

Bracket bond strength and cariostatic potential of an experimental resin adhesive system containing Portland cement

Masahiro Iijima^a; Masanori Hashimoto^b; Susumu Nakagaki^c; Takeshi Muguruma^d; Naohisa Kohda^d; Kazuhiko Endo^e; Itaru Mizoguchi^f

ABSTRACT

Objective: To determine if a new experimental resin-based material containing Portland cement (PC) can help prevent enamel caries while providing adequate shear bond strength (SBS).

Materials and Methods: Brackets were bonded to human premolars with experimental resin-based adhesive pastes composed of three weight ratios of resin and PC powder (PC 30, 7:3; PC 50, 5:5; PC 70, 3:7; n = 7). Self-etching primer (SEP) adhesive (Transbond Plus) and resin-modified glass ionomer cement (RMGIC) adhesive (Fuji Ortho FC Automix) were used for comparison. All of the bonded teeth were subjected to alternating immersion in demineralizing (pH 4.55) and remineralizing (pH 6.8) solutions for 14 days. The SBS for each sample was examined, and the Adhesive Remnant Index (ARI) score was calculated. The hardness and elastic modulus of the enamel were determined by a nanoindenter at 20 equidistant depths from the external surface at 100 µm from the bracket edge. Data were compared by one-way analysis of variance and a chi-square test.

Results: PC 50 and PC 70 showed significantly greater SBS than Fuji Ortho FC Automix, although Transbond Plus showed significantly greater SBS than other bonding systems. No significant difference in the ARI category was observed among the five groups. For specimens bonded with PC 50 and PC 70, the hardness and elastic modulus values in most locations were equivalent to those of Fuji Ortho FC Automix.

Conclusions: Experimental resin-based bonding material containing PC provides adequate SBS and a caries-preventive effect equivalent to that of the RMGIC adhesive system. (*Angle Orthod.* 2012;82:900–906.)

KEY WORDS: Adhesive; Portland cement; Shear bond strength; Nanoindentation; Demineralization

^a Associate Professor, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Hokkaido, Japan.

^b Associate Professor, Division of Biomaterials and Bioengineering, Department of Oral Rehabilitation, School of Dentistry, Health Sciences University of Hokkaido, Hokkaido, Japan.

^c Postgraduate student, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Hokkaido, Japan.

^d Instructor, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Hokkaido, Japan.

^e Professor, Division of Biomaterials and Bioengineering, Department of Oral Rehabilitation, School of Dentistry, Health Sciences University of Hokkaido, Hokkaido, Japan.

^f Professor, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Hokkaido, Japan.

INTRODUCTION

An increased prevalence of the formation of white spots in orthodontic patients has been reported¹ due to the irregular surfaces of fixed orthodontic appliances, which create areas of stagnation for plaque, make tooth cleaning more difficult, and limit natural self-cleaning mechanisms.¹ While glass ionomer cements have high levels of fluoride-releasing ability, which

Corresponding author: Dr Masahiro Iijima, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Kanazawa 1757, Ishikari-tobetsu, Hokkaido 061-0293, Japan
(e-mail: iijima@hoku-iryo-u.ac.jp)

Accepted: November 2011. Submitted: September 2011.
Published Online: January 9, 2012

© 2012 by The EH Angle Education and Research Foundation, Inc.

plays an important role in preventing the demineralization of enamel, they show poor bracket bond strength.² Over the past decade, resin-modified glass ionomer cements (RMGIC) with improved bracket bond strength have been developed.^{3,4} The cariostatic potential of adhesives adjacent to brackets has been investigated both in vitro^{1,5,6} and in vivo^{7,8} by quantifying the depth of demineralization and mineral loss by various evaluation methods. Most studies have shown that RMGIC are more effective for preventing the demineralization of enamel than both fluoride-releasing and non-fluoride-releasing composite resin adhesive systems,^{1,5,6} although RMGIC was shown to have a weaker bond strength than composite resin adhesives.^{3,4}

Mineral trioxide aggregate (MTA), which is composed of tricalcium silicate (53%), dicalcium silicate (23%), bismuth oxide (22%) and small amounts of tricalcium aluminate and calcium sulphate, was introduced to dentistry in the mid-1990s by Dentsply Tulsa Dental (Tulsa, Okla).⁹ According to the manufacturer, MTA can be used for root-end filling, apexification, repair of perforations, and direct pulp capping. MTA is a bioactive material that can produce hydroxyapatite or carbonate apatite in the presence of phosphate-buffered saline.^{10,11} Portland cement (PC) has also been investigated as a substitute for MTA because of its chemical similarity and low cost.¹² Since PC releases ions and forms crystals, resin-based bracket-bonding materials that contain PC may also be useful for preventing caries.¹³

The purpose of this in vitro study was to investigate if this new experimental resin-based material containing PC can provide adequate shear bond strength (SBS) for bonding orthodontic brackets and also help to prevent the formation of enamel caries. We hypothesized that a resin-based bracket bonding material containing PC would have adequate bracket bond strength and superior caries-preventive effects.

MATERIALS AND METHODS

Materials

Thirty-five human noncarious upper premolars, obtained by extraction from patients who were to undergo orthodontic treatment, were allocated into five groups of seven each for measurements of SBS and cross-sectional nanoindentation. This in vitro study was approved by the ethics committee at the Health Sciences University of Hokkaido. The buccal surfaces of all teeth were cleaned using nonfluoridated pumice. The teeth were subsequently polished using a rubber cup and thoroughly washed and dried using a moisture-free air source.

PC Powder and Acrylic Resin Matrix

White PC (3CaO/SiO_2 , 2CaO/SiO_2 , $3\text{CaOAl}_2\text{O}_3$, $\text{CaSO}_4/2\text{H}_2\text{O}$; Ube-Mitsubishi Cement, Tokyo, Japan) was used in this study. Experimental light-cured resins were prepared with 80 mass% Bis-GMA (2, 2-bis-[4-(2-hydroxy-3-methacryloyloxypropoxy)-phenyl] propane; Polysciences, Warrington, PA) and 20 mass% triethylene glycol dimethacrylate (Wako Chemical, Kanagawa, Japan). To make the experimental resins light-curable, 0.005 mass% camphorquinone (Wako Chemical) and 0.01 mass% 2-(dimethylamino) benzoate (Wako Chemical) were added to the resin monomers. Experimental resin-based adhesive pastes with three weight ratios of resin and PC powder (W[R]:W[P]; 7:3; 5:5; 3:7) were prepared.

Groups Tested

Group 1: Transbond Plus SEP adhesive system. Transbond Plus SEP (3M Unitek, Monrovia, Calif) was applied to and rubbed on the enamel surface for 3 seconds. Air was lightly applied to the enamel, and metal brackets for upper premolars (Victory Series, 3M Unitek) with a base area of 10.0 mm^2 were bonded with Transbond XT composite.

Group 2: Fuji Ortho FC Automix RMGIC adhesive system. Fuji Ortho Gel Conditioner (20% polyacrylic acid) was applied to the enamel surface for 10 seconds, followed by thorough washing and drying. Metal brackets were then bonded with Fuji Ortho FC Automix paste.

Groups 3–5: PC-containing resin-based adhesive systems. Clearfil Mega Bond Primer (Kuraray Medical, Tokyo, Japan) was applied to the enamel surface for 20 seconds and dried with a gentle air flow. Clearfil Mega Bond (Kuraray Medical) was then applied to the enamel surface. Metal brackets were then bonded with experimental resin-based adhesive pastes with three weight ratios of the resin and PC powder (group 3, 7:3; group 4, 5:5; group 5, 3:7).

Excess bonding material was removed with an explorer, and the enamel surface around the bracket was carefully wiped off with cotton. Samples in groups 1 and 2 were light-cured according to the instructions from each manufacturer (Figure 1a). Samples bonded with PC-containing resin-based pastes (groups 3–5) were light-cured for 20 seconds (10 seconds for each proximal side). Acid-resistant nail varnish was next applied to each tooth, leaving a 1-mm rim of exposed sound enamel surrounding the bracket (Figure 1b). All bonded teeth were immersed individually in a plastic vial with 2 mL of demineralizing solution (2 mmol/L calcium chloride and 2 mmol/L sodium dehydrogen phosphate, with 50 mmol/L acetic acid added to give a pH of 4.55) for 4 hours at 37°C as described

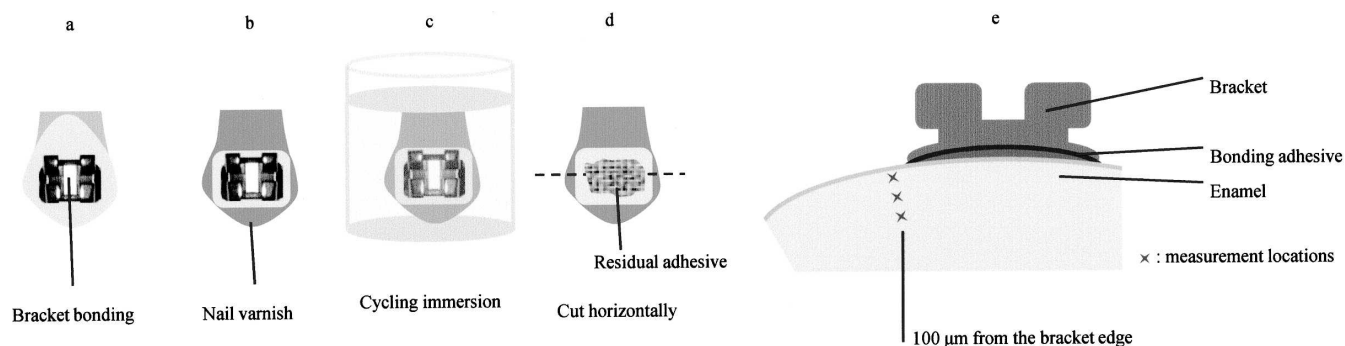


Figure 1. Schematic illustration of specimen preparation and regions of tooth enamel investigated with the nanoindentation test.

previously.¹⁴ After being rinsed with deionized water and then dried, the specimens were immersed in 2 mL remineralizing solution (2 mmol/L CaCl_2 and 2 mmol/L NaH_2PO_4 with 0.1 mol/L of NaOH added to give a pH of 6.8) for 20 hours at 37°C.¹⁴ This process was repeated for 14 days (Figure 1c).

Debonding Procedure

After cycling immersion, the SBS in each group was measured. The specimens were fixed to a custom-fabricated acrylic resin block using Model Repair II, and the block was fixed to a universal testing machine (EZ Test, Shimadzu, Kyoto, Japan). A knife-edged shearing blade was secured to the crosshead with the direction of force parallel to the buccal surface and the bracket base. Force was applied directly to the bracket-tooth interface. The brackets were debonded at a crosshead speed of 0.5 mm/min. After bond failure, the bracket bases and enamel surfaces were examined with a stereoscopic microscope at a magnification of 10 $\times$. ARI scores were used to assess the amount of adhesive left on the enamel surface.¹⁵

Cross-Sectional Enamel Microhardness Measurements With a Nanoindentation Test

After the debonding procedure, five specimens from each group were cut with a slow-speed water-cooled diamond saw (Isomet, Buehler, Lake Bluff, Ill) so that they were divided into occlusal and cervical halves (Figure 1d); one of the sectioned specimens (transverse planes) was then encapsulated in epoxy resin (Epofix, Struers, Copenhagen, Denmark) for the nanoindentation test. All samples were ground (600-grit sandpaper) and polished using diamond suspensions (3-, 1-, and 0.25- μm particle sizes) to obtain a suitable surface for nanoindentation. All nanoindentation testing (ENT-1100a, Elionix, Tokyo, Japan) was carried out at 28°C with a peak load of 5 mN using a Berkovich indenter. The indentations were placed at depths of 1–96 μm (20 locations spaced 5- μm apart)

from the external surface at approximately 100 μm from the bracket edge (Figure 1e). The hardness and elastic modulus were calculated by software provided with the nanoindentation apparatus.

Statistical Analysis

The experimental results were analyzed using PASW Statistics software (version 18.0J for Windows, IBM, Armonk, NY). The mean SBS, along with the standard deviation ($n = 7$), for the groups of bonding materials was compared by one-way analysis of variance (ANOVA), followed by the Tukey Kramer honestly significant difference test. The chi-square test was used to evaluate the significance of differences in the ARI scores among the different groups. For the statistical analysis, ARI scores of 0 and 1 were combined, as were ARI scores of 2 and 3. The mean hardness and elastic modulus values, along with the standard deviation ($n = 15$), for the groups with different bonding materials were compared by one-way ANOVA, followed by the Tukey test. For all statistical tests, significance was predetermined at $P < .05$.

RESULTS

The results regarding SBS are shown in Figure 2. Based on the results of one-way ANOVA, significant differences in SBS were found among the five specimen groups ($P = .000$). Group 1 (Transbond Plus SEP adhesive system) showed a significantly greater mean SBS (9.4 MPa) than groups with other bonding systems (3.7 MPa, Fuji Ortho FC Automix; 4.9 MPa, 30% PC-containing resin-based adhesive; 6.0 MPa, 50% PC-containing resin-based adhesive; 5.1 MPa, 70% PC-containing resin-based adhesive). Groups 4 and 5 (PC-containing resin-based adhesives; 30% and 50%) showed a significantly greater mean SBS than groups with the Fuji Ortho FC Automix adhesive system. A chi-square analysis that compared the ARI scores for the three adhesives found no significant difference in the distribution of frequencies

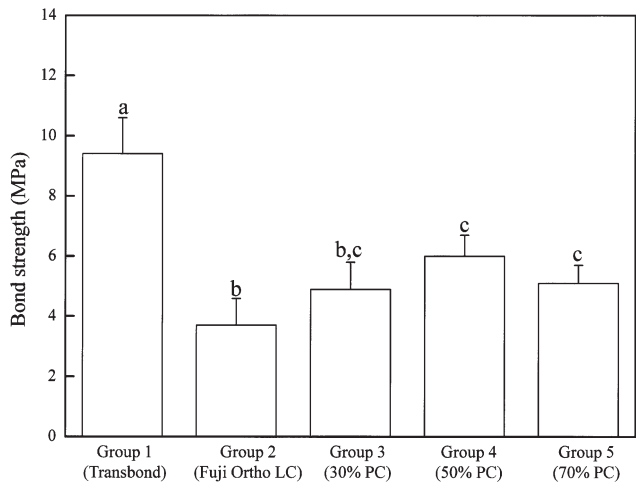


Figure 2. Mean and standard deviation for bond strength (MPa) in the five specimen groups. For bars with identical letters, the average values are not significantly different ($P < .05$, Tukey test).

among the ARI categories for the five adhesive groups (Table 1).

Figure 3 shows the mean values of hardness and the elastic modulus of cross-sectioned polished specimens (transverse planes) obtained by the nanoindentation test; the results of the statistical comparisons of the five specimen groups are summarized in Table 2 and 3. There was no significant difference in hardness among the five specimen groups at depths of 86 μm to 96 μm from the enamel surface. The hardness in group 1 (Transbond SEP adhesive system) was significantly lower at a depth of 1–81 μm from the enamel surface than those in other groups, except a depth of 71 μm . For specimens bonded with 50% and 70% PC-containing resin-based adhesive, the hardness values at most locations were equivalent to those of the specimens bonded with RMGIC. There was no significant difference in the elastic modulus among the five specimen groups at a depth of 1 μm and 66–96 μm from the enamel surface, except at a depth of 76 μm . The elastic modulus in group 1 (Transbond SEP adhesive system) was significantly lower than those in the other groups at a depth of 6–61 μm from the enamel surface, except at a depth of 51 μm . For specimens bonded with 50% and 70% PC-containing resin-based adhesive, the elastic modulus values at most locations were equivalent to those in the specimen bonded with RMGIC.

DISCUSSION

In this study, the mean bond strength obtained with the Transbond Plus SEP adhesive system was 9.4 MPa. This value is similar to published values for the Transbond Plus SEP adhesive system, which have ranged from 6.1 MPa to 10.4 MPa, although these

Table 1. Frequency Distribution of ARI Scores of Tested Groups^a

ARI Score	0	1	2	3
Transbond Plus SEP	0	3	3	1
Fuji Ortho LC	0	2	5	0
30% PC adhesive	0	4	3	0
50% PC adhesive	0	1	3	3
70% PC adhesive	0	1	4	2

^a ARI indicates Adhesive Remnant Index; PC, Portland cement. 0 = no adhesive left on the tooth surface, the failure site was between the adhesive and enamel; 1 = less than half of the adhesive was left on the tooth surface; 2 = half or more of the adhesive was left on the tooth; 3 = all of the adhesive was left on the tooth surface, the failure site was between the adhesive and bracket base. ARI scores of 0 and 1 were combined (less than 50% adhesive left on the tooth surface) as were 2 and 3 (50% or more of the adhesive left on tooth surface) for the chi-square analysis. There were no significant differences among the groups.

studies used different experimental conditions.^{16,17} On the other hand, the mean bond strength obtained with the Fuji Ortho LC Automix in this study was 3.7 MPa, which was lower than previously published values (3.7–12.6 MPa) for other types of RMGIC (Fuji Ortho LC and Photac-Fil Aplicap). Most studies refer to an article by Reynolds (1975), who proposed that 6 to 8 MPa was a clinically acceptable value for the bracket bond strength.¹⁸ Although it is difficult to compare the values in the orthodontic literature because of the multiple test configurations and the assumptions and approximations integrated into the experimental methodologies,¹⁹ new experimental resin-based bracket bonding materials containing PC powder (groups 4 and 5) showed significantly greater SBS (5.1–6.0 MPa) than RMGIC (Fuji Ortho FC Automix), suggesting that this PC-containing resin-based adhesive has clinically acceptable bracket bond strength.

A comparison of the ARI scores revealed no significant differences among the ARI categories for the five adhesive groups. These results suggest that the addition of PC powder to the resin matrix does not offer any benefits with regard to removing residual adhesive after a debonding procedure.

The use of cross-sectional microhardness measurements with a Knoop indenter has been a popular method for investigating the demineralization of enamel quantitatively,³ since there is high correlation between enamel microhardness and the percentage of mineral in carious lesions.²⁰ The traditional Knoop microhardness test generally produces a large indentation (indentation length equivalent to the width of 5–20 enamel prisms) and is also influenced by the substrate.²¹ In contrast, recent advances in the nanoindentation test have allowed the analysis of nano-scale mechanical properties.²² In addition, the elastic modulus for this very small volume of material can be obtained mathematically from a load-displacement curve. The results of the cross-sectional nanoindentation

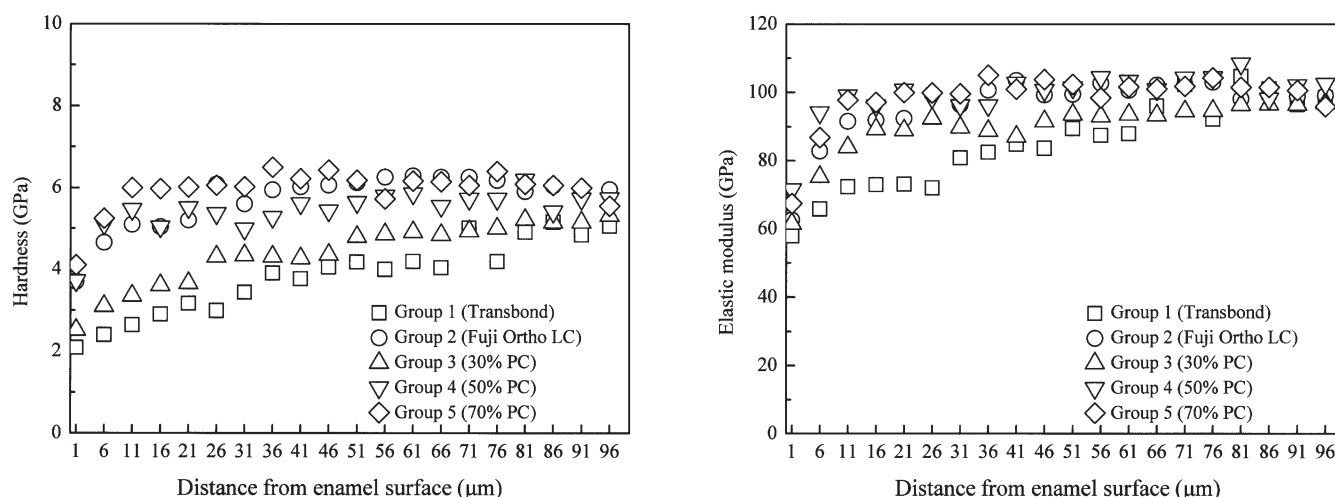


Figure 3. Mean values of the hardness of enamel at approximately 100 μm from the bracket edge at different depths from the surface after immersion in demineralizing and remineralizing solutions. Group 1, Transbond Plus SEP adhesive system; group 2, Fuji Ortho FC Automix RMGIC adhesive system; group 3, experimental resin-based adhesive system containing 30% of Portland cement powder; group 4, experimental resin-based adhesive system containing 50% of Portland cement powder; group 5, experimental resin-based adhesive system containing 70% of Portland cement powder.

analysis in this study showed that the values of hardness and the elastic modulus for Transbond Plus SEP without fluoride-releasing ability were decreased at depth of up to 81 μm (hardness: 2.09–4.91 MPa) and 61 μm (elastic modulus: 57.77–87.90 MPa), suggesting that these regions of the enamel structure were demineralized by the cycling immersion process. For the surface region (up to 6 μm) of the specimens bonded with RMGIC and PC-containing resin-based adhesive (50% and 70%), the values of hardness and

the elastic modulus decreased due to demineralization by cycling immersion and an etching effect by the SEP and polyacrylic acid conditioner. However, for specimens bonded with RMGIC and PC-containing resin-based adhesive (50% and 70%), the values at depths exceeding 11 μm ranged from 5.10 to 6.43 MPa for hardness and from 91.52 to 105.08 MPa for the elastic modulus, which show that material at these depths may not be demineralized by cycling immersion and were similar to recently published values (4.7–5.7 MPa

Table 2. Mean Values and Standard Deviation of Cross-Sectional Hardness (GPa) of Enamel at Approximately 100 μm From the Bracket Edge at Different Distances From the Surface After Cycling Immersion^a

Depth, μm	Transbond Plus SEP		Fuji Ortho LC		30% PC adhesive		50% PC adhesive		70% PC adhesive		One-Way ANOVA <i>P</i> Value
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
1	2.09 ^a	1.01	3.71 ^{b,c}	1.59	2.52 ^{a,b}	1.55	3.73 ^{b,c}	1.41	4.11 ^c	1.62	.001
6	2.4 ^a	1.43	4.66 ^b	1.44	3.11 ^a	1.62	5.11 ^b	1.72	5.25 ^b	0.89	.0001
11	2.64 ^a	1.45	5.1 ^b	1.24	3.36 ^a	1.64	5.47 ^b	1.86	6 ^b	0.79	.0001
16	2.9 ^a	1.93	5.04 ^{b,c}	1.35	3.61 ^{a,b}	1.93	5.04 ^{b,c}	1.64	5.97 ^c	0.82	.0001
21	3.17 ^a	2.38	5.2 ^{b,c}	1.64	3.67 ^{a,b}	1.84	5.51 ^c	1.76	6.02 ^c	0.78	.0001
26	2.99 ^a	2.07	6.08 ^c	0.75	4.32 ^{a,b}	1.99	5.37 ^{b,c}	1.69	6.06 ^c	0.44	.0001
31	3.44 ^a	1.73	5.6 ^{b,c}	1.12	4.34 ^{a,b}	1.85	4.99 ^{a,b,c}	2.14	6.02 ^c	0.59	.0001
36	3.91 ^a	2.15	5.95 ^{b,c}	0.7	4.32 ^{a,b}	1.65	5.28 ^{a,b,c}	1.98	6.5 ^c	1.25	.0001
41	3.77 ^a	2.04	6.02 ^c	0.6	4.27 ^{a,b}	1.6	5.61 ^{b,c}	1.71	6.21 ^c	0.57	.0001
46	4.06 ^a	2.15	6.06 ^b	0.57	4.36 ^a	1.5	5.44 ^{a,b}	1.6	6.43 ^b	0.37	.0001
51	4.18 ^a	1.99	6.12 ^b	0.82	4.8 ^{a,b}	1.36	5.65 ^b	1.64	6.18 ^b	0.44	.0001
56	4 ^a	2.11	6.26 ^b	1.08	4.86 ^{a,b}	1.24	5.8 ^b	1.45	5.73 ^b	1.28	.001
61	4.2 ^a	2.24	6.29 ^b	0.71	4.92 ^{a,b}	1.12	5.86 ^b	1.49	6.16 ^b	0.31	.0001
66	4.05 ^a	2.35	6.26 ^b	0.54	4.85 ^{a,b}	1.27	5.56 ^b	1.56	6.14 ^b	0.4	.0001
71	5.01 ^{a,b}	2.38	6.26 ^b	0.39	4.94 ^a	0.87	5.73 ^{a,b}	1.31	6.06 ^{a,b}	0.86	.025
76	4.2 ^a	2.14	6.18 ^{b,c}	0.76	5.01 ^{a,b}	1.07	5.73 ^{b,c}	1.31	6.4 ^c	0.55	.0001
81	4.91 ^a	1.81	5.91 ^{a,b}	1.07	5.22 ^{a,b}	0.95	6.19 ^b	1.39	6.08 ^{a,b}	0.62	.02
86	5.16	1.81	6.04	0.87	5.16	0.98	5.41	1.91	6.06	0.65	.156
91	4.85	1.82	5.95	0.84	5.16	1.02	5.73	1.65	5.99	0.78	.066
96	5.06	1.9	5.96	0.91	5.32	1	5.74	1.57	5.55	1.6	.484

^a One-way ANOVA followed by the Tukey multiple range test. PC indicates Portland cement; ANOVA, analysis of variance. Identical letters indicate that the mean values were not significantly different ($P < .05$).

Table 3. Mean Values and Standard Deviations of Cross-Sectional Elastic Modulus (GPa) of Enamel at Approximately 100 μm From the Bracket Edge at Different Distances From the Surface After Cycling Immersion^a

Depth, μm	Transbond Plus SEP		Fuji Ortho LC		30% PC Adhesive		50% PC Adhesive		70% PC Adhesive		One-Way ANOVA P Value
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
1	57.88	16.9	62.59	22.8	61.56	15.94	71.53	16.08	67.46	21.52	.32
6	65.83 ^a	11.76	82.83 ^{b,c}	19.79	75.25 ^{a,b}	12.32	94.07 ^c	10.55	86.75 ^{b,c}	9.38	.0001
11	72.31 ^a	14.57	91.52 ^{b,c}	12.88	83.97 ^{a,b}	12.78	99 ^c	13.78	97.74 ^c	11.88	.0001
16	72.98 ^a	17.3	91.85 ^b	13.49	89.25 ^b	13.15	96.15 ^b	11.07	97.12 ^b	7.4	.0001
21	73.12 ^a	29.25	92.36 ^b	14.44	88.92 ^{a,b}	10.81	100.78 ^b	10.71	99.98 ^b	6.92	.0001
26	72.03 ^a	24.83	99.69 ^b	7.28	92.4 ^b	10.37	98.54 ^b	10.52	99.89 ^b	6.43	.0001
31	80.79 ^a	15.52	96.43 ^b	7.26	89.73 ^{a,b}	12.07	96.23 ^b	17.75	99.57 ^b	6.49	.001
36	82.46 ^a	19.51	100.66 ^{b,c}	6.87	88.77 ^{a,b}	13.41	96.21 ^{a,b,c}	13.7	105.08 ^c	15.61	.0001
41	84.78 ^a	18.59	103.46 ^b	11.15	87.07 ^a	15.33	102.77 ^b	9.76	100.98 ^b	7.08	.0001
46	83.69 ^a	19.96	99.24 ^b	7.97	91.58 ^{a,b}	12.18	100.68 ^b	8.3	103.77 ^b	6.44	.0001
51	89.4 ^a	24.15	99.46 ^{a,b}	8.42	93.51 ^{a,b}	11.17	101.41 ^{a,b}	7.98	102.34 ^b	5.23	.039
56	87.39 ^a	23.06	102.74 ^b	10.91	93 ^{a,b}	12.58	104.4 ^b	8.53	98.4 ^{a,b}	14.19	.013
61	87.9 ^a	19.26	100.57 ^b	6.9	93.49 ^{a,b}	9.58	103.35 ^b	12.19	101.6 ^b	4.59	.002
66	96	33.8	102.15	5.5	93.29	13.13	100.86	12.03	101.01	5.2	.578
71	102.27	17.77	102.14	4.85	94.51	9.98	104.24	11.37	101.82	7.78	.165
76	92.13 ^a	20.7	103 ^{a,b}	8.18	94.64 ^{a,b}	11.97	104.42 ^b	11.4	104.27 ^b	6.01	.018
81	104.66	29.16	98.1	10.68	96.32	8.93	108.44	12.7	101.49	6.72	.23
86	100.93	9.39	99.24	7.49	96.54	9.88	98.12	15.38	101.53	8.37	.686
91	96.43	10.71	98.82	6.85	96.4	10.51	102.06	10.68	100.6	7.22	.373
96	98.45	15.92	99.11	5.95	96.88	10.44	102.49	13.09	95.86	19.85	.733

^a One-way ANOVA followed by Tukey multiple range test. PC indicates Portland cement; ANOVA, analysis of variance. Identical letters indicate that the mean values were not significantly different ($P < .05$).

for hardness; 87.3–100.3 MPa for elastic modulus) for noncarious human premolars.²³

For bracket-bonding materials, surface characteristics such as surface roughness should be considered since they affect dental film accumulation. Van de Sande et al.²⁴ reported on the surface roughness of orthodontic band cements and found that storage time and immersion in lactic acid solution increased the surface roughness of the majority of the tested cements. The surface characteristics of resin-based bracket-bonding adhesive containing PC powder should be studied in future research.

One of the concerns about PC powder is the amount of lead and arsenic in its composition, although mixed results have been reported for the release of the elements.¹² Since some types of PC powder show high solubility,¹² the composition should be modified for biological safety.

Over the past decade, RMGIC with improved bracket bond strength have been developed.^{3,4} Most studies have shown that RMGIC are more effective for preventing the demineralization of enamel than both fluoride-releasing and non-fluoride-releasing composite resin adhesive systems,^{1,5,8} although previous studies have shown that RMGIC has a lower bond strength than composite resin adhesives.^{3,4} Recently, the release of calcium ion and changes in pH with MTA have been studied²⁵; the bioactivities of MTA and PC have been attributed to their ability to produce hydroxyapatite¹⁰ or carbonate apatite¹¹ in the presence of phosphate-buffered saline. In the present study, new resin-based bracket-bonding adhesive containing

PC powder successfully inhibited the demineralization of enamel in vitro, and its remineralization ability was equivalent to that of RMGIC. Further research is necessary to elucidate the detailed mechanism of the enamel caries-preventive effects of resin-based bracket-bonding adhesive containing PC powder.

CONCLUSIONS

- Experimental resin-based bonding materials containing PC powder provide adequate SBC for bonding orthodontic brackets.
- The caries-preventive effect of experimental resin-based materials containing PC powder was equivalent to that of resin-modified glass ionomer cement adhesive.

REFERENCES

1. Sudjalim TR, Woods MG, Manton DJ, Reynolds EC. Prevention of demineralization around orthodontic brackets in vitro. *Am J Orthod Dentofacial Orthop.* 2007;131:705.e1–9.
2. Basdra EK, Huber H, Komposch G. Fluoride released from orthodontic bonding agents alters the enamel surface and inhibits enamel demineralization in vitro. *Am J Orthod Dentofacial Orthop.* 1996;109:466–472.
3. Owens SE Jr, Miller BH. A comparison of shear bond strengths of three visible light-cured orthodontic adhesives. *Angle Orthod.* 2000;70:352–356.
4. Sfondrini MF, Cacciafesta V, Pistorio A, Sfondrini G. Effects of conventional and high-intensity light-curing on enamel shear bond strength of composite resin and resin-modified glass-ionomer. *Am J Orthod Dentofacial Orthop.* 2001;119:30–35.

5. Hu W, Featherstone JD. Prevention of enamel demineralization: an in-vitro study using light-cured filled sealant. *Am J Orthod Dentofacial Orthop*. 2005;128:592–600.
6. Paschos E, Kleinschrodt T, Clementino-Luedemann T, Huth KC, Hickel R, Kunzelmann KH, Rudzki-Janson I. Effect of different bonding agents on prevention of enamel demineralization around orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 2009;135:603–612.
7. Gorton J, Featherstone JD. In vivo inhibition of demineralization around orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 2003;123:10–14.
8. Pascotto RC, Navarro MF, Capelozza Filho L, Cury JA. In vivo effect of a resin-modified glass ionomer cement on enamel demineralization around orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 2004;125:36–41.
9. Camilleri J. Characterization of hydration products of mineral trioxide aggregate. *Int Endod J*. 2008;41:408–417.
10. Sarkar NK, Caicedo R, Ritwik P, Moiseyeva R, Kawashima I. Physicochemical basis of the biologic properties of mineral trioxide aggregate. *J Endod*. 2005;31:97–100.
11. Reyes-Carmona JF, Felipe MS, Felipe WT. Biomineralization ability and interaction of mineral trioxide aggregate and white Portland cement with dentin in a phosphate-containing fluid. *J Endod*. 2009;35:731–736.
12. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review—part I: chemical, physical, and antibacterial properties. *J Endod*. 2010;36:16–27.
13. Lee YL, Lee BS, Lin FH, Yun Lin A, Lan WH, Lin CP. Effects of physiological environments on the hydration behavior of mineral trioxide aggregate. *Biomaterials*. 2004;25:787–793.
14. Chin MYH, Sandham A, Rumachik EN, Ruben JL, Huysmans MDNJ. Fluoride release and cariostatic potential of orthodontic adhesives with and without daily fluoride rinsing. *Am J Orthod Dentofacial Orthop*. 2009;136:547–553.
15. Årtun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. *Am J Orthod Dentofacial Orthop*. 1984;85:333–340.
16. Yamamoto A, Yoshida T, Tsubota K, Takamizawa T, Kurokawa H, Miyazaki M. Orthodontic bracket bonding: enamel bond strength vs time. *Am J Orthod Dentofacial Orthop*. 2007;130:435.e1–e6.
17. Bishara SE, Ostby AW, Laffoon JF, Warren J. Shear bond strength comparison of two adhesive systems following thermocycling. *Angle Orthod*. 2007;77:337–341.
18. Reynolds I. Comparing bond strengths. *Br J Orthod*. 1975;2:171–178.
19. Eliades T, Bourauel C. Intraoral aging of orthodontic materials: the picture we miss and its clinical relevance. *Am J Orthod Dentofacial Orthop*. 2005;127:403–412.
20. Hu W, Featherstone JD. Prevention of enamel demineralization: an in-vitro study using light-cured filled sealant. *Am J Orthod Dentofacial Orthop*. 2005;128:592–600.
21. Featherstone JD, ten Cate JM, Shariati M, Arends J. Comparison of artificial caries-like lesions by quantitative microradiography and microhardness profiles. *Caries Res*. 1983;17:385–391.
22. Marshall GW Jr, Balooch M, Gallagher RR, Gansky SA, Marshall SJ. Mechanical properties of the dentinoenamel junction: AFM studies of nanohardness, elastic modulus, and fracture. *J Biomed Mater Res*. 2001;54:87–95.
23. Kohda N, Iijima M, Brantley WA, Muguruma T, Yuasa T, Nakagaki S, Mizoguchi I. Effects of bonding materials on the mechanical properties of enamel around orthodontic brackets. *Angle Orthod*. In press.
24. van de Sande FH, da Silva AF, Michelon D, Piva E, Cenci MS, Demarco FF. Surface roughness of orthodontic band cements with different compositions. *J Appl Oral Sci*. 2011;19:223–227.
25. Tanomaru-Filho M, Chaves Faleiros FB, Saçaki JN, Hungaro Duarte MA, Guerreiro-Tanomaru JM. Evaluation of pH and calcium ion release of root-end filling materials containing calcium hydroxide or mineral trioxide aggregate. *J Endod*. 2009;35:1418–1421.