Evaluation of the Bonding Mechanism of an Adhesive Material to Primary Teeth

Gaia Gotti, DDS Cecilia Goracci, DDS, MS
Franklin García-Godoy, DDS, MS Marco Ferrari, MD, DDS, PhD

ABSTRACT

Purpose: The aim of this study was to evaluate with scanning electron microscopy (SEM) the bonding mechanism of the one-bottle adhesive Prime&Bond NT (Dentsply, PBNT) to enamel and dentin of deciduous teeth, following different methods of substrate treatment.
Methods: Eighteen extracted posterior deciduous teeth were randomly divided into 6 groups (N=3). The experimental groups differed for substrate and method of substrate conditioning prior to bonding with PBNT. Group 1: 36% phosphoric acid (PA)/PBNT on dentin; group 2: PA/PBNT on enamel; group 3: non-rinsing conditioner (NRC) (Dentsply)/PBNT on dentin; group 4: NRC/PBNT on enamel; group 5: PBNT on dentin without any previous conditioning; group 6: PBNT on enamel without any previous conditioning. On all the specimens, following the application of the adhesive solution, Dyract AP was layered on top and light-cured. The bonded specimens were processed for SEM observations.
Results: When used in combination with 36% phosphoric acid, PBNT was able to form a hybrid layer with resin tags on both enamel and dentin. Following conditioning with NRC, a thinner hybrid layer with shorter resin tags was developed on dentin; on enamel an etching pattern was still detectable. When only PBNT was applied without any previous conditioning, on dentin neither hybrid layer nor resin tags were visible; no sign of micromechanical bonding could be seen on the untreated enamel.
Conclusions: The bonding mechanism of the one-bottle adhesive Prime&Bond NT on enamel and dentin of deciduous teeth is effective only following substrate conditioning with 36% phosphoric acid or NRC. (*J Dent Child*. 2004;71:54-60)

KEYWORDS: DECIDUOUS ENAMEL, DECIDUOUS DENTIN, BONDING, SIMPLIFIED ADHESIVIES, SCANNING ELECTRON MICROSCOPY

In recent years, the field of adhesive dentistry has concentrated on developing simplified systems that also allow for a reduced chair time. Self-etching primers, for instance, affect both the conditioning and priming action in a single step. One-bottle systems, on the other hand, combine the priming and bonding action into one solution, which has to be applied after etching the substrate with phosphoric acid.[1]

Current research standards require that the new materials proposed for use as dental adhesives be evaluated through microscopic observations, bond strength tests, and leakage tests.

Miss Gotti is an undergraduate student and Miss Goracci is a PhD student, Department of Dental Materials, Dr. Ferrari is professor, College of Dental Medicine, University of Siena, Italy; Dr. García-Godoy is professor, Bioscience Research Center, College of Dental Medicine, Nova Southeastern University, Fort Lauderdale, Fla.
Correspond with Dr. Ferrari at ferrarimar@unisi.it

Scanning electron microscope (SEM) observations have clearly shown that the bonding mechanism of adhesive systems to enamel and dentin is substantially of a micromechanical nature. Researchers agree that the development of a hybrid layer at the interface between resin and dental substrate, as well as the presence of resin tags with adhesive lateral branches, are typical microscopic aspects of an effective bond.

In one study,[2] Prime&Bond NT (DeTrey/Dentsply, Konstanz, Germany) was evaluated through SEM observations in its ability to create a micromechanically retentive lock to enamel and dentin of permanent teeth. Prime&Bond NT can be used in 3 different ways:

1. combination with phosphoric acid;
2. combination with a nonrinsing acid conditioner (NRC, Dentsply DeTrey, Konstanz, Germany);
3. without any conditioner.

The manufacturer's suggestion is to apply a (NRC) when using a compomer restorative material. Phosphoric acid should instead be preferred for substrate conditioning when utilizing resin-based composites. In the same study, Prime&Bond NT was tested in each of the 3 described ways on enamel and dentin samples. The results of this investigation suggested that when adhesive was directly applied on the dental surface without any previous etching, the degree of demineralization was unsatisfactory and apparently not retentive enough.

At the dentin site, a distinct hybrid layer could not be detected. Very few resin tags were present, suggesting that the tubules had not been completely cleared from smear plugs. A gap between dentin and resin was visible in several areas along the interface. Such microscopic features led the authors to conclude that Prime&Bond NT alone was not successful in preparing the dental substrate for an effective micromechanical bond with resin. A previous study with Prime&Bond,[3] an earlier version of the adhesive, also showed that when phosphoric acid was used, dentin bond strength increased significantly.

The simplification of the clinical procedure allowed by the use of a self-etching primer, such as Prime&Bond NT, would be especially useful when working on young, possibly uncooperative patients. The adhesion of Prime&Bond NT to enamel and dentin of primary teeth has been measured with shear[4] and tensile[5] bond strength tests, as well as with the microtensile method.[6] However, in order to completely evaluate the quality of bonding of an adhesive material, the results of bond strength tests should be complimented with the findings of microscopic observations. Therefore, the purpose of the present study was to evaluate by means of SEM the ability of Prime&Bond NT to produce a micromechanically retentive lock, through the formation of a hybrid layer and resin tags with adhesive lateral branches, when the adhesive system was used on enamel and dentin of primary teeth, with or without previous conditioning of the dental substrate.

METHODS

Eighteen human posterior primary teeth extracted for orthodontic reasons were selected for the study. The teeth were free from caries and any previous restoration. Each tooth was randomly assigned to 1 of 6 groups.

In groups 2, 4, and 6, a flat enamel surface was prepared on the occlusal surface of the tooth with a diamond bur mounted on a high-speed handpiece under abundant water spray. In groups 1, 3, and 5, a flat dentin surface was obtained by sectioning the teeth parallel to their long axes, with a low-speed diamond saw (Buehler Ltd, Lake Bluff, Ill).

SPECIMEN PREPARATION

GROUPS 1 AND 2

The "total etch technique" was followed using 36% phosphoric acid (De Trey Etch, Dentsply De Trey, Konstanz, Germany, batch No. 9801001217) for 20 seconds on the enamel as well as on the dentin samples. After the application of the acid gel, the substrate was washed with a water spray for 15 to 20 seconds. Then, to maintain the substrate moist, as suggested by Kanca,[7] the tooth was gently dried for 1 to 2 seconds with an air syringe from a distance of 2 cm. A layer of the adhesive Prime&Bond NT (Dentsply De Trey, Konstanz, Germany, batch No. KP2-172-1) was applied and left on the surface for 30 seconds. The solvent was removed by blowing it with an air syringe, and the residual resin layer was light cured for 20 seconds with a Visilux 2 (3M ESPE, St. Paul, Minn) light source (intensity >500 mW/cm^2). A layer of Dyract AP (Dentsply De Trey, Konstanz, Germany, batch No. 97070000413), a polyacid-modified resin composite, approximately 1 mm thick was applied and light-cured for 20 seconds.

GROUPS 3 AND 4

The substrates were treated with NRC (Dentsply DeTrey, Konstanz, Germany). The conditioner was applied for 30 seconds. As rinsing is not required with this material, NRC was simply dried with the air syringe for 1 to 2 seconds. Then, Prime&Bond NT was applied and left in place for 20 seconds. After blowing away the solvent and curing the resin for 20 seconds, a layer of Dyract AP was applied on top and light-cured for 20 seconds.

GROUPS 5 AND 6

The substrates were not treated with any conditioner. Prime&Bond NT was directly applied on the prepared surface, as previously described. Then, Dyract AP was placed over the adhesive.

The specimens were stored for 3 days in distilled water at room temperature (22°C) before being processed for microscopic observations. Each tooth was split-fractured along its long axis and through the center at the surface.

One section of the specimen was gently decalcified with 36% silica-free phosphoric acid for 10 seconds and deproteinized with a 2% sodium hypochlorite solution for 120 seconds at the interface between resin and dentin. The other section was kept for 2 days in a 30% hydrochloric acid solution to completely dissolve the dental tissues, thus obtaining resin replicas. Then, all of the sections and replicas were mounted on aluminum stubs, sputter-coated with gold

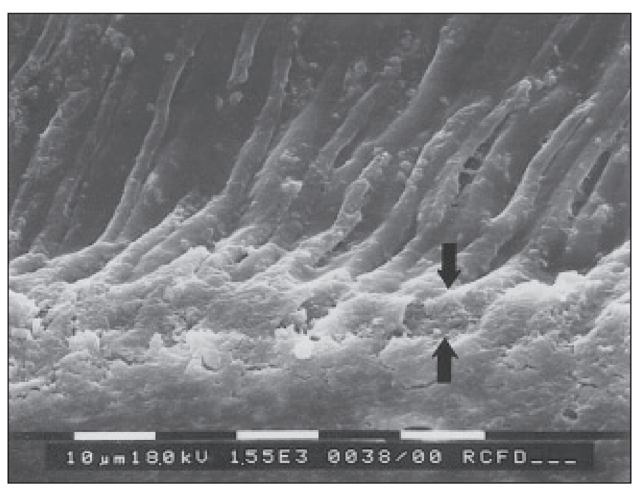

Figure 1. The hybrid layer created by Prime&Bond NT on phosphoric acid etched dentin (group 1). The thickness of the hybrid layer (between arrows) was 7- to 8-μm (×1,550).

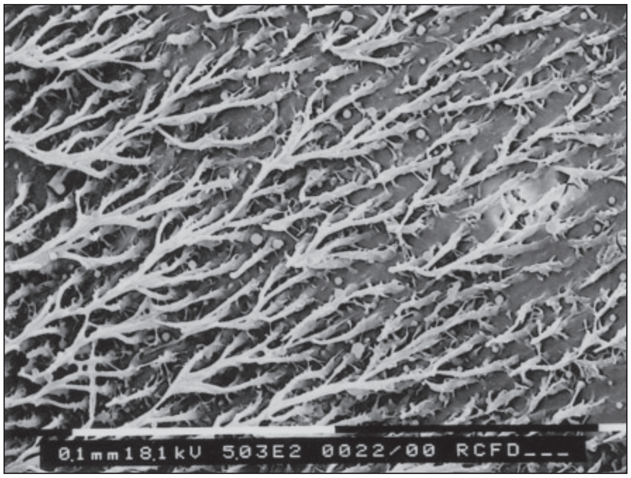

Figure 2. In dentin samples treated with phosphoric acid (group 1), resin tags were uniformly distributed and deeply penetrating (×500).

Figure 3. High magnification of Figure 2, showing the reverse-cone shape of the resin tags and presence of adhesive lateral branches (×3,500).

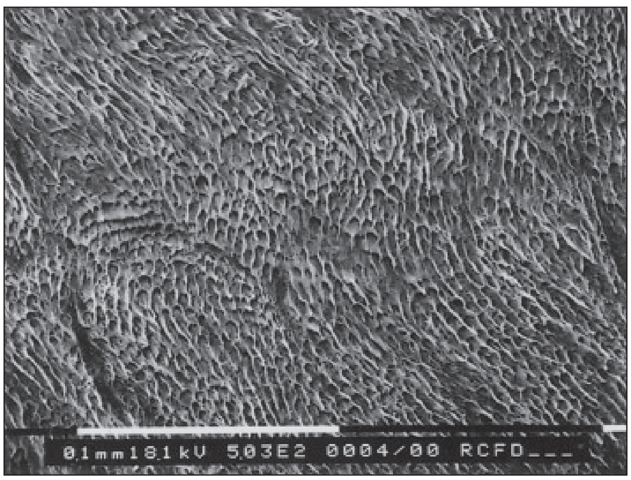

Figure 4. Etching pattern after phosphoric acid treatment of the enamel (group 2). The surface is uniformly and deeply demineralized (×500).

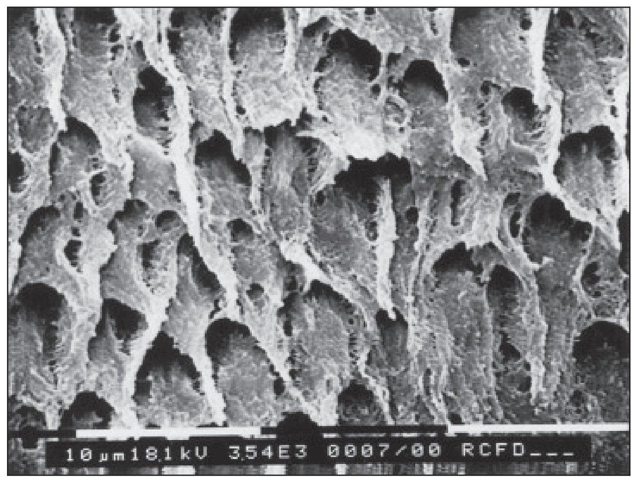

Figure 5. Figure 4 at a higher magnification (×3,500).

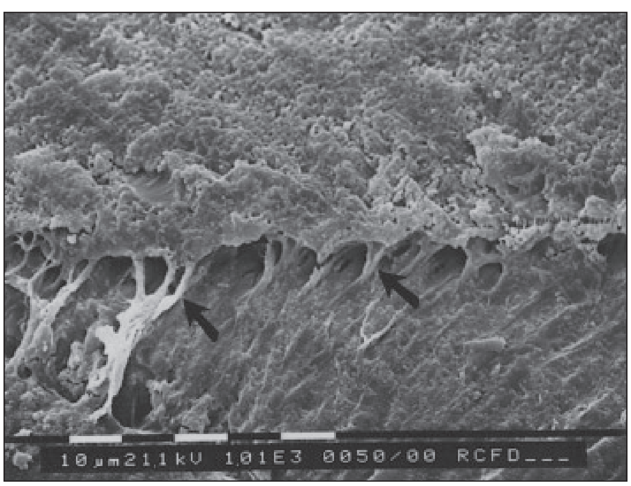

Figure 6. Resin tags formation can be noted at the interface between etched enamel and resin (group 2; ×1,010).

Figure 7. The hybrid layer (between arrows) created at the interface between resin and dentin conditioned with NRC was only few microns thick (group 3; ×2,000).

using the Edward's Coater S150B (London, England), and observed under a Philips 515 SEM (Philips, Eindhoven, The Netherlands). Micrographs were taken for every specimen at the interface between resin and dental substrate to observe the differences in hybrid layer morphology (magnification ×1,000-2,000). Micrographs of the resin replicas were taken

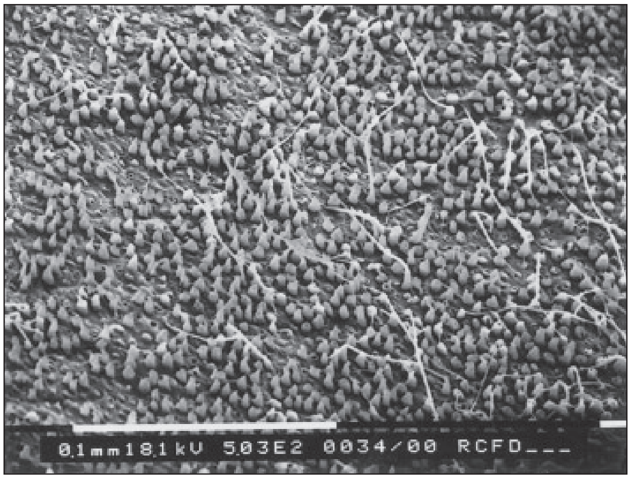

Figure 8. A resin replica observed at a low magnification shows the scattered distribution of relatively short resin tags on the dentin surface conditioned with NRC (×500).

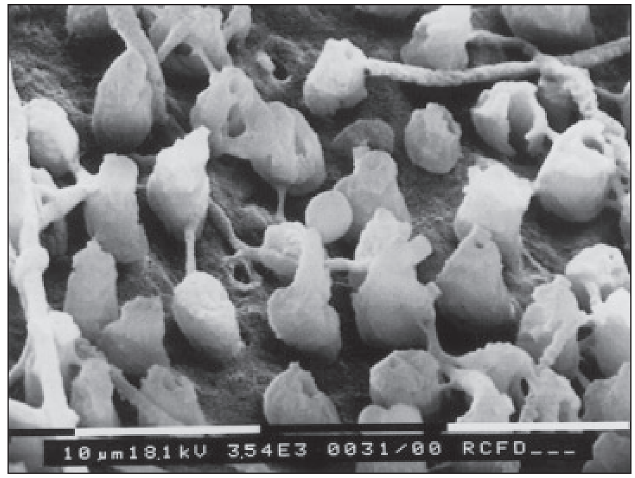

Figure 9. Higher magnification of Figure 8. Resin tags exhibited a reverse-cone shape (×3,500).

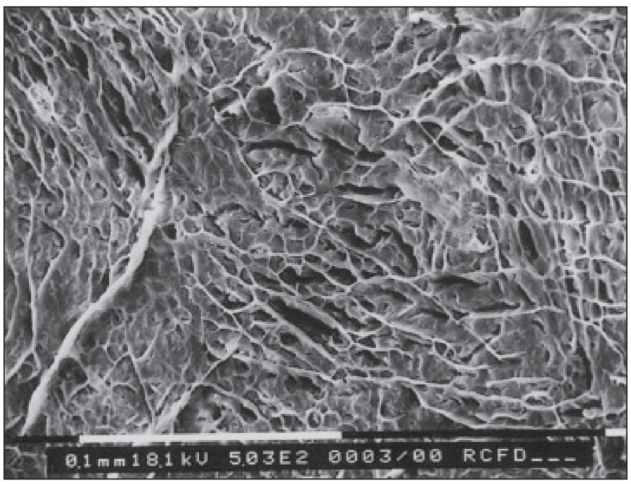

Figure 10. Resin replica of the etching pattern produced by NRC (group 4). Enamel demineralization is less deep than that produced by phosphoric acid (×500).

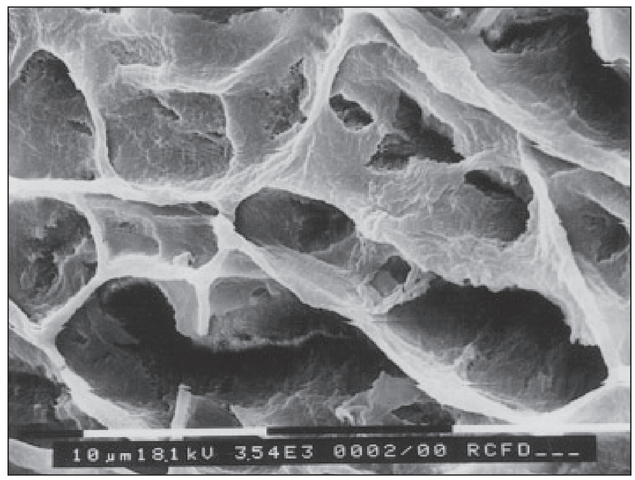

Figure 11. Figure 10 at a higher magnification (×3,500).

at 2 magnifications (×500 and ×3,500) in the center of the specimen for enamel samples and in an area close to the pulp in dentin samples.

RESULTS

On the dentin samples etched with 36% phosphoric acid (group 1), the demineralized surface was impregnated by the resin, giving rise to a 7- to 8-µm thick hybrid layer and to resin tags penetrating into the dentinal tubules to a depth of 10- to 30-µm. The area of resin infiltrating the etched peritubular dentin exhibited a rough texture (Figure 1). On the replicas, resin tags appeared numerous and uniformly distributed. Several adhesive lateral branches were clearly visible at a low (Figure 2, ×500) and high magnification (Figure 3, ×3,500). The latter observations also revealed that resin tags had a typical reverse-cone shape (Figure 3).

On the enamel samples (group 2), phosphoric acid effectively demineralized the substrate (Figure 4), creating surface irregularities into which the resin could flow (Figure 5).

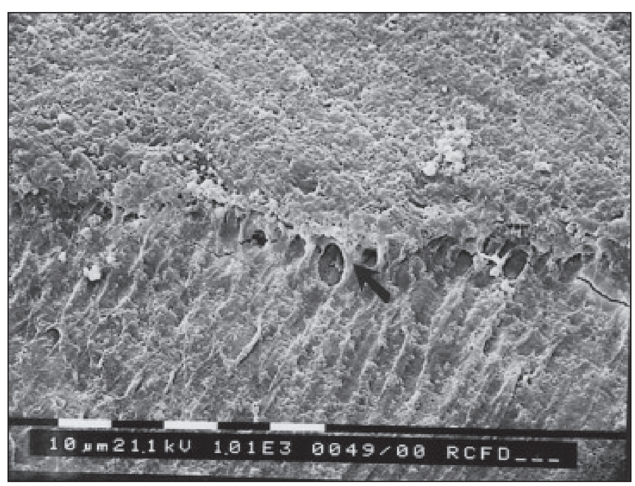

Figure 12. Resin tags (arrow) are visible at the interface between resin and enamel treated with NRC (group 4; ×1,010).

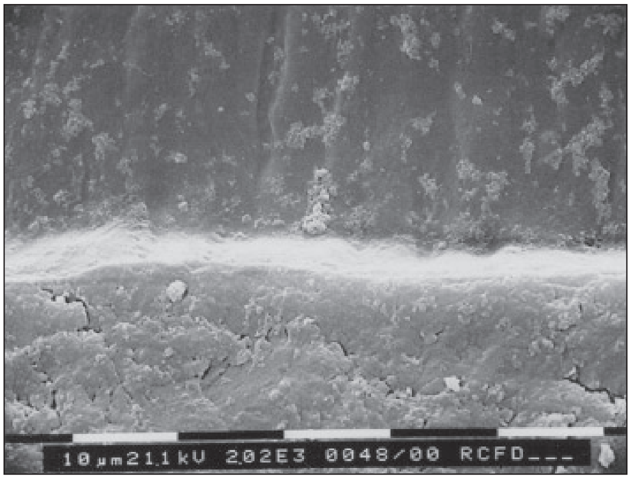

Figure 13. Interface between unconditioned dentin and Prime&Bond NT (group 5). Resin tags and hybrid layer are not evident (×2,000).

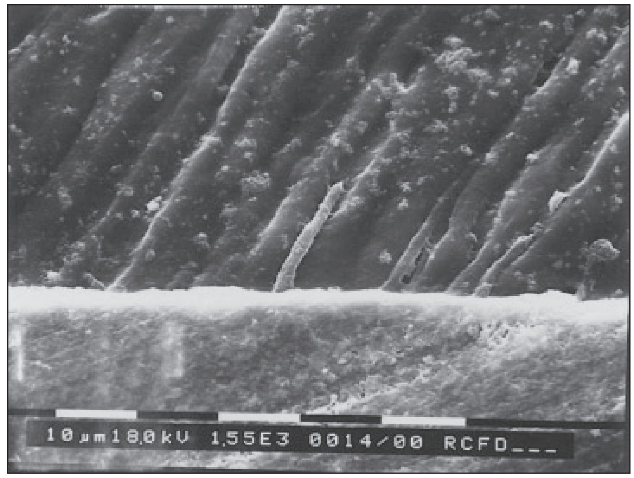

Figure 14. On the specimens of group 5 resin tags, when present, appeared narrow and smooth (×1,500).

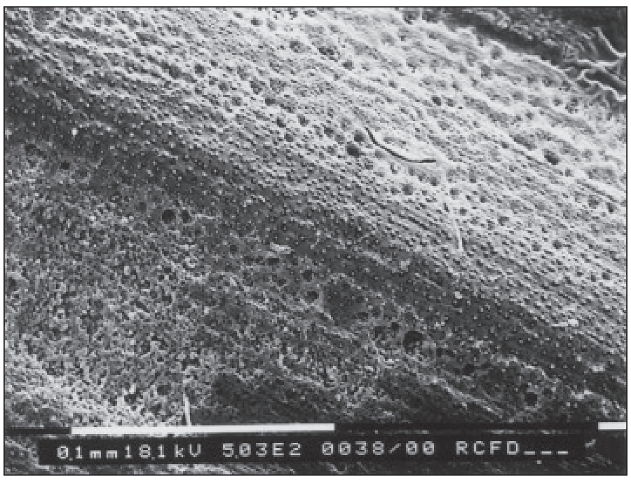

Figure 15. Resin replica of dentin on which Prime&Bond NT had been applied without any previous conditioning (group 5). Only few areas showed resin tags formation (×500).

Figure 16. Figure 15 at a higher magnification. In some areas of the resin replicas, the tubular orifices were closed by globules (×3,500).

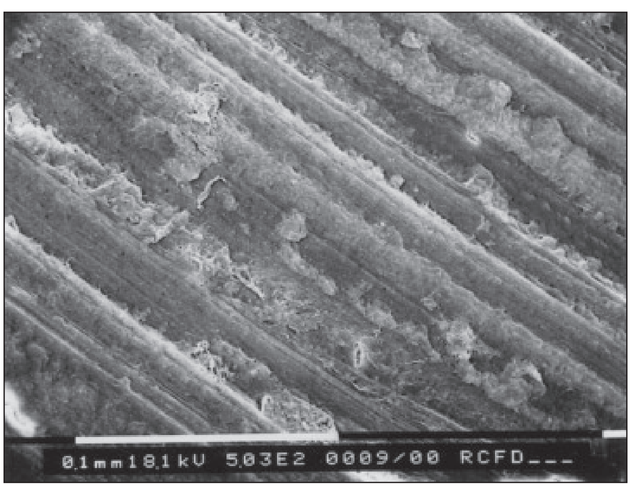

Figure 17. Resin replica of enamel on which Prime&Bond NT had been applied without any previous conditioning (group 6). No etching pattern was visible (×500).

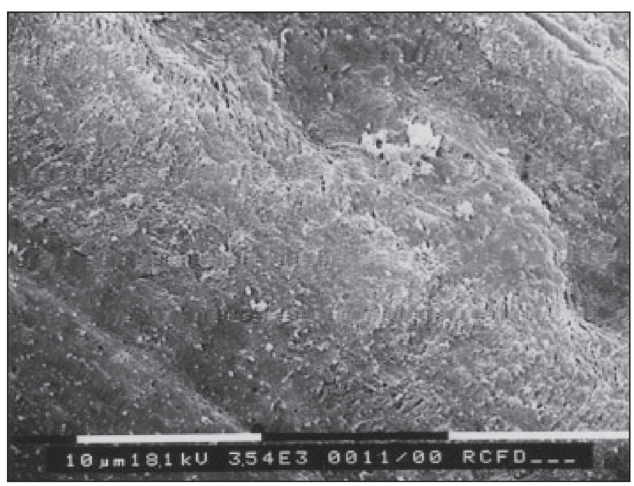

Figure 18. Figure 17 at a higher magnification. No micromechanical retention was produced by the action of Prime&Bond NT alone (×3,500).

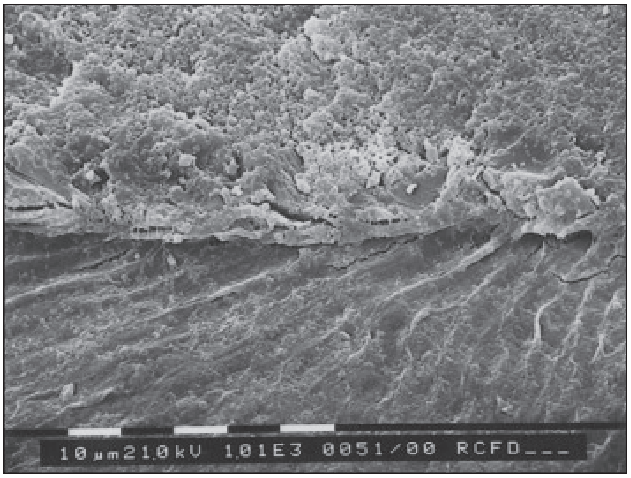

Figure 19. The interface between unconditioned enamel and resin. No mechanical interlock was created between the 2 substrates (×1,010).

Micrographs taken at the interface documented the penetration of resin tags into the demineralized enamel layer, ensuring a mechanical interlock between the 2 substrates (Figure 6).

On the dentin samples on which Prime&Bond NT had been applied in combination with NRC (group 3), a hybrid layer was formed. However, the layer of demineralized dentin infiltrated by resin was less thick than on samples treated with phosphoric acid (group 1), and in the range of few μm (Figure 7). The observation of resin replicas revealed that resin tags with adhesive lateral branches were developed in some areas, but they were more scattered, shorter, and thinner than in group 1 (Figures 8 and 9).

The etching pattern created by NRC on enamel samples (group 4) is shown in Figures 10 and 11. It appears less uniform and retentive than that produced with phosphoric acid. A limited penetration of resin tags into the demineralized enamel layer was revealed by micrographs taken at the interface (Figure 12).

On the dentin samples on which Prime&Bond NT had been applied without any previous conditioning of the substrate (group 3), a distinct hybrid layer could not be detected at the interface. A gap was indeed present between resin and dentin along the greatest part of the interface (Figure 13). Only few resin tags developed, and they appeared narrow and smooth, suggesting poor adhesion into the tubules (Figure 14). No adhesive lateral branches were detectable. As the resin replicas showed, the formation of globules, rather than tags, at the tubular orifices was a common finding, indicating that the tubules were still obstructed by smear plugs (Figures 15 and 16).

The enamel surface on which Prime&Bond NT had been directly applied did not exhibit the typical morphology of etched enamel (Figures 17 and 18). No resin tags were seen to penetrate into the enamel layer at the interface (Figure 19).

DISCUSSION

In the preparation of dentin samples, discs can be cut at different levels of depth into the thickness of the tissue to expose superficial or deep dentin. In the present study, it was decided to test the adhesive system on an area of dentin close to the pulp, about 1 mm above it. In this area, the density of tubules is high and they have a larger diameter, thus favoring resin penetration. This situation can be considered ideal and possibly more reproducible for adhesion testing.

Enamel samples were prepared on the occlusal surface of posterior primary teeth. In this area, the direction of enamel prisms is usually perpendicular to the surface. For this reason, enamel can be etched favorably and with a predictable result.

Sample preparation at the adhesive interface for the microscopic observation of the hybrid layer can be done with or without polishing. Polishing would yield a smooth and uniform surface to be observed. However, that would require a specific device and some extra time. Different acids, such as phosphoric acid or hydrochloric acid, an Argon ion beam, and/or a 2% sodium hypochlorite solution, could be applied at the interface for different times.[1,2] In this study, it was decided to follow the most common as well as the easiest procedure. The interface was etched with 37% silica-free phosphoric acid for 10 seconds, without any previous polishing. Then, a 2% sodium hypochlorite solution was applied for 120 seconds to deproteinize the demineralized structures, thus exposing hybrid layer and resin tags.

The morphologic features of the hybrid layer and resin tags are likely to be different at different distances from the pulp, cementum-dentin junction, and enamel-dentin junction. For this reason, it is advisable to use the same sample to evaluate the hybrid layer at the interface and resin tags on the replica. In the present study, each specimen was split into halves, and one of them was used to evaluate the hybrid layer. On the other half, resin tags could be observed as replicas after complete demineralization of the dental structures. The method of replicas is very useful to evaluate resin tags and adhesive lateral branches.[9,11] A great deal of information can be collected if the same area of a resin replica is observed at different magnifications. At a low magnification, it is possible to assess the density of resin tags, their length, and the increase in surface area as a result of conditioning. At high magnifications, the shape and surface roughness of resin tags is revealed.

When used in combination with 36% phosphoric acid, Prime&Bond NT was able to effectively bond to enamel and dentin of primary teeth. The microscopic evidence for this claim was provided by the ability of the system to form a hybrid layer, resin tags, and adhesive lateral branches.

When Prime&Bond NT was applied in combination with a NRC, the hybrid layer at the interface between resin and dentin appeared thinner than in the samples on which phosphoric acid had been used. In addition, the density of resin tags was found to be lower than in the samples treated with phosphoric acid. These mophological aspects are probably due to the less aggressive action of NRC.

Regarding the effects on enamel, the present study showed that, when the NRC was applied, or when no conditioning of the primary enamel was performed prior to the application of the bonding agent, the demineralization was shallower and the surface less retentive, as compared with primary enamel etched with phosphoric acid. These findings are in agreement with the results of the aforementioned study, in which Prime&Bond NT had been tested following the same protocol on permanent teeth.[1]

CONCLUSIONS

This study has proved at a microscopic level the ability of Prime&Bond NT to effectively bond to enamel and dentin of primary teeth, provided that the adhesive is applied after etching the substrate with phosphoric acid. The evidence that a less reliable adhesion is obtained when the substrates are treated with a NRC or are not conditioned at all was also provided by this investigation. The microscopic features of the bond produced by Prime&Bond NT on primary teeth were similar to those observed when the same system had been used on permanent teeth. Also, the morhological aspects of the adhesion created by Prime&Bond NT in combination with phosphoric acid were similar to those obtained with 3-step enamel-dentin bonding systems. As compared with the latter materials, Prime&Bond NT would offer the advantage of an easier handling and reduction in chair time. This property should be particularly useful when working on pediatric patients.

The present study suggests that, from a microscopic standpoint, Prime&Bond NT is a reliable adhesive on primary teeth. However, for a more complete assessment of the performance of the material on primary teeth, the findings of this microscopic investigation should be complimented by the results of bond strength tests and clinical evaluations.

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