

Acid Resistance Induced by a New Orthodontic Bonding System *in vitro*

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The effect of fluoride-containing orthodontic resin bonding systems on acid resistance of adjacent enamel was evaluated using transversal microradiography.

Four fluoride-containing orthodontic resin bonding systems : BeautyOrtho Bond (BO), BeautyOrtho Bond+Salivatect (BOS), Kurasper F (KP), Transbond XT (TB) and a resin-modified glass-ionomer (Fuji Ortho LC (FO)) were used. Superbond Orthomite (SB) was used as a non-fluoride material. Rectangular bovine enamel specimens (10×6 mm) were prepared. After curing the materials, nail varnish was applied to the enamel surfaces, leaving a gap of 1 mm from the cured material's periphery. The specimens were demineralized with 8% Methocel MC gel and 0.1 M lactic acid. BO, BOS, and FO revealed shallow lesions and distinct surface layers. The mineral losses of BO, BOS, and FO were significantly lower than those of TB, KP, and SB ($p < 0.05$). In conclusion, the new system induced superior acid resistance in enamel surrounding orthodontic brackets.

Key words: Demineralization, Microradiography, S-PRG filler

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INTRODUCTION

The direct bonding of orthodontic brackets has been frequently used for the purpose of resolving esthetic problems. On the other hand, there are some clinical concerns: the occurrence of caries or the detachment of brackets during orthodontic treatment. Generally, most orthodontic bonding materials consist of a resin composite based on Bis-GMA formula, and it is indispensable to perform acid etching on enamel¹⁻⁴. However, the direct bonding of orthodontic brackets using acid etching increases the accumulation of plaque around the brackets. This phenomenon consequently increases the risk of caries formation at these sites, since plaque removal is difficult in these areas^{5,6} during the period of orthodontic treatment. It has been indicated that decalcification or white spot lesions often developed around bracket margins within a few weeks^{7,8}.

Many studies have shown irreversible alterations in enamel arising from the retention of resin tags and the potential risk of enamel damage during debonding⁹⁻¹¹. Any remnants of composites around bracket periphery may predispose the patient to decalcification after debonding¹². To circumvent these debonding problems, Hotz *et al.*¹³ and Powis *et al.*¹⁴ recommended citric acid for enamel pretreatment before bracket bonding. In addition, Miguel *et al.*¹⁵ reported that there were no significant differences in bonding strength between citric acid-pretreated and phosphoric acid-pretreated enamel. On the other hand, to resolve these problems in relation to bracket bonding, the development of novel

self-etching primers and bonding systems without the need for acid etching has seized the attention of workers in various dental fields¹⁶⁻¹⁸.

With a view to reducing — and even inhibiting — demineralization around orthodontic brackets without acid etching, various types of glass ionomer cements¹⁹⁻²³ or fluoride-releasing bonding systems^{24,25} have been developed. In this respect, several studies have favorably reported on reduction in demineralization following the use of bonding orthodontic brackets with fluoride-releasing capability^{26,27}. Recently, a new orthodontic adhesive material, BeautyOrtho Bond, has been developed. It consists of a self-etching primer and a fluoride-containing paste. The paste contains Surface Pre-reacted Glass-ionomer (S-PRG) filler. It was reported that a resin composite with an S-PRG filler released fluoride at a higher rate than fluoroaluminosilicate glass filler materials²⁸. Further, S-PRG filler was also found to release inorganic elements such as Al, Si, and Sr²⁹.

The aim of this *in vitro* study was to evaluate the effect of a newly developed fluoride-releasing orthodontic bonding system for the inhibition of demineralization of adjacent enamel in comparison with other bonding systems.

MATERIALS AND METHODS

Materials

Table 1 lists the adhesive materials used in this study. Five of which were fluoride-releasing bonding systems, namely BeautyOrtho Bond (BO), BeautyOrtho Bond+Salivatect (BOS), Kurasper F

Table 1 Adhesive materials used in this study

Trade name	Code	Lot No.	Composition	Manufactures
BeautyOrtho Bond	(BO)			Shofu Inc, Kyoto, Japan
Primer A		03111301	Water, Acetone, Others	
Primer B		03041101	Phosphonic acid monomer, Ethanol, Dyes, Others	
Paste		02040901	Bis-GMA ¹⁾ , TEGDMA ²⁾ , S-PRG filler ³⁾ , Others	
BeautyOrtho Bond +Salivatect	(BOS)			Shofu Inc, Kyoto, Japan
Salivatect ⁴⁾		01041401	Bis-GMA, TEGDMA, S-PRG filler, Others	
Transbond XT	(TB)			3M Unitek Corp., Calif, USA
Transbond Plus Self-etching primer		200089-L5C	Methacrylated phosphoric acid esters, Amino benzoate, Camphorquinone	
Paste		5NA	Bis-GMA, TEGDMA, Silane-treated quartz, Amorphous silica, Camphorquinone	
Kurasper F	(KP)	11241		Kuraray Medical Inc., Tokyo, Japan
K-etchant		00334A	37% phosphoric acid	
F-bond		00033B	TEGDMA, 2-HEMA, Bis-GMA, Methyl methacrylate-methacryloyl fluoride copolymer, Sodium fluoride, Silanated silica filler, Initiators	
Paste		00024E	Bis-GMA, TEGDMA, Silanated glass filler, Initiators	
Fuji Ortho LC	(FO)			GC Corp., Tokyo, Japan
Conditioner		0407231	10% polyacrylic acid	
Powder		0408091	Fluoro-alumino-silicate glass, Pigment	
Liquid		0408041	Polyacrylic acid, Distilled water, 2-hydroxyethyl-methacrylate, Dimethacrylate	
Superbond Orthomite ⁵⁾	(SB)			Sun Medical, Co. Ltd., Moriyama, Japan
Red activator		KK1	65% phosphoric acid	
Powder		KF2	PMMA	
Liquid		KK1	MMA, 4-META	
Catalyst		KG21	Tri-n-butylbolane	

1) Bis-GMA: 2, 2-Bis[4-(2-hydroxy-3-methacryoxypropoxy) phenyl] propane

2) TEGDMA: Triethylene glycol-dimethacrylate

3) S-PRG filler: Surface reaction type pre-reacted glass-inomer filler based on fluoroboroaluminosilicate glass, Paste filler contents: 76.8wt%

4) Salivatect filler contents: 67.8wt%

5) Non fluoride-releasing material

(KP), Transbond XT (TB), and Fuji ORTHO LC (FO). One non-fluoride-releasing system, Superbond Orthomite (SB), was used as a control. BO, BOS, and TB are self-etching adhesive systems, and KP and SB are phosphoric acid-etching adhesive systems. As for FO, it is a resin-modified glass ionomer cement.

BOS and BO paste consist of Bis-GMA, TEGDMA, and 76.8 wt% S-PRG filler. Salivatect consists of the same components as the BOS and BO pastes, except for 67.8 wt% S-PRG filler. Salivatect was used only for the BOS system for the purpose of excluding salivary contamination from the surface of the bonding agent. Details of all the materials used are also listed in Table 1.

Preparation of specimens

A total of 30 extracted defect-free bovine lower incisors were obtained. A diamond-coated wire sectioning machine (Well Type 3242, Walter Ebner, Mannheim, Germany) was used to separate crowns from roots. After the debridement of visible soft tissues, each crown was cut and prepared to sizes of 6 mm in width, 10 mm in length, and 3 mm in thickness. Surfaces of specimens were finished with a 2,000-grit waterproof abrasive paper (Sankyo Rikagaku Co. Ltd., Saitama, Japan) to obtain flat surfaces. Following which, a masking tape of approximately 90 μm thickness with a 2×6 mm window was put on the enamel surface. Thirty enamel samples were thereby obtained and randomly distributed into six experimental groups ($n=5$ per group).

Treatment of experimental surfaces

The experimental surfaces were treated with each

adhesive system according to the manufacturers' instructions. All bonding procedures were performed by the same operator and briefly described as follows:

- For BO, an enamel window was conditioned with an accompanying self-etching primer for three seconds, and then gently air-dried. After the paste was applied, it was cured with a visible light-curing unit (Optilux 400, Demetron Research Corp., USA) for 20 seconds with a pressed polystyrene sheet.
- For BOS, the same primer and paste were used. In addition to the same procedure as for BO, Salivatect was applied to the enamel surface before applying the paste.
- For TB, an enamel window was scrubbed with an accompanying self-etching primer for three seconds, and then gently air-dried. The paste was applied and cured for 10 seconds with a pressed polystyrene sheet.
- For KP, an enamel window was applied with a 37% phosphoric acid for 40 seconds followed by thorough rinsing and drying. After F-bond was applied, the paste was immediately placed on the tooth and cured for 20 seconds with a pressed polystyrene sheet.
- For FO, 10% polyacrylic acid was applied for 20 seconds followed by thorough rinsing and drying. A mixture of powder and liquid was placed on the tooth and cured for 40 seconds with a pressed polystyrene sheet.
- For SB, 65% phosphoric acid was applied for 30 seconds followed by thorough rinsing and drying. The liquid, catalyst, and powder were mixed, and then the mixture was placed on the tooth and chemically cured for eight minutes.

After curing, the thickness of all materials was

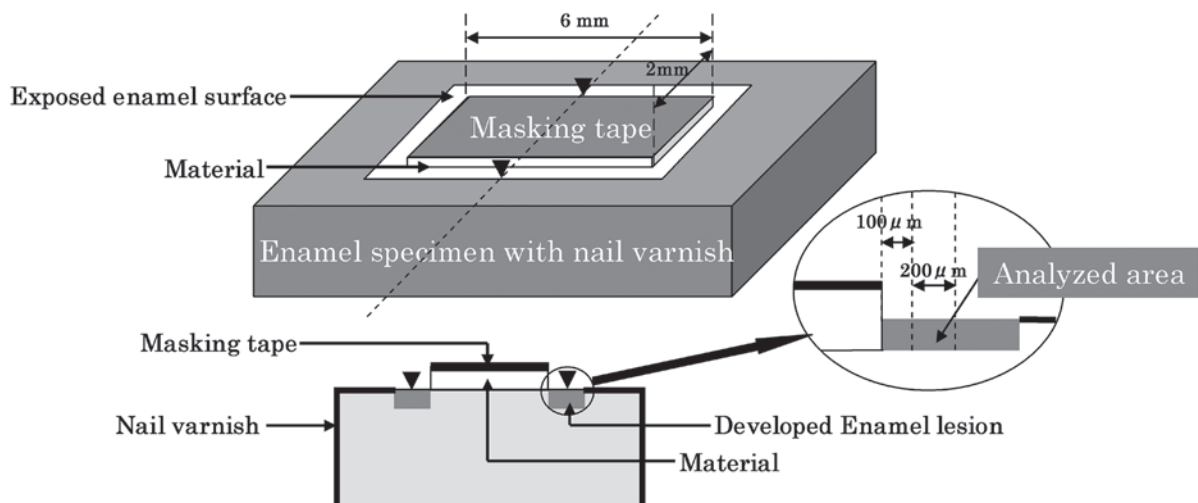


Fig. 1 Schematic drawing of material on the enamel specimen.

90 μm . After the cured samples were left for 24 hours at room temperature in 100% humidity, an adhesive masking tape of the same size was put on the cured materials to simulate brackets. This tape limited fluoride-releasing sites to the borders of the cured materials. Cured materials that exceeded the enamel windows were completely removed, and then the masking tapes with the windows were peeled off. Subsequently, the enamel surfaces of specimens were painted with an acid-resistant nail varnish, leaving a gap of 1 mm from the cured material's periphery (Fig. 1).

Acid resistance testing

Each sample was fixed to the bottom of a plastic container and covered with 20 ml of 8% Methocel MC gel (Fluka, Buchs, Switzerland), followed by pouring 20 ml of 0.1 M lactic acid at pH 4.6 at 37°C. After 10 days of incubation, all samples were taken from the containers and gently washed with deionized water.

Microradiography

After acid resistance test, the specimens were embedded in an epoxy resin (Spurr Low-Viscosity Embedding Media, Polysciences Inc., Warrington, PA, USA). Then, three 150- μm -thick sections from each specimen were cut perpendicularly to the surface using a diamond-coated wire sectioning machine. Each section was placed on a Perspex holder and covered with a thin polyester sheet. Together with an aluminum step wedge of 13 steps, the sections were radiographed on a high-resolution

glass film plate (High-Resolution Glass Plate, Konica, Tokyo, Japan) with a nickel-filtered Cu-K α source operating at 25 kV and 15 mA for 20 minutes (PW3830, Spectris, Surrey, UK). At a distance of 100 μm away from each cured material, only an area within 200 μm was analyzed. This analysis was done to investigate whether fluoride released from the material affected the enamel structure positioned away from the material.

The radiographic images and aluminum step wedge were analyzed with a microscope-video camera-microcomputer set-up with dedicated software (TMR 2000, Inspektor Research Systems, Amsterdam, The Netherlands). Data obtained pertained to the mineral content profiles of the lesions, integrated mineral loss (IML), and lesion depth (LD).

Statistical analysis

Differences between the groups were tested for significance at $p < 0.05$ level by one-way ANOVA followed by Duncan's multiple range test using SPSS version 10.1.

RESULTS

Figures 2 and 3 show the representative TMR images and the mean mineral profiles of the specimens of each treatment group. The TMR images of TB, KP, and SB revealed mineral profiles which showed thin surface layers and a severe body of lesions (Fig. 2). On the other hand, BO, BOS, and FO showed thick surface layers and a slight body of lesions (Fig. 3).

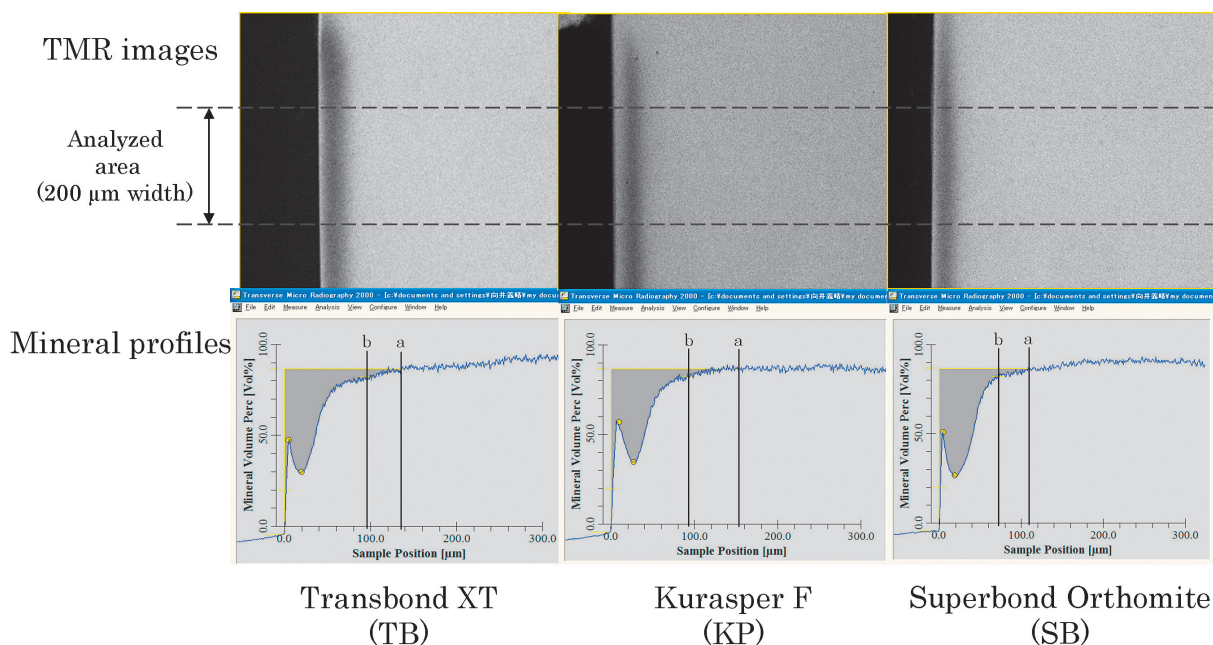


Fig. 2 Representative TMR images and mineral profiles of TB, KP, and SB.

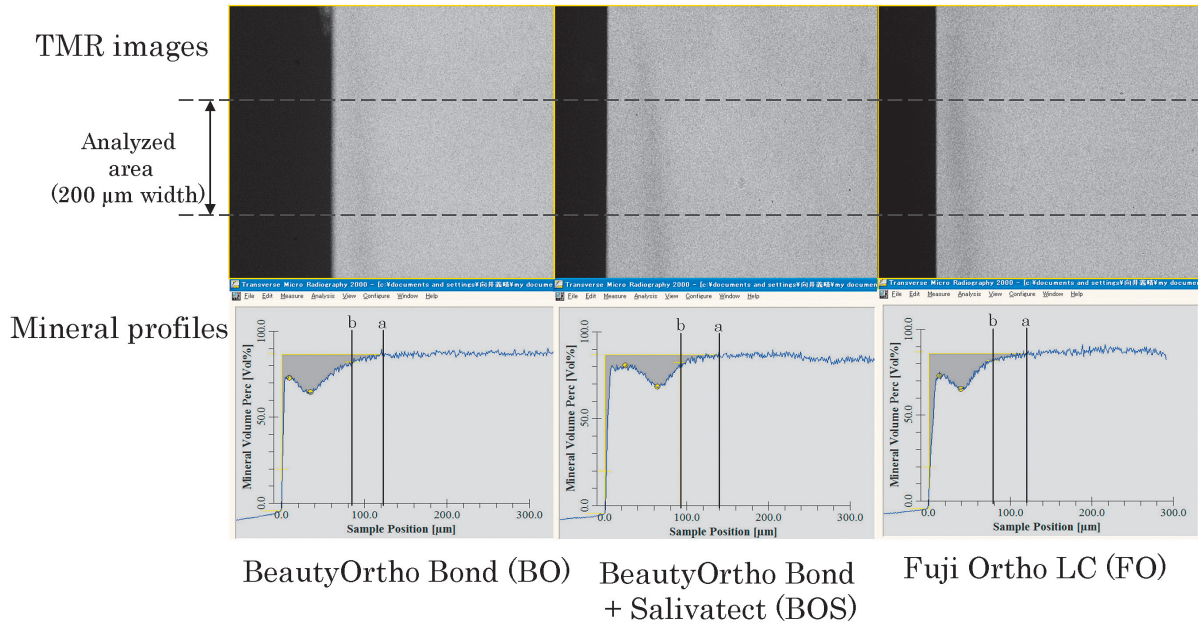


Fig. 3 Representative TMR images and mineral profiles of BO, BOS, and FO.

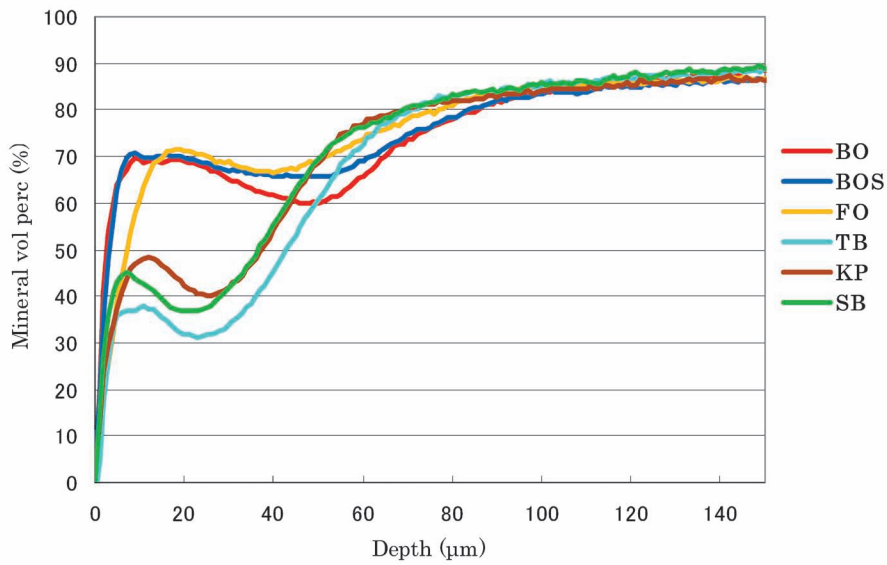


Fig. 4 Averaged mineral profiles of all experimental groups.

Table 2 IML and LD data of all experimental groups

Group	IML (vol% · μm)	ID (μm)
TB	2835.8 (407.6) a	75.8 (5.5) a
KP	2382.6 (400.8) a	76.4 (11.3) a
SB	2414.5 (358.2) a	78.5 (12.8) a
BO	1832.6 (604.2) b	88.7 (12.0) a
BOS	1689.8 (369.8) b	88.0 (16.7) a
FO	1620.9 (285.0) b	81.6 (6.5) a

n = 5, Mean (±SD)

Figure 4 shows the averaged mineral profile of each group. BO, BOS, and FO revealed distinct surface layers with a high volume% of minerals.

Table 2 summarizes the IML and LD data. The IML values of TB, KP, and SB were 2835.8, 2382.6, and 2414.5 vol%· μm , while 1832.6, 1689.8, and 1620.9 were detected in BO, BOS, and FO. These results indicated that the IML values of BO, BOS, and FO were significantly lower than those of TB, KP, and SB ($p < 0.05$). On the other hand, the LD values of the experimental groups were not significantly different from each other.

DISCUSSION

A new bonding system containing S-PRG filler was developed for the purpose of producing a material which would exhibit both structural strength and fluoride-releasing ability. Mukai *et al.*³⁰ reported that a newly developed all-in-one adhesive system which contained S-PRG filler was able to form inhibition layers, and that the latter provided acid resistance around restorative materials. Similarly, in this study, analysis of the IML data showed that the fluoride-releasing materials contained in S-PRG filler contributed to inhibiting enamel demineralization. However, no differences were observed for LD. ten Cate *et al.*³¹ demonstrated that LD was controlled by the pH of the solution *versus* the fluoride concentration. Furthermore, Mukai *et al.*³² and Suga *et al.*³³ suggested that fluoride contained in demineralization solutions suppressed mineral dissolution and that a distinct surface layer was formed within the lesion. Consequently, the demineralization potential in the resultant solution that penetrated to the inner enamel might remain, due to the rich hydrogen ions which were not consumed for surface demineralization.

Beauty Ortho Bond (BO and BOS) used in this experiment contained S-PRG filler as a fluoride-releasing source. The filler particles were produced using pre-reacted glass-ionomer (PRG) technology^{34,35}. With this technology, a glass-ionomer phase is formed on glass particles through the reaction of fluoroaluminosilicate glass and a polycarboxylic acid in the presence of water.

Underwood *et al.*²⁶ demonstrated the durability and anticariogenic potential of a fluoride-exchanging resin as an orthodontic adhesive. In addition, much of the published literature has dealt with varnishes and fluoride-releasing materials to inhibit microbial colonization around brackets and reduce the risk of demineralization. On the other hand, at present, many clinicians use phosphoric acid as an etching gel to secure adhesive strength between brackets and enamel surfaces. Furthermore, many studies have suggested that the bond strengths of brackets bonded

with self-etching adhesive systems were lower than those bonded with phosphoric acid-etching adhesive systems³⁶⁻³⁸. However, the application of phosphoric acid-etching adhesive systems has resulted in an increase in caries activity, whereby white and brown spots were observed around or under the brackets after debonding³⁹.

Recently, Kitayama *et al.*⁴⁰ reported that a shear bond strength of 17.7 MPa was present after thermal cycling in the SB (Superbond) group, as well as 18.8 MPa in the BO group. However, SEM observation after debonding revealed that BO resulted in less enamel dissolution compared with KP and SB. This indicated that self-etching adhesive systems caused less damage to the enamel surface compared with phosphoric acid-etching adhesive systems. Furthermore, Scougall Vilchis RJ *et al.*⁴¹ also reported that no enamel fracturing was observed during shear bond testing when the self-etching primer accompanying BO was used. Based on these reports, it was clearly shown that the application of the self-etching primer accompanying BO resulted not only in sufficient shear bond strength, but that it was also non-destructive to enamel. In addition, it was implied from this study that differences in acidity of the etching agents would not affect mineral loss and IML values.

It has been extensively reported that fluoride release from glass-ionomer cements may result in the reduction or elimination of enamel demineralization around bonded brackets in an environment conducive to acid attack⁴². However, in terms of mechanical behavior, different results were yielded with different laboratory studies^{43,44}. Some studies showed that the bond strengths of glass-ionomer cements appeared to be too low for use as routine orthodontic bonding agents^{45,46}. Moreover, some reports showed that the shear bond strengths of resin cements were significantly higher than those of resin-modified glass-ionomer cements when brackets were bonded to enamel⁴⁷.

In the present experiment, BO, BOS, and FO exhibited a significantly lower degree of demineralization than TB, KP, and SB ($p < 0.05$). In the context of the present study, BO, BOS, and FO were able to inhibit the demineralization of enamel substructure even with an intervening gap of 100 μm from the materials. These findings indicated that the S-PRG filler-containing orthodontic bonding agents imparted an acid resistance capability to the enamel around the orthodontic brackets — an effect equal to that rendered by resin-modified glass-ionomer systems.

The KP system included methyl methacrylate-methacryloyl fluoride copolymer and sodium fluoride as the fluoride sources. However, there were no statistically significant differences between the IML values of KP and SB. This result of KP might have

been affected by the thin layer of F-bond applied according to manufacturer's instructions. In pitting conventional fluoride-containing composite resins against S-PRG filler, some studies have shown that fluoride released from S-PRG was connected with the prevention of plaque accumulation⁴⁸. In this respect, this antibacterial benefit might serve to eliminate the risk of dental caries.

CONCLUSION

Based on the observations in this study, it was concluded that the newly developed fluoride-releasing orthodontic bonding system has a potential to inhibit demineralization around orthodontic brackets. This beneficial effect originates from the S-PRG filler, a newly developed filler component.

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