Scientific Article

Fluoridated light-activated bonding resin adhesion to enamel and dentin: primary vs. permanent

Yumiko Hosoya, DDS, PhD Yumiko Kawashita, DDS, PhD Makoto Yoshida, DDS Chikako Suefuji, DDS
Grayson W. Marshall Jr, DDS, MPH, PhD

Dr. Hosoya is an associate professor; Dr. Kawashita is an instructor; Makoto Yoshida is a post graduate student; and Dr. Suefuji, is an instructor, all at the Department of Pediatric Dentistry, Nagasaki University School of Dentistry, Sakamoto, Nagasaki, Japan; and Dr. Marshall Jr. is a professor, Department of Restorative Dentistry, Division of Biomaterials and Bioengineering, University of California, San Francisco, California. Correspond with Dr. Hosoya at hosoya@net.nagasaki-u.ac.jp

Abstract

Purpose: *This study compared fluoridated bonding resin adhesion to primary enamel (Group 1), primary dentin (Group 2), permanent enamel (Group 3), and permanent dentin (Group 4).*

Methods: *The buccal surfaces of 24 primary molars and 24 premolars were used. The bonding system and resin composite used in this study were Imperva Fluorobond® and Lite-Fil II A® (Shofu Inc., Kyoto, Japan). Effects of tooth surface conditioning by FB primer were observed using SEM (N=2/group). Shear bond strengths (SBS) were tested, and the test surfaces of enamel, dentin, and resin specimens were observed using SEM (N=10/group). Data was statistically analyzed using one-way ANOVA with subsequent post hoc Duncan's new multiple range test at* $P<0.05$.

Results: *Effects of tooth surface conditioning by FB primer were appropriate to dentin but low to enamel. Means and standard deviations of the SBS for each group were: Group 1 (16.34 , 5.53 MPa), Group 2 (15.06, 7.02 MPa), Group 3 (14.39, 6.52 MPa) and Group 4 (15.45, 5.35 MPa). There was no significant difference of SBS among Groups 1, 2, 3, and 4.*

Conclusion: *Imperva Fluorobond® system gave the same level of bond strength to primary enamel, primary dentin, permanent enamel, and permanent dentin. (Pediatr Dent 22:101-106, 2000)*

It has been reported by many in vivo and in vitro studies that fluoride-releasing restorative materials may prevent the development of secondary caries in the restored tooth and the initiation of primary caries in adjacent tooth tissue.[1-3] Glass ionomer cements have been used as materials for a fluoride source.[4-6] Resin composites have been effective as restorative materials and the release of fluoride from several fluoride containing composites have been evaluated.[7-13] Several reports have compared the bond strength between primary dentin and permanent dentin using different bonding systems.[14-18] The results have varied with findings indicating no significant difference of bond strength between primary dentin and permanent dentin,[14] significantly lower bond strengths to primary dentin,[15-17] and higher bond strengths to primary dentin.[18] Bond strength to primary dentin and permanent dentin might vary according to the morphological, physiological, and chemical differences between primary and permanent dentin.[19-22] From the clinical perspective, bond strengths to primary enamel and dentin should be equivalent to permanent enamel and dentin.

Recently, Imperva Fluorobond® (Shofu Inc., Kyoto, Japan) a fluoride-releasing bonding resin for composite restoration that contains pre-reacted glass ionomer fillers, was developed. Release of fluoride from this material,[11,13] cavity adaptation to bovine permanent teeth using this material,[23] and bond strengths to bovine permanent enamel and dentin[24-27] or to human permanent enamel and dentin[25,28] using this material have been reported. However, no information on bonding to primary teeth using this material has been reported.

The purpose of this study was to compare the bond strengths to enamel and dentin for human primary and permanent teeth using the Imperva Fluorobond® system.

Methods

Buccal surfaces of 24 caries-free human primary molars and 24 human premolars that had been exfoliated or extracted for orthodontic reasons and frozen in physiologic saline soon after extraction were used. Informed consent was obtained from parents and patients for collecting the teeth. All teeth were used within 6 months of extraction. To obtain flat enamel or dentin surfaces, the buccal surfaces of all teeth were ground with a water-cooled air turbine using a 301 diamond bur (Shofu Inc., Kyoto, Japan) then abraded with 400, 600, 800, and 1000 grit wet silicone carbide papers. The depth of the central area of the dentin specimens was prepared in the middle region between the dentino-enamel junction and pulp chamber wall. After surface preparation, specimens were ultrasonically washed in de-ionized water for one minute.

The teeth were divided into four groups: Group 1—primary enamel; Group 2—primary dentin; Group 3—permanent enamel; Group 4—permanent dentin.

The bonding system and resin composite used in this study were Imperva Fluorobond® and Lite-Fil IIA.

Imperva Fluorobond (FB) is a self-etching primer system. The primer of FB utilizes an adhesive promoting monomer, 4-AET (4-acryloxyethyltrimellitic acid) with 2-HEMA (2-hydroxyethyl methacrylate) and water. The bonding resin of FB utilizes an adhesive promoting monomer, 4-AET, 2-HEMA, UDMA (urethane dimethacrylate), TEGDM (triethylene glycol dimethacrylate), and SiO_2 microfillers. Lite-Fil IIA is a hybrid light-cured resin composite.

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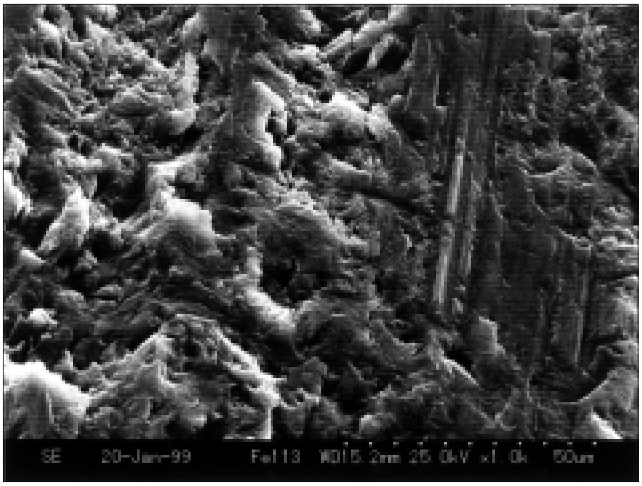

Fig 1. Primary enamel surface primed with FB primer for 10 seconds, gently air dried for 1 second, and then soaked in acetone solution for 2 minutes. The enamel prism structure was only partially evident.

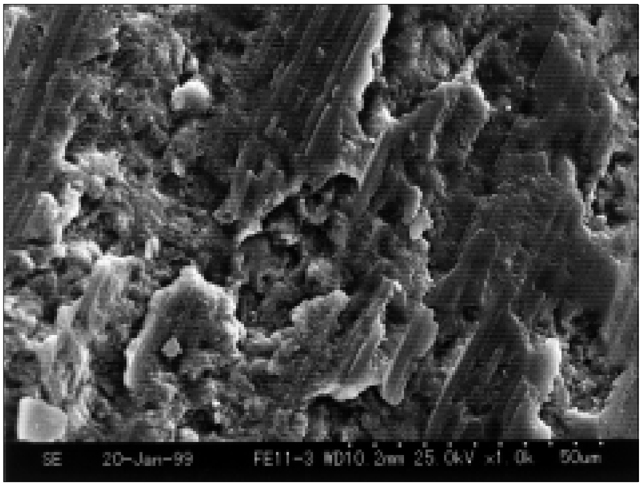

Fig 2. Permanent enamel surface primed with FB primer for 10 seconds, gently air dried for 1 second, and then soaked in acetone solution for 2 minutes. Enamel prism structure was not evident.

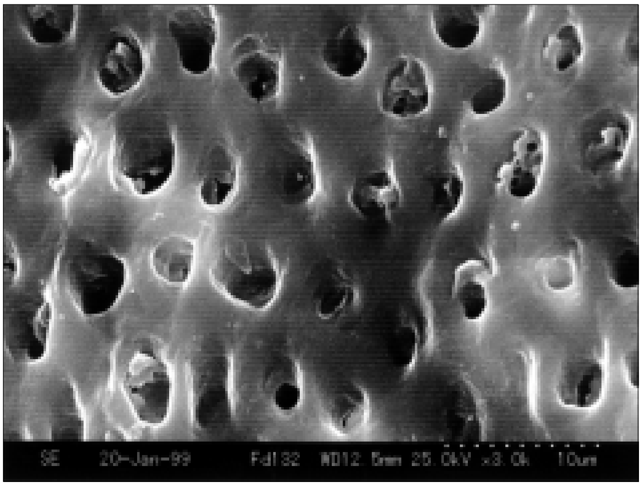

Fig 3. Primary dentin surface primed with FB primer for 10 seconds, gently air dried for 1 second, and then soaked in acetone solution for 2 minutes. Smear layer was removed and dentin tubules were opened.

Efficacy of tooth surface conditioner

The buccal surfaces of 4 primary molars and 4 premolars (N=2/group) were used. FB primer was applied on the enamel or dentin surfaces for 10 seconds and gently air-dried for 1 second. According to the method of previous studies,[27,29] the specimens were soaked in acetone solution for 2 minutes to dissolve the monomer of FB primer. In our pilot study, the primed enamel or dentin surfaces without acetone treatment were covered with the resinous component of FB primer and the structure of the enamel or dentin was unclear. The specimens were dehydrated with 50, 70, 80, 90, 95, and 100% ethyl-alcohol and dried using 100% HMDS (Hexamethyldisilazane). The primed enamel or dentin surfaces were observed by scanning electron microscopy (SEM), Hitachi S-3500N (Hitachi Inc., Tokyo, Japan) following gold coating. The efficacy of FB primer to enamel or dentin was compared with the results of the previous studies,[30-33] in which phosphoric acid was used as the enamel or dentin conditioner.

Bond strength

Buccal surfaces of 20 primary molars and 20 premolars (N=10/group) were used for the bond strength test. All specimens were subjected to a single-plane shear test (SPST) that was designed by Watanabe et al.[34] The sample tooth was held in plate 1 of the SPST with New Plastone® dental stone (GC Co., Tokyo, Japan). Mylar tape was mounted on the enamel or dentin to mask off a circular area 3 mm in diameter. FB primer was applied for 10 seconds and gently air dried for 1 second. FB bond was applied on the primed enamel or dentin and light irradiated for 10 seconds. Then plate 2 of the SPST was attached to plate 1. Two layers of Lite-Fil IIA (shade: A2) resin composite, each 1.5 mm in thickness, were placed on the primed and bonded enamel or dentin through a counter sunk hole of plate 2. Each layer was light cured for 40 seconds. The visible light activation unit used in this study was Visilux 2® (3M Dental Products, St. Paul, MN, USA). All specimens were stored wet in a box at room temperature for 24 hours.

The shear bond strength (SBS) was tested with an autograph DCS-500™ (Shimazu Product Inc., Kyoto, Japan) at a cross-head speed of 2.0 mm/min. All data were statistically analyzed using one-way ANOVA with subsequent to post hoc Duncan's new multiple range test at $P<0.05$.

Fracture mode

After the shear bond strength test, the test surfaces of the enamel, dentin, and resin were observed using SEM. The SEM views were studied under 25x-20,000x magnifications. The modes of fracture were designated: enamel or dentin fracture if 100% of the bonded enamel or dentin was fractured; adhesive fracture if 100% of the bonded interface failed between the enamel/dentin, and the bonding resin; cohesive resin fracture if 100% of the failures was in the resin composite: or mixed fracture if the failures were partially adhesive and partially cohesive resin fracture and/or enamel or dentin fracture.

Resin tag formation was also observed on cross-sectioned samples through the bonded region after SBS testing. The degree of the resin tag formation was classified into 3 categories: ++ if numerous and distinct long resin tags were observed; + if a limited number of short resin tags were observed; and if no resin tags was observed. It was impossible to measure the length of resin tags because fracture of resin tags occurred during SBS

Table 1. Shear Bond Strengths to the Primary Enamel and Dentin and Permanent Enamel and Dentin (Unit:MPa)

Group	Mean(SD)	Number of Cases
Primary enamel	16.34 (5.53)•	10
Primary dentin	15.06 (7.02)•	10
Permanent enamel	14.39 (6.52)•	10
Permanent dentin	15.45 (5.35)•	10

• No significant difference at *P*<0.05.

testing. The relationships between the fracture mode and the SBS were determined according to the previously reported method,[34-36] and the data were analyzed using the chi-square test at *P*<0.05.

Results

Efficacy of tooth surface conditioner

Figures 1-4 are SEM micrographs of the primed enamel or dentin surfaces after soaking in acetone solution for 2 minutes.

Fig 1 shows the primary enamel surface following application of the primer and its removal by dissolution in acetone. The enamel prism structure was only partially evident. Figure 2 shows the permanent enamel surface after treatment. The enamel prism structure was not evident and striations from the surface grinding procedure were apparent. The efficacy of the FB primer for enamel conditioning was uncertain, since the enamel prism structure was not well etched for either primary or permanent enamel.

Fig 3 shows the primary dentin surface following application of the primer and its removal by dissolution in acetone. The smear layer was removed and dentinal tubules were widely opened. Fig 4 shows the permanent dentin surface after treatment. Small particles remained on the dentin, but smear layer was almost removed and dentinal tubules were opened. The FB primer appeared to effectively condition both primary and permanent dentin.

Bond strength

Table 1 shows the SBS testing for the primary enamel, primary dentin, permanent enamel, and permanent dentin groups. There was no significant difference found for the bond strength among any of the groups (ANOVA, *P*<0.05).

Fracture mode

Table 2 shows the observed fracture modes between primary and permanent enamel and the bonded material. Fracture modes that showed the highest percentage were mixed fracture in the primary enamel and adhesive fracture in the permanent enamel. Enamel fracture was observed in only one mixed fracture case in the primary enamel. Cohesive resin fracture was observed for only one sample in each of the primary enamel and permanent enamel. There was no significant difference of the fracture modes between primary enamel and permanent enamel.

Table 3 shows the observed fracture modes between primary and permanent dentin and bonded material. Most cases showed adhesive fracture or mixed fracture. In the primary dentin, dentin fracture was observed in two cases and cohesive resin fracture was observed in one case. There was no significant difference of the fracture modes between primary dentin group and permanent dentin group.

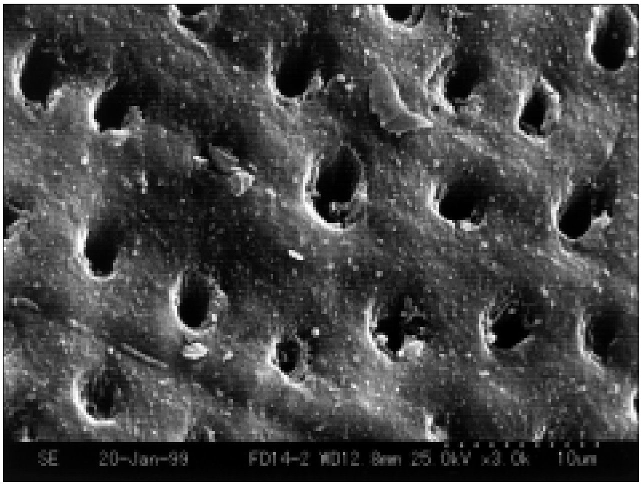

Fig 4. Permanent dentin surface primed with FB primer for 10 seconds, gently air dried for 1 second, and then soaked in acetone solution for 2 minutes. Small particles remained on the dentin, but smear layer was almost removed and dentin tubules were opened.

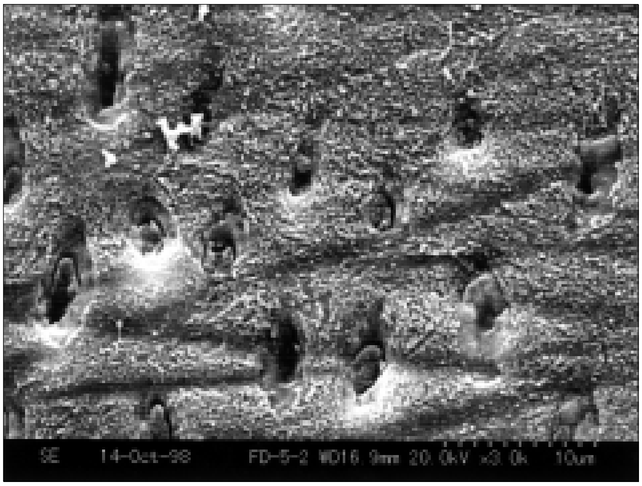

Fig 5. Permanent dentin specimen after SBS test. The bond strength was 28.43 MPa. Mesh-like structure was observed on the intertubule dentin and fracture resin tags remained in many of the dentin tubules.

Fig 5 shows a fracture surface for the highest strength sample from the permanent dentin group after SBS testing (SBS 28.43 MPa). A mesh-like structure was observed in the intertubule dentin and fractured resin tags remained inside many of the dentin tubules. Both for primary and permanent dentin, mesh-

Table 2. Fracture Modes between Enamel and Resin (%)

Group	Enamel Fracture	Adhesive Fracture	Cohesive Resin Fracture	Mixed Fracture	Number of Cases
Primary enamel	1•	3•	1•	6•	10
Permanent enamel	0•	6•	1•	3•	10

• No significant difference at *P*<0.05.

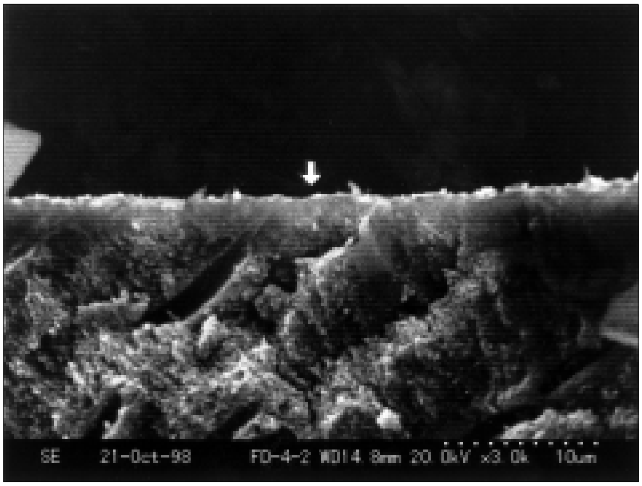

Fig 6. Cross-section of permanent dentin specimen after SBS test. The bond strength was 12.48 MPa. Hybrid layer was about 2 μm thick (arrow).

like or amorphous structures were observed in some of the specimens, but other specimens showed glossy or smooth surface.

Fig 6 shows a typical cross-section through the bonded region from the permanent dentin group after SBS testing. Both for primary and permanent dentin, about 2 μm thick hybrid layer was observed on the dentin surface. Table 4 shows the classification of resin tags formation on the enamel and dentin.

Resin tags were poorly developed for both forms of enamel and almost equally classified into + and - scores. For primary dentin and permanent dentin, resin tags were well developed with 4/10 cases rated ++ for primary dentin and 6/10 cases rated in the best category for permanent dentin. Chi-square testing showed no significant difference using these criteria for resin tag formation for either enamel or dentin between primary and permanent teeth.

Discussion

Resin composites are widely used as restorative materials and the release of fluoride from several of these materials has been evaluated[7-10] based on various fluoride-releasing systems that have been developed. Some formulations adopted a fluoride containing compound as the resin monomer,[38, 39] while others have used a microencapsulated soluble salt[40, 41] or dispersion of sparingly soluble fluoride salts[42] as their inorganic fillers. In this study, a fluoride-releasing bonding resin, Imperva Fluorobond,® which contains pre-reacted glass ionomer fillers was used. Hotta et al.[11] analyzed the fluoride uptake into bovine dentin from Imperva Fluorobond® by electron probe x-ray microanalyzer (EPMA). They reported that 0.77 ppm fluoride released after 30 days and this value was 1/80 of the glass ionomer cement, Base Cement® (Shofu Inc., Kyoto, Japan) used as control. The presence of fluoride was detected about 2-3 μm into the dentin wall that was in contact with Imperva Fluorobond.^{R} Han et al.[13] found sustained fluoride release from Imperva Fluorobond® in contact with human enamel and dentin for up to 2 months, and demonstrated acid resistance of both the enamel and dentin specimens. They concluded that the fluoride-releasing bonding resin was useful to prevent primary and secondary caries.

Tensile (TBS) and shear (SBS) bond strengths to permanent enamel and dentin using Imperva Fluorobond® system have been reported.[24-28] In this study, there was no significant difference of SBS between permanent enamel and dentin (Table 1), in support of TBS findings by Yoshikawa et al.[25] and SBS results by Iwasaki.[27]

Bond strengths to primary enamel and dentin using Imperva Fluorobond® system have not been previously reported. In this study, there was no significant difference of SBS between primary enamel and dentin.

Several reports have compared the bond strength between primary dentin and permanent dentin using other bonding systems.[14-18] The results have varied with findings indicating no significant difference of bond strength between primary dentin and permanent dentin,[14] significantly lower bond strengths to primary dentin,[15-17] and higher bond strengths to primary dentin.[18] In this study, there was no significant difference of SBS between primary enamel and permanent enamel, and primary dentin and permanent dentin. Bond strength to enamel and dentin varies according to several factors. The differences of dentin hardness,[19] dentin permeability,[21] and the degree of mineralization[20, 22] between primary dentin and permanent dentin might influence the bond strength. Depth of dentin influences resin adhesion. It has been shown that the bond strengths of some dentin adhesives decrease with depth from the occlusal dentin to the pulp.[43, 44] In this study, the depth of primary and permanent dentin specimens were similar, but the dental age (degree of calcification and time after eruption) of the primary teeth and permanent teeth differed. However, bond strength tests tend to have relatively high variability and the kinds of bond strength tests and the methods of holding specimens in place during bond strength tests are important factors. In shear bond strength tests, a wide variety of configurations has been used including loops, points, and knife edges to apply the shearing force. Clearly different methods of load application lead to differing stress distributions. The single-plane shear test[34] used in this study avoids applying torque to the specimens during loading as is common with other shear tests.

In comparing the conditioning efficacy of FB primer and that of 10% and 35% phosphoric acid etchant, which were used in our previous studies,[19-22,36,37] there was little difference between FB primer and phosphoric acids. However, for enamel, the efficacy of FB primer was significantly lower than that of phosphoric acids and it was not effective in etching the enamel rods so as to reveal the enamel prism structure. In this study, primed enamel or dentin were soaked in ac-

Table 3. Fracture Modes between Dentin and Resin (%)

Group	Dentin Fracture	Adhesive Fracture	Cohesive Resin Fracture	Mixed Fracture	Number of Cases
Primary dentin	2[*]	3[*]	1[*]	4[*]	10
Permanent dentin	0[*]	5[*]	0[*]	5[*]	10

[*] No significant difference at $P<0.05$.

Table 4. Resin Tags Formation on the Enamel and Dentin

Group	Resin Tags Formation ++	+	-	Number of Cases
Enamel				
Primary enamel	0•	4•	6•	10
Permanent enamel	0•	5•	5•	10
Dentin				
Primary dentin	4•	4•	2•	10
Permanent dentin	6•	2•	2•	10

• No significant difference at *P*<0.05.

etone solution to dissolve the monomer of FB primer so that the underlying partially demineralized calcified tissues could be observed. Since smear layer was removed, it was concluded that the smear layer could be dissolved by the acidic component of the self etching primer. However, this is not the procedure that is used clinically, since the primer remains on the surface and the dissolved smear layer is incorporated into the bonded structure. Thus, the smear layer on primed dentin without acetone treatment might be different from the SEM views in this study, but we carried out this procedure to determine whether or not the self etching primer was capable of dissolving smear layer.

Yoshikawa et al.[25] reported that, in comparison with 10 seconds etching, 15 seconds etching by 7% phosphoric acid did not significantly increase TBS to bovine enamel and dentin mediated by the Imperva Fluorobond® system. However, Shinkai et al.[26] reported that, in comparison with 10 seconds etching, 15 seconds etching by 7% phosphoric acid significantly increased the SBS to bovine enamel and dentin for the same system. Yoshiyama et al.[28] measured the micro-TBS of Imperva Fluorobond® to human cuspid teeth and reported that this bonding system produced good adhesion to dentin by creating a thin hybrid layer and transitional layer, but bonding to enamel needed to be improved.

Our results suggest that the FB primer effectively conditioned dentin but the efficacy of enamel conditioning was low (Figures 1-4) and resin tags were poorly developed for the enamel (Table 4).

Typical fracture modes for primary or permanent enamel or dentin and the resin were adhesive fracture and mixed fracture (Tables 2 and 3), generally in agreement with Shinkai et al.[26] In this study and our previous ones,[33,36,37,45] there was no correlation between the enamel-resin fracture mode and the bond strength in any of the adhesive systems. The Imperva Fluorobond® system exhibited a thin hybrid layer to dentin (Fig 6) and short resin tags to enamel (Table 4).

Miyazaki et al.[24] compared the influence of the primer drying time on SBS to bovine enamel for three self-etching primer bonding systems, including Imperva Fluorobond®. They reported that the SBS to enamel could be influenced by the drying time of the primer with effects that differed among the systems. At short drying times, primer solvents such as water and ethanol might not be removed. Therefore they might act as inhibitors for the polymerization of the bonding agent[46] or may lead to imperfect coverage of the etched enamel surface by the bonding agent and decrease micromechanical retention with a subsequent decrease in bond strength. The benefit of using a self-etching primer, in terms of simplifying the clinical procedure, might be negated by the technique-sensitive factors that can lead to reduced bond strengths. No conclusive evidence for or against a treatment effect of inhibition of secondary caries by the glass-ionomer restorations has been reported.[47] Despite its promise, long-term clinical observation is required to evaluate the effectiveness of this self-etching fluoridated restorative resinous material.

Conclusions

1. Effects of tooth surface conditioning by FB primer were appropriate to dentin but low to enamel.
2. There was no significant difference among the SBS to the primary enamel, permanent enamel, primary dentin, and permanent dentin.

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