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Effect of the Bonding Technique on the Morphology of Dentin/Adhesive Interfaces Bonded with Various Adhesive Systems. SEM Investigation*

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PALMA-DIBB, R.G.; ESPER, H.R.; RAMOS, R.P. Efeito da técnica adesiva na morfologia da interface adesivo/dentina de vários sistemas adesivos. Estudo em MEV. **Rev. Odontol. UNESP,** v. 33, n. 1, p. 33-40, jan./mar. 2004.

Resumo: Este estudo analisou, em MEV, o efeito da técnica de adesão na morfologia das interfaces adesivas de cinco sistemas *total-etch* (Scotchbond MP, Single Bond, Optibond FL, Stae, Unibond) e um *self-etching* (Etch&Prime 3.1). As técnicas *úmida* e *seca* foram comparadas. Dezoito discos de dentina foram preparados, *smear layer* padronizada foi produzida e os discos divididos ao meio. Os seguintes protocolos de secagem foram realizados: num hemi-disco, a dentina foi mantida úmida; no outro foi seca. Os sistemas adesivos foram aplicados e uma camada da resina Z250 foi acomodada na superfície dentinária e polimerizada. Os espécimes foram seccionados, polidos e preparados para MEV. As interfaces resina-dentina foram analisadas quanto à formação de camada híbrida, enfocado suas características e qualidade. O sistema *self-etching* não foi capaz de produzir uma camada híbrida consistente, independente da técnica de adesão. As camada híbridas produzidas pelos adesivos *total-etch*, foram mais espessas e homogêneas quando a técnica úmida foi realizada.

Palavras-chave: *Adesivos; colagem dentária; microscopia eletrônica de varredura.*

Abstract: This study analyzed, by SEM, the effect of bonding technique on the morphology of dentin/adhesive interfaces of five total-etch (Scotchbond MP, Single Bond, Optibond FL, Stae and Unibond) and one self-etching system (Etch&Prime 3.1). *Moist* and *dry* bonding techniques were compared. Eighteen dentin disks were prepared, standardized smear layer was produced and the disks were bisected. The following drying protocols were accomplished: on one half, dentin was kept moist; on the other, dentin was dried. The adhesive systems were applied and a layer of Z250 resin was placed on dentin surface and light-cured. Specimens were sectioned, smoothened and prepared for SEM. The resin-dentin interfaces were analyzed as to the formation of a hybrid layer, focusing on its characteristics and quality. The self-etching system did not produce a consistent hybrid layer, regardless of the bonding technique. The hybrid layers produced by the total-etch adhesives, were always thicker and more homogeneous when the *moist* technique was performed.

Keywords: *Adhesives; dental bonding; scanning electron microscopy.*

Introduction

Adhesion of restorative materials to tooth structure has been a subject of great interest for dental research since Buonocore[2] proposed the acid etching of enamel in an attempt to optimize the adhesion of restorative materials to tooth substrate. Recent advances in the chemistry of adhesive systems have improved the short-term bond strength, mainly in dentin[20]. For most conventional bonding systems, the preparation of dentin substrate prior to the placement of resin composites is a multi-stage process that integrates the application of an acidic conditioner, a priming agent and adhesive[10].

Current dentin adhesives employ two different means

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to achieve micromechanical retention between resin and dentin substrate. The first method removes the smear layer completely and demineralizes the subsurface of intact dentin *via* acid-etching using a strong inorganic acid[19], thereby promoting an increase in the permeability and decalcification of both inter and peritubular dentin[3]. These systems utilize hydrophilic monomers, which, upon polymerization into the interstitial spaces between collagen fibers, create a resin-dentin inter-diffusion zone or hybrid layer, which is believed to be the main source of bonding to acid-etched dentin[11,12]. Additionally, penetration of the monomers deep into the dentinal tubules, results in the formation of resin tags thereby improving bond strength. These systems are available in two distinct presentations. The first consists of complete systems indicated for various purposes, with a three-step protocol comprising etching with an acidic conditioner, priming with a hydrophilic resin in a solvent and bonding with an unfilled or lightly-filled resin. The other consists of simplified, one-bottle adhesive systems that incorporate the primer and the bonding resin in a single container[1].

The second method uses the smear layer as bonding substrate. A self-etching primer, rather than an acidic etchant, is applied to the smear layer-covered dentin without further rinsing and the adhesive resin is further applied to the treated dentin. In these systems, the smear layer is substituted, modified or incorporated in the hybrid layer[19]. The advantage of such adhesives is to provide simultaneous demineralization of tooth structure and impregnation of the etched area by hydrophilic monomers, thus preventing the existence of extensive demineralized areas not impregnated by the resin monomers[11,13].

It has been widely reported that the drying of dentin substrate is a critical step in the adhesive protocol, which may substantially influence the morphology of the hybrid layer. Excessive drying of dentin can cause the collapse of collagen fibers, a decrease in permeability and hence lead to lower penetration of the adhesive in the interstitial spaces between the fibers[4,6,15,21]. On the other hand, a moist dentin surface is reported to prevent the collapse of the collagen-rich demineralized dentin after etching, thus improving the penetration of hydrophilic monomers[23].

In view of the great variability in formulation, required pretreatment and acting mechanism of the commercially available bonding agents, it is of extreme interest for adhesive dentistry to investigate whether the integrity of the adhesive interface may be compromised by the drying technique accomplished on the dentin substrate. Therefore the aim of this study was to assess, by means of scanning electron microscopy, the effect of *moist* and *dry* bonding techniques on the morphology of dentin/adhesive interfaces bonded with several one- and multiple-step total-etch systems and one self-etching primer adhesive, as regards the formation of a hybrid layer, focusing on its characteristics, quality and integrity.

Material and method

Eighteen extracted caries-free, non-restored human third molars stored in a 0.5% chloramine solution at 4 °C for up to one month after extraction were used in this study. Teeth were cleaned with scaler and water/pumice slurry and the occlusal overlying enamel layer was removed. Then, teeth were individually fixed in a sectioning machine (Minitom, Struers A/S, Copenhagen, DK-2610, Denmark) with a water-cooled diamond saw positioned parallel to the occlusal surface and a 1 mm thick (± 0.1 mm) disk was obtained from middle dentin (n = 18). The bottom surface and borders of each disk was carefully coated with two layers of a cosmetic nail varnish.

The disks were randomly assigned to six groups of equal size (n = 3), according to the adhesive system used: I - Scotchbond MP; II - Single Bond; III - Etch & Prime 3.1; IV - Optibond FL; V - Stae; VI - Unibond (UB). The tested materials with their compositions, specifications and manufacturers are displayed in Table 1.

Afterwards, the disks were bisected and each half was manually ground under water cooling using #120- to #400-grit silicon carbide paper to provide a flattened, smooth dentin surface. Complementary grounding was performed with #600-grit SiC paper for 60 seconds to produce standardized smear layers[17].

Each half of a same disk was treated according to one of the following drying protocols: *moist* - after acid-etching, the dentin surface was gently dried with absorbent paper to remove excess water and keep tooth surface moist; *dry* - drying of etched dentin was performed by a mild oil-free air-stream for 5 sec at a 10 cm distance from dentin surface.

The adhesive systems were applied strictly following manufacturers' instructions. For SBMP, SB, OB, ST and UB, dentin was etched with 35-37% phosphoric acid gel (Gel Etchant, Kerr Corporation, Orange CA, 92667 USA) for 15 seconds, rinsed thoroughly for 20 seconds with an air/water spray and dried by the above described techniques. The adhesive systems were applied to the etched dentin surface and light-cured with a visible-light curing unit with a 450 mW/cm^2 output (XL 3000, 3M/ESPE, St Paul MN 55144, USA). For EP self-etching primer adhesive system, dentin surface was thoroughly rinsed after grinding and then dried according to either the *moist* or the *dry* technique. Then, drops of catalyst and universal were mixed and applied with a light scrubbing motion for 30 seconds, slightly air-thinned for 5 seconds and light-cured for 20 seconds. After the bonding protocols were completed, a 1 mm thick layer of a hybrid light-activated composite resin (Z250, 3M/ESPE, St Paul, MN, 55144, USA) was placed on dentin surface and light-cured for 40 seconds.

Table 1. Tested adhesive systems

Adhesive system	Composition	Batch number	Manufacturer
Scotchbond MP (SBMP) (3 steps)	Primer: HEMA, polyalkenoic acid, water; Adhesive: HEMA, Bis-GMA	Primer: 6LB Adhesive: 7HT	3M/ESPE St Paul MN 55144 USA
Single Bond (SB) (2 steps)	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, ethanol and water	9DK	3M/ESPE St Paul MN 55144 USA
Etch&Prime 3.1 (EP) (1 step)	Catalyst: pyro-phosphate, HEMA; Universal: HEMA, ethanol, distilled water	Catalyst: 059811 Universal: 059810	Degussa S.A., Guarulhos, SP, 07042, Brazil
Optibond FL (OB) (3 steps)	*Primer:* HEMA, GPDM mono (2-methacryloxy ethyl phthalate, ethanol, water; *Adhesive:* BIS-GMA, HEMA, filler	Primer: 25881 Adhesive: 25882	Kerr Corp., Orange CA 92667, USA
Stae (ST) (2 steps)	Acetone; Acrylic monomer; Butylated hydroxy toluene; Sodium fluoride; water	0322	Southern Dental Industries, Bayswater 3153, Australia
Unibond (UB) (2 steps)	Methacrylic acid ester, tertiary amine aliphatic polyacrylate, acetone	05510059	Vigodent S/A Rio de Janeiro, RJ, 21041, Brazil

Then, the specimens were serially sectioned at a 90° angle to the bonding interface, thereby providing three fragments *per* specimen. The interfaces to be analyzed were manually smoothened with #1000- to #4000-grit SiC paper to obtain even and smooth surfaces. The dentin/adhesive interface was etched with a 37% phosphoric acid gel for 5 seconds, rinsed and the samples were ultrasonicated for 10 minutes, thoroughly washed with distilled water and immediately immersed in 2.5% glutaraldehyde in 0.1 M cacodylate buffer, pH 7.4, for 12 hours at 4 °C. After fixing, the samples were washed with cacodylate buffer several times, sequentially dehydrated in an increasing alcohol solution series and then immersed in 100% hexamethyldisizilane (Electron Microscope Sciences, Washington, PA, USA) for 10 minutes and left drying in an exhaust system.

Specimens were mounted on stubs with their treated surfaces face up, using a double-faced carbon tape and sputter-coated with gold. The adhesive/dentin interfaces were throughout examined with a JSM 5410 scanning electron microscope (JEOL Ltd, Tokyo 190-0012, Japan,) operating at 15 kV and a standardized series of photomicrographs was taken only from the most representative areas. Two previously calibrated examiners performed the analysis of the interfaces, independently and a consensus was always reached between them. The adhesive interfaces were observed as regards the formation or not of a hybrid layer, focusing on its integrity, homogeneity and continuity along the interface, as well as on the arrangement, uniformity of size and characteristics of hybridization of resin tags. The morphological aspects were assessed, comparing both drying techniques and adhesive systems.

Result

Scanning electron microscopy revealed different interfacial morphology for the adhesive systems tested in the study.

For Etch&Prime 3.1 self-etching primer, it was observed the formation of not consistent, irregular hybrid layers, with various disruptions along the adhesive interface, regardless of the bonding technique. However, no tag formation was observed. In several specimens, no evidence of hybridization could be noticed (Figures 1 and 2).

All the total-etch adhesive systems produced a resin-dentin inter-diffusion zone, with penetration of resin monomers into the dentinal tubules, regardless of the bonding technique. However, it was evident that the hybrid layer formed was consistently thicker and more homogeneous when the *moist* bonding technique was performed after acid-etching.

The simplified systems (Single Bond, Stae and Unibond) produced thinner and less homogeneous hybrid layers, mostly with the *dry* bonding technique (Figures 3, 5 and 7). Single Bond formed a layer with the presence of uniformly-sized tags throughout its extension, mainly for the *moist* technique (Figure 4).

For Stae, a homogeneous hybrid layer and few resin tags were found with the *moist* technique (Figure 6). Unibond showed a great number of very shiny and profusely anastomosed resin tags at the base of the interface, and this characteristic was more evident with the *moist* bonding technique (Figure 8)

For Optibond FL, some of the dry-bonded specimens exhibited thin hybrid layers but no tag formation was noticed (Figure 9). Nevertheless, for the moist-bonded specimens, uniform, well-defined hybrid layers were observed (Figure 10).

For Scotchbond MP, the resin tags did not appear uniformly distributed when the dry technique accomplished (Figure 11). On the other hand, homogeneous hybrid layers and tags were noticed for the *moist* bonding technique (Figure 12).

Discussion

The findings of the reported study disclosed that, for all adhesive systems tested, the bonding technique influenced, to some extent, the formation of the hybrid layer. For the specimens in which the *moist* bonding technique was accomplished, morphologically more consistent and uniform hybrid layers were observed, also exhibiting good resin tags formation. A feasible explanation for such characteristic may be the fact that the *moist* bonding prevents the collapse of collagen fibers on the etched and demineralized surface of the dentin and provides a surface onto which the hydrophilic resins contained in the dentin bonding agents may become more easily entangled[9,16]. Moreover, the findings of a recent study[7] showed that the amount of resin impregnation within the hybrid layer formed with dry bonding was significantly lower (approximately 50%) than the one formed with wet bonding. The drying technique has also been claimed to affect marginal infiltration[14,16].

In addition to the analysis of the conditions of the dental substrate, it is of paramount importance to assess the type of solvent used in the primer or in the primer/adhesive, to determine its effect on hybrid layer formation. Adhesive systems with water-based primers should be preferably used with a dry substrate, since water is assumed to play an important role in re-hydrating the air-dried etched surface, thus allowing resin monomers to interdiffuse still efficiently[22]. Therefore, the water added to the adhesive system could re-hydrate collapsed collagen fibers and facilitate the infiltration of hydrophilic monomers[12]. Never-

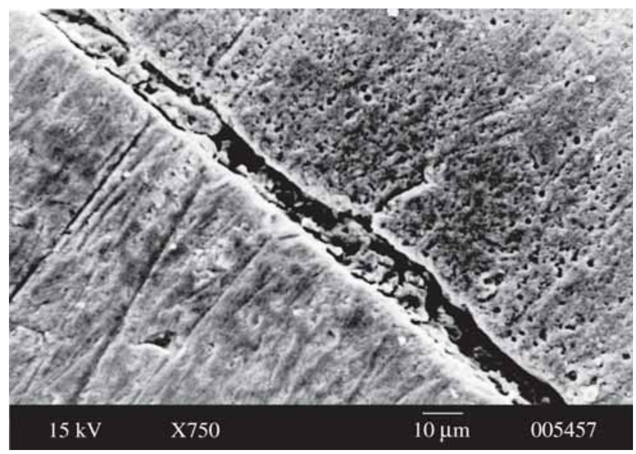

Figure 2. Scanning electron micrograph of dentin/adhesive interface. Etch&Prime 3.1 self-etching system + Moist bonding technique. Absence of hybrid layer and resin tags formation. (Original magnification X750).

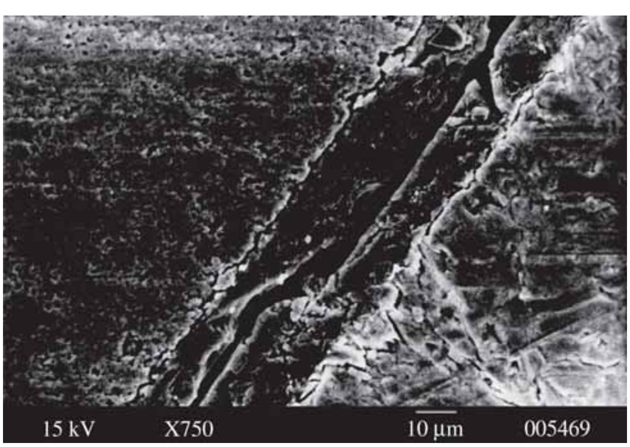

Figure1. Scanning electron micrograph of dentin/adhesive interface. Dry bonding technique + Etch&Prime 3.1 self-etching system. Absence of hybrid layer and resin tags formation. (Original magnification X750).

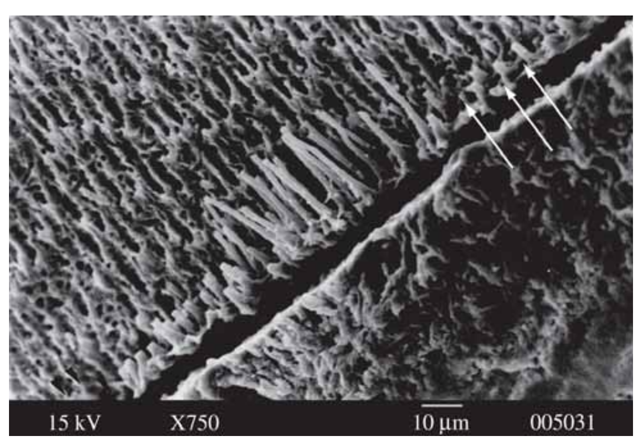

Figure 3. Scanning electron micrograph of dentin/adhesive interface. Dry bonding technique + Single Bond (Original magnification X750). Areas with lack of resin tags formation (arrows).

theless, it is widely accepted that solely the water contained in the primer would not be sufficient to completely re-hydrate the collagen network[21]. This assumption can be supported by the results reached in the present study. Single Bond, which is an ethanol- and water-based system, formed a more uniform hybrid layer with the *moist* bonding technique. Similar results were reached by Scotchbond MP. On the other hand, the acetone-based primers are more technique sensitive and require a moist dentin substrate[1,4,22]. Presumably, these systems are not able to re-hydrate a desiccated collapsed collagen layer to allow resin penetration and good hybrid layer formation[5]. The small amount of water available in these systems may not be sufficient to act as a re-hydrating agent[15]. Due to their relatively high volatility, solvents such as acetone and, to a lesser degree, ethanol, may displace surface moisture and serve as a better vehicle to carry the primer monomers into the microspaces of the exposed collagen network[9]. This was quite evident in this study, in which Unibond and Stae, both acetone-based primer/adhesive systems, produced hybrid layers with

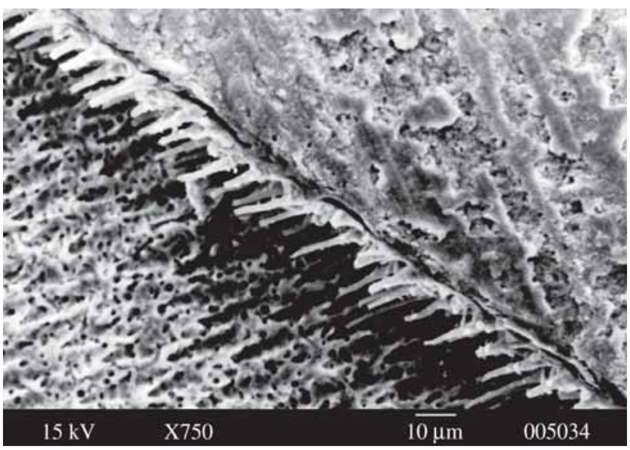

Figure 4. Scanning electron micrograph of dentin/adhesive interface. Moist bonding technique + Single Bond (Original magnification X750). Well-defined hybrid layer and uniformly-sized tags throughout its extension.

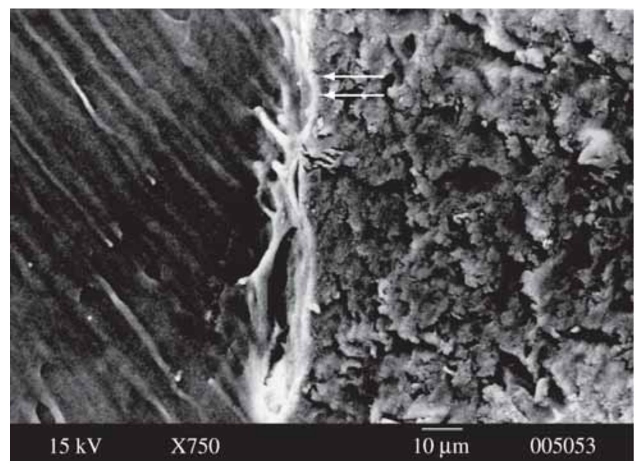

Figure 5. Scanning electron micrograph of dentin/adhesive interface. Dry bonding technique + Stae (Original magnification X750). Non-homogenous hybrid layer with variable thickness (arrows).

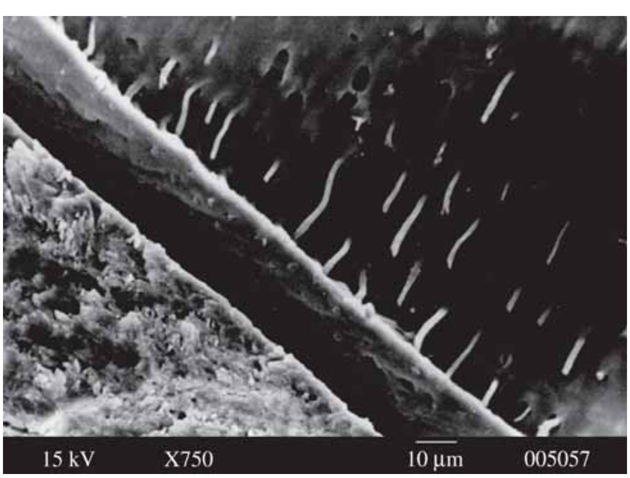

Figure 6. Scanning electron micrograph of dentin/adhesive interface. Moist bonding technique + Stae (Original magnification X750).

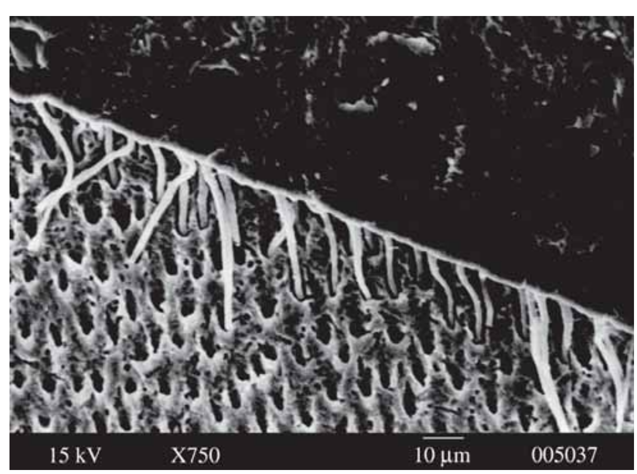

Figure 7. Scanning electron micrograph of dentin/adhesive interface. Dry bonding technique + Unibond (Original magnification X750). Well-defined hybrid layer with non-uniform formation resin tags.

good formation of tags in the *moist* technique.

With respect to the multiple-step adhesives (Scotchbond MP and OptiBond FL), the interfacial morphology was more homogeneous as compared to the other one-bottle and self-etching systems. This behavior may probably be ascribed to the fact that the one-bottle systems have a higher solvent-to-monomer ratio, and therefore there is always a possibility that, during the adhesive protocol, such adhesives are applied in a too thin layer. For those systems to achieve adequate bonding it is necessary that the solution be richly applied to facilitate the saturation of the exposed collagen fibril network and establish a satisfactorily thick resin layer on top of the hybrid layer. The glass filler added to Optibond FL may also help to provide a uniform resin film that stabilizes the hybrid layer. Nevertheless, it is mandatory that this agent be carefully applied, because a thickened adhesive layer may possibly affect esthetics.

Among the tested materials, Etch&Prime 3.1 self-etch-

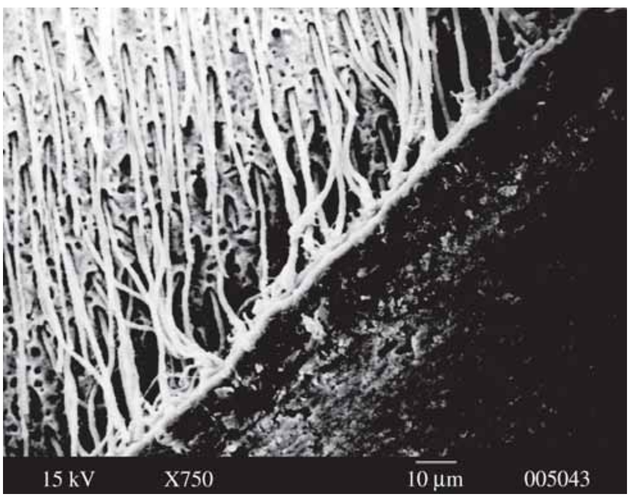

Figure 8. Scanning electron micrograph of dentin/adhesive interface. Moist bonding technique + Unibond (Original magnification X750). Great amount of very shiny and profusely anastomosed resin tags at base of the interface.

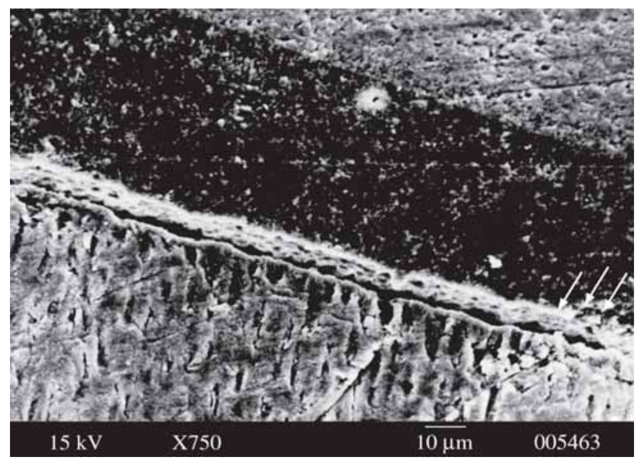

Figure 9. Scanning electron micrograph of dentin/adhesive interface. Dry bonding technique + Optibond FL (Original magnification X750). Areas with thin hybrid layer (arrows) but lack of resin tags.

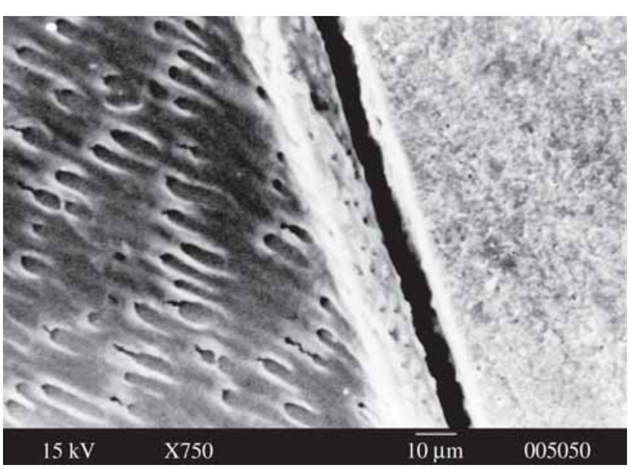

Figure 10. Scanning electron micrograph of dentin/adhesive interface. Moist bonding technique + Optibond FL (Original magnification X750). Well-defined hybrid layer without an evident formation of resin tags.

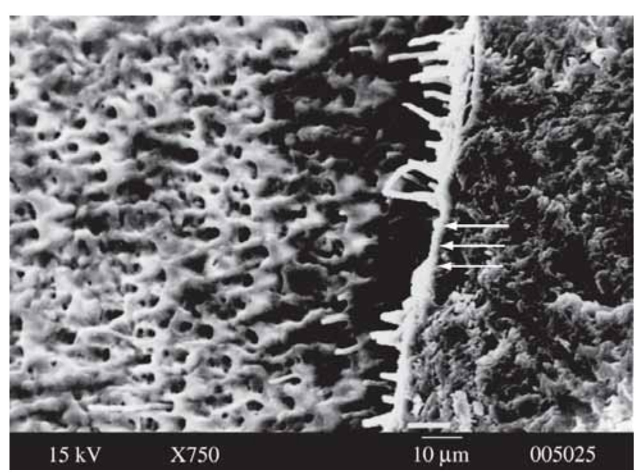

Figure 11. Scanning electron micrograph of dentin/adhesive interface. Dry bonding technique Scothbond MP (Original magnification X750). Areas with lack of resin tags formation (arrows).

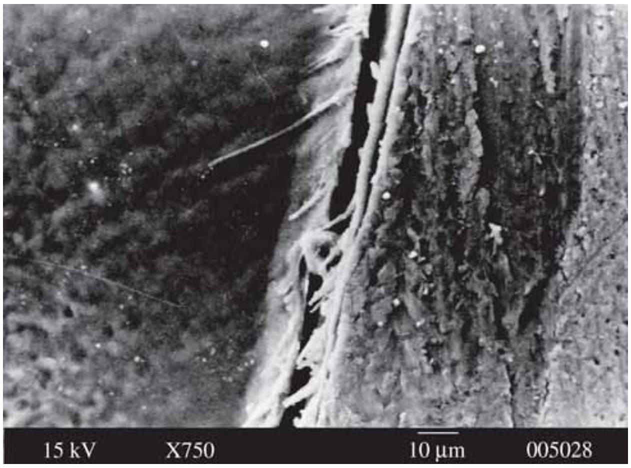

Figure 12. Scanning electron micrograph of dentin/adhesive interface. Moist bonding technique + Scothbond MP (Original magnification X750). Hybrid layer and uniformly-sized tags throughout its extension.

ing system was the less affected by the experimental conditions evaluated in the study. Even though, it was not able to yield the formation of adequate dentin hybridization. The bonding mechanism of EP relies on the application of a mild self-etching primer, which probably did not result in complete surface demineralization. In the present study, it was observed the formation of thin, ill-defined hybrid layers, with various disruptions along the adhesive interface, for both experimental conditions proposed. In several specimens, no consistent hybrid layer could be noticed.

The findings of the conducted research disclosed that, for all types of adhesives tested, it is crucial that the dentin substrate not be dehydrated after surface conditioning. Moist dentin allows an improved penetration of the hydrophilic monomers on etched dentin substrate, thus resulting in a more homogeneous hybrid layer and well-defined tags. However, it must be emphasized that the *moist* bonding can only warrant efficient resin interdiffusion if all the remaining water on dentin surface is completely eliminated and replaced by monomers during a subsequent priming step, since water can compete with resin for space inside the demineralized dentin[8]. It seems appropriate to highlight that, despite the notable and unquestionable advances in dental research, there is too much to be investigated with respect to the ultimate efficiency of bonding on dental substrate

Further investigation is certainly required to corroborate the findings of the conducted research as well as to establish the basis for rational assessment of adhesion to dentinal substrate focusing on enhancing the quality of resin-dentin interface thereby providing an optimized bonding protocol.

Conclusion

Based on the data obtained from this *in vitro* investigation, and within the limitations of an *in vitro* study, the following conclusions may be drawn:

- For all the total-etch adhesive systems, more homogeneous and well-defined hybrid layers were observed when the dentinal substrate remained moist after acid conditioning;
- The self-etching system did not yield the formation of consistent hybrid layers or resin tags, regardless of the bonding technique;
- The three-step adhesive systems produced thicker and more uniform hybrid layers than those obtained with one-bottle systems.

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