

IN VIVO BRACKET BOND STRENGTH USING TWO ADHESIVE SYSTEMS APPLIED UNDER WET AND DRY CONDITIONS

Elida N. Ciola¹, Alicia M. Picco¹, Ana M. Sois², Mercedes H. Lucena³, Verónica Alonso¹,
Maella Valvo¹, Luis García², Ariel Geazzi²

¹Department of Orthodontics, School of Dentistry, National University of Rosario, Argentina. ²Institute of Applied Mechanics and Structures, School of Engineering, National University of Rosario, Argentina. ³Department of Biochemistry, School of Dentistry, National University of Rosario, Argentina.

ABSTRACT

The purpose of this study was to investigate, *in vivo*, the bond strength of two adhesive materials: a moisture insensitive primer (MIP)* and a one step self etching primer (SEP)*, both used with Transbond XT* on dry and wet enamel and an adhesion time of 10-15 minutes. First or second upper and/or lower bicuspid (n=124), to be extracted for orthodontic reasons, were used. A comparison of the materials' behavior was conducted under four different situations: 1) MIP on enamel etched and dry; 2) MIP on a surface etched and wetted with patient's saliva; 3) SEP on a dry field, 4) SEP on a saliva-wet enamel. For statistical analysis, Dunn-Sidak's multiple comparison test was applied with a probability of less than 0.05 (before correction). Stainless

steel brackets with mesh-backed pads were bonded to the teeth. Bond strength was tested with modified orthodontic pliers on which a strain-gage was fixed to measure handle deformation while debonding. Moisture insensitive primer tested on wet enamel showed the highest mean bond strength outcomes (8.98 MPa) compared to one step etching primer (5.81 MPa). Statistical difference between these groups was significant ($p = 0.000$). Standard deviation was lower for the one-step technique, under dry and wet conditions. Since the media bond strength of SEP proved sufficient for clinical purposes and its behavior tended to be more homogeneous, this was considered the best choice.

Key words: brackets, resins, adhesion, strength, humidity.

COMPARACIÓN IN VIVO DE LA FUERZA DE DESPEGUE DE DOS SISTEMAS ADHESIVOS EN CONDICIONES SECAS Y HÚMEDAS

RESUMEN

El propósito de este estudio fue investigar *in vivo* la efectividad de dos materiales adhesivos: un Primer insensible a la humedad (MIP) y otro, autograbante en un solo paso (SEP). Ambos materiales fueron utilizados con Transbond XT sobre esmalte seco y húmedo de los primeros premolares superiores e inferiores (n=124) que debían ser extraídos por razones ortodóncicas.

La comparación del comportamiento de los mismos fue realizada bajo cuatro situaciones diferentes 1) MIP sobre esmalte grabado y seco 2) MIP sobre superficie grabada y humedecida con saliva del paciente 3) SEP sobre campo seco, y 4) SEP sobre esmalte húmedo con saliva.

Sobre la cara vestibular de cada unidad se adhirieron brackets de acero inoxidable APC con resina incorporada. La fuerza de despegue fue medida con un alicate de Ortodoncia modificado, en uno de cuyos brazos fue adosado un transductor extensométrico que permitía medir la deformación del mismo durante el desprendimiento del bracket.

Para el análisis estadístico se aplicó Análisis de la Variancia (ANOVA) y el test de comparación múltiple Dunn-Sidak

con una probabilidad menor al 0,05 (antes de la corrección).

El Primer insensible a la humedad evaluado sobre esmalte húmedo mostró la mayor fuerza de despegue (MIP: 8,98MPa) comparado con el de grabado en un sólo paso (SEP: 5,81MPa). La diferencia estadística entre ambos grupos resultó altamente significativa ($p=0.000$).

La variabilidad en los resultados fue menor utilizando la técnica de un sólo paso (SEP), bajo condiciones secas y con la técnica (MIP) bajo condiciones húmedas.

Basándonos en el hecho de que el SEP requiere una técnica de un solo paso y que la media para la fuerza de despegue (en ambas condiciones) resultó suficiente para los propósitos clínicos, además de un comportamiento homogéneo de sus resultados (menor variabilidad), fue considerada una opción válida entre los materiales comparados, aún cuando el MIP en condiciones de humedad presentó la mayor fuerza de despegue y valores similares de variabilidad.

Palabras clave: brackets, resinas, adhesión, fuerza, humedad.

* 3M Unitek. Monrovia, California

INTRODUCTION

Direct bracket bonding based on acid-etching was first suggested by Newman (1) in 1964 and it has been in use for almost four decades because of its many advantages compared to former methods. However, orthodontists know that its disadvantage lies in managing a moisture-sensitive technique in a highly humid environment (2).

Saliva contamination after etching reduces surface energy and fills enamel porosities, diminishing the number and length of resin tags, thus resulting in poor mechanical retention (3,4). For Zachrisson (5), moisture is the most common reason for bond failure. Although orthodontists are aware of this, they have to deal with a variety of clinical conditions that do not allow ideal isolation (6).

Manufacturers have tried to offer a solution to this problem, introducing hydrophilic bonding materials. It has been reported that these hydrophilic primers (first designed to adhere resin to dentin), when used on enamel, seem to have reduced bond strengths (7) and that this effect is enhanced when enamel is contaminated by saliva (8).

New systems are based on resin-modified glass ionomers that accept humidity better than their resin-based counterparts.

This study was designed to test in vivo the shear bond strength of two of them.

Transbond XT was tested with a moisture insensitive primer (MIP), recommended to be used with chemical or light-activated resins, either on dry- or wet-etched enamel although Grandy R et al. (9) have suggested that (MIP) should be used only with light-activated composite resins.

Adhesion of brackets to enamel using MIP is a process that includes several steps: etching with phosphoric acid to obtain higher bond strengths (10), washing with water and drying. The result is a high surface energy.

The other material tested was a non washing self etching primer combination of phosphoric acid and a methacrylate group (SEP). According to the manufacturer's laboratory research, this system enables the primer to penetrate the whole depth of the etch in just one step. This approach is intended to improve retention, shorten chair-side time and simplify the technique.

MIP basic physico-chemical processes are similar to those of SEP, except during polymerization.

The aim of this study was to compare in vivo the shear bond strengths of two composite hydrophilic materials right at the time when clinical work should be resumed after adhesion. One of them (MIP), used on phosphoric acid etched enamel, and the other (SEP), employed as a one step etching-primer as recommended by manufacturer, were used with Transbond XT, under wet and dry conditions.

MATERIALS AND METHODS

This experiment included one hundred and twenty four (124) teeth belonging to 44 patients who needed 2/4 bicuspid extractions, and provided their written consent to participate in this investigation. APC's Victory MBT* brackets with bases of 10.61 sq mm (0.01644 sq in) were bonded on first or second upper and/or lower bicuspid before removal. The type of bracket was selected in order to avoid technical difficulties in obtaining a well spread and homogeneous coating.

Bicuspid were selected as units because they had to be extracted, although clinical experience shows premolars are one of the most difficult pieces on which to perform direct bonding (11). Their changing sizes and curvature ratios may prevent correct bracket positioning or adaptation.

For measuring shear bond strength of both resins, a debonding device was developed, based on modified band remover pliers, whose jaws were trimmed for a better bracket embracement. A strain gage was fixed to one of the handles of the pliers (Figs. 1 and 2).

A constant pressure system was applied to give a value of deformation independent of the operator (Fig. 3).

Tightening the pliers deforms the handles. The deformation sensed by the strain gage is transformed into an electrical signal by means of a strain indicator (Fig. 4).

The numerical tension value recorded is the resulting maximum deformation value corresponding to the debonding stress. Then, using known masses in a laboratory simulation, the same value of deformation is reproduced, to obtain the actual debonding stress.

The instrument was chosen and modified in order to produce a shear stress as pure as possible, ignor-

* 3M Unitek, Monrovia, California

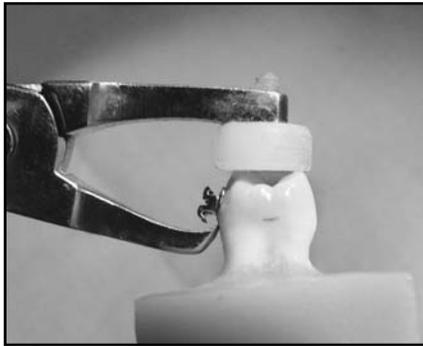


Fig. 1.
Modified jaws
of orthodontic
band removing
pliers.

Fig. 2.
Strain gage
fixed under
sleeve of right
handle.

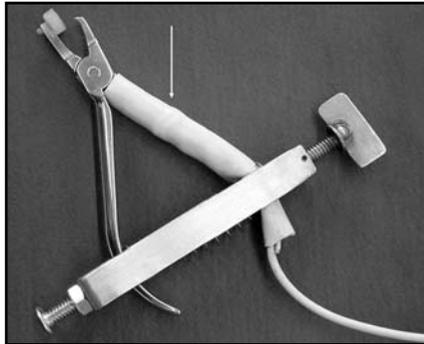


Fig. 3.
System selected
to produce
deformation.

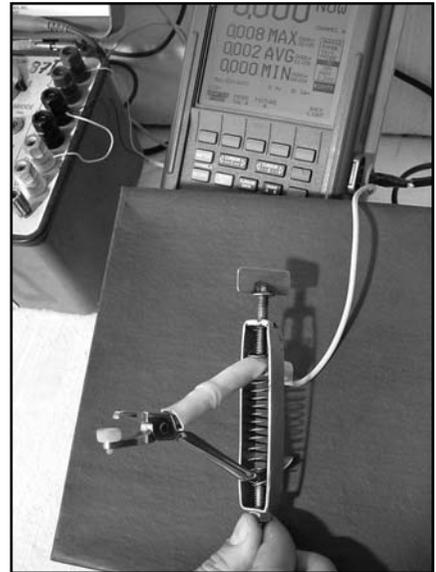
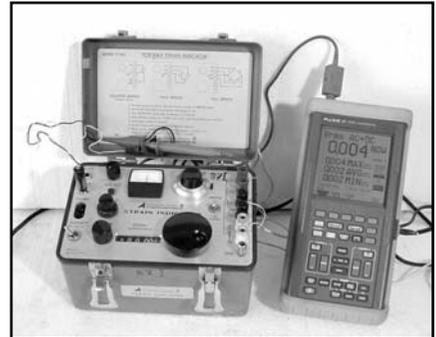


Fig. 4. Strain
indicator.



ing the moment that could exist between the acting forces. Shear debonding strength was calculated using the nominal area given by the brackets' manufacturer because of the impossibility of determining the actual adhesion surface. Frictional force had a constant influence on all measurements, and lastly, the pliers positioning angle depended on the dental morphology (Fig. 1), so that all factors involved in the shear bond strength measurement were considered as confounding variables since they had a random and non-controllable effect on each observed unit.

Bracket debonding was performed by the same operator at all times.

Patients were attended one by one and randomly numbered from 1 to 44.

They were instructed not to eat or drink for 1 hour before bracket adhesion. Teeth were cleaned and polished with a nonfluoride pumice paste with a rubber cup for 10 seconds. Lips and cheeks were held apart with spandex and field-dried with a high-suction device.

Buccal surfaces of 1st or 2nd upper and lower right bicuspid (A – C, Table I) were etched for 15 sec with a 37% phosphoric acid gel supplied by the

manufacturer. Afterwards, teeth were rinsed thoroughly with distilled water for 30 sec and dried with oil and moisture free air. The enamel surface was then inspected to check the dull, white appearance of the etching.

The buccal surfaces of upper bicuspid (A – B, Table I) corresponding to those patients filed with an odd number, were then brushed with one coat of the patient's saliva. The same procedure was followed for even-numbered patients, although on these saliva was brushed on lower bicuspid (C –

TABLE I. Experimental design.

| Wetted upper buccal surfaces: odd numbers | | | |
|---|--|--|-----|
| (A) | 2 nd bi or 1 st bi | 1 st bi or 2 nd bi | (B) |
| (C) | 2 nd bi or 1 st bi | 1 st bi or 2 nd bi | (D) |
| Wetted lower buccal surfaces: even numbers | | | |
| n = 124 | | | |

TABLE II. Two factor ANOVA table. Analysis of Variance to compare bond strength of the resins and conditions (wet-dry) of dental enamel.

| Source of Variation | Degrees of freedom | Mean squares | F statistics | Associated probability for F |
|---------------------|--------------------|--------------|--------------|------------------------------|
| RESINS | 1 | 155.834 | 1.52 | 0.0002 |
| CONDITION | 1 | 1.1302 | 0.11 | 0.7461 |
| Interactions | 1 | 26.486 | 2.47 | 0.1188 |
| Residual | 120 | 10.731 | | |
| Total | 123 | 11.929 | | |

TABLE III. Result of estimators by group (In MPa).

| Estimators | Group | | | |
|---------------------|-----------|-----------|-----------|------------|
| | SEP Dry | SEP Wet | MIP Dry | MIP Wet |
| Mean | 6.54 | 5.81 | 7.86 | 8.98 |
| Standard Deviation | 2.60 | 2.43 | 4.24 | 3.49 |
| Coef. Variation (%) | 40% | 42% | 54% | 40% |
| Conf. int. | 5.38-7.70 | 4.69-6.93 | 6.72-9.01 | 7.74-10.23 |
| Max. | 12.52 | 10.31 | 18.66 | 16.94 |
| Min. | 1.23 | 1.47 | 0.98 | 3.93 |

D, Table I) in order to alternate higher moisture possibilities.

Then, usual procedures for adhesion were followed, using MIP for the right and SEP for the left sides in all patients.

Brackets were removed from their light-sealed packages at the moment of use, then placed on the buccal side of bicuspids and pressed in the center with a setting force of approximately 0.250 / 0.300 kgf with a Dontrix gage*, modified for this purpose. Excess adhesive was removed with a scaler. Brackets were bonded with a light-curing unit* for 10 sec on distal sides and 10 sec on mesial sides.

The other primer (SEP) was used on the patients' left side. Considering the characteristics of one step self-etching primers, the second adhesion step (phosphoric acid application and water rinsing) was not performed. Lower premolars of even-numbered patients were again wetted with saliva. The same procedure was performed on upper bicuspids in odd-numbered patients. All others steps were reproduced.

* 3M Unitek. Monrovia, California

The pliers were placed as shown in Fig. 1. The same operator removed all the brackets in the same way between 10 and 15 minutes after adhesion. The time in which the test should be completed was carefully decided based on the following aims:

- 1) To evaluate degree of adhesion in a time close to that recommended by the manufacturer for restarting clinical procedures.
- 2) To limit discomfort to the patient.

The data were recorded and processed for later statistical analysis.

To compare the bond strength of both resins (MIP and SEP) and the conditions of the environment (dry and wet), a two-factor analysis of variance (ANOVA) was used.

RESULTS

Based on these results (Table II), it is considered that both resins used affect the response variable "debonding strength" (P=0.0002).

There is no statistical evidence that the condition of the environment (wet or dry) affects debonding strength (P=0.7461) 10-15 minutes after adhesion

TABLE IV. Analysis of variance (ANOVA) for bond strength values in the resin response groups under different enamel conditions.

| Source of Variation | Degrees of freedom | Mean squares | F statistic | Associated probability for F |
|---------------------|--------------------|--------------|-------------|------------------------------|
| Between groups | 3 | 59.835 | 5.576 | 0.001 |
| Within groups | 120 | 10.731 | | |
| Total | 123 | | | |

TABLE V. Dunn-Sidak multiple comparison test for the analysis of difference between groups ($\alpha = 0.05$).

| Categories | Mean | Deviation | Sample | Test Hyp |
|------------|------|-----------|--------|----------------|
| SEP Dry | 6.54 | 2.60 | 31 | p=0.367 |
| SEP Wet | 5.81 | 2.43 | 34 | not sig |
| MIP Dry | 7.86 | 4.24 | 32 | p=0.194 |
| MIP Wet | 8.98 | 3.49 | 27 | not sig |
| SEP Dry | 6.54 | 2.60 | 31 | p=0.112 |
| MIP Wet | 7.86 | 4.24 | 32 | not sig |
| SEP Dry | 5.81 | 2.43 | 34 | p=0.000 |
| MIP Wet | 8.98 | 3.49 | 27 | sig dif |

or that there is interaction between the factors resin and condition (P=0.1188).

Multiple comparison tests show highly significant differences for the two resins, but not for the condition, where the null hypothesis of equality cannot be rejected, coinciding with the ANOVA table.

For this reason it was thought best to make a one-factor analysis of variance, considering the situations (levels) as four treatments.

Data on the debonding strength of the materials studied, under wet and dry conditions, are shown in Table III.

This set of estimators shows the behavior of debonding strength in the different groups.

MIP, under saliva-wetted enamel conditions, showed the highest value (8.98 Mpa).

When used under dry conditions, bonding strength decreased to 7.86 MPa.

Conversely, SEP showed slightly better results on dry surfaces (dry SEP 6.54 MPa / wet SEP 5.81 MPa).

In all cases, the mean debonding strength for SEP was below the values for MIP, in both wet and dry conditions.

The analysis of the one-factor ANOVA table showed that not all the debonding strength averages are the same (F test with P = 0.001) (Table IV).

To determine where the difference laid, the Dunn-Sidak multiple comparisons test was applied (Table V) with a significance level of 5% ($\alpha = 0.05$). After comparisons, different behaviors were evaluated. There were no statistically significant differences when each material was considered individually, on dry or wet enamel (SEP: P=0.367 and MIP: P=0.194). When the mean for the debonding strength of the two resins was compared under identical enamel conditions, highly significant differences (P=0.000) were only found under wet conditions (MIP 8.98 /SEP 5.81 MPa).

The Dunn-Sidak test showed the existence of three significantly different groups (Table VI).

Group 1: Resin presenting lower debonding strength (SEP Wet and SEP Dry), identified with letter A.

TABLE VI. Grouping of treatments according to the Dunn-Sidak test ($\alpha = 0.05$).

| Treatments | Conf. Int. | Groups | |
|------------|-------------|--------|---|
| SEP Wet | 5.81 ± 0.82 | A | |
| SEP Dry | 6.54 ± 1.03 | A | |
| MIP Dry | 7.86 ± 1.43 | A | B |
| MIP Wet | 8.98 ± 1.29 | | B |

Group 2: Resin MIP under dry enamel conditions, presenting an intermediate strength, identified with the letter combination AB.

Group 3: Resin MIP under wet enamel conditions, presenting the greatest debonding strength, assigned letter B.

DISCUSSION

The debonding strengths of the humidity insensitive Primers obtained *in vivo* were lower than those reported in *in vitro* studies. It should be remembered that the observations in this study were made on live teeth and in periods varying between 10 and 15 minutes after bracket placement. At that time the material achieves enough adhesion to continue clinical procedures, although full polymerization is reached 24 hours later. Our expectations –according to studies published by the manufacturer– indicated that this strength should be some 60% of the total debonding strength achieved in that interval.

Webster et al. (6) working *in vitro* with identical material but under different conditions obtained strengths within a range of 28.12 to 15.28 MPa. Grandhi's (9) research showed values of 10.14 and 8.90 MPa for the same adhesive system, under dry and wet conditions.

The highest debonding stress obtained in this study was for Transbond XT/MIP used on wet enamel (8.98 MPa). The differences obtained for the same system applied on dry enamel were not significant ($P=0.194$).

Pickets (12) reported differences in the behavior of the same resin *in vitro* and *in vivo* (debonding stress values of 12.82 and 5.47 MPa respectively), while working with the conventional Transbond XT system.

It is important to point out that in this investigation resins remained adhered to the bracket, remaining separate from the enamel. It should also be remembered that this work was done in the lateral zones of the dental arches, where vision is hampered and it is difficult to avoid humidity. Crevicular exudate is usually present and eliminating it is not an easy task, especially in short-crowned premolars. It is probable that the debonding stress reached after 10-15 minutes would have been greater if a dental piece with a different location in the arch had been chosen, or if the debonding of the bracket had been made after 24 hours.

These factors may have partially masked the true performance of the resin.

The comparative results of the studied materials may be seen in Table V.

In all cases the debonding stress of SEP was lower than that of MIP.

The heterogeneous performance of the latter material may have been due to the number of clinical steps involved in its processing, thus increasing the possibility of error. The handling of one step SEP enables some of these to be eliminated, which may account for its more homogeneous behavior.

The reasons why the self etching material presented a lesser debonding strength than MIP may be that the demineralization process of the former is chemically controlled and is regulated so as to produce a certain degree of enamel decalcification. This fact should be related with the period of activity of the radical phosphate on the enamel, which may be different from that chosen for MIP, which requires human intervention at this stage.

Comparison of the debonding force for both resins on wet enamel proved highly significant ($P=0.000$). Despite the results obtained, it should be considered that the debonding strength of SEP was sufficient (6.54/5.81 MPa) for clinical use, especially bearing in mind that the debonding tests were carried out 10-15 minutes after polymerization. It has been reported (13) that debonding strength is sufficient when it is within a range between 5.9 and 7.8 MPa. These levels should be enough to withstand the masticatory and orthodontic forces.

CONCLUSIONS

This study was performed *in vivo* to compare the debonding strengths of a Moisture Insensitive Primer (MIP) and a one step self etching primer (SEP), both used with Transbond XT. Resins were tested on wet and on dry enamel, 10 to 15 minutes after bracket adhesion.

The results obtained showed that:

- 1) MIP had the highest mean in terms of debonding strength.
- 2) The mean for the debonding strength of SEP was clinically acceptable.
- 3) Working under wet conditions, the comparison of means for the debonding strength of both materials revealed statistically significant differences.

- 4) MIP showed greater variability in the values of debonding strength under dry conditions as compared to the more homogeneous behavior of SEP, under the same conditions. Comparison of means for debonding strengths did not reveal statistically significant differences.
- 5) The high coefficient of variability found in the behavior of MIP when dry may generate uncertainty at clinical level. These findings together with the simplicity and speed of clinical handling of SEP led us to conclude that this material has more advantages than MIP.

ACKNOWLEDGEMENTS

We are grateful to 3M Unitek for their generous donation of material.

We also thank Dr. Ricardo Macchi, for his comments and advice.

CORRESPONDENCE

Dra. Elida G. de Ciola
Rivadavia 2147 5° 1
Rosario 2000
elidaciola@arnet.com.ar
ciola@arnet.com.ar

REFERENCES

1. Newman GV. Bonding plastic orthodontic attachments to tooth enamel. *J NJ Dental Soc* 1964; 35:346-58.
2. Gwinnett AJ. Bonding of restorative resins to enamel. *Int Dent J* 1988; 38:91-96.
3. Silverstone LM. State of arts on sealant research; *J Dent Educ* 1984; 48 (supp): 107-18.
4. Hormati AA, Fuller JL, Denehy GE. Effects of contamination and mechanical disturbance on the quality of acid etched enamel. *J Am Dent Assoc* 1980; 100:34-8.
5. Zachrisson BJ. A posttreatment evaluation of direct bonding in orthodontics. *Am J Orthod* 1997; 71:173-89.
6. Webster MJ, Nanda R, Duncanson MG Jr, Khajotia S, Sinha P. The effect of saliva on shear bond strengths of hydrophilic bonding systems. *Am J Orthod Dentofacial Orthop* 2001; 119:54-58.
7. Woronko GA Jr, Germain HA Jr, Meiers JC. Effects of dentin primer on the shear bond strength between composite and enamel. *Operative Dentistry* 1996; 21:116-21.
8. Xie J, Powers JM, Mc Guckin RS. In vitro bond strength of two adhesives to enamel and dentin under normal and contaminated conditions. *Dental Materials* 1993; 9:295-9.
9. Grandy RK, Combe EC, Speidel TM. Shear bond strength of stainless steel orthodontic brackets with a moisture-sensitive primer. *Am J Orthod Dentofacial Orthop* 2001; 119:251-255.
10. Bishara S, Gordan V, Von Wald L, Olson ME. Effect of an acidic primer on shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop* 1998; 114: 243-247.
11. Miller J. Basic concepts concerning brackets failure research. *Angle Orthod* 1997; 3:167-168.
12. Pickett KL, Sadowsky PL, Jacobson A, Lacefield W. Orthodontic in vivo bond strength: comparison with in vitro results. *Angle Orthod* 2001; 141-148.
13. Reynolds IR. A review of orthodontic bonding. *Br J Orthod* 1975; 2:171-8.