Bond strength and interfacial morphology of orthodontic brackets bonded to eroded enamel treated with calcium silicate–sodium phosphate salts or resin infiltration

Aline Costenoble, Elsa Vennat, Jean-Pierre Attal, Elisabeth Dursun

Original Article

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Aline Costenoble[a]; Elsa Vennat[b]; Jean-Pierre Attal[c]; Elisabeth Dursun[d]

ABSTRACT

Objective: To investigate the shear bond strength (SBS) of orthodontic brackets bonded to eroded enamel treated with preventive approaches and to examine the enamel/bracket interfaces.

Materials and Methods: Ninety-one brackets were bonded to seven groups of enamel samples: sound; eroded; eroded+treated with calcium silicate–sodium phosphate salts (CSP); eroded+infiltrated by ICON®; eroded+infiltrated by ICON® and brackets bonded with 1-month delay; eroded+infiltrated by an experimental resin; and eroded+infiltrated by an experimental resin and brackets bonded with 1-month delay. For each group, 12 samples were tested in SBS and bond failure was assessed with the adhesive remnant index (ARI); one sample was examined using scanning electron microscopy (SEM).

Results: Samples treated with CSP or infiltration showed no significant differences in SBS values with sound samples. Infiltrated samples followed by a delayed bonding showed lower SBS values. All of the values remained acceptable. The ARI scores were significantly higher for sound enamel, eroded, and treated with CSP groups than for all infiltrated samples. SEM examinations corroborated the findings.

Conclusions: Using CSP or resin infiltration before orthodontic bonding does not jeopardize the bonding quality. The orthodontic bonding should be performed shortly after the resin infiltration. (*Angle Orthod.* 2016;86:909–916.)

KEY WORDS: Bonding; Erosion; Enamel

[a] Graduate Orthodontic Resident, Department of Orthodontics, Unité de Recherches en Biomatériaux, Innovations et Interfaces, Université Paris Descartes, Montrouge, France; and Hôpital Albert Chenevier, Créteil, France.

[b] Associate Professor, MSSMat, CNRS, CentraleSupélec, Université Paris-Saclay, Châtenay-Malabry, France.

[c] Associate Professor, Department of Dental Materials, Unité de Recherches en Biomatériaux, Innovations et Interfaces, Université Paris Descartes, Montrouge, France; and Hôpital Charles Foix, Ivry-sur-Seine, France.

[d] Associate Professor, Department of Dental Materials, Unité de Recherches en Biomatériaux, Innovations et Interfaces, Université Paris Descartes, Montrouge, France; Hôpital Albert Chenevier, Créteil, France; and MSSMat, CNRS, CentraleSupélec, Université Paris-Saclay, Châtenay-Malabry, France.

Corresponding author: Dr Elisabeth Dursun, Faculté de Chirurgie Dentaire, 1 rue Maurice Arnoux 92120 Montrouge, France
(e-mail: elisabethdursun@gmail.com)

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INTRODUCTION

Dental erosion comprises an acid-induced wear, without the involvement of bacteria. In the first stages, a superficial demineralization characterized by a softening of the enamel surface occurs, with subsequent wear occurring layer by layer until the erosion reaches the dentin, in more advanced stages.[1] The softened surface is also more susceptible to mechanical stress, such as that involved in tooth brushing or bracket debonding, that may contribute to the loss of tooth structure.

The increased consumption of acid drinks and food has resulted in a rising prevalence of erosive tooth wear. Gastroesophageal reflux and eating disorders also lead to erosive lesions. According to several recent studies,[2–5] erosion affects at least 30% of the population and about half of adolescents.

The best way to stop the progression of erosive lesions is to reduce the direct contact of exogenous or endogenous acids with the tooth surface.

Table 1. Materials, Manufacturers, Batch Numbers, and Chemical Composition of Each Products, Solution, or Device Used

Materials	Manufacturer	Composition
Regenerate Enamel Science™	Unilever	Calcium silicate–sodium phosphate–fluoride-based system
Icon®	DMG	TEGDMA-based resin
Experimental infiltrant resin	DMG	Bis-GMA– and Bis-EMA–based resin
Scotchbond™ Universal etchant	3M ESPE	Orthophophoric acid (37%), water (50–60%), synthetic amorphous silica, polyethylene glycol, aluminum oxide
Cavity conditioner	GC	Distilled water (77%), polyacrylic acid (20%), aluminum chloride hydrate (3%)
Ortho Solo™ Primer	Ormco	Alkyl dimethacrylate resins (60–80%), barium aluminoborosilicate glass (14–24%), fumed silica (2–10%), sodium hexafluorosilicate (1–5%)
Transbond™ XT	3M, Unitek	Silane-treated quartz (70–80%), bisphenol A diglycidyl ether dimethacrylate (10–20%), bisphenol A bis(2-hydroxyethyl ether) dimethacrylate (5–10%), silane-treated silica (<2%)
Microarch®	Gac, Dentsply	Metal orthodontic brackets

TEGDMA = Triethyleneglycol dimethacrylate.
Bis-GMA = Bisphenylglycidyl dimethacrylate.
Bis-EMA = Ethoxylated bisphenol-A dimethacrylate.

However, this is not always possible, and noninvasive strategies have been suggested to inhibit enamel loss at early stages.

One chemical approach consists of increasing the enamel resistance by mineral precipitations. Several formulations with fluoride and/or other beneficial agents have been suggested, providing relative protection (as stannous fluoride, titanium tetrafluoride, casein phosphopeptide–amorphous calcium phosphate, arginine, chitosan and tin). Recently, a novel calcium silicate–sodium phosphate–based system (CSP) has been reported[6–8] to reharden acid-challenged enamel to a greater extent. Another mechanical approach suggests the penetration of and protection against incipient erosive lesions using a resin infiltration technique,[9,10] in the same way that infiltration of noncavitied carious lesions may stop the diffusion of bacteria.[11]

Adolescents are most frequently affected by initial erosion and, thus, are most frequently exposed to preventive treatments, and this age group constitutes the candidates for orthodontic treatment. Few studies have focused on orthodontic bonding to eroded enamel or to eroded enamel strengthened by chemical or mechanical approaches.

This study aimed to investigate the shear bond strength (SBS) and failure mode of orthodontic brackets on eroded enamel subjected to applications of CSP toothpaste/gel or to resin infiltration. The enamel/bracket interfaces were also examined using scanning electron microscopy (SEM).

MATERIALS AND METHODS

Sample Preparation

Ninety-one freshly extracted human teeth were collected, cleaned of soft tissue, stored at 4°C in a solution of 1% chloramine, and used within 3 months. The teeth were intact and extracted for reasons unrelated to the objectives of this study, with the patients' informed consent. The project was approved by the scientific council of the Faculty of Dental Surgery of Paris Descartes University.

Tooth roots were removed with a cutting machine, and crowns were then roughened on their buccal surface with water-cooled sandpaper (400, 800, and 1200 grit) using a polishing machine (Planopol 3, Struers, Kobenhavn, Denmark) to obtain a plane enamel surface. These residual crowns were embedded in self-cured acrylic resin (Plexcil-Escil, Chassieu, France), exposing the flat enamel surface. Each sample was inspected under 40× magnification to ensure that the enamel was intact and free of debris.

Samples were then randomly allocated into seven groups ($n = 13$). For each group, 12 teeth were used for SBS testing and one tooth for SEM observation.

Groups Tested

The materials used and their compositions are given in Table 1.

Enamel Erosion

Except for the control group (group 1), all teeth were exposed to erosion challenge (immersion in a lemon juice solution for 15 minutes at pH 2.6), then rinsed with water for 5 seconds and subjected to a remineralization procedure (storage in human saliva for 30 minutes—the saliva of the operator, taken at least 3 hours after a meal), then rinsed with water for 5 seconds. The enamel treatments are summarized in Table 2.

Five enamel surface conditions and two time delays for bracket bonding after resin infiltration were studied, with groups designated as follows:

Table 2. The Different Procedures of Enamel Erosion Treatment

Group	Abbreviation	Enamel Treatment Procedures
Group 1	SE	No erosion Bracket bonding (etching with phosphoric acid)
Group 2	EE	Enamel erosion (demineralization, remineralization) Bracket bonding
Group 3	REG	Enamel erosion (demineralization, remineralization) First application of Regenerate™ toothpaste (toothpaste/water in 1:2 ratio) for 1 min, then rinsing for 5 s; Remineralization with saliva for 6 h in a stove at 37°C, then rinsing for 5 s. Second application of Regenerate™ toothpaste (toothpaste/water in 1:2 ratio) for 1 min, then rinsing for 5 s; Application of Regenerate™ serum (serum NR5/enhancer 1:1 ratio) for 3 min, then rinsing for 5 s. Remineralization with saliva for 18 h in a stove at 37°C, then rinsing for 5 s All of these steps were repeated the two following days Bracket bonding
Group 4	IRI	Enamel erosion (demineralization, remineralization) Icon infiltration: application of polyacrylic acid (Cavity Conditioner®) for 15 s, then rinsing for 5 s and air-drying for 5 s. Application of Icon Dry for 30 s, then air-drying for 10 s. Application of Icon infiltrant for 3 min, then light-curing for 40 s. Application of Icon infiltrant for 1 min, then light-curing for 40 s Bracket bonding
Group 5	IRI+1	Enamel erosion (demineralization, remineralization) Icon infiltration: application of polyacrylic acid (Cavity Conditioner®) for 15 s, then rinsing for 5 s and air-drying for 5 s. Application of Icon Dry for 30 s, then air-drying for 10 s. Application of Icon infiltrant for 3 min, then light-curing for 40 s. Application of Icon infiltrant for 1 min, then light-curing for 40 s Bracket bonding 1 mo after
Group 6	ERI	Enamel erosion (demineralization, remineralization) Experimental resin infiltration: application of polyacrylic acid (Cavity Conditioner®) for 15 s, then rinsing for 5 s and air-drying for 5 s. Application of Icon Dry for 30 s, then air-drying for 10 s. Application of the experimental infiltrant resin for 3 min, then light-curing for 40 s. Application of the experimental infiltrant resin for 1 min, then light-curing for 40 s Bracket bonding
Group 7	ERI+1	Enamel erosion (demineralization, remineralization) Experimental resin infiltration: application of polyacrylic acid (Cavity Conditioner®) for 15 s, then rinsing for 5 s and air-drying for 5 s. Application of Icon Dry for 30 s, then air-drying for 10 s. Application of the experimental infiltrant resin for 3 min, then light-curing for 40 s. Application of the experimental infiltrant resin for 1 min, then light-curing for 40 s Bracket bonding 1 mo after

1. group 1: sound enamel (SE);
2. group 2: eroded enamel (EE);
3. group 3: eroded enamel treated with application of CSP (REGENERATE Enamel Science™) (REG);
4. group 4: eroded enamel treated with Icon® resin infiltration (IRI);
5. group 5: eroded enamel treated with Icon® resin infiltration, with bracket bonding 1 month after infiltration (IRI+1);
6. group 6: eroded enamel treated with an experimental infiltrant (ERI); and
7. group 7: eroded enamel treated with an experimental infiltrant, with bracket bonding 1 month after infiltration (ERI+1).

Bracket Bonding

Enamel surface was etched with 37% phosphoric acid gel (Scotchbond™, Universal Etchant, eM ESPE, St Paul, Minn, USA) for 30 seconds, washed with water spray for 10 seconds, and dried with an air syringe until a chalky white appearance was obtained. The adhesive (OrthoSolo™ Primer, Ormco, Orange, Calif) was applied with a microbrush and air-thinned with a gentle air blow. A small amount of bonding resin (Transbond™ XT, 3M Unitek, Monrovia, Calif) was placed onto the bracket, which was positioned on the flat surface and pressed using a shure. Excess resin were removed and light-curing was performed (Radii Plus, SDI, Victoria, Australia) for 40 seconds (20 seconds in the cervical direction and 20 seconds in the occlusal direction).

For groups 1, 2, 3, 4, and 6, the bonding procedure was carried out just after enamel surface treatment, whereas for groups 5 and 7, it was performed 1 month after infiltration.

SBS Testing and Failure Mode Determination

SBS was tested with a universal testing machine (LRX, Lloyd Instruments, Fareham, UK). The shear force was applied at the enamel/bracket interface with a chisel-shaped blade parallel to the enamel surface. A cross-head speed of 0.5 mm/min was chosen.

The debonded specimens were observed in a binocular microscope (Olympus Europe SZH10, Hamburg, Germany) and scored according to the adhesive remnant index (ARI) to define the site of bond failure.[12] The scores range from 0 to 3 and are defined as follows: 0 = no adhesive remained on the enamel surface; 1 = less than half of the adhesive remained on the enamel; 2 = more than half of the adhesive remained on the enamel; and 3 = all of the adhesive remained on the enamel, with an impression of the bracket base.

SEM Examination

One specimen from each group was sectioned perpendicularly to the bonded interface using a low-speed diamond saw (Isomet, Buehler, IL, USA)

Table 3. Mean and Standard Deviations of Shear Bond Strength (SBS) for the Various Groups Tested[a]

Group	SBS. MPa
SE	21.1 ± 7.8 AB
EE	26.2 ± 8.6 A
REG	19.2 ± 5.4 B
IRI	20.4 ± 5.0 AB
IRI+1	16.6 ± 5.7 B
ERI	21.9 ± 7.9 AB
ERI+1	16.1 ± 6.4 B

[a] Values with the same small capital letter are not significantly different at $P < .05$. Definitions for groups are located in Table 2.

with water cooling, as near as possible to the center of the bracket.

The sections obtained were polished with abrasive discs of decreasing grit size (400-, 800-, 1200-, 2400-, and 4000-grit silica-carbide), followed by diamond particles of 3.1 and 0.25 μm. The samples were cleaned after each polishing step by ultrasonication in ethanol. A 15-second acid attack in 0.1% hydrochloric acid was carried out, followed by dehydration in a 100% ethanol bath for 2 hours. The samples were then placed in a solution of hexamethyldisilazane for 10 minutes and were left to dry in the open air.

The samples were then placed in a brass test sample-holder with a conducting solution (silver lacquer), rendered conductive by metallization for 3 minutes, forming a layer of gold of approximately 20 nm and deposited under vacuum using a metallizer (Sputter Coater SC 500 Bio Rad Microscience Division Elexience, Verrieres-le-buisson, France). SEM (Jeol, JSM 6400, Tokyo, Japan) observations were undertaken with 1000× and 3000× magnifications. The voltage used was 15 kV, and the working distance was 9 to 13 μm.

Statistical Analysis

The assumptions of equality of variances and normal distribution of errors were checked for all of the variables tested. An analysis of variance and Tukey post hoc tests were performed for statistical comparisons of SBS values. The ARI values were analyzed with the Fisher's exact test. In all tests, the significance level chosen was at $P < .05$.

Table 4. Adhesive Remnant Index (ARI) Scores for the Various Groups Tested[a]

Group	No. of Samples	ARI 0	ARI 1	ARI 2	ARI 3
SE	12 A	0	6	4	2
EE	12 AB	0	1	8	3
REG	12 B	0	0	5	7
IRI	12 C	9	1	1	1
IRI+1	12 ABC	6	0	2	4
ERI	12 C	7	1	3	1
ERI+1	12 C	6	6	0	0

[a] Values with the same small capital letter are not significantly different at $P < .05$. Definitions for groups are located in Table 2.

RESULTS

SBS and Failure Analysis

SBS values for all experimental groups are summarized in Table 3. There were no significant differences in bonding to sound (21.1 MPa) and eroded enamel (26.2 MPa), despite the higher value on eroded enamel. The SBS value of the REG group (19.2 MPa) showed no significant difference when compared to that of sound enamel, but this value was significantly lower than that associated with eroded enamel. The SBS value of infiltrated samples, the IRI (20.4 MPa) or ERI groups (21.9 MPa), showed no significant differences with sound or eroded enamel. When the bonding was delayed after infiltration, the SBS values of the IRI+1 (16.6 MPa) or ERI+1 groups (16.1 MPa) showed no significant differences compared with the SBS values of the IRI, ERI, or ES groups; however, the values were lower and also significantly different than the SBS values for the ER group.

Significant differences in ARI values were observed between the various groups (Table 4): the SE, EE, and REG groups presented significantly more high scores

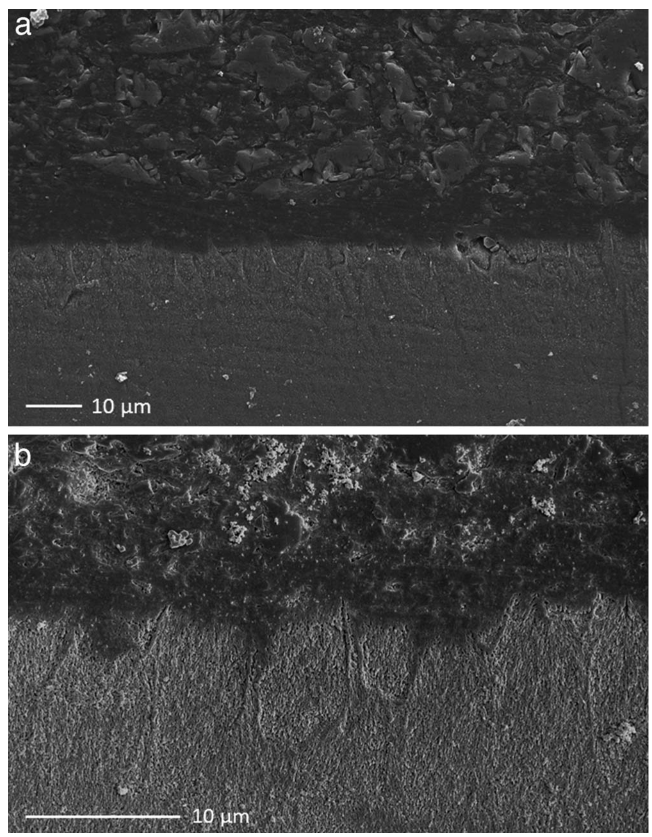

Figure 1. SEM of the interface between sound enamel and adhesive (bracket not visible) at 1000× (a) and 3000× (b) magnification.

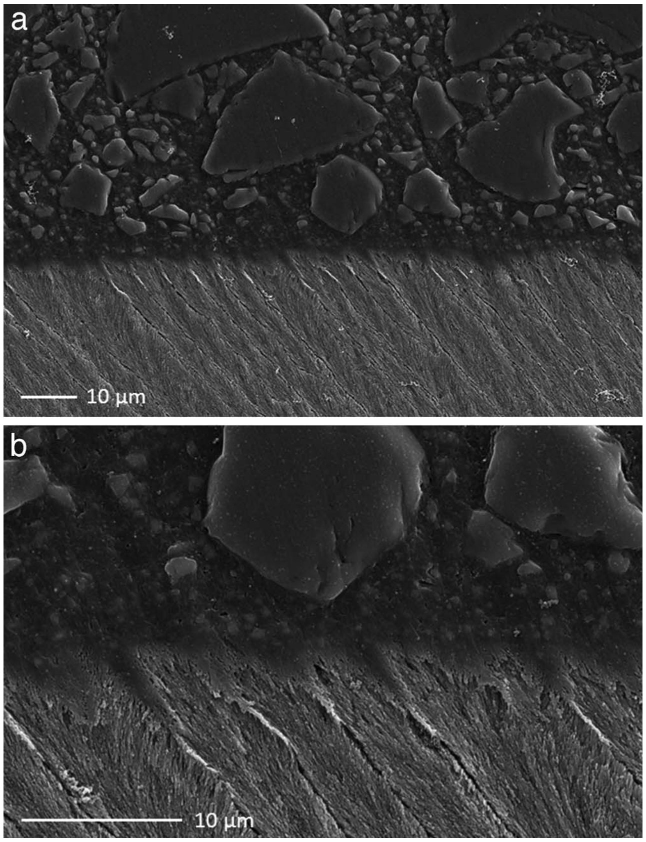

Figure 2. SEM of the interface between eroded enamel and adhesive (bracket not visible) at 1000× (a) and 3000× (b) magnification.

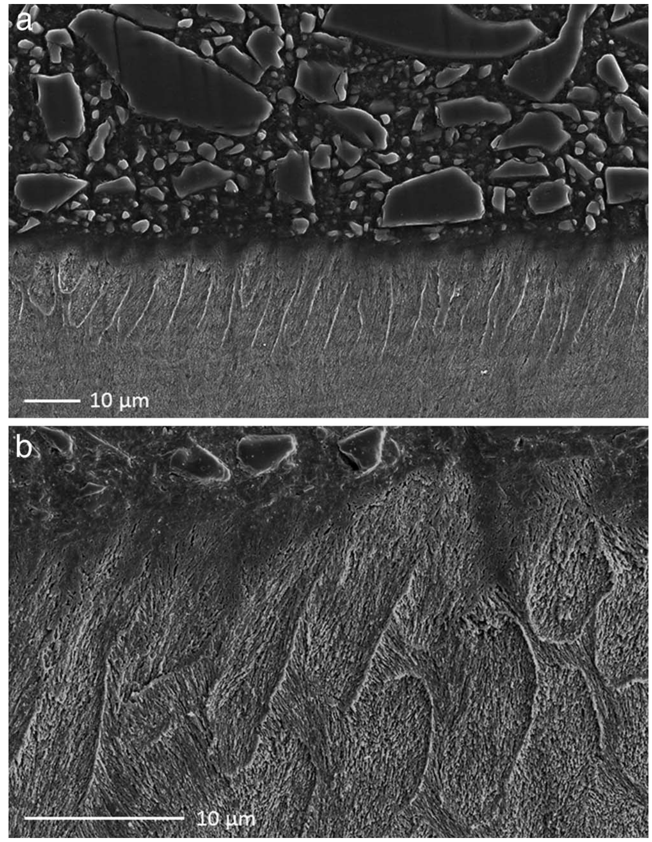

Figure 3. SEM of the interface enamel/adhesive (bracket not visible) for the REG group at 1000× (a) and 3000× (b) magnification.

(ARI values of 2 and 3, and no 0 values), while all infiltrated samples (IRI, ERI, IRI+1, ERI+1 [regardless of the resin type and the moment of bonding]) presented more low scores (ARI values of 0 or 1). No cohesive failures in enamel were registered.

SEM Examinations

Figures 1 to 7 show, respectively, the SEM images for the groups tested, with 1000× ("a" values) and 3000× magnifications ("b" values).

The control pattern (ES) showed uniform and regular resin tags in thickness and in depth. Regardless of the enamel surface treatment, an intimate contact between the substrate and the bonded bracket was found.

SEM images of SE, EE, and REG groups (Figures 1 through 3) presented a homogeneous hybrid layer and regular tags. The enamel surface seemed rougher for the EE and REG groups. SEM images of the IRI and ERI groups (Figures 4 and 6) presented a homogeneous infiltrant penetration covering the enamel surface and well copolymerized with the adhesive, whereas SEM images of the IRI+1 and ERI+1 groups (Figures 5 and 7) presented a homogeneous infiltrant penetration, but without covering the enamel surface. In addition, gaps were observable between the infiltrant and the adhesive.

DISCUSSION

The measures to strengthen eroded enamel imply microstructural changes, which might jeopardize orthodontic bonding. Few studies have focused on the bonding to the reinforced eroded enamel.

Demineralizing Agent and Remineralization Solution

To simulate initial enamel erosion, samples were immersed in lemon juice for 15 minutes and then in saliva for 30 minutes. Lemon juice was chosen because citric acid is usually found in acidic soft drinks and is the main cause of erosion because of its high erosive potential.[13] The saliva aimed to reflect the clinical conditions, with remineralization after the acid exposure. Indeed, Meurman et al.[14] showed less erosion for samples exposed to an erosive solution when they were stored in saliva. The soaking times were based on the fact that the consumption of acidic beverages might take about 15 minutes and that

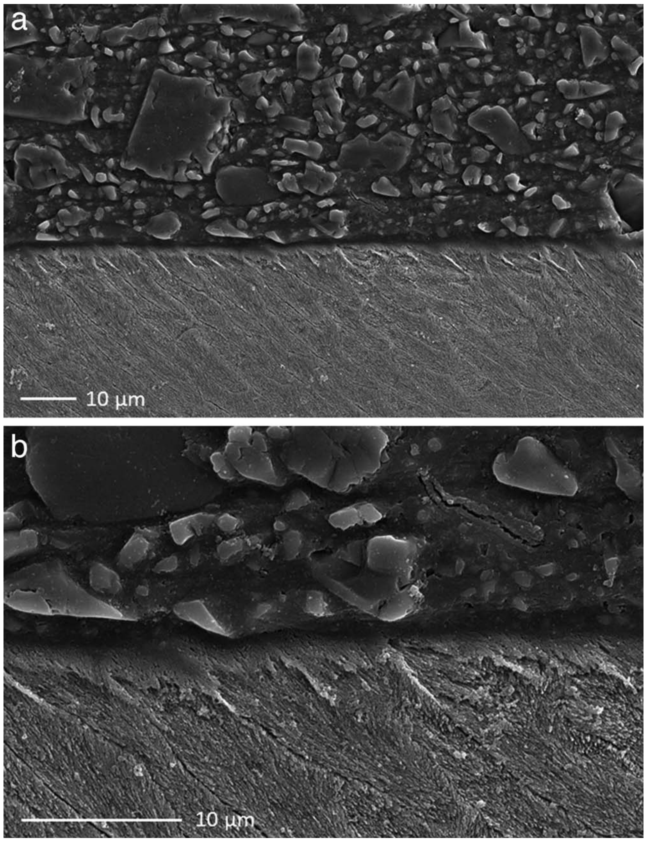

Figure 4. SEM of the interface enamel/infiltrant/adhesive (bracket not visible) for the IRI group at 1000× (a) and 3000× (b) magnification.

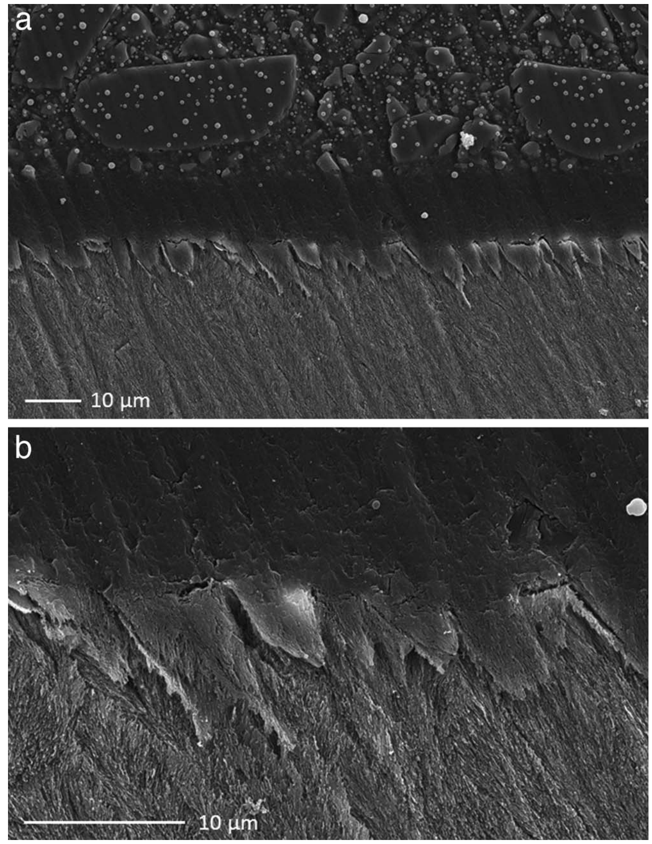

Figure 5. SEM of the interface enamel/infiltrant/adhesive (bracket not visible) for the IRI+1 group at 1000× (a) and 3000× (b) magnification.

30 minutes would be the time necessary for saliva to achieve remineralization of the softened surface layer.[15] Finally, human saliva was used to best mimic the oral environment.

Protocol of Resin Infiltration

The Icon® protocol begins with an etching step of hydrochloric acid at 15% for 2 minutes. This application causes a strong demineralization (≈58 µm),[16] higher than that associated with phosphoric acid at 37% (≈18 µm) and, in turn, higher than that of citric acid. Eroded enamel exhibits crevices likely to be penetrated by infiltrant resin. de Olivera et al.[10] have also shown that infiltration without enamel etching can penetrate and protect enamel from dental erosion. Given these data, and in order to be noninvasive in our treatment, we chose to replace hydrochloric acid with polyacrylic acid for cleaning the substrate and optimizing the resin penetration. In addition, the softened enamel layer might be penetrated by resin infiltration.

SBS Testing

There were no significant differences between SBS values on sound and eroded enamel, even if the value was higher on eroded enamel. These results support those of Navarro et al.,[17] who reported no significant differences in SBS values between sound and eroded enamel and whose results are in line with those of Lenzi et al.,[18] who reported an increased bond strength of etch-and-rinse adhesives to eroded enamel. The erosion process may result in higher mineral loss with deeper demineralized layer, which may contribute to higher SBS values. However Casas-Apayco et al.[19] reported lower bond strength on eroded enamel, but they focused on eroded enamel without saliva remineralization.

The SBS values of the REG group showed no significant differences compared to that of sound enamel, but these values were significantly lower than the SBS values on eroded enamel. Sun et al.[6] claimed that calcium silicate can be deposited onto enamel surfaces and then form hydroxyapatite. Thus, CSP may contribute to recovering a structure that is close to sound enamel. Phosphoric acid application could also remove this thin deposit layer (≈750 µm). The CSP may be used without compromising the orthodontic bonding.

Using infiltration just before orthodontic bonding did not significantly changed the SBS values compared to

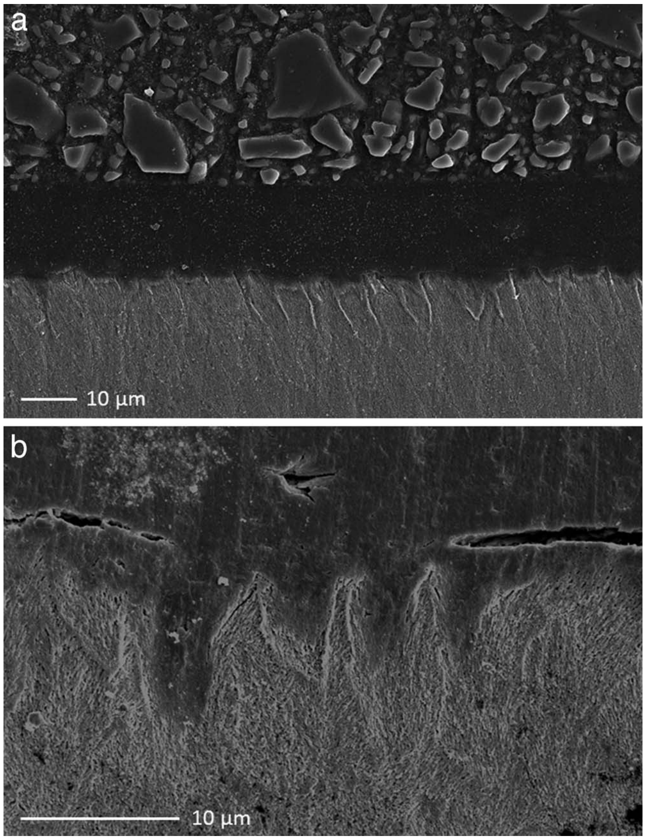

Figure 6. SEM of the interface enamel/infiltrant/adhesive (bracket not visible) for the ERI group at 1000× (a) and 3000× (b) magnification.

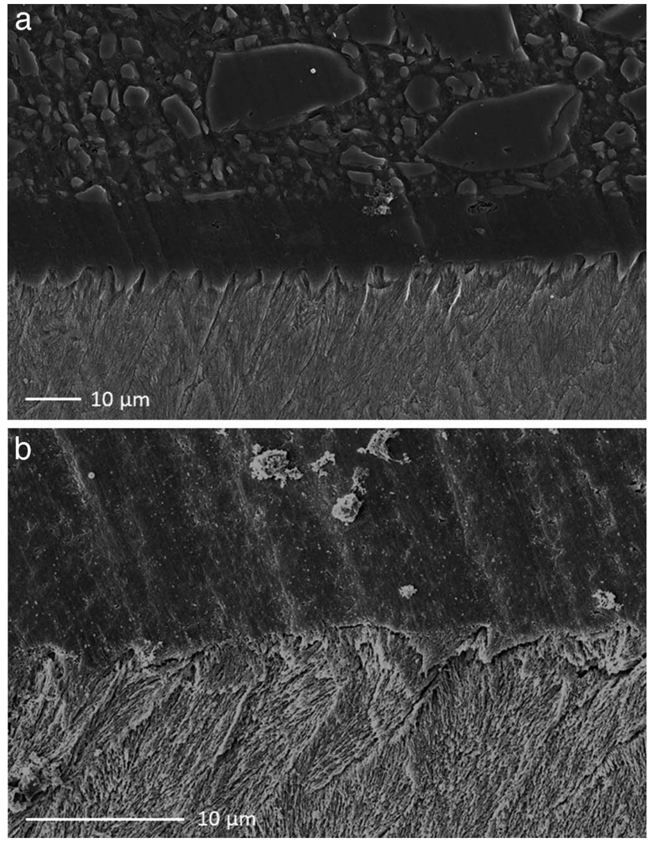

Figure 7. SEM of the interface enamel/infiltrant/adhesive (bracket not visible) for the ERI+1 group at 1000× (a) and 3000× (b) magnification.

those obtained for sound or eroded enamel, regardless of the resin used. Moreover, Hammad and Enan[20] showed that Icon® infiltration improved the SBS values of bonded brackets submitted to acidic soft drinks. However, when the bonding was delayed, SBS was lower compared to that of immediate bonding and of bonding to sound enamel, and significantly lower than that of eroded enamel. Thus, orthodontic bonding should be performed immediately or shortly after resin infiltration. Sandblasting of the infiltrated enamel surface could improve the SBS. No previous studies have investigated the effect of the bonding delay. Other studies on the long-term aging of these resins should be undertaken. However, all of these values could be acceptable for reliable orthodontic bonding.[21,22] The longest period between infiltration and bonding was 56 hours.[23]

Failure Analysis

The predominant occurrence of quite-high ARI scores for the SE, EE, and REG groups, in particular for the EE and REG groups, underlines the bonding effectiveness related to the irregularities of surface, which may promote interlocking to enamel. These results corroborate those of Navarro et al.,[17] who reported no significant differences in failure mode between sound and eroded enamel, with a slight increase for erosion with Coca-Cola.

The low ARI scores for all of the groups treated by infiltration, regardless of the resin type or the bonding delay, suggest less interlocking with enamel. It was not possible, however, to know whether the failure occurred in the infiltrant, between the infiltrant and the composite, or in the composite.

No cohesive failure in enamel were registered. Thus, these preventive treatments have no detrimental effects on orthodontic bonding.

SEM Examination

The SEM images are in accordance with the SBS and ARI results: all groups showed an intimate contact between adhesive and enamel, confirming the reliability of orthodontic bonding.

SEM images of the SE, EE, and REG groups presented a homogeneous hybrid layer and regular tags, with a more pronounced roughness for the EE and REG groups. The CSP could allow reinforcing enamel in restoring its structural composition but not its surface aspect. No previous studies have examined interfaces of adhesive bonded on eroded enamel, nor are there any studies that have focused on enhanced enamel by CSP. Casas-Apayco et al.[19] have studied such interfaces with confocal laser scanning microscopy (CLSM) and reported irregular formation of hybrid layers, related to lower SBS, but they did not consider the benefit of saliva. Oncag et al.[15] have observed the enamel surface after bracket debonding and reported defects around the brackets, at 50 μm from the adhesive/enamel border, due to the protective effect of the adhesive.

SEM images of the IRI and ERI groups presented a homogeneous infiltrant penetration, covering the enamel surface and well copolymerized with the adhesive. No previous studies have examined interfaces of resin infiltration on eroded enamel, but de Olivera et al.[10] showed also the deep penetration of Icon with CLSM. In addition, Hammad and Enan[20] reported a smoother enamel surface after infiltration, almost as sound enamel.

SEM images of the IRI+1 and ERI+1 groups presented a homogeneous infiltrant penetration, but without covering the enamel surface, and gaps were observable between the infiltrant and the adhesive. These phenomena may be explained by the aging.

Further investigation is required to evaluate the SBS values over time. Other adhesive systems, such as self-etching adhesives or resin-modified glass ionomer (RMGI) and other acid attacks using higher erosive challenges may be also tested.

CONCLUSIONS

- Using CSP or resin infiltration to stop the erosion process before orthodontic bonding will not jeopardize the bonding quality.
- The orthodontic bonding should, however, be performed immediately or shortly after resin infiltration.

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