Clinical dental adhesive application: the influence on composite–enamel interface morphology

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Abstract
Although the adhesion phenomenon is crucial in achieving and maintaining a composite building on dental structure, this phenomenon is not completely understood. On the other hand, adhesion is dependent on the interface quality (the interface between enamel and adhesive). In this study, the authors approached the subject of the influence of adhesive clinical application on the composite–enamel interface, which was less investigated by the scientists. On intact extracted human teeth were prepared enamel areas, and then filled with light-curing composite. The teeth were sectioned and prepared for microscopic investigation, at 10×, 100× and 200× magnifications.

Keywords: dental composites, adhesion, enamel–composite interface, adhesive application technique.

Introduction
Numerous clinical studies on dental structure were focused on the study of surface characteristics of enamel or dentin etched with ortho-Phosphoric Acid, and so making important observations and results of this phenomenon [1–5]. We have noticed a weaker focus of researchers on the influence of adhesive application techniques on adhesive bond strength. Many experimental investigations relied on guidelines supplied by producers of materials studied. Thus, we intend to focus on issues arising from modification of applying technique for adhesive materials, considering that they have a major impact on dental adhesive restorative clinic, but also in the quality of adhesion.

Objective
Theoretical conditions required for achieving resistant adhesive joints with acid etched enamel or dentin are already known [6–11]. They state that the resin must moisten better acid attacked surface, so the contact angle adhesive–enamel or adhesive–dentine to be as small as possible – ideal 0°. It is also known the fact that the strength of adhesive joints depends on the conditions in which ab initio acid etching runs [12–15]. Therefore, for an effective adhesive, it is necessary that the etching model would be as retentive as possible.

In addition to these issues, we believe that clinical technique and the type of the adhesive application exert great influence (for three steps adhesives, two steps adhesives or self-etching adhesives) [16–19]. The purpose of this study was to investigate this hypothesis.

Materials and Methods
Were treated 16 upper premolars extracted for orthodontic purposes, free of dental caries or cavities. The teeth were kept in a solution of 2% Chloramine T before ultrasonic cleaning for organic residues. Then the teeth were stored in saline solution at room temperature (25°C) up to the execution of the examination samples. We chose the adhesion technique in two steps. The etching was obtained by using 34% ortho-Phosphoric Acid for 30 seconds, followed by washing for 30 seconds and drying the enamel surface. The adhesive used was a “one bottle” type (Gluma®, Heraeus-Kulzer, Germany). Its application was carried out according to the manufacturer’s instructions, with the curing time of 20 seconds using a LED curing light (Dentmate®, Korea).

The teeth were divided into four groups, and treated on the buccal sides as follows:

- Group 1:
  - two teeth: etching, composite in layers, and adhesive applied by brushing (usual technique);
  - two teeth: etching, composite in layers, and adhesive applied by rotation.

- Group 2:
  - two teeth: etching, composite in contouring crown, and adhesive applied by brushing (usual technique);
  - two teeth: etching, composite in contouring acetate crown, and adhesive applied by rotation.

- Group 3:
  - two teeth: enamel additional preparation with burs, etching, composite in layers, and adhesive applied by brushing (usual technique);
  - two teeth: enamel additional preparation with burs, etching, composite in layers, and adhesive applied by rotation.

- Group 4:
  - two teeth: enamel additional preparation with burs, etching, composite in contouring acetate crown, and adhesive applied by brushing (usual technique);
two teeth: enamel additional preparation with burs, etching, composite in contouring acetate crown, and adhesive applied by rotation.

The rotating application of adhesive was made with an application tip (modified and adapted for the contra-angle hand-piece in personal concept) at a speed of 90–125 rpm.

Then the teeth were sectioned using a diamond disc with active edge at conventional speed and running water, along to the vertical axis of the tooth, on buccal-oral direction. The buccal faces were sectioned into two halves to expose the stratification of the investigated area. The root portion has also been removed.

For a good handling and processing, specimens were embedded in cold curing acrylic resin (Duracryl®, Spofa, Czech Republic) leaving the surface investigated free. The samples were stored in a container in sterile saline solution at 4–5°C, until the surface examination.

The samples were prepared in a specialized rotary facility using P800, P1200 and P2200 SiC sandpaper. Grinding was carried out under running water. To avoid prolonged contact of the sample with the air, between the three rounds of abrasive paper, the samples were washed thoroughly and were stored in a glass vessel with water at 25°C.

After the polishing with P2200 SiC paper, the samples were washed with tap water and were stored for microscopic examination in a container with distilled water at 25°C for 24 hours (Figure 1).

Surface examination was qualitative, being achieved by high power light microscopy (10×, 100× and 200×) with a Neophot 21 type microscope (Microstructures Investigations Laboratory, Research Center, INTEC S.A., Bucharest), aiming uniformity and continuity of the hybrid layer and mass characteristics of the composite.

Results

Group 1: acid etching and composite in layers

Enamel–adhesive interface following brushing adhesive

On some portions, the composite has nearly homogeneous structure, but in the contact with the enamel stands a sinuous structure, sometimes in the form of bundles, implying an irregular infiltration of the adhesive on the surface adherent (red area). We believe this is due to the technique of brushing the adhesive, that “sweeps” enamel debris and thus resulting “clean” surface portions and areas of clumps of detritus mixed with adhesive (Figure 2).

Enamel–adhesive interface following rotating adhesive

We present a 10× magnification portion of the sample examined, marking the dental hard tissues with D – dentin, S – enamel and C – composite attached to the tooth. We can see dentin fracture (at the border with enamel, and dentin mass). The enamel prisms can be identified. The arrows indicate the portion where the composite was attached.

Although the two interfaces are almost equal in thickness, there are cracks in the composite mass both in the case of the usual adhesive techniques (by brushing), and in the case of rotating technique, more likely from the polymerization shrinkage. However, their appearance differs: in the case of using rotating technique for adhesive – the cracks are beginning from interface and enter into the composite mass; are parallel to each other and having a nearly linear path. They are also much shorter than the cracks occurring in brushing technique (Figure 3).

Group 2: acid etching and composite in contouring acetate crown

Enamel–adhesive interface following brushing adhesive

The analysis of the picture obtained from the enamel level with brushing adhesive and application of composite in a contouring crown shows cracks in the composite mass as in the previous experiment, in terms of the waves or undulations because of the polymerization shrinkage. However, their appearance differs: in the case of brushing technique – the cracks are beginning from interface and enter into the composite mass; are parallel to each other and having a nearly linear path. They are also much shorter than the cracks occurring in brushing technique (Figure 4).

Enamel–adhesive interface following rotating adhesive

The technique of rotation application of the adhesive allowed the installation of a composite system with enhanced appearance than the brushing technique. The use of the contouring acetate crown has led to a decrease in the polymerization shrinkage cracks, which have short route and are almost parallel to each other (Figure 5).

Comparative analysis of the microscopy pictures of
the enamel–adhesive interface, with acid etching of the enamel, and with various clinical techniques for the application of adhesive (brush or rotation) shows that both in the case of layering composite, and in the case of its application with the contouring crown, results in adhesive complexes with better quality (almost no cracks in the mass of the composite and continuous hybrid layer) for adhesive rotational application technique.

**Figure 3** – Interface aspect of demineralized enamel (S) and complex composite (C) using rotation adhesive and composite layers: (a) The area of interest (10×); (b) Detail of the previous image (100×).

**Figure 4** – Interface aspect of demineralized enamel (S) and complex composite (C) using brushing adhesive and composite in acetate contouring crown (100×).

**Figure 5** – Interface aspect of demineralized enamel (S) and complex composite (C) using rotating adhesive and composite in contouring acetate crown (100×).

**Group 3: enamel preparation with burs, acid etching, and composite in layers**

**Enamel–adhesive interface following brushing adhesive**

The enamel prepared with diamond burs has led to an increased retentivity for adhesive (composite material) comparing to simple etching. This is revealed by the uniform and homogeneous aspect of the hybrid intermediate layer (boundary between the S and C region). The composite layering led to the emergence of low retentivity areas at the interface (circled area). We assume that this has occurred due to the presence of strongly adherent enamel strips on underlying structures (possibly prism-less enamel). Instead, the composite shows a homogeneous structure, almost with no cracks (Figure 6).

**Enamel–adhesive interface following rotating adhesive**

The next figure illustrates the manifestation of a microscopic qualitative interface of composite resin (adhesive) and enamel when the adhesive was applied on the enamel surface by mechanical rotational technique. In terms of preparing for increasing enamel retentivity (mechanical preparation with diamond burs) plus acid etching, the distinction between adherent and adhesive is quasi-continuous and uniform (red border) (Figure 7).

Thus, the shaded area was studied microscopically, revealing the above. This entitles us to believe that rotation causes a more uniform distribution of the adhesive layer and thus a better link with enamel.

**Group 4: enamel preparation with burs, acid etching, and composite in contouring acetate crown**

**Enamel–adhesive interface following brushing adhesive**

Processing enamel surface with diamond burs to obtain an additional retentivity has proven reliable and in the teeth of Group 4, restored with composite in contouring acetate crown. Optical microscopy images showed a
quasi-uniform and homogeneous intermediate hybrid layer, with areas of low retentivity or overlapping material (arrow). The portion of the composite is a homogeneous, with no cracks (C area) (Figure 8).

Thus, in this case the acetate-contouring crown permits the application of the composite material with a relatively evenly distributed and constant pressure by the operator. It results a more uniform hybrid layer in opposite to the method of stratification. In this case, the fighting against polymerization shrinkage seems more effective. One adjuvant factor is represented by acid attack on enamel surface (acid etching).

**Enamel–adhesive interface following rotating adhesive**

After additional processing of the enamel with burs, along with rotational technique for adhesive and composite material with contouring crown the image obtained is special, almost competing with the previous one. The interface shows here more homogeneous. In contrast to previous image, the intimacy between enamel and composite is higher in the second case (red frame) (Figure 9).

Our assumption is that in this case the contouring crown (allowing the application of composite material with a relatively uniform and constant pressure), and the rotary adhesive technique lead to superior quality composite systems.

**Figure 6** – Interface aspect between prepared and demineralized enamel (S) and composite complex (C) following brushing application of adhesive and composite in layers (100×).

**Figure 7** – Interface aspect between prepared and demineralized enamel (S) and composite complex (C) using rotating adhesive and composite layers: (a) The area of interest (10×); (b) Detail of the previous image (100×).

**Figure 8** – Interface aspect between prepared and demineralized enamel (S) and composite complex (C) using brushing adhesive and composite in contouring crown (100×).

**Figure 9** – Interface aspect between prepared and demineralized enamel (S) and composite complex (C) using rotating adhesive and composite in contouring acetate crown (100×).
Discussion

Enamel substrate can vary, being present in the following forms:
• intact (permanent and temporary teeth), and
• modified by the action of dental burs (prepared/cut).

This substrate has a different behavior to acid etching and composite material application. Consequently that will influence operator protocol when are used adhesive techniques. The tooth enamel demineralization obtains micromechanical retentivity, which can be supplemented by cutting the enamel prisms with burs, and thus a high receptivity to adhesion. Treatments for increasing enamel surface retentivity lead to durable adhesive structures, but many authors consider this to be insufficient [20].

In terms of correct handling, the composite materials strength will not be a problem, making them virtually “inseparable” with the tooth, both by acid etching or the adhesive used. In this way can be avoided traumatic prosthetic solutions especially in growth age. Prosthetics works will remain an alternative therapeutic solution for plastic reconstruction in case of composite restoration failure [21–23].

Due to their physical structure and mechanical properties [24–26], direct composite materials present certain advantages for dental clinical operative, and so becoming effective solutions. This category presents some technological advantages for direct application:
• ease of treatment, which can be executed in a single session, reducing working time;
• exclusion of dental laboratory;
• achievement of an esthetic and cheap restoration, in the medium and long term.

The disadvantages could be represented in terms of optical and mechanical resistance less than other esthetic restorative materials (e.g., ceramics). These micro-imperfections often appear after overlapped layering of composite.

By means of an adhesive, two different materials can be joined together to give a new set – the composite system. The components of this system are: tooth surface (represented by enamel and/or dentin) as a substrate; adhesive; and resin composite. This new system will have different properties and behavior from its components [27, 28].

Whatever is the applicator tip, with a brush the adhesive is spread like paint.

In contrast to the brush, the rotational movement for adhesive applicator determines a better spreading and smoothing of the dental substrate, thus locally providing an adhesive layer and therefore a hybrid layer with improved characteristics. We observed that the mechanical rotational application of the adhesive allows the adhesive to cover dental hard tissue surface with a layer almost uniformly and continuously.

In dental clinical adhesive techniques protocols, the operator presses through modeling instrument with a unidirectional and perpendicular, or a bidirectional force on the dental substrate. This “clinic” force is also moving parallel with the composite–hard dental substrate, for morphology modeling of dental restoration. This movement allows the emergence of “micro bumps” (undulations/waves) of the composite layers. The next composite layer filled after the curing of the previous one will present dehiscence at the interface with his predecessor. This will be manifested by low resistance in the oral environment of the adhesive restoration. Also, it is difficult to maintain clinically a quasi-uniform thickness of light-curing composite resin (1.5–2 mm) [29–31] to ensure complete polymerization by volume of the composite. In our study, the contouring crowns were used as supplements and alternatives to modeling instruments, and for increasing adhesion to dental hard tissues.

Another aspect that we would like to emphasize is the construction of dental composite restorations on damaged substrate during processing/operative steps. We hope that the images obtained by optical microscopy are an argument for using dental composite materials in different purposes and with different dental restorative techniques (including the new rotational one). The resistance failure of these constructions is not exclusively dependent on “quality” of the composite (commonly blamed in the dental environment). It is rather using an underlined dental substrate during its preparation to receive adhesive “treatment”.

The interface between enamel and dental adhesive/composite (hybrid layer) must have a structure (and therefore such properties) that will enable to resist to oral stress factors (e.g., mechanical, thermal, chemical, etc.). From the optical microscopy point of view, this means an intermediate layer that must have a quasi-homogeneous aspect. In our study, this layer appears alongside with a new clinical adhesive technique – rotational. The best results were obtained when this technique was used in combination with contouring crowns.

Conclusions

The condition of building strength during time depends on the quality of composite substrate adhesion achieved between enamel and dental composite. From image comparative analysis of clinical techniques achieved through adhesive brush application and rotational technique, we found that the latter is an enabling element in improving the final composite system (adhesion), and could be a key to clinical success along with existing techniques.

Author contribution
All authors have equal contributions to the study and the publication.

References


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