Evaluation of the Interfacial Morphology between a Single Component Adhesive and Dentin with or without Preliminary Acid Etching

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The purpose of this study was to analyze the interfacial morphology between dentin and a single-component dental adhesive system (Single Bond Universal, 3M ESPE), applied with or without preliminary phosphoric acid etching. In this study, 20 cavities were prepared in extracted teeth and restored with composite resin, using the tested adhesive. The axial sections were observed by scanning electron microscopy. When the adhesive was applied without etching, the hybrid layer was thin and not uniform. The resin tags could rarely be observed within the tubules. Preliminary acid etching of dentin resulted in thicker hybrid layers and augmented resin tags. However, for the tested adhesive, preliminary etching with phosphoric acid did not seem to significantly improve the quality of the dentin sealing. The EDX determination using the Line mode of the elements variation indicated a more severe drop of calcium in dentin when preliminary etching with phosphoric acid was applied comparing to the situations when the adhesive was applied in self-etching procedures.

Keywords: bonding system, phosphoric acid, hybrid layer, dentin, self-etch, total-etch

The adhesive systems have been developed in order to mediate the bonding between the hydrophilic dental tissues and the hydrophobic composite resins used for dental restorations. Simplified systems are available today, combining the hydrophilic primer and the hydrophobic resin in one single bottle. Depending on the underlying strategies, any contemporary adhesive can be classified as an “etch-and-rinse” or a “self-etch” system. Both systems create a hybrid layer consisting of partially-demineralized dentine impregnated with resin. Etch-and-rinse systems involve a preliminary stage of etching with phosphoric acid while the mono-component self-etching adhesives use acidic monomers to dissolve the smear layer, demineralize dentin and simultaneously impregnate it [1]. However the elimination of the acid etching, particularly for one-bottle systems, seems to adversely affect the quality of adhesion [2-7]. The attempts to combine the self-etching systems with phosphoric acid etching have produced conflicting results, with studies showing improved bond strength [8], no effect [9] or reduced bond strength [10].

The purpose of this study was to analyze the interfacial morphology for one of this simplified system, which is recommended by the manufacturers to be used with or without preliminary phosphoric acid etching.

Experimental part

The study group included 10 caries-free third molars, extracted for orthodontic reasons. Standardized class V cavities were prepared in both lingual and facial surfaces with the gingival margin bellow the enamel-cement junction. The cavities were cleaned with water and lightly air-dried using cotton pellets. For restorations we used a composite resin (Filtek Z550, 3MESPE) and the adhesive Single Bond Universal (3M ESPE) in a bulk-technique. For the buccal cavities, the adhesive was applied using a total-etch procedure. Phosphoric acid (Scotchbond Universal Etchant – 3M ESPE) was applied on both dentin and prepared enamel for 15s, than rinsed thoroughly with water and dried with cotton pellets. In lingual cavities no etching gel was applied. In all the cavities the adhesive was applied on the entire prepared surface and rubbed for 20s. Subsequently, a gentle stream of air was directed over the adhesive for about 5s, than it was polymerized using a curing LED light for 10s, according to manufacturer instructions. The cavities were filled with the composite resin and light-activated for 40s.

The prepared teeth were split in an axial lingual-buccal direction. The sections were observed by scanning electron microscopy using a VEGA II LMH (TESCAN) microscope. We have also used EDX detector, XFlash 6/10 model, for chemical determinations. Beside standard chemical composition determinations, using PB-ZAF data base on a selected area, the Esprit software of the equipment can determine the variations of selected chemical elements on a line.

Results and discussions

Most images obtained by scanning electron microscopy confirmed an acceptable adhesion to dentin, regardless of the chosen technique (with or without etching).

When the adhesive was applied without etching, the hybrid layer was thin with small adhesive failures in the interface (fig. 1a). The resin tags were very short and rarely could be observed within the tubules. In limited areas of several specimens, neither the hybrid layer nor the resin tags could be observed (fig. 1b). When gaps were observed between the composite resin and dentin, the failure seemed to be adhesive in most situations, with a very thin layer of adhesive preserved on the top of the dentine (fig.

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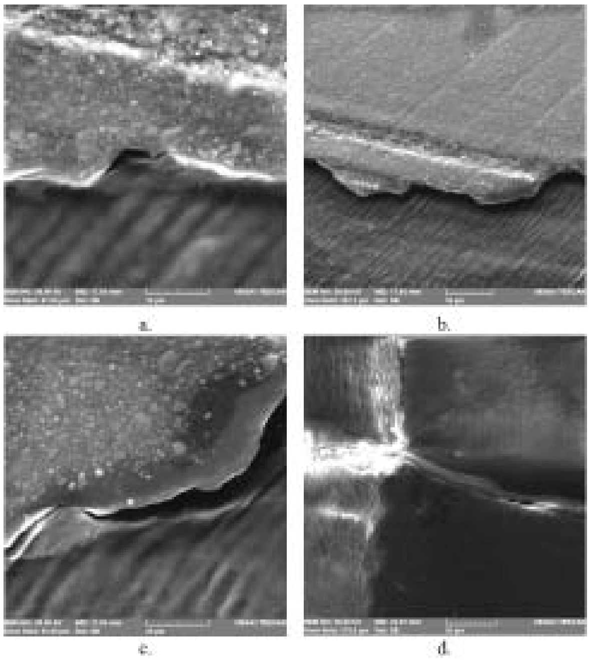

Fig. 1. SEM images for interfacial morphology of Single Bond Universal applied in self-etching procedure: a. 3000X, b. 1000X, c. 3000X, d. 1000X

1c). The gaps did not involve the margin of the restorations in any of the smecimens investigated (fig. 1d).

When preliminary acid etching was applied on dentin, the resin layer at the interface had an uneven thickness (fig. 2a). Failure was rarely noticed at the joint between the composite resin and the dentine.

The thickness of the hybrid layer appeared to be increased and consistent resin tags could be observed in the dental tubule (fig. 2b).

The dentine seemed to remain sealed with adhesive resin at the interface in most images. However the acid etching did not completely eliminate the risk for failures, several gaps being observed between the composite resin and dentine (fig. 2c). Cohesive failures seemed to involve both composite and adhesive. In most situations, the adhesive remnants seemed to seal the dentine surface (fig. 2d).

The EDX determination using the Line mode of the elements variation is more a qualitative analyze and less a quantitative one (fig.3).

However the charts indicated a more severe and deeper (up to 10 microns) drop of calcium in dentin when preliminary etching with phosphoric acid was applied (fig. 3a) comparing to the situations when the adhesive was applied in self-etching procedures (fig. 3b), while phosphorus seemed to be less influenced by the preliminary etching step.

These finding supports the concerns about the risk of dentin over-etching when systems use preliminary acid-etching procedures.

The bonding failure may be the result of either adhesive failure between the composite resin and the dental structure or cohesive failure of one of them. Of all the elements, the more complex and difficult to control is the hybrid layer.

Single Universal Bond is a self-etching adhesive, whose peculiarity is that manufacturers recommend it also for total and selective etching techniques according to the practitioner option. The adhesive has a mild acidity (pH = 2.7), which should yield only superficial demineralization, normally no deeper than 1μm [3]. The manufacturers claim that it is sufficient for etching the dentin and the prepared

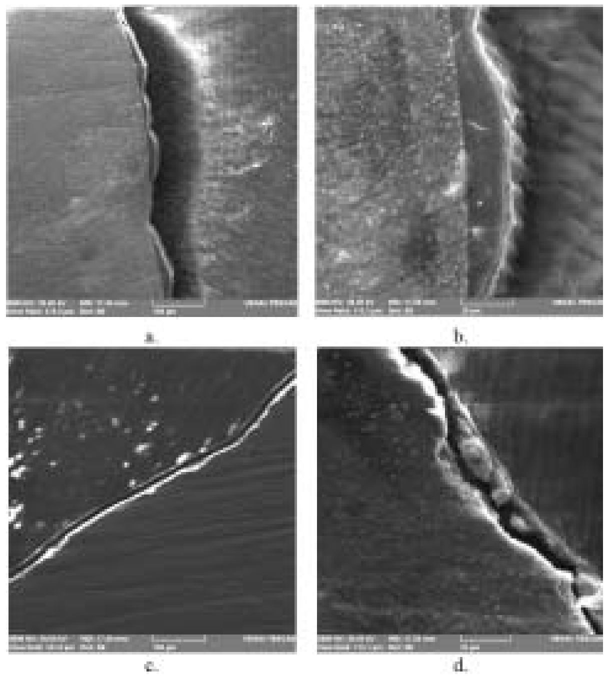

Fig. 2. SEM images for interfacial morphology of Single Bond Universal applied on pre-etched dentin: a. 500X, b. 2000X, c. 500X, d. 2000X

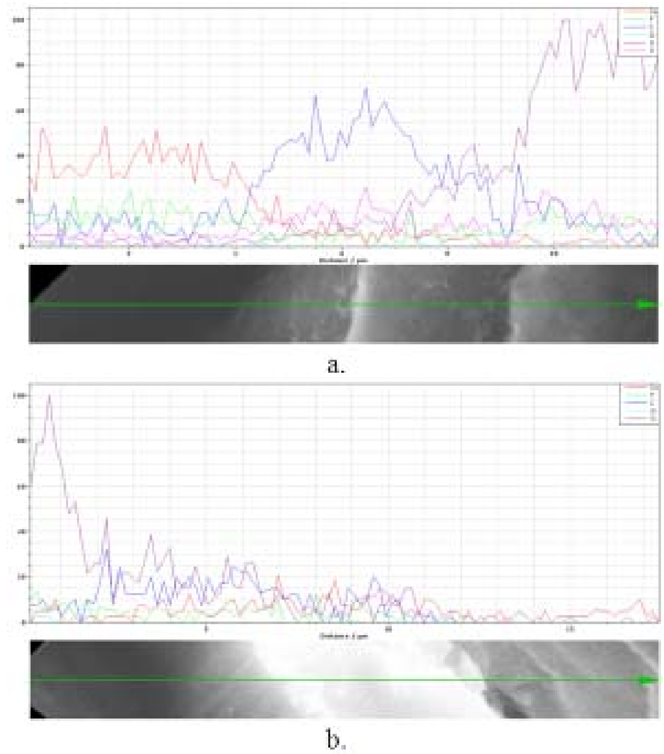

Fig. 3. Line mode of the elements variation of the interface between dentin and composite resin for Single Bond Universal applied in self-etching procedure (a) and total-etching procedure (b).

enamel in self-etching procedures although the preliminary etching with phosphoric acid could be also applied.

Our study aimed to observe the interfacial morphology when this system was used with and without acid etching and to notice if any of this procedure was more effective in sealing the dentin and eliminating the failure.

The appearance of the adhesive junction varied even within the same type of procedure. The thickness of the hybrid layer and residual adhesive resin was inconsistent in several specimens. The main reasons for this variability may be related to the mechanisms of adhesive infiltration into dentin, and the way in which the solvent is evaporated. The increased viscosity (the solvent used is alcohol) may cause some difficulty in wetting and preserving a uniform thickness after the application of the air spray. The experiment condition could be partially responsible for this problem, as extracted teeth could be more prone to desiccation and distortion of dentin collagen mesh than vital teeth. Further studies may clarify this issue and provide the clinician a clear and reproducible protocol. It is also true that direct processing of dental samples for scanning electron microscopy, which involved dehydration, caused high forces that probably exceeded those that occur through polymerization shrinkage.

Several gaps were observed at the interfaces of some specimens. The failures seemed to involve the adhesive interface for both techniques; cohesive failures were not found in dentin and rarely involved the composite resin. Adhesive failures of self-etching adhesives were also found by previous studies [11, 12], suggesting lower bond strength values comparing to etch-and rinse adhesives [8].

Some morphological differences of the adhesive joint were reported when using etch-and-rinse bonding systems or self-etch adhesives, the most remarkable being related to the thickness of the hybrid layer [13]. In our study in the absence of preliminary acid etching, the hybrid layer seemed to be thinner and fewer resin tags could be observed, which is consistent with previous results of other studies [1, 14-18]. The penetration of these systems is limited by the buffering capacity of dentine, resulting in uneven adhesion to dentin related to the variable thickness of the smear layer [1, 12, 14, 19-22].

In our study, the preliminary acid etching did not seem to significantly improve the quality of the dentine sealing (adhesive failures were present regardless of the protocol) although it discreetly changed the morphology of the interface. Hybrid layer was thicker and the presence of the resin plugs more frequent. However, one of the most extended gaps was found for one of the pre-etched specimens. Our observations support the results of another study which found indications of low-quality hybridization following acid etching prior to application of a one-step self-etching adhesive, in the form of a porous and poorly resin-infiltrate collagen mesh [23]. Another study found that the inclusion of dentin pre-etching resulted in deeper decalcification in etched versus non-etched dentine [24], which is consistent with our results following the EDAX study. Sabatini found the lowest values of bond strength and a constant pattern of adhesive failure for the self-etching adhesive applied with preliminary acid etching. The negative effect of phosphoric acid in dentin may be the combined result of over-etching, incomplete adhesive infiltration and compromised potential for the chemical adhesion [11] as specific functional monomers within dental adhesives can interact with hydroxyapatite [25]. In this case the preliminary etching could impinge on the qualities of adhesive joint, even if the thickness of the hybrid layer would increase. This mechanism would explain the conflicting results of several studies that evaluated the bond strength of such adhesive applied in total-etch procedures [11, 16, 24].

Conclusions

The SEM analyze of the interfacial morphology between a self-etching, single-component adhesive (Single Bond Universal, 3M ESPE) and dentine, has shown the formation of a thin hybrid layer and very inconsistent short resin tags within the dentinal tubules. When applying the adhesive with preliminary acid etching of dentin, the hybrid layer was thicker, and more resin tags were observed. However, for the tested adhesive, preliminary etching did not seem to significantly improve the quality of dentin sealing. For both techniques, limited gaps were observed at the adhesive interface. EDAX analysis showed a more significant demineralization of dentin when preliminary acid-etching had been used. The combination of single-component systems with selective etching or total etching techniques should be limited to products that have been tested and validated for use in such conditions. The potential benefits of additional etching with phosphoric acid should also be evaluated in terms of bond strengths and resistance to biodegradation.

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