

Etching Adhesives versus Classical Adhesives Concerning the Phenomenon of Dentin Hybridization

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An important feature of modern adhesive systems is their ability to modify the properties of dentin. We compared the appearance and properties of the hybrid layer generated at the dentin level dentin caused due to the application of a classic adhesive, with a self-etching adhesive. Scanning Electron Microscopy (SEM) revealed the detailed ultra-structure of dental tissues restored with the help of the adhesive systems. The disadvantage of classical adhesive systems is the difficulty of obtaining a degree of moisture content in the dentin. Advantages of etch adhesive systems are the inclusion of the smear-layer, the reduction of working time and the possibility of drying of dentin surface without risking its collapse in the collagen fibrillar network.

Keywords: dentin hybridization, smear layer, bonding, etching

Studies over recent years have shown that failures of dental restorations are due to secondary caries and pulpal complications [1] through the phenomenon of marginal bacterial micro-infiltration [2]. Choosing the type of pulpo-dentin protection depends on the thickness of the remaining dentin. Thus, in the shallow cavity, protection is provided by the thickness of enamel, but in the deep cavities this is no longer true and it resorts to a series of materials for pulp protection. At present it is considered that deep cavities hybridization is the best method to avoid pulp inflammation consecutive to bacterial-type infiltration [3-5].

The hybridization of the dentine wound is a process that creates at the molecular level a hybrid, i.e. an area with physico-chemical properties different than the original local structure through partial demineralization and impregnation of the collagen fibers exposed with polymer resin adhesive after acid etching [6]. The dentin detritus called smear layer covers the surface of any dentin wounds and it is the result of the process of physico-chemical degradation of the proteins from the heterogeneous structure of the hard dental tissues.

The hybridization of the dentine wound can be achieved by two therapeutic strategies: either by impregnation with adhesive primers and monomers while maintaining the smear layer on the dentin, or by complete or partial removal of the smear layer through acid etching of the dentin wound followed by the conditioning of the dentine wound.

The adhesive systems have known several improvements over the years in order to increase durability and resistance of the esthetic dental restorations. The new generation of dentin etching adhesives gained more ground compared to classical dentin adhesives that are applied following an etching because of their quality and advantages. An important feature of modern adhesive systems is their ability to modify the properties of dentin, turning it from a hydrophilic surface into a hydrophobic one.

Analyzing the ultra-structural appearance of the hybrid layer, Tay and Brajdic emphasized three characteristic aspects [7, 8]. The first aspect is the appearance of "shag-carpet" of the surface hybrid layer which means loss of the organization of collagen fibrils directed towards the

resin adhesive. The second aspect is the hybridization of the canalicular walls and represents the extension of the hybrid layer inside the dentinal tubules. Therefore, the so-called adhesive tags (adhesive retentions) that are formed in the dentinal tubules are circular and are surrounded by a hybrid layer at the canalicular opening. These adhesive tags that penetrate to a distance of 5-10 μ from the canalicular opening contributes in the greatest way to the achievement of an effective retention and seal. The third aspect is the so-called "side canalicular hybridization" that has been described as the forming of a thin hybrid layer in the lateral canalicular walls, called "micro-tag", which surround the core of an adhesive extension. Scanning Electron Microscopy (SEM) enables a detailed outline of dental hard tissue ultrastructure restored with adhesive systems.

The purpose of this study is to compare the appearance and properties of the hybrid layer generated at the dentin level as a result of the application of a classical adhesive after the demineralization of the dentin surface with 37% phosphoric acid, with an etching adhesive applied directly to the mineralized dentin.

Experimental part

For this study we used a total of 20 intact premolars without caries or fillings processes, extracted for orthodontic purposes. After extraction the teeth were kept in a 0.4% chloramine solution until their preparation and preparation for the SEM exam. For a better visualization of the hybrid layer aspect, deep cavities were prepared in which the dentin structure with numerous and large dentinal ducts favours an appropriate adhesive bond.

We used two types of adhesive systems:

- A classic adhesive (C-Bond adhesive);
- An etching adhesive (Futura-Bond adhesive).

Two study groups were formed as follows:

- Group 1, consisted of preparations obtained from teeth to which classic adhesives were applied in the deep cavities in the following manner: after the tooth was isolated, demineralization was done with 37% phosphoric acid which was initially applied on the enamel edges for 15 s and then for another 15 s on the entire dentin surface. In this way the excessive demineralization of dentin resulting in the collagen warping was avoided.

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Subsequently, the teeth were washed with a jet of water, dried with a cotton swab applied by dabbing it on the dentin surface, and then, the adhesive has been applied with a brush in a quantity enough to saturate the dentin surface and to facilitate the infiltration in the fibrillar collagen network. To accomplish the evaporation of the solvent the adhesive surface was dried by air jet for 5 s followed by curing the surface for 20 s. The operation was repeated by brushing it with a second layer of adhesive followed by removal of the solvent with a soft air jet for 5 s followed by curing the surface for 20 s.

- Group 2, consisted of preparations obtained from teeth to which etch adhesives were applied in the deep cavities as follows: after the cavity was isolated and the surface was dried with a jet of air, the adhesive was brushed directly to the dentin wound according to the manufacturer instructions, followed by air drying and curing the surface for 20 s. On top of the polymerized adhesive layer, a second adhesive layer was applied in the following the same steps as above.

Finally, each tooth was coronary restored with physiognomic composite.

The prepared teeth were embedded in self-curing acrylic in order to achieve vertical sections in the longitudinal axis of the tooth with a 20 cm diameter diamond disc. The resulting sections were kept in an ultrasonic bath for 10 min to remove any impurities from their surfaces. The preparations thus obtained were kept in adequate humid conditions until their preparation for SEM examination.

For the SEM examination there were selected only those preparations which showed no cracks or fractures in the studied surfaces. For the electronic microscopy SEM analysis, each section was affixed to the metal stand with adhesive carbon discs and the surfaces were metalized with platinum-palladium of 6 nm in argon atmosphere. The SEM analysis was performed on two types of scanning electron microscopes each with a different resolution.

Results and discussions

At the preparations of Group 1 there can be seen the appearance of the "shag-carpet" characteristic to the hybridization phenomenon which denotes the formation of a hybrid layer in accordance to the loss of organization

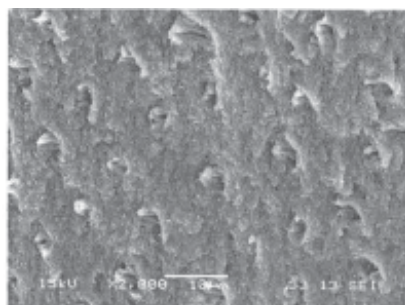


Fig. 1. The appearance of "shag-carpet" in the hybrid layer is observed in Group 1 preparations resulting from directing the collagen fibers towards resin adhesive

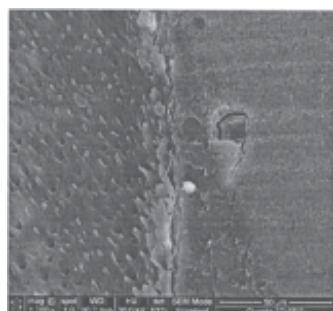


Fig. 2. The aspect of the dentin tags formed in the dentinal tubules of Group 1

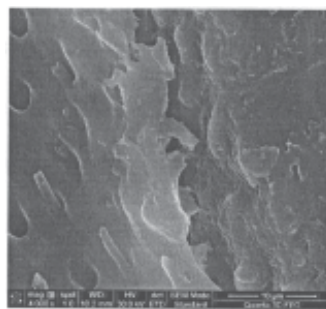


Fig. 3. The aspect of the dentin tags formed in the dentinal tubules of Group 2

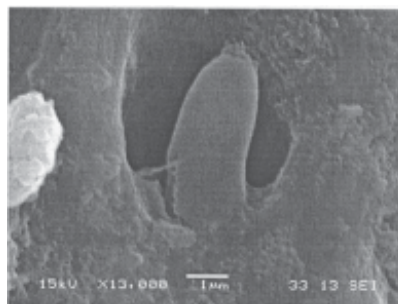


Fig. 4. Aspects of the dentin tags formed by the infiltration of the Optibond Solo Plus adhesive system

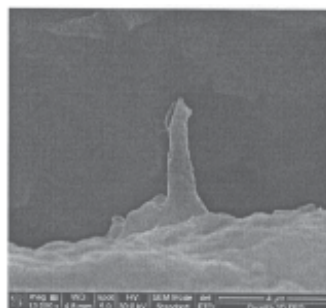


Fig. 5. Aspects of the dentin tags formed by the infiltration of the etching adhesive Futura-Bond

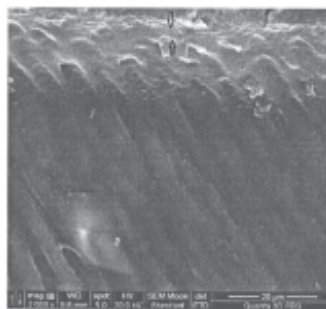


Fig. 6. The appearance and thickness of the hybrid layer in Group 1

of the collagen fibrils and their direction towards the adhesive resin (fig. 1).

In regards to the formation of dentin tags through the resin infiltration in the dentinal tubules, the results of this study revealed their presence in both types of the adhesive systems applied (fig. 2, 3), but their appearance varies. Thus, Group 1 found the presence of intra-canalicular tags thicker looking, with a conical shape and relatively long (fig. 4) while in the preparations from Group 2 the dentin tags formed are shorter and thinner (fig. 5).

The two types of adhesive systems used caused the development of some hybrid layers that are different in terms of quality and quantity of infiltration in the dentin structure. Therefore in Group 1, the conditioning of the dentin from the deep cavities prepared with 37% phosphoric acid for 15 s and the application of the mono-component resin adhesive causes the development of a medium thickness hybrid layer (fig. 6), with the formation of a greater number of dentinal tags compared to Group 2.

The Group 2 preparations show a hybrid layer of uniform thickness, also including in its structure the smear layer (fig. 7), which in the case of Group 1, is removed by etching.

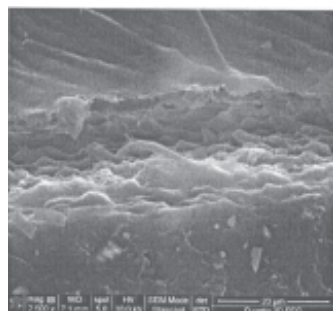


Fig. 7. The appearance and thickness of the hybrid layer in 2; the arrow indicates the appearance of a dentin tag characteristic to etching adhesives

The weak adhesive bond achieved by earlier dentinal adhesives was due to their inability to penetrate the smear layer which blocks the dentinal tubules and covers the exposed dentin surface. Microscopically, the smear layer is composed of an amorphous organic film, relatively smooth, with a maximum thickness of 1-2 μ m in which the 0.5 to 15nm hydroxyapatite crystals are incorporated [9, 10].

Most modern adhesive systems are based on the removal of the smear layer according to the global engraving principle. The technique was introduced by Nakajima [11, 12] who simultaneously etched both the enamel and dentin with a 40% phosphoric acid to completely remove the smear layer. Fontanari et al., testing the ability of substances to remove the smear layer, showed that the 24% EDTA and the 25% citric acid solutions resulted in a hyper-demineralization of the root surface where it is applied [13].

The impregnation with resin adhesive of the dentin wound occurs through the infiltration of the dentinal tubules and pores situated along the collagen fibers of the matrix of demineralized dentin. The permeability of the fibrillar collagen network plays an important role in achieving a quality hybrid. Therefore, if the fibers shrink (by air drying or by using organic solvents), the pores are dilated, and if the fibers expand (by being saturated with water or pH acid) the pores shrink and the adhesive penetration is limited [14, 15].

The acid-etching, which is applied on the dentin surface in order to remove the smear layer and to expose the collagen fibers, reduces the elasticity modulus of the dentin from 14-19GPa only 5MPa, from a rigid tissue to a soft and elastic tissue. This sharp decrease in elasticity modulus can have negative consequences on the quality of the hybridization if an incorrect technique is applied (unexpected drying) facilitating the collapse of the collagen fibers and compromising the resin retention. A demineralization with phosphoric acid for 15 s which does not distort the collagen will dissolve only the hydroxyapatite crystals between the fibers, and not those within the fibers [16, 17].

Normally, the application of a dentin adhesive requires three steps: in the first step the acid-etching is performed, which removes the smear layer and, at the same time, the mineral substances within the dentin; in the second step the primer is applied, which aims to re-expand the collagen network after its collapse produced by the drying of the

Study Groups	Type of dentinal adhesive	Chemical Composition	Application Technique	Smear Layer	Hibrid Layer	Dentinal Tags
Group 1	<i>Optibond Solo Plus</i> Classic Adhesive	Bisphenol -A diglycidyl methacrylate, 2-hydroxyethyl methacrylate, glycerol dimethacrylate, glycerol phosphate dimethacrylate, ethanol, camphorquinone, 2-(ethylhexyl)-4-(dimethylamino)benzoate, butylhydroxytoluene, coupling factor A174, filler (fumed SiO ₂ , barium aluminoborosilicate, Na ₂ SiF ₆).	Etching Acid Technique	Absent	Present Medium Thickness Uniform	Present Numerous Conical shape, long
Group 2	<i>Futura-Bond Self-etching Adhesive</i>	Acidic adhesive monomer 50-100%, bisphenol -A diglycidyl methacrylate 5-10%, 2-hydroxyethyl methacrylate 5-10%, fluorides, camphorquinone, siliciumdioxide nanoparticles.	Self-etching Technique	Present Disolved	Present Large Thickness Less Uniform	Present Numerous Thin, short

Table 1
RESULTS OBTAINED IN THE STUDY GROUPS

demineralized dentin [18]. The third step is the application of the resin adhesive which will infiltrate the network of collagen fibers and generate the hybrid layer. In the case of self-etching adhesives, the acid monomer concentration increases from 5-10% to 30-40%. By dissolving it in 30-40% HEMA (hydroxyl-ethyl methacrylate), a water-soluble primer is obtained whose very low pH ($pH=1-2$) allows the penetration of the smear layer [19]. Thus, the composition of the self-etching adhesive system allows, in a single application, the production of a hybrid layer with adhesive properties similar to the hybrid layer produced consecutively with the total etching technique.

The self-etch adhesive systems have a major advantage because they are applied to a mineralized dentin surface which may be dried enough without the danger of collapsing the fibrillar collagen network. Distortion of the collagen translates into loss of fibrillar appearance and its transformation into an amorphous mass of gelatin. Simultaneously there is the occurrence of the plucking of the debris of the hydroxyl-apatite crystals from the surface of the dentin wound, followed by their redistribution and cementation in the organic mass. Also, another advantage is the reduced treatment time through a single step application ("all in one") or a two-step application ("self-etching primers") of all components of the adhesive system.

In the case of self-etch systems, the smear layer serves as a substrate of adhesion; on the inside of the dentinal tubules, not being completely removed, the plug of the remaining smear layer is responsible for the absence of postoperative sensitivity [7, 20].

In our study, the preparation of cavities in the dentin where the adhesive systems were applied, was made keeping in mind the structural characteristics of the dentinal tissue. Thus, the diameter of the dentinal tubules gradually increases from 0.5 to 0.9 μ m at the enamel-dentine junction, reaching up to 2-3 μ m at the dental pulp level. The number of the dentinal tubules increases from 7000-15000/mm² near the enamel-dentine junction to 45000-65000/mm² near the dental pulp. The number and diameter of the dentinal tubules exposed represents a great clinical significance to the extent that the opening of dentinal tubules exposed represents the biological substrate of the hybrid layer by forming intra-canalicular adhesive extensions called tags.

In both study groups we found the formation of dentin tags within the dentinal tubules but each with a different aspect. Similar results were mentioned by Kukletova et al. [21] which compared the aspect of the adhesive extensions formed consecutively with the using of mono-component adhesive systems and those of the self-etch adhesive and they showed no significant differences in dentin-resin interface. The study done by Albaladejo showed that the mono-component self-etching adhesives "all in one" are able to dissolve the smear layer ($pH=0.6$) and to form a relatively thick hybrid complex [22].

We also found differences in the appearance and thickness of the hybrid layer generated in the two study groups. Therefore, in Group 1 we have revealed a hybrid layer of medium thickness and uniform aspect, while in Group 2 the hybrid layer is thicker and less uniform. Regardless of the classical adhesive applied, the adhesive bond decreases with time due to the degradation of collagen fibers that serve as support and are included in the hybrid layer [23].

Conclusions

The classical adhesive systems applied consecutively to self-etching are able to achieve a quality hybrid layer if the correct approach is applied. The disadvantage is in the difficulty of obtaining appropriate moisture content in the dentin.

The self-etch adhesive systems can achieve a strong adhesive bond by dissolving and by its inclusion in the smear layer. The major advantage is given by the reduction of working time and by the possibility of drying of the dentin surface without the risk of the collapse of the fibrillar collagen network.

Regardless of the adhesive system used and its application technique, the restoration of dental structures must respect the principle of proper conservation and protection of the pulpo-dentinal complex.

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