

# Effect of moisture on dental enamel in the interaction of two orthodontic bonding systems

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**Objective:** The purpose of this study was to assess by means of scanning electron microscopy (SEM) the remaining adhesive interface after debonding orthodontic attachments bonded to bovine teeth with the use of hydrophilic and hydrophobic primers under different dental substrate moisture conditions.

**Material and Methods:** Twenty mandibular incisors were divided into four groups (n=5). In Group I, bracket bonding was performed with Transbond MIP hydrophilic primer and Transbond XT adhesive paste applied to moist substrate, and in Group II a bonding system comprising Transbond XT hydrophobic primer and adhesive paste was applied to moist substrate. Brackets were bonded to the specimens in Groups III and IV using the same adhesive systems, but on dry dental enamel. The images were qualitatively assessed by SEM.

**Results:** The absence of moisture in etched enamel enabled better interaction between bonding materials and the adamantine structure. The hydrophobic primer achieved the worst micromechanical interlocking results when applied to a moist dental structure, whereas the hydrophilic system proved versatile, yielding acceptable results in moist conditions and excellent interaction in the absence of contamination.

**Conclusion:** The authors assert that the best condition for the application of primers to dental enamel occurs in the absence of moisture.

**Keywords:** Dental primer. Scanning electron microscopy. Orthodontic brackets.

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## INTRODUCTION

Bonding procedures are based on histomorphological changes occurring in the enamel due to the action of surface treatments initially proposed by Buonocore in 1955,<sup>5</sup> who noted that the action of acid substances on the adamantine structure enables a micromechanical interaction between resinous materials and etched/conditioned dental enamel.

Subsequently, findings disclosed by Buonocore were used in Orthodontics (1970), producing undeniable clinical benefits.<sup>2,5</sup> This bonding mechanism remains in use to this day despite numerous changes undergone by orthodontic attachments as well as adhesive systems.

Such benefits led to some unfavorable clinical situations, namely: increased likelihood of injuries to dental enamel structures occurring after bracket removal, since this procedure may damage the enamel surface due to great bond strength.<sup>1-5</sup> Furthermore, resin remnants remain bonded to the enamel and may not only induce plaque accumulation, but also undermine esthetics.<sup>2,5</sup>

Therefore, when choosing a bonding system one should consider the type, concentration and etching/conditioning time, selecting the less deleterious systems,<sup>4,5</sup> thereby preserving clinical performance.

New bonding systems which blend into one single product both etching and primer materials, have been proposed. With the development of one-step self-etching systems,<sup>1-5</sup> an estimated reduction of 65% in the time required for bonding orthodontic brackets was achieved,<sup>1,2</sup> thus, decreasing the risk of surgical field contamination by oral fluids. Nevertheless, the effects of these bonding agents on tooth enamel have been questioned by lab and clinical studies.<sup>8-11</sup>

New hydrophilic bonding systems have been launched into the dental market. These products address substrate contamination without interfering in the orthodontic bonding process.<sup>6,18,23,25,28</sup>

The foregoing considerations emphasize the importance of shedding light on the true nature of the interaction between hydrophilic primers and dental enamel, and comparing it with the performance of a conventional bonding system under various substrate conditions.

## Objective

This research aims at assessing — by means of scanning electron microscopy (SEM) — the remain-

ing adhesive interface after debonding orthodontic attachments bonded to bovine teeth with the use of hydrophilic and hydrophobic primers under different dental substrate moisture conditions: a) Moisture interference in the systems used in this study; b) Advantages of the micromechanical interlocking system under different conditions; c) Systems that provide the best results.

## MATERIAL AND METHODS

The sample for this *in vitro* study consisted of 20 permanent bovine incisors<sup>18</sup> freshly extracted from three-year old heifers or steers confined in slaughterhouses. After cleaning the teeth, they were stored in aqueous 0.1% thymol until use.<sup>5-12,17,26</sup>

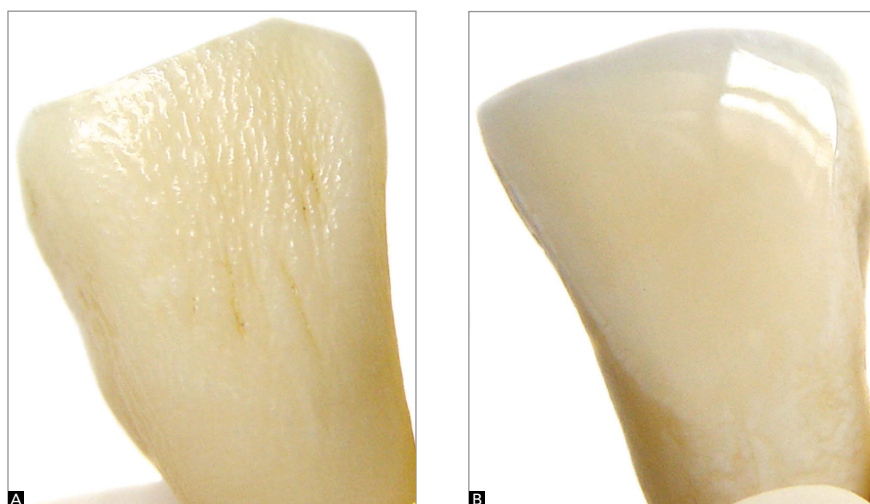
Cracks were detected in the enamel by transilluminating the buccal surface of the teeth<sup>12</sup> with the aid of a stereomicroscope with 1.2 x magnification. Only those teeth which were free from cracks and carious lesions on all surfaces, were selected.

In order to render the buccal surfaces more regular, flat and smooth, they were polished for 10 seconds with a rotary polisher (Buehler Ecomet 3) using silicon carbide discs (Buehler) at 150 rpm in decreasing grain size order (320, 600, 800, 1200).

Afterwards, the crowns were polished with felt discs (Buehler) Texmet 1000 soaked in 1  $\mu\text{m}$  diamond suspension, followed by 0.3  $\mu\text{m}$  suspension (Fig 1).

Subsequently, teeth were randomly divided into four groups according to bonding system and substrate condition (Table I). Pre-adjusted stainless steel maxillary central incisor brackets (Abzil.Lancer) with mesh bases (two-point welding) were bonded to all specimens.

**Group I:** Specimens had Transbond MIP adhesive system applied according to the manufacturer's recommendations, followed by prophylaxis of the enamel buccal surface carried out by means of pumice, water and a Robinson brush at low speed. In the following step, the enamel surface was etched with phosphoric acid at 37% for 15 seconds. The entire substrate was then washed with distilled water and excess was removed with absorbent paper, always preserving tissue moisture. Primer was then applied using the brush that comes with the product, followed by gentle jets of compressed air for about 5 seconds.



**Figure 1** - Bovine teeth before (A) and after (B) polishing the buccal crown surfaces.

**Table 1** - Experimental groups, orthodontic bonding material and substrate condition.

Group	n	Bonding material	Manufacturer	Batch	Substrate
I	5	Transbond MIP (Moisture Insensitive Primer)	3M/Unitek Dental Products	6KN 2008-10	Moist
II	5	Transbond XT	3M/Unitek Dental Products	6CW/6WB 2007-09	Moist
III	5	Transbond MIP (Moisture Insensitive Primer)	3M/Unitek Dental Products	6KN 2008-10	Dry
IV	5	Transbond XT	3M/Unitek Dental Products	6CW/6WB 2007-09	Dry

Transbond XT light cure adhesive was subsequently applied to the bracket bases under a pressure of 300g, measured with a tension gauge (Dontrix, ETM Corporation, Monrovia, CA),<sup>18</sup> to determine the minimum compression weight and ensure similar paste flow on all teeth. Excess was removed with the aid of an explorer #5 (Duflex SSWhite) accurately circumscribing the Orthodontic Bonding Area (OBA).

Soon afterwards, the materials were light cured for 10 seconds on the mesial and 10 seconds on the distal surface by means of an Ultralux (Dabi Atlante, Ribeirão Preto, Brazil) curing light of 500mW/cm<sup>2</sup> intensity. The specimens were then immediately debonded with debonding pliers/AEZ (Ormco Corp. Glendora, CA, USA). The pliers tips were positioned at the bracket/adhesive interface and brackets were pulled off (Fig 2).

**Group II:** A conventional bonding system was applied to the specimens in this group (Table 1), which underwent the following steps: prophylaxis, etching, substrate moistening, excess removal and light curing, performed in the same manner as described for

Group I, except that for the latter group bonding was performed with Transbond XT adhesive system associated with acrylic resin cement. The specimens were then immediately debonded using the technique previously described.

**Group III:** Brackets were bonded to these specimens by means of the Transbond MIP bonding system. The same steps described for Group I were performed, but after etching, washing and drying the tooth surface, the enamel remained completely dry.

**Group IV:** Brackets were bonded using the conventional bonding system (Transbond XT - 3M Unitek Dental Products) following the same procedures performed for Group II, but under the same substrate conditions adopted for Group III.

All brackets were bonded by a single operator to minimize random errors.

### Preparing the specimens for electron micrograph

In order to assess the adhesive interface, two teeth from each group were cut with a diamond disc

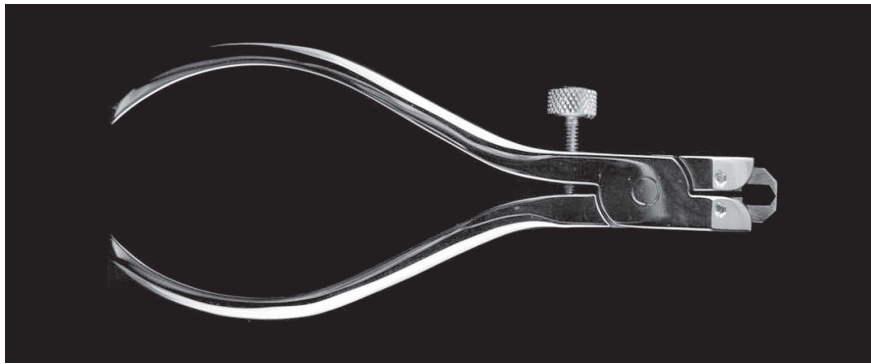


Figure 2 - Debonding plier.

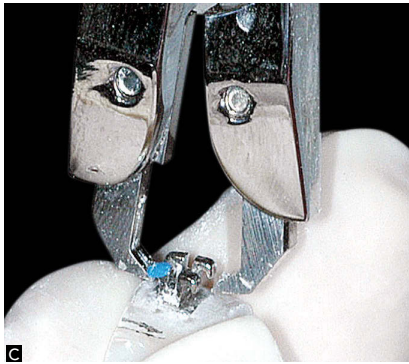


Figure 3 - A) Specimen cut in the orthodontic bonding area. B) Sectioning nearly as far as the buccal enamel. C) Debracketing using debonding pliers/AEZ, D) Specimen after debonding.

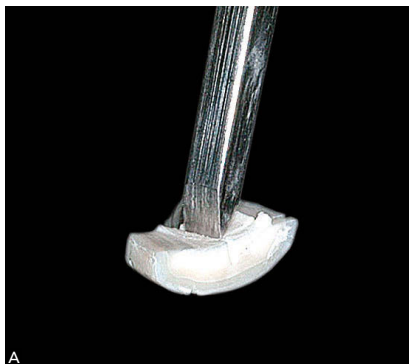


Figure 4 - A) Chisel in position to perform fracture. B) Specimen transversely fractured for SEM assessment.

(Buehler, Lake Bluff, IL, USA), yielding small dental fragments where the brackets had been previously bonded (Fig 3).

The area was subsequently fractured with a gouge chisel tip (Duflex SSWhite) to enable microscopic analysis of the adhesive interface (Fig 4). To highlight such an interface, etching was carried out with phosphoric acid for 5 seconds and rinsed with water for 20 seconds.

The fragments obtained from each specimen were placed on stubs, identified and metallized with gold alloy to render the surface electrically conductive (Fig 5).

### Microscopic analysis of the adhesive interface

The SEM device (JEOL JSM5600 LV, Japan) was set at 13KV voltage acceleration at a working distance of 30 mm. Images were obtained at 1300, 3000, 6000-times magnification. The adhesive interface underwent qualitative analysis and the photographed regions were representative of each specimen.

## RESULTS

The most representative regions of each specimen were evaluated, taking into account the adhesive interface (assessment at 1400 X) and the quality of the micromechanical interlocking (assessment at 1300 X, 3000 X and 6000 X magnification).

In Group I, the adhesive interface showed good micromechanical interlocking, developing tags (T) and few gaps (L), indicative of decalcified regions not fully filled with adhesive material, possibly due to the presence of water. In general, these characteristics indicate that Transbond MIP hydrophilic adhesive system had a satisfactory interaction with the enamel, even in the presence of moisture (Fig 6A).

This interaction was better assessed after removing the mineral content, assessing the micromechanical interlocking and the surface topography of adhesive remnants at 1300X, 3000X and 6000X magnification (Figs 6B, 6C and 6D).

Figure 6B reveals that there was homogeneous penetration of the adhesive system across the study area (Ad), showing that the presence of moisture did not prevent the adhesive from penetrating the etched area. Here, the presence of Gaps (L) in etched areas are more evident, which enables increased penetration of the adhesive material.

At higher magnifications (Figs 6C and 6D), it appears that there was in-depth micromechanical inter-

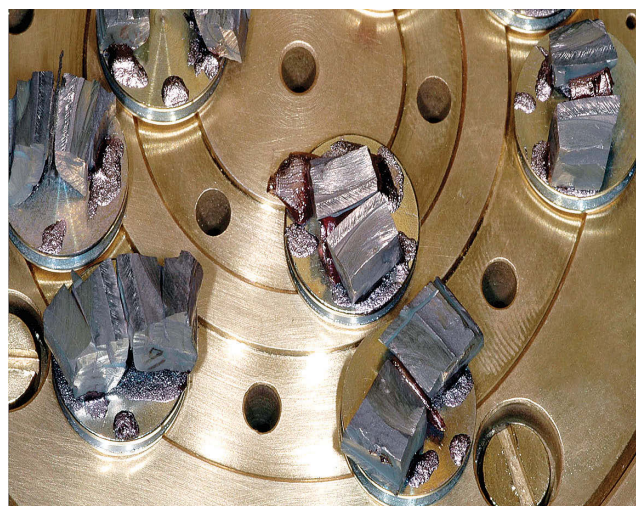


Figure 5 - Metallized specimens placed on stubs.

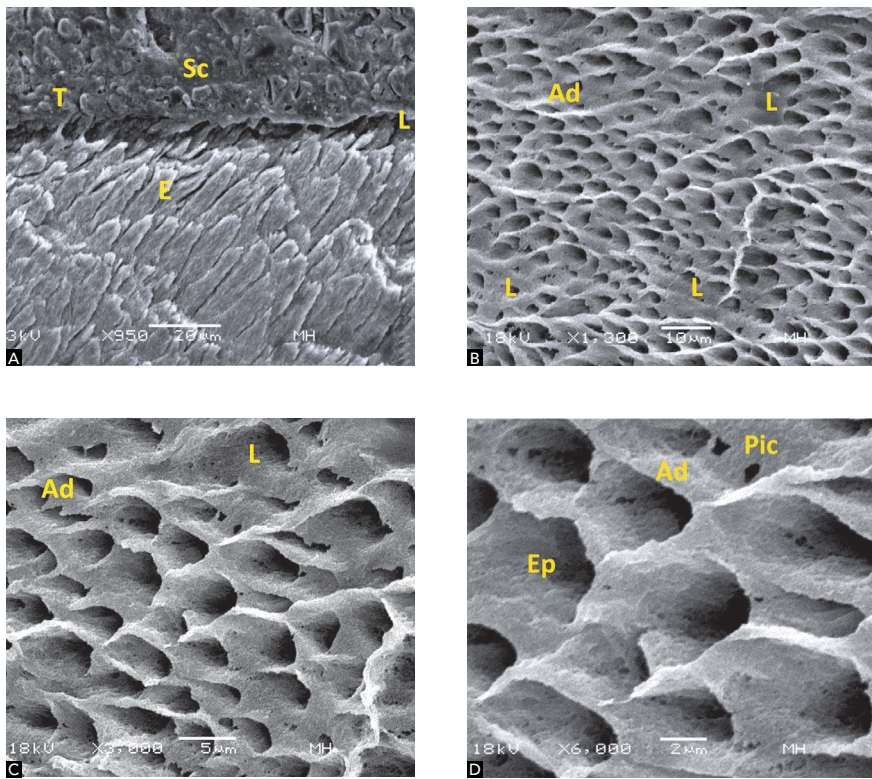
action in the enamel structure, preserving much of the mineralized prismatic area (Ep), although in some places there was partial dissolution of the rods generating nano interlocking areas with inter-crystallite adhesive penetration (Pic).

Group II showed (Fig 7A) the presence of clear, extensive and wide gaps (L) all over the bonding interface. These observations confirm that the presence of moisture, to some extent, insulated the substrate and did not allow satisfactory micromechanical interlocking to take place, since irregular and scarce tags (T) became visible in restricted and isolated areas.

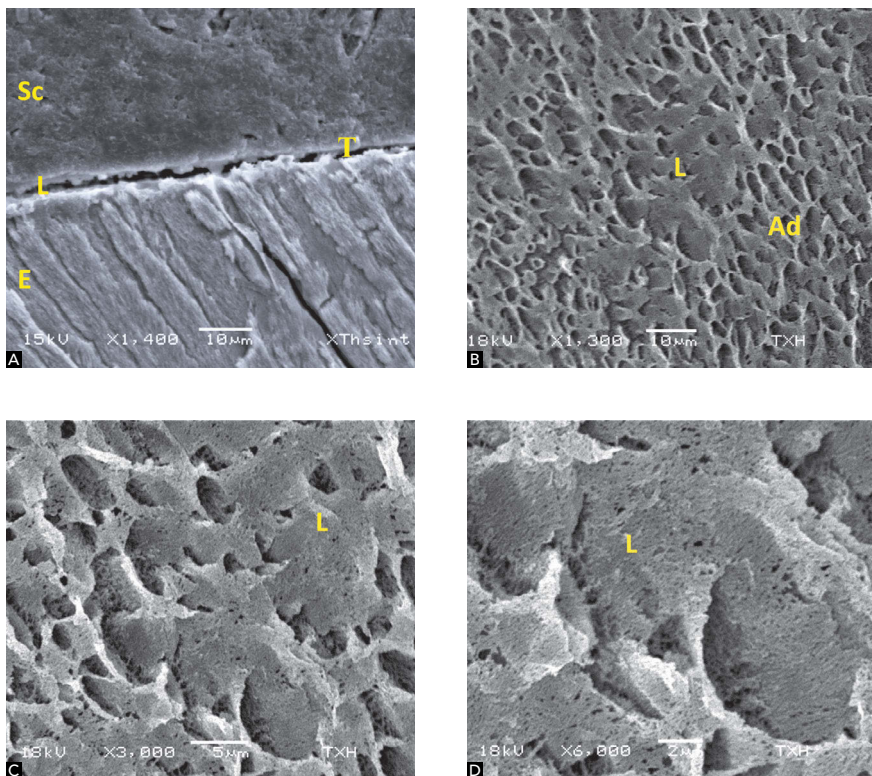
With the removal of the mineral content (Figs 7B, 7C and 7D), the bonding system (Sc) showed irregular topography and the presence of gaps (L). This means that water filled most of the area that had been demineralized by the phosphoric acid, which not only prevented the inter prismatic spaces from being filled up, but also hindered inter crystallite penetration by the adhesive system.

As for Groups III and IV, (teeth treated with Transbond MIP hydrophilic primer and hydrophobic Transbond XT Primer (Sc), respectively) the enamel/adhesive interface (Figs 8A and 9A) showed good micromechanical interaction with the enamel (E), absence of cracks and the development of numerous and continuous tags (T). These characteristics demonstrate that the application of phosphoric acid followed by rinsing and drying of the surgical field, produces excellent adhesive interfaces regardless of the primer used for bonding.

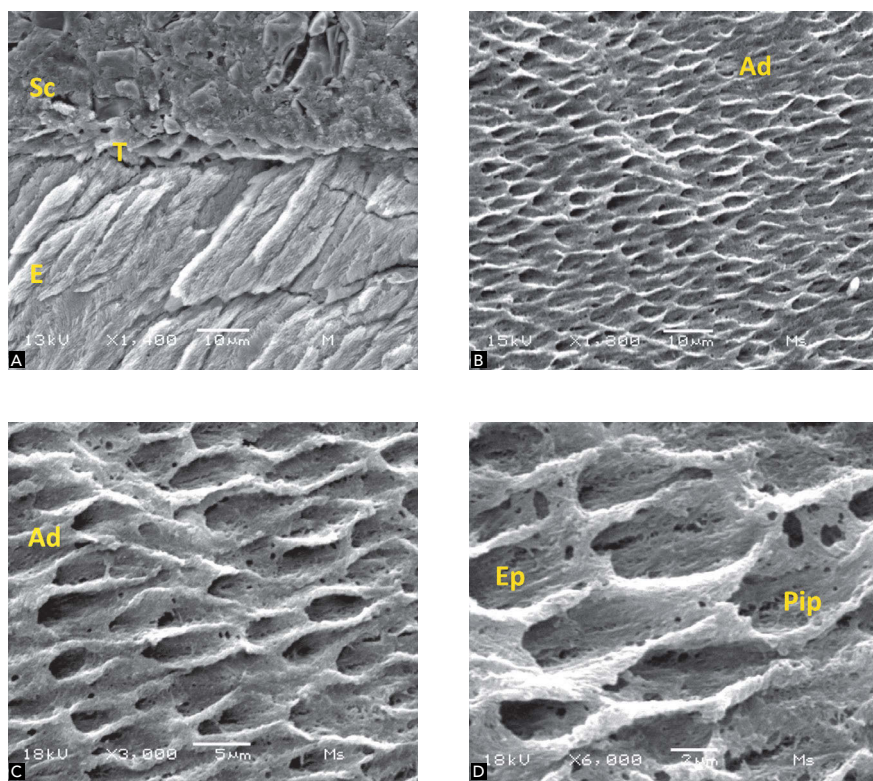
At larger amplitudes and after removal of the mineral content (Figs 8B and 9B), it became apparent that



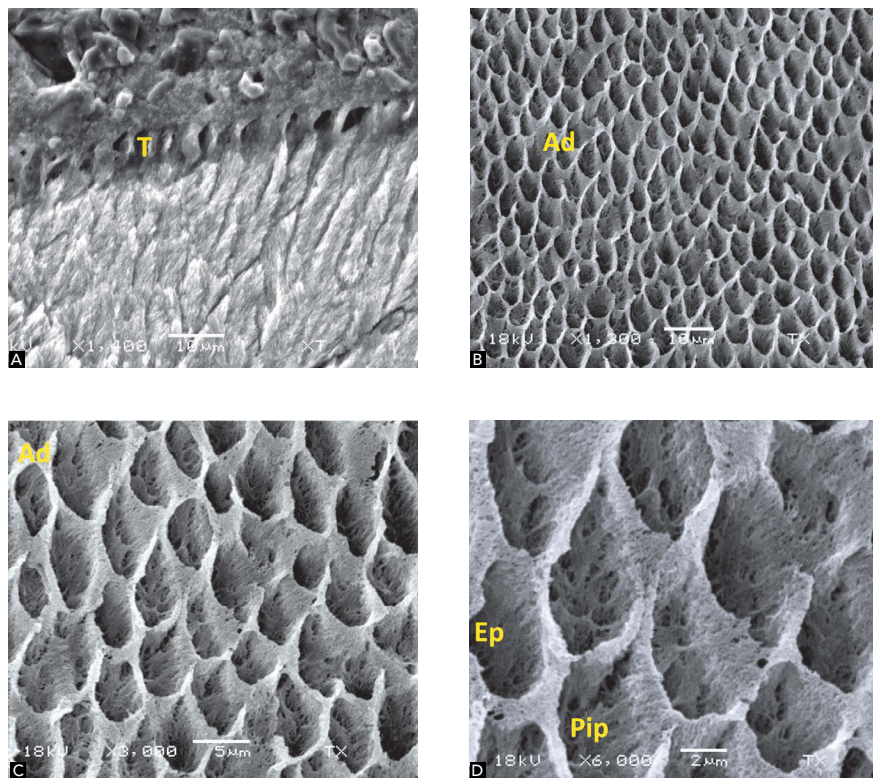
**Figure 6** - Electron micrographs: Moist MIP and enamel bonding interface. **A)** Relationship between the material and the enamel surface (950X) showing irregularities in the interaction between primer/enamel, areas with gaps (L) and presence of tags (T). **B, C and D)** Irregular primer surface after enamel dissolution. Images at 1300X, 3000X and 6000X.



**Figure 7** - Electron micrographs: Adhesive interface and interlocking achieved by the Transbond XT bonding material in moist conditions. T-Tags, Sc-Bonding system, L-Gaps, E-Enamel, Ad- Primer. Images at 1400X, 1300X, 3000X and 6000X magnification.



**Figure 8** - Electron micrographs: Bonding interface and interlocking achieved with Transbond MIP in the absence of moisture. T-Tags, Sc-Bonding system, E-Enamel, Ad-Primer, Ep-Space occupied by the rods, Pip-Inter-crystallite penetration. Images at 1400X, 1300X, 3000X and 6000X magnification.



**Figure 9** - Electron micrographs: Bonding interface and interlocking achieved with Transbond XT bonding material in the absence of moisture. T-Tags, Sc-Bonding system, E-Enamel, Ad-Primer, Ep-Space occupied by the rods, Pip-Inter-crystallite penetration. Images at 1400X, 1300X, 3000X and 6000X magnification.

interlocking was consistent in the interprismatic spaces without the presence of gaps, which had been previously occupied by water. This fact indicates that the primer (Ad) penetrated into all previously etched areas.

Figures 8C, 8D, 9C and 9D show excellent penetration of both primers. Likewise, the spaces occupied by the rods (Ep) are clear, with the primer also penetrating the inter-crystallite spaces (Pip), thus, highlighting excellent bond strength.

## DISCUSSION

Hydrophilic adhesive systems are used in restorative dentistry to ensure interaction with the dentin tissue, which is inherently moist. In this structure, etching — in association with these systems — forms a hybrid adhesive layer and dentin tags, i.e., structures that play an essential role in successful direct and indirect restorations.

During bracket bonding processes, enamel surface contamination, by blood, saliva or oral cavity moisture, is a major concern. Therefore, moisture insensitive primers are also suitable for bonding orthodontic attachments.

This research analyzed the adhesive interface of hydrophilic and hydrophobic bonding materials under different substrate conditions using scanning electron microscopy (SEM). This method provides good image resolution, allowing detailed observation of the substrate and the interfaces produced by adhesive systems.<sup>2,4,5,8,16,18,19,23,25,28</sup>

The results demonstrate that the specimens in Group I developed adequate micromechanical interlocking between adhesive system and enamel, forming resin tags in the interprismatic spaces, although cracks and/or gaps were also observed in some regions. This performance, even in the presence of moisture, is possibly related to the composition of the primer.

The outcome of this study may explain the findings of Cacciafesta et al,<sup>18</sup> who achieved the highest bond strength in contaminated enamel using hydrophilic systems. This study emphasizes the limitations of using hydrophobic systems in contamination cases in which the substrate had been previously etched.

No adhesion relation was observed in specimens of Group II, which showed low interaction between enamel and primer, allowing the formation of gaps in the adhesive/enamel interface and a lack of tags

which, even when present, were restricted to small areas. These observations are also justified by the composition of the primer, which contains no carrier or hydrophilic resin monomers. Thus, once the etched area is moist, a reliable bonding interface is unlikely to develop if moisture control is not absolute. Webster et al<sup>30</sup> compared the effectiveness of these materials in moist conditions, and concluded that when hydrophobic primers are used in moist conditions they provide low bond strength.

The results yielded in Groups III and IV showed that, in the absence of moisture, all bonding systems used in this investigation displayed micromechanical interaction with the enamel structure and the development of numerous, continuous, gap-free tags. Undoubtedly, successful bonding could only be achieved through the demineralizing action of phosphoric acid, which removed the aprismatic layer and exposed the spaces between the enamel rods. When these spaces were present on a surface with high surface energy and no contamination, they were easily filled by the primers, which also exhibited low viscosity. The findings of this study corroborate those of Cal-Neto et al,<sup>19</sup> who used SEM to assess the behavior of conventional hydrophobic primers in dry conditions, noting that there was adequate bonding between adhesive and enamel with the development of long, uniform tags and adequate micromechanical interlocking.

Data from this study associated with those obtained from the literature allow the authors to assert that the best condition for the application of primers to dental enamel occurs in the absence of moisture, regardless of bonding system features. However, the use of moisture insensitive (hydrophilic) systems is recommended, given the clinical impossibility of ensuring that the application areas are 100% dry.

## CONCLUSIONS

This study led the authors to conclude that: (a) the absence of moisture in etched enamel enabled better interaction between bonding materials and the adamantine structure; (b) the hydrophobic primer achieved the worst micromechanical interlocking results when applied to a moist dental structure; and (c) the hydrophilic system proved versatile, accomplishing acceptable results in moist conditions and excellent interaction in the absence of contamination.

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