MTBS than adhesion to dry dentin did. These results agree with other *in vitro* studies²⁰⁻²⁴. Nevertheless Miers et al.²⁵ reported that dry and moist adhesion techniques are not a determining factor for bond strength. These discrepancies are owed to the type of test, which was done using the shearing mode, or that the adhesive used (Scotchbond Multi-Purpose 3M/ESPE) for the study may have re-moistened the collagen fibers, favoring monomer infiltration, as shown by Dal-Bianco et al.²⁶ when they compared the effects of moisture and rubbing action on dry dentin in the MTBS test for adhesive systems based on water/ethanol (Single Bond 3M/ESPE) and acetone (One Step BISCO).

When demineralized dentin is air-dried, the water within the collagen matrix is eliminated and collagen fibers are attracted until they achieve close contact, forming a weak inter-peptide link, shrinking, hardening and becoming practically impermeable to the adhesive resin^{27,28}. This leads to poor hybridization, reducing the infiltration ratio of the adhesive resin within the hybrid layer.

In agreement with other studies²⁹⁻³¹, another finding of the present research was that the Prime & Bond NT adhesive system with acetone as a solvent produced higher MTBS results in moist dentin than Excite, which uses ethanol, did.

In this regard, Reis et al.²³ state that a quantity of water is required to maximize the bond strength in adhesives available on the market. While acetone-based adhesives require a moister surface, water-based systems attain higher bond strength on dry surfaces.

Along the same lines, other authors found maximum hybridization and resinous tag formation when they used acetone-based adhesives in the moist adhesion technique, drying with cotton swabs^{32,33}. This might explain the slightly higher

bond values we obtained with Prime & Bond NT on moist dentin, even though they were not statistically significant when compared to Excite.

Nevertheless, other studies³⁴ have found that ethanol-based adhesive systems have higher bond strength than acetone-based systems. This might be due to the fact that acetone as a solvent has high volatilization and rapid evaporation from the open bottle and is therefore considered to be a less favorable option as a solvent^{35,36}.

Other studies^{37,38} using methodology similar to ours found equivalent results for Excite and Prime Bond NT, in agreement with the results of our study. Nevertheless, the values obtained for Prime bond NT on dry dentin were lower. According to Perdigao, this is due to the accumulation of nanofilling deposited at the entrance to the dentinal tubules, which interferes with the entry of the adhesive when the dentin is dried³⁹. This might explain Xp Bond's sensitivity, as its composition includes nanofilling.

Our study found that the Xp Bond adhesive system with tert-butyl alcohol as a solvent, had higher MTBS than the Prime & Bond NT and Excite, which use acetone and ethanol as solvents, respectively, on both moist and dry dentin.

To date we have not found any previous studies measuring bond strength of Xp Bond to compare with our results. Although the bond strategy of Xp Bond is similar to the adhesive systems tested in this study, the bond values obtained in this study for moist and dry dentin appear to be due to the nature of the solvent.

Quantum mechanics, used in other fields for understanding specific phenomena, may explain the results of this research at molecular level. Quantum mechanics is the branch of physics that explains very small-scale behavior of matter. In this case, we used software (MOPAC) to understand the molecular relationship of adhesive systems with the collagen of partly demineralized dentin. The results we obtained show that there is a chemical reaction between collagen and Xp Bond and Excite adhesives in gaseous state, since enthalpic calculations (see Fig. 2 and 3) show a spontaneous reaction of the components of the adhesives with collagen. No spontaneous reaction was observed for Prime & Bond NT (see Fig. 4), suggesting that there is no kind of chemical bond between the adhesive and the collagen.

The adhesion values obtained with an acetone-based adhesive could probably be explained by its high vapor pressure, which enables rapid evaporation, and the ease with which it divides water molecules in liquid state, producing more

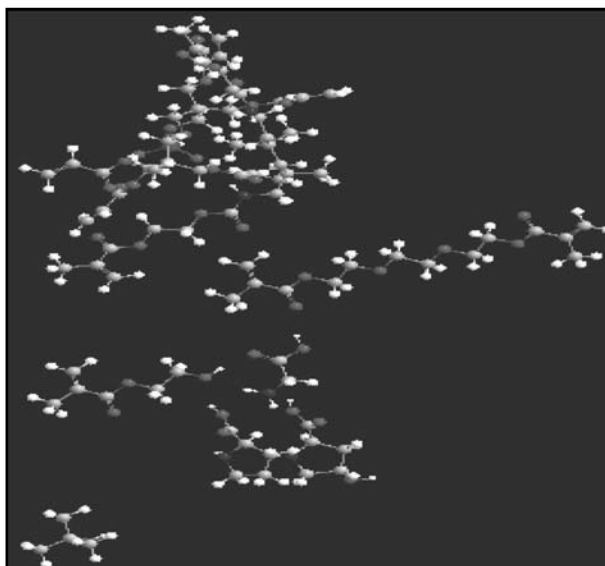


Fig. 2: Theoretical calculation of ΔH for Xp Bond + collagen $\Delta H = -1722.69565$ kcal/mole using Mopac with (AM1).

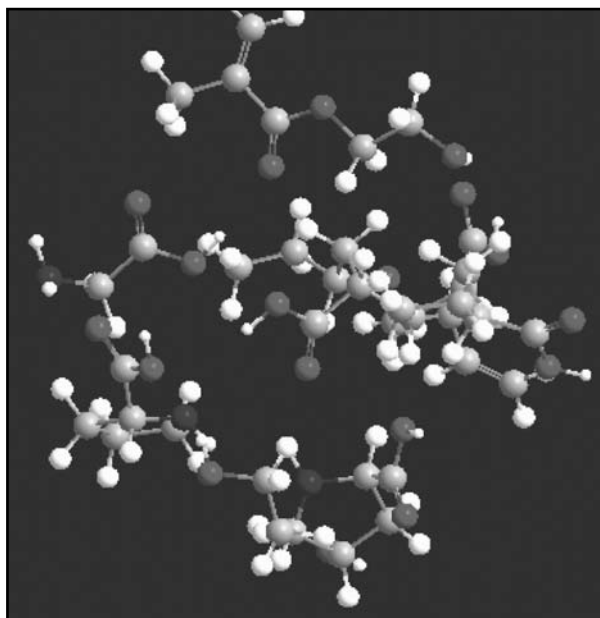


Fig. 3: Theoretical calculation of ΔH for Prime Bond NT + collagen $\Delta H = 9427.88272$ kcal/mole using Mopac with (AM1).

penetrating infiltration of monomers, which produce a larger, deeper mechanical block on hardening. On the other hand, the lack of chemical reactivity might also explain why it is susceptible to failing in microfiltration tests, since the bond would leave nanometric spaces through which fluids could leak and produce the degradation of the interface^{40,41}. The aforementioned spontaneous reaction might be inversely proportional to the degree of microfiltration. In his regard, Rosales⁴² found that XP Bond performed well regarding sealing the gingival wall in dentin, compared to self-etch systems such as Clearfil SE Bond (Kuraray Dental), XENO III (Dentsply) and i-Bond (Haraeus Kulzer).

Within the limitations of this study, we can infer that dentin must remain under ideal moisture conditions when total etch adhesives are used. The single dose commercial presentation of these adhesive systems should be preferred to the conventional bottle in order to reduce the risk of solvent evaporation, which leads to poor hybridization of these systems. All the adhesives used in this study can be used in daily practice. Aging studies must be awaited to determine whether those values endure over time or are affected by hydrolytic degradation, sudden temperature changes or mechanical fatigue.

Considering all of the above, we reject the null hypothesis and accept the alternative hypothesis, since XP Bond bond strength was higher than the bond strength of other one-bottle total etch bonding

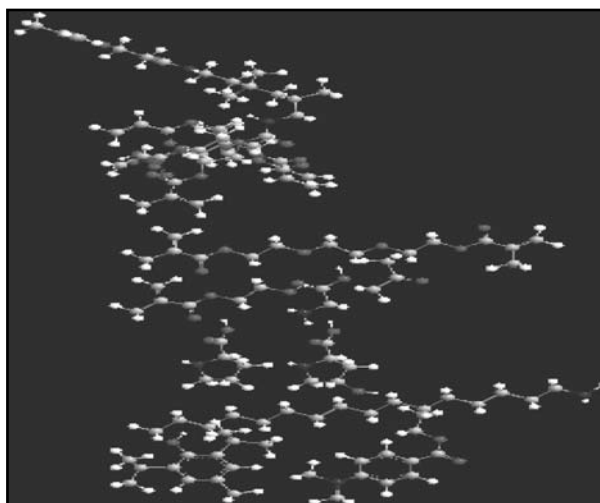


Fig. 4: Theoretical calculation of ΔH for Excite + collagen $\Delta H = -919.75766$ kcal/mole using Mopac with (AM1).

systems when applied to dry dentin and moist dentin.

CONCLUSIONS

Adhesion to moist dentin resulted in greater tensile bond strength than adhesion to dry dentin. Xp Bond applied to moist dentin resulted in greater bond strength than the other groups, attaining the highest bond values. Its bond strength was superior to Excite and Prime Bond NT.

ACKNOWLEDGMENTS

We would like to thank International Catalonia University, DENSTPLY and Los Andes University CDCHT (0-219-08-07-A) for the funding and support that enabled this research.

CORRESPONDENCE

Noé Orellana.
Universidad de Los Andes, Facultad de Odontología.
Calle 24 entre Av. 2-3,
Edificio El Rectorado Cátedra de Operatoria Dental,
(5101) Mérida, Venezuela.
Phone: 582742402394.
e-mail: noeorellana@yahoo.com

REFERENCES

- Buonocore, MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955;34:849-853.
- Schwartz R, Summitt J, Robbins W. *Fundamentos en Odontología Operatoria*. Caracas: Actualidades Médico Odontológicas; 1999.
- Molla K, Park HJ, Haller B. Bond strength of adhesive/composite combinations to dentin involving total- and self-etch adhesives. *J Adhes Dent* 2002;4:171-180.
- Tay FR, Pashley DH. Water treeing-a potential mechanism for degradation of dentin adhesives. *Am J Dent* 2003;16:6-12.
- Cardoso Pde C, Lopes GC, Vieira LC, Baratieri LN. Effect of solvent type on microtensile bond strength of a total-etch one-bottle adhesive system to moist or dry dentin. *Oper Dent* 2005;30:376-381.
- De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, Van Meerbeek B. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005;84:118-132.
- Eckert GJ, Platt JA. A statistical evaluation of microtensile bond strength methodology for dental adhesives. *Dent Mater* 2007;23:385-391.
- Oilo G. Bond strength testing-what does it mean?. *Int Dent J* 1993;43:492-498.
- Cavalcante LM, Erhardt MC, Bedran-de-Castro AK, Pimenta LA, Ambrosano GM. Influence of different tests used to measure the bond strength to dentin of two adhesive systems. *Am J Dent* 2006;19:37-40.
- Yoshiyama M, Carvalho RM, Sano H, Horner JA, Brewer PD, Pashley DH. Regional bond strengths of resins to human root dentin. *J Dent* 1996;24:435-442.
- Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM, Pashley DH. Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives. *J Dent Res* 1995;74:1679-1688.
- Phrukkanon S, Burrow MF, Tyas MJ. The influence of cross-sectional shape and surface area on the microtensile bond test. *Dent Mater* 1998;14:212-221.
- Shono Y, Ogawa T, Terashita M, Carvalho RM, Pashley EL, Pashley DH. Regional measurement of resin-dentin bonding as an array. *J Dent Res* 1999;78:699-705.
- Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA, Tay F. The microtensile bond test: a review. *J Adhes Dent* 1999;1:299-309.
- Meira JB, Souza RM, Driemeier L, Ballester RY. Stress concentration in microtensile tests using uniform material. *J Adhes Dent* 2004;6:267-273.
- Kerby RE, Knobloch LA, Clelland N, Lilley H, Seghi R. Microtensile bond strengths of one-step and self-etching adhesive systems. *Oper Dent* 2005;30:195-200.
- Can Say E, Nakajima M, Senawongse P, Soyman M, Ozer F, Ogata M, Tagami J. Microtensile bond strength of a filled vs unfilled adhesive to dentin using self-etch and total-etch technique. *J Dent* 2006;34:283-291.
- Uekusa S, Yamaguchi K, Miyazaki M, Tsubota K, Kurokawa H, Hosoya Y. Bonding efficacy of single-step self-etch systems to sound primary and permanent tooth dentin. *Oper Dent* 2006;31:569-576.
- Yamaguchi K, Miyazaki M, Takamizawa T, Tsubota K, Rikuta A. Influence of crosshead speed on micro-tensile bond strength of two-step adhesive systems. *Dent Mater* 2006;22:420-425.
- Gwinnett AJ, Kanca JA 3rd. Micromorphology of the bonded dentin interface and its relationship to bond strength. *Am J Dent* 1992;5:73-77.
- Perdigao J, Swift EJ, Cloe BC. Effects of etchants, surface moisture, and resin composite on dentin bond strengths. *Am J Dent* 1993;6:61-64.
- Abdalla AI, Davidson CL. Bonding efficiency and interfacial morphology of one-bottle adhesives to contaminated dentin surfaces. *Am J Dent* 1998;11:281-285.
- Reis A, Loguercio AD, Azevedo CL, de Carvalho RM, da Julio Singer M, Grande RH. Moisture spectrum of demineralized dentin for adhesive systems with different solvent bases. *J Adhes Dent* 2003;5:183-192.
- Cardoso Pde C, Lopes GC, Vieira LC, Baratieri LN. Effect of solvent type on microtensile bond strength of a total-etch one-bottle adhesive system to moist or dry dentin. *Oper Dent* 2005;30:376-381.
- Miears JR Jr, Charlton DG, Hermes CB. Effect of dentin moisture and storage time on resin bonding. *Am J Dent* 1995;8:80-82.
- Dal-Bianco K, Pellizzaro A, Patzlaft R, de Oliveira Bauer JR, Loguercio AD, Reis A. Affects of moisture degree and rubbing action on the immediate resin-dentin bond strength. *Dent Mater* 2006;22:1150-1156.
- Pashley DH, Carvalho RM. Dentin permeability and dentin adhesion. *J Dent* 1997;25:355-372.
- Mohan B, Kandaswamy D. A confocal microscopic evaluation of resin-dentin interface using adhesive systems with three different solvents bonded to dry and moist dentin in vitro study. *Quintessence Int* 2005;36:511-521.
- Pflug K, Gomes G, Perdigao J, Swift EJ. Bond strengths of acetone-based one-bottle adhesives. En: IADR. [Abstract 2290], *J Dent Res* 1999;78.

30. Sang J, Hammesfahr PD, Hayes LC. Adhesion comparison of current dual cure dental adhesive systems. [Abstract 3005], *J Dent Res* 1999;78.
31. Munoz CA, Dunn N, Jessop J, Munoz SY. Shear bond strength to enamel and dentin using three one-bottle filled adhesives. [Abstract 1704], *J Dent Res* 2000;79.
32. Gregoire GL, Akon BA, Millas A. Interfacial micromorphological differences in hybrid layer formation between water- and solvent-based dentin bonding systems. *J Prosthet Dent* 2002;87:633-641.
33. Bhuvaneshwaran Mohan, Deivanayagam Kandaswamy. A confocal microscopic evaluation of resin-dentin interface using adhesive systems with three different solvents bonded to dry and moist dentin. An in vitro study. *Quintessence Int* 2005;36:511-521.
34. Lopes GC, Cardoso PC, Vieira LC, Baratieri LN, Rampinelli K, Costa G. Shear bond strength of acetone-based one-bottle adhesive systems. *Braz Dent J* 2006;17:39-43.
35. Gallo JR, Burgess JO, Xu X. Effect of delayed application on shear bond strength of four fifth-generation bonding systems. *Oper Dent* 2001;26:48-51.
36. Perdigão J, Swift EJ Jr, Lopes GC. Effects of repeated use on bond strengths of one-bottle adhesives. *Quintessence Int* 1999;30:819-823.
37. Perdigão J, Frankenberger R. Effect of solvent and rewetting time on dentin adhesion. *Quintessence Int* 2001;32:385-390.
38. Perdigão J, Geraldeli S, Carmo AR, Dutra HR. In vivo influence of residual moisture on microtensile bond strengths of one-bottle adhesives. *J Esthet Restor Dent* 2002;14:31-38.
39. Perdigão J, Lopes M. Dentin bonding-questions for the new millennium. *J Adhes Dent* 1999;1:191-209.
40. Duarte S Jr, Perdigão J, Lopes MM. Effect of dentin conditioning time on nanoleakage. *Oper Dent* 2006;31:500-511.
41. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 2003;28:215-235.
42. Rosales Juan. XP Bond y el sellado de la interfase de unión. *Dentsply Noticias*. 2007;36:18-20.