

Comparative Study of Acid Etching on Dental Enamel

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This experimental study aims to analyse the effectiveness of various materials used in demineralisation of dental enamel. This work aims to create a mechanical bond by filling the pegs with sealing material. In order to achieve this goal, five teeth were compared using different concentrations of orthophosphoric acid and exposure times. In this regard, five different tests were performed and the results were analysed using the SEM technique (scanning electron microscopy). These comparative analyses revealed that etching using 35% orthophosphoric acid for one minute and etching using Icon Etch for two minutes were the most effective.

Keywords: acid etching, dental enamel, electronmicroscopy, adhesion

Adhesive techniques may represent the greatest progress known by restorative dentistry, especially in the last half of the 20th century. Both adherence to dental rigid structures and adherence to artificial structures such as metal, ceramics or polymers have revolutionized the principles and methods by which we are currently guiding our work.

Michael Buonocore's work (1955) in adhesive restorative dentistry was pioneering. Inspired by the practices employed in the naval industry to ensure better adherence of lacquers and paints to metallic surfaces, he suggested ever since those times to prepare dental enamel for adhesion by treating it with phosphoric acid. We can only talk about the real utility of acid etching beginning with 1962, when Rafael Bowen patented Bis-GMA resins. This material, the formula of which is 2.2-bis [p-(20-hydroxy-30-metha cryloxypropoxy)phenyl] propane, is widely used as a base monomer in polymeric dental materials [1]. Since then, thanks to the achievement of significant results in terms of low susceptibility to technical errors, adhesion to enamel has undergone few changes in principle and methods. If the concentration of orthophosphoric acid originally proposed by Michael Buonocore was 85%, it subsequently was reduced to 30% -40%. Other changes involved reducing the application time from 60 to 15 s or the presentation of the demineralizer as gel [2].

When a coronary lesion is restored, one of the working principles to take into account is to use materials adhering to the dental surfaces. This avoids the phenomenon of marginal infiltration (percolation), as well as the onset of dentinal hypersensitivity or postoperative pain [3]. For this reason, in restorative dentistry practice, the mere *juxtaposition* of a filling material in a cavity is no longer accepted. It can no longer represent a final therapeutic solution for the repair of dental lesions (the most common of which are coronary) manifested by the lack of hard dental substance.

Current studies [4-7] have shown that effective adhesion can be achieved by using much lower concentration acids. These acids, applied on the surface of the enamel, are able to demineralize and dissolve the inorganic hydroxyapatite matrix from the structure of enamel prisms,

forming micropores and microcracks. These acids transform the surface of the intact enamel, which has low energy and various degrees of impurity, into an active, clean, demineralised surface, with high superficial energy. In the same manner, acids also act on enamel processed with different types of rotary instruments. Following this processing, the enamel is covered with a layer of residual debris, consisting of inorganic residues and smear layer [2]. Before washing the demineralized surfaces, it is advisable to aspirate the conditioning agents to prevent it from coming into contact with adjacent surfaces [8]. The type of washing in the case of classical acid etch involves the need to completely remove the precipitate consisting of calcium salts to achieve a high superficial energy. Insufficient washing may allow acid to act in certain areas more than it is allowed [9].

In current dental practice, when applying 37% phosphoric acid as a gel, a 15s wash time is sufficient if pressurized water is used. When self-etching adhesives are applied subsequently to etching with high-concentration acids, the wash time can be reduced to 5 s. Conditioned and washed enamel should be dried by classical methods for 3-5 s with a dry and uncontaminated air jet. The type of drying will be reduced if hydrophilic / hydrophobic monomers are used, which require a slight degree of moisture of the substrate for effective adhesion. Conditioned enamel allows its wetting and impregnation micropores with resin-monomer, which once polymerized will generate *tab-like extensions* at micro-cavities level that will retain the adhesive layer, forming a hybrid layer between the enamel and the adhesive [2,10].

The goal of acid etching is to create *retentive micro-craters* for better adhesion of enamel and dentine fill materials. At the same time, acid etching is preceded by a step consisting in the mechanical preparation of the enamel by means of diamond drills (carries removal; bevelling of cavity edges, etc.). Both procedures make their mark on the tooth load-bearing structure, creating injuries at this level, injuries that, *in vivo*, need months to be remedied [3, 8, 10]. All these make it possible to impregnate the tissue with a monomer that will be retained inside the micropores created when it changes its aggregation state [8]. By

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means of acid etching, about 10µm of the enamel surface is removed and a microporous layer of 5-50µm depth is created.

The latest trends in dentistry are based on reducing the so-called *chair-time* (time spent by the patient in the dental chair). A study published on 12 December 2015 in the *Journal of Dentistry of Tehran University of Medical Sciences* has proved that the high temperature induced by the light emitted by the light-curing lamp may shorten the acid etching time. In order to prove this relationship, three groups were used in the study: G1 - acid etching for 15 s; G2 - acid etching for 5 seconds; G3 - acid etching for 5 seconds + light of the light-curing lamp. The result of the study showed that using the light-curing lamp technique, the acid etching time is reduced to just 5 s without endangering the quality of the work done [10, 11].

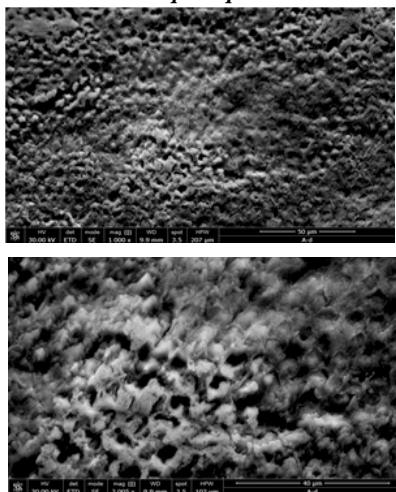
Experimental part

This experimental study aims to analyse the effectiveness of various materials used in demineralisation of dental enamel. This work aims to create a mechanical bond by filling the pegs with sealing material.

In order to achieve this goal, two patients were involved and five teeth affected by periodontal disease were extracted, i.e. bottom central incisors and bottom lateral incisors. The two patients expressed their consent in writing for the use of the teeth for research purposes. The extraction was performed in a delicate manner so that the enamel surface remains undamaged. After extraction, the teeth were stored in holders containing physiological serum, thus avoiding the dehydration phenomenon. Each vestibular surface was divided into two halves. One-half was isolated using a liquid dam to prevent the dispersion of the acid etching material at this level, and a second surface was left free to be demineralized. Next, each non-insulated surface was etched using the following materials: 35% orthophosphoric acid; Gaenial bonding self-etch; Icon-Etch. Further on, the samples were compared by the scanning electron microscopy (SEM) method, using magnifications from 250x-3000x.

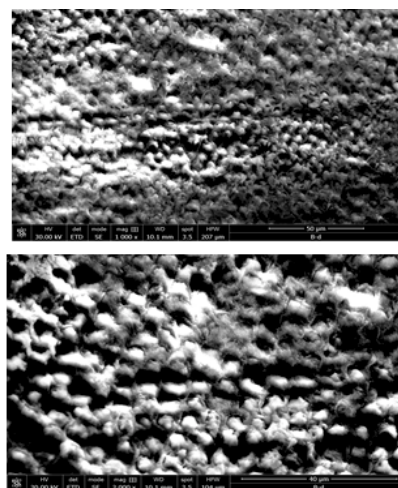
In this regard, five samples were applied, as follows:

Sample 1: 35% Orthophosphoric acid - 60 s



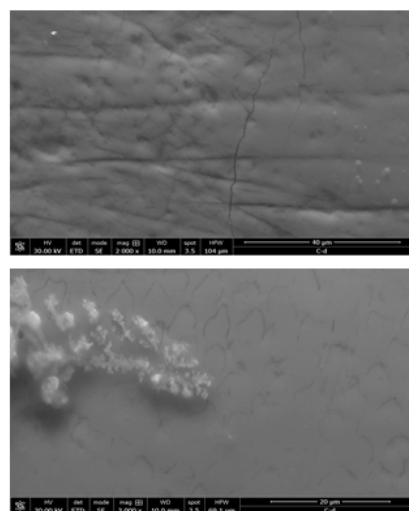
In this sample, the enamel demineralization was performed using orthophosphoric acid for 60 s. Subsequently, the acid was removed by washing under a water jet using the air/water syringe of the dental unit, and then the tooth surface was dried. After the liquid dam was removed, the tooth was put under the microscope to see the difference between the etched area and that not subject to acid attack.

Sample 2: 35% Orthophosphoric acid - 30 s



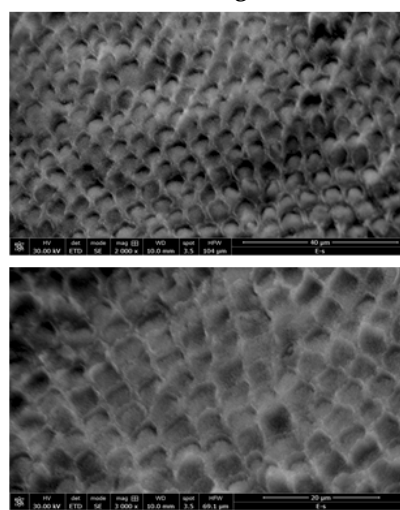
The same Gluma orthophosphoric acid (35% concentration) was also used in Sample 2, but this time the etching time was reduced to 30 s.

Sample 3: Gaenial Self-Etch Adhesive -10 s



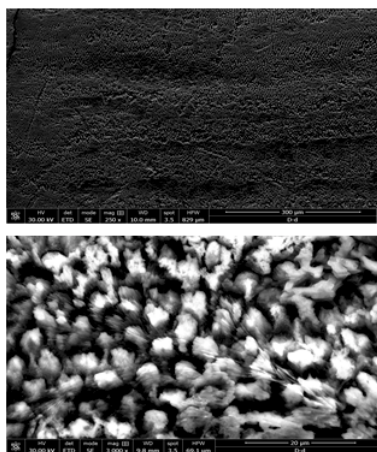
Sample 3 consists of demineralizing the enamel using Self-Etch Gaenial Adhesive for 10 s, the period indicated by the manufacturer (leaflet). After the 10 s (etching time), the tooth was washed with water under pressure and dried using the air/water syringe of the dental unit.

Sample 4: Icon Etch, etching time 2 min



In Sample 4, the incision was treated with Icon Etch for two minutes, the working time indicated by the manufacturer (leaflet). The tooth was then washed with water under pressure and dried using the air/water syringe of the dental unit.

Sample 5: 35% orthophosphoric acid + light-curing lamp for 10 s



On this sample, we wanted to test whether the high temperature induced by the light emitted from the lamp that photopolymerizes the composite can shorten the acid etching time. After the acid was applied to the tooth, we exposed the tooth-acid complex to the blue light for 10 s.

Results and discussions

Sample 1: 35% Orthophosphoric acid - 60 s. At a 250x magnification, the vestibular half was subjected to acid etching (35% orthophosphoric acid - 60 s) with non-etched surface. Compared with the etched surface of the enamel that was smooth, the etched surface showed a change in the enamel structure, which became more porous. The analysis at a 1000x magnification revealed that, unlike the magnification achieved at a 250x coefficient, the enamel porosity can be observed in detail after demineralization with 35% orthophosphoric acid - 60 s. Moreover, at a magnitude of 2000x, microretention caused by orthophosphoric acid 35% for 60 s can be clearly seen. Microretention areas are complete, uniform and stretched to the surface. At the end of this test, the magnification coefficient on the demineralised area was 3000x. The honeycomb-like texture of enamel is very well represented in this image, the microretentions are well individualized, accentuated, and their shape is continuous. The tooth is prepared for the next step in the obturation process (application of adhesive).

Sample 2: 35% Orthophosphoric acid - 30 s. Differences between Sample 1 and Sample 2 are not significant under small magnifications, but under great magnifications incomplete microretention can be noted. Thus, at a magnitude of 250x, the etched surface with 35% orthophosphoric acid (for 30 s) begins to show future microretentions, as the appearance of the rigid substance is porous. In this picture, the demineralised half of the tooth appears with the same honeycomb-like aspect as in Sample 1, but, since the etching time is smaller, incompletely demineralised areas are noticed and microretentions are not evenly spread. Compared to Sample 1, at the same magnification of 2000x, more faded *honeycomb* areas, incompletely formed microretentions can be seen, a consequence of the shorter demineralisation time. The latest test of Sample 2 is conducted on etched enamel, and the magnification set-up reaches the maximum of 3000x. We can observe superficially demineralised areas and sometimes incompletely formed porosities.

Sample 3: Gaenial Self-Etch Adhesive - 10 s. In this sample, the results of microscope are not very relevant because the material produces during the 10 s (action time) both demineralization, creation of microretention and their filling with liquid resin (adhesive).

Sample 4: Icon Etch, etching time 2 min. Following this test, results similar to the situation in Sample 1 can be

observed. At a magnitude of 250x, porosity traces can be noted, while honeycomb-like form occur at a magnitude of 3000x.

Sample 5: 35% orthophosphoric acid + light-curing lamp for 10 s. At a magnitude of 250x, traces of porosity can be identified, while at a magnitude coefficient of 3000x microretention can be noted.

Conclusions

Acid etching is very important in the filling process, as it has the role of forming the pegs in which the sealing material will flow and will form a mechanical bond between the conditioned surface of the tooth and the composite material. Of the chosen versions, etching using 35% orthophosphoric acid for one minute and etching using Icon Etch for two minutes were the most effective. In both samples, microretention was completely formed, spread over the entire enamel area and uniform. The rest of the samples showed an incompletely formed *honeycomb*, with rare pegs and areas of non-etched enamel. Self-Etch Bonding proved to be the least relevant; in this sample, it was not possible to detect the acid-attacked area compared to the non-etched area, because at the same time with demineralization filling of microretentions with the liquid resin occurs.

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