### ORIGINAL PAPER



### Microscopic aspects of junction between dental hard tissues and composite material depending on composite insertion: layering *versus* bulk-fill

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### **Abstract**

Polymerization stresses in adhesive structure could determine an adhesive or cohesive failure and interfacial gaps forming. Some clinical procedures – as light curing composite resin layering in 2–3 mm increments (especially for dental fills) or using dental reconfiguring clear acetate crowns – are sat up to combat (in some way) the polymerization shrinkage. This study approaches the manner how clinical dental adhesive application could influence the hard dental tissues–composite materials interface. The sample studied consisted of 12 upper bicuspids, extracted for orthodontic reasons. In our study, we chose the adhesion technique in "two steps". We prepared enamel and dentine areas, and then filled them with light-polymerization composite. After that, the teeth were subject of microscopic investigations, at 100× and 200× magnification. As conclusion of our study, we sustain that light curing composite resin applying clinical technique (by layering or with acetate crown) might influence a good dental restoration clinical performance.

Keywords: adhesion, light curing, composite, stratification (layering), acetate crown (bulk).

### ☐ Introduction

Bonding adhesion has two components: an adhesive and a least an adherent surface on which the adhesive is bonded. For dental hard tissues, such as enamel and dentine, there is a chemical-like bonding through van der Waals forces [1] and micromechanical retention between two elements – dentine/adhesive or enamel/adhesive [2, 3]. This is realized in the following sequence: substrate acid etching, adhesive infiltration, and polymerization. In the case of dental enamel, due to its highly mineralized structure, the facts are quite clearly established about composite adhesion. Instead, for dentine – a less mineralized dental tissue, the phenomenon is particular and named as hybridization by Nakabayashi et al., since 1982 [4]. The term was extended for dental enamel. In current speech, it is used to designate a special layer resulted from composite (adhesive) infiltration into enamel and dentine superficial zone. The main function of a dental adhesive is to create a bonding between itself and dentine collagen fibers and/or remaining enamel prisms.

The dental bonding effectiveness depends on adherence etching model [5–10]. There are already stipulated the theoretical conditions for acid etched enamel or dentine [11–27]. We believe that clinical technique of composite insertion, layering or bulk-fill, could influence the strength of adhesive joints. For this purpose, we analyzed the microscopic morphology aspects of junction between enamel and composite material and between dentine and composite material.

### Materials and Methods

The study was performed in Microstructures Investigations Lab, Research Centre, INTEC SA, Bucharest, Romania, and was based on previous study published in 2014 [28]. For enamel, we used the same materials and methods as in the study mentioned above, but in addition, we also prepared and examined the dentine surface.

Were used 12 upper premolars extracted for orthodontic purposes, free of dental caries or cavities. The teeth were kept in a 2% solution of Chloramine T before ultrasonic cleaning for organic residues. We chose the adhesion technique in "two steps". The etching was obtained by using total etch technique, with 34% ortho-Phosphoric Acid for 30 seconds, followed by washing for 30 seconds and drying the enamel and dentine 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 light-emitting diode (LED) curing light (Dentmate®, Korea). We used the same adhesive application technique – by manual brushing.

The teeth were divided into three groups, and treated as follows:

- Group #1 (enamel):
- (a) Two teeth: acid etching, adhesive applied by manual brushing (usual technique), and composite in *layers*;
- (b) Two teeth: acid etching, adhesive applied by manual brushing, and composite in *contouring/acetate crown*.

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- Group #2 (enamel):
- (a) Two teeth: enamel surface additional preparation (milling) with diamond burs, acid etching, adhesive applied by manual brushing (usual technique), and composite in layers;
- (b) Two teeth: enamel surface additional preparation (milling) with diamond burs, acid etching, adhesive applied by manual brushing composite in contouring/acetate crown.
  - Group #3 (dentine):
- (a) Two teeth: acid etching, adhesive applied by manual brushing (usual technique), and composite *in layers*;
- (b) Two teeth: acid etching, adhesive applied by manual brushing, and bulk-fill composite.

In fact, in Groups 1 and 2 were included teeth with enamel surface prepared for microscopic examination, and in Group 3 were included teeth with dentine surface prepared for microscopic examination. Also, there were differences between composite application on enamel or dentine due to specific preparation of these dental hard tissues:

- on enamel simply acid etching or surface preparation with diamond burs (as supplementary mechanical retention) and acid etching;
- on dentine to expose dentine, we performed crown class I cavities with standard dimensions (2 mm depth, 2 mm wide, 2 mm length) [29, 30].

The composite material was applied *on* the enamel surface (hand layering or with contouring/acetate crowns). For dentin, the composite material was applied *into* cavities (layering or bulk insertion).

The samples preparation method was as follows:

- Step #1: teeth sectioning. We used an active edge diamond disc under running water and at conventional speed (dental straight handpiece). First, we cut the teeth into two halves along vertical axis, from coronal to apical and from buccal to oral direction. Then, we sectioned the buccal portions again in two parts, in order to expose the stratification of the investigated area. After that, we removed the dental roots.
- Step #2: sample acrylic embedding (for a good handling and processing). We used a cold curing acrylic resin (Duracryl®, Spofa, Czech Republic). We leaved free the areas for investigations.
- Step #3: storage of the samples. We put the samples into a container with sterile saline solution, at 4–5°C, until the surface examination. This method allows eliminating the accumulated stresses during dental hard tissue preparation and acrylic polymerization.
- Step #4: preparation of the samples for optical microscopy investigation. We used a specialized rotary device and P800, P1200 and P2200 SiC sandpaper, under running water. After each grinding/polishing round, we washed thoroughly the samples and we stored them in a glass vessel with water, at 25°C. The motivation was to avoid the prolonged contact of the samples with the air. For the final polish, we used a diamond paste (without water). We washed again the samples with running water and we stored them for microscopic examination in a container with distilled water, at 25°C, for 24 hours (Figure 1).
- Step #5: optical microscopy investigation. We chose a qualitative surface examination, using a high power light microscopy (100× and 200× magnification), with a

microscope type Neophot 21 (Microstructures Investigations Lab, Research Centre, INTEC SA, Bucharest). We aimed to see the characteristics of the hybrid layer and composite restoration.

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For enamel–composite interface (layering), we partially used the photos/figures from our previous study, but with different interpretations. For dentine–composite interface (bulk-fill), we obtained new images.

### Group #1 (enamel)

# 1.a. Acid etching, adhesive applied by manual brushing (usual technique), and composite in layers

Despite its nearly homogeneous structure, the composite has a sinuous structure towards enamel contact. These bundles express the irregular infiltration of the adhesive (Figure 2a, red area). The brushing clinical technique sweeps the enamel debris and makes a discontinuous hybrid layer.

## 1.b. Acid etching, adhesive applied by manual brushing, and composite in contouring/acetate crown

The analysis of the microscopic morphology aspect after using the composite material in a modeling crown emphasizes slight fissures into the composite material. These fissures are wave-like (undulations) and we consider that because of the polymerization shrinkage. The hybrid layer in this situation is thin and irregular (Figure 2b, circled area).

#### Group #2 (enamel)

# 2.a. Additional preparation with diamond burs (increased retention), acid etching, adhesive applied by manual brushing (usual technique), and composite in layers

Comparing to simple acid etching, the diamond bur enamel preparation has led to an increased mechanical retention for adhesive composite material. The microscopic images of the interface between enamel and dental adhesive show a well-constructed hybrid intermediate layer at the boundary zone between the E and C regions. There are also areas of bad retention at the interface between enamel and layered composite (Figure 3a, circled area). We believe that this is a result of a particular enamel structure: enamel prisms strongly bonded on subjacent structures or possibly prism-less enamel. Instead, the dental composite restoration shows an almost homogeneous structure.

# 2.b. Additional preparation with diamond burs (increased retention), acid etching, adhesive applied by manual brushing composite in contouring/acetate crown

The optical investigation (Figure 3b) revealed a mostly well-configured intermediate hybrid layer. However, there were some areas of bad retention or super positioning (Figure 3b, arrow). The composite restoration showed a homogeneous structure (Figure 3b, C area). Therefore, we believe that the additional enamel preparation has led to an increased adhesive retention. So this clinical method has proven reliable also in the teeth of subgroup 2b.

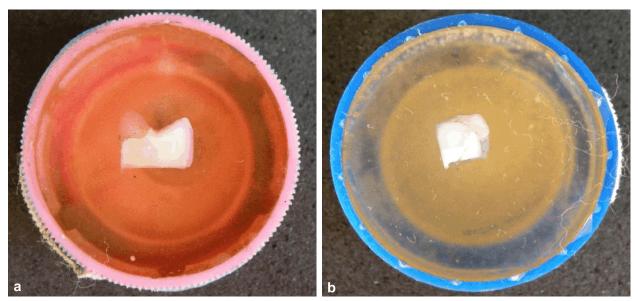


Figure 1 – The appearance of a specimen for microscopic investigation: (a) For enamel [28]; (b) For dentine.

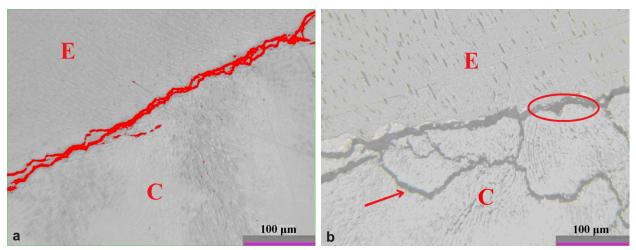


Figure 2 – Interface aspect of demineralized enamel (E) and complex composite (C) with adhesive applied by brushing (100×): (a) Composite in layers; (b) Composite in contouring acetate crown [28].

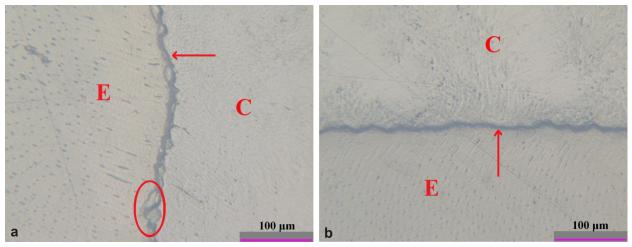


Figure 3 – Interface aspect between diamond bur prepared and demineralized enamel (E) and composite complex (C) following brushing application of adhesive (100×): (a) Composite in layers; (b) Composite in contouring acetate crown [28].

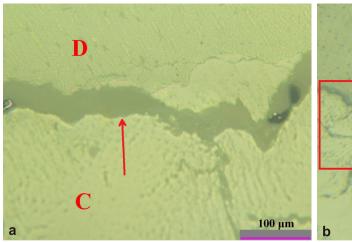
### Group #3 (dentine)

# 3.a. Acid etching, adhesive applied by manual brushing (usual technique), and composite in layers

In this case, we noticed a large dehiscence between dentine, adhesive and composite material restoration (Figure 4a, arrow). In our opinion, this is due to composite polymerization shrinkage. The clinical layering technique was not able to prevent this phenomenon.

## 3.b. Acid etching, adhesive applied by manual brushing, and bulk-fill composite

Using bulk insertion technique led to interesting results. We observed on sections of dentin–composite interface a relatively uniform contact between the two components, at different magnifications –  $100\times$  (Figure 4b) and  $200\times$  (Figure 5). In the same time, we noticed cracks into the composite structure. We believe that is also a result of polymerization shrinkage and a consequence of bulk insertion – an excessive quantity of composite material inserted at once.



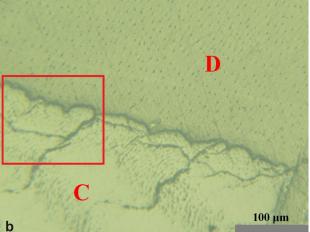


Figure 4 – Interface aspect of dentine (D) and composite complex (C) following brushing application of adhesive (100×): (a) Composite in layers; (b) Bulk-fill composite.

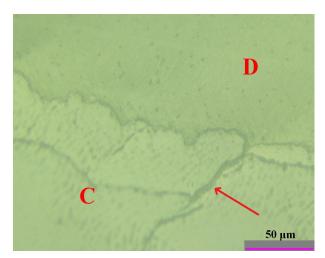


Figure 5 – Interface aspect of dentine (D) and composite (C) following brushing application of adhesive for bulk-fill composite (200×).

### **₽** Discussions

Generally speaking, in terms of dental adhesion, two different materials can be put together resulting a new "product" – the *composite system*. This new system will have three elements: (*i*) the tooth surface (represented by enamel and/or dentine) as a substrate; (*ii*) the adhesive; and (*iii*) the resin composite. Instead, unlike its components, this new system will have other properties and behaviors [23, 24].

In dental adhesive therapy, the main problems faced

are polymerization shrinkage and consequently accumulated stresses.

The dental enamel and dentine have different behaviors to acid etching. In the same time, composite material application will be different for intact enamel, prepared enamel or dentine. That will modify the adhesive operating protocols for each dental hard tissue.

The enamel demineralization leads to micromechanical retention. This can be precedent increased by clinical enamel cutting. In this way, the treatments for increasing enamel surface retention could lead to reliable adhesive structures, but there are many authors that consider these conditions to be insufficient [8].

Adhesion to dentine involves especially a hybrid layer. This particular layer has quality properties linked to adhesion strength and sealing capacity.

The use of contouring acetate crown on enamel could be similar to composite bulk-fill insertion for dentine.

On enamel, the acetate-contouring crown emphasized some advantages: the constant operator's hand/finger pressure through modeling crown could lead to an evenly distributed composite. In optical microscopy terms, the images revealed a more uniform hybrid layer besides stratification technique. The acid attack on enamel surface (acid etching) or special surface preparation with diamond burs are also increasing reliability factors.

On the other hand, in case of dental dentine and prepared restoration cavities, the layering method is more accurate. It can prevent discontinuities between the indications of the dental composite material and where it is used. Despite of its many disadvantages presented by

different authors, such as difficulty of placing the multiple increments, consecutive increased working time or polymerization shrinkage and marginal leakage [25], when it is performed in an appropriate manner, the layering technique can prevent the polymerization shrinkage acting like a buffer through its architecture. Of course, the cavity configuration factor definitely plays a role. This is why various incremental techniques are still used, such as: faciolingual layering (vertical), gingiva—occlusal layering (horizontal), three site technique, wedge-shaped layering (oblique), successive cusp build-up technique, bulk technique, and centripetal build-up [26].

Direct composite materials present certain advantages for dental clinical operative, relying on their physical structure and mechanical properties [27, 29, 30]. They also have disadvantages, mainly represented in less aesthetic than other restorative materials (e.g., ceramics) or in less mechanical resistance (e.g., dental alloys).

There are contradictory results in the studies performed on bulk-fill composites, about polymerization shrinkage, gap formation, and voids [31, 32], so we recommend the bulk-fill composite insertion technique when the operator knows very well what kind of composite he/she is using, when the dental restoration is limited to one dental tissue (in the most cases – the dental enamel), or when the restored dental structure is not submitted to excessive occlusal forces.

The uneven pressure of the modeling instrument (oral spatula) leads to the apparition of "microbumps" (undulations/waves) into the composite layers. It is an operator sensitive factor. It is relatively difficult for a clinician to maintain a quasi-uniform thickness of light-curing composite resin (1.5–2 mm) [33], to respect the standardized terms for a complete polymerization of the composite. In our study, we intended to use the contouring crowns or bulk insertions as supplementary techniques and/or alternative methods to modeling instruments.

### → Conclusions

The optic microscopy allowed us to observe quite pertinent the morphological aspects of the dental hard tissues and the composite material interface. We conclude that the designing and effective build-up of dental composite restorations are often realized on damaged dental substrate. Even we obtain a very good adhesion, the life of a dental restoration is tributary to a sound foundation. Layering technique is useful in case of large dentine restorations whereas the bulk insertion could achieve structural internal defects. In case of enamel, using contouring crowns could produce more accurate restorations.

### **Conflict of interests**

The authors declare that they have no conflict of interests.

### **Authors' contribution**

Ruxandra-Ilinca Matei and Emilia Albiniţa Cuc are authors with equal contributions to the study and the publication.

All authors of this research paper have directly participated in the planning, execution, or analysis of this study, and also all authors of this paper have read and approved the final version submitted.

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