Original Article

Shear Bond Strength of Calcium Phosphate Ceramic Brackets to Human Enamel

Daijiro Meguro[a]; Tohru Hayakawa[b]; Mayuko Kawasaki[c]; Kazutaka Kasai[d]

ABSTRACT
The aim of this study was to examine the shear bond strength between Hyaline brackets, a new type of calcium phosphate ceramic bracket, and human enamel using various types of adhesive resin and to investigate the effectiveness of a silane-coupling agent to bond Hyaline to human enamel. Kurasper F, Light Bond, Super Bond C&B, and Transbond XT were used as adhesive resins, and Porcelain Liner M was used as the silane-coupling agent. The Hyaline bracket was bonded to human enamel using one of the above adhesive resins according to the manufacturer's instructions. After applying the Porcelain Liner M to Hyaline, the Hyaline bracket was also bonded to enamel using one of the above adhesive resins according to the manufacturer's instructions. The shear bond strengths were measured after immersion in water at 37°C for 24 hours. Three types of adhesive resin, Kurasper F, Light Bond, and Super Bond C&B, produced clinically acceptable shear bond strength with and without Porcelain Liner M. Transbond XT produced significantly lower bond strength to enamel with or without Porcelain Liner M ($P < .05$). The application of Porcelain Liner M was not useful for improving the bond strength of Hyaline to enamel. The adhesive remnant indices were not significantly different among four adhesive resins. In conclusion, adhesive resins such as Kurasper F, Light bond, and Super Bond C&B are useful for bonding esthetic Hyaline brackets to human enamel. (*Angle Orthod* 2006;76:301–305.)

KEY WORDS: Shear bond strength; Calcium phosphate ceramic brackets; Adhesive remnant index; Silane-coupling agent

INTRODUCTION

The direct bonding of orthodontic brackets to enamel is widely accepted because of its ease, efficacy, and improved esthetics. The phosphoric acid–etching technique, introduced by Buonocore,[1] produces a reliable mechanical bond between the bracket and the enamel surface.

Ceramic brackets were introduced into orthodontic clinics in an attempt to meet the increasing demand for more esthetic appliances. Ceramic brackets resist staining and discoloration and are chemically inert to oral fluids. A disadvantage of ceramic brackets is their failure to bond with adhesive dental resins and their clinical complications, including increased friction and a higher chance of enamel fractures or cracks during debonding procedures.[2,3]

Most ceramic brackets are made from alumina. Alumina exists as single crystal or polycrystal units and is therefore known as monocrystalline or polycrystalline.[4] Klocke et al[5] reported that the bond strength to monocrystalline brackets was significantly higher than that to polycrystalline brackets. Several studies have been investigated into clinically acceptable bond strength of ceramic brackets to human enamel, including the application of silane-coupling agents.[6–8] Silane-coupling agents, for example γ-methacryloxypropyl trimethoxysilane, were originally developed for bonding glass fillers into polymers in dental composite.[9]

Recently, a new type of calcium phosphate ceramic bracket, Hyaline (Tomy International Inc, Tokyo, Japan), was introduced for orthodontic treatment. Hya-

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Accepted: April 2005. Submitted: March 2005.

line has excellent biocompatibility, low friction properties, and compatible hardness with human enamel. The aim of this study was to examine the shear bond strength between Hyaline brackets and human enamel using various types of adhesive resin and to investigate the effectiveness of a silane-coupling agent to bond Hyaline to human enamel.

MATERIALS AND METHODS

A total of 120 human premolar teeth were randomly allocated to eight protocols of 15 teeth each. The teeth were embedded in acrylic resin with the buccal surfaces available for bonding. After curing the acrylic resin, the tooth surfaces to be bonded were cleansed and polished with pumice and rubber prophylactic cups for 10 seconds to simulate a routine clinical procedure.

Orthodontic calcium phosphate ceramic bracket (Hyaline, Tomy International Inc) was used in this study. The average bracket surface area, which was supplied by the manufacturer, was 11.188 mm^2. Kurasper F (Kuraray Medical Inc, Okayama, Japan), Light Bond (Reliance Orthodontic Products, Itasca, Ill), Super Bond C&B (Sunmedical Co Ltd, Shiga, Japan), and Transbond XT (3M Unitek, Monrovia, Calif) were used for bonding. Porcelain Liner M (Sunmedical Co Ltd) was used as the silane-coupling agent. Liquid A of Porcelain Liner M contained a silane compound, and liquid B contained carboxylic acid.[10]

Bonding procedures

Kurasper F group. A total of 30 teeth were etched with phosphoric acid gel (K-etchan, Kuraray Medical Inc, Okayama, Japan) from the Kurasper F kit for 40 seconds. After rinsing and drying, the bonding agent, F bond (Kuraray Medical Inc, Okayama, Japan), was applied to the etched teeth and light cured for 10 seconds.

The 30 teeth were divided into two groups. Hyaline brackets, which were not treated with the silane-coupling agent, were bonded to 15 teeth using Kurasper F paste (Kuraray Medical Inc, Okayama, Japan), using 20 seconds of photoirradiation according to the manufacturer's instruction. To bond the remaining 15 teeth, Hyaline brackets were treated with a silane-coupling agent. A and B liquids of Porcelain Liner M were mixed thoroughly before application, and the mixed solution was placed directly onto the Hyaline brackets. The silane-treated Hyaline brackets were bonded with Kurasper F paste, using 20 seconds of photoirradiation according to the manufacturer's instruction.

Light Bond group. A total of 30 teeth were etched with phosphoric acid gel from the Light Bond kit for 30 seconds. After rinsing and drying, the bonding agent, Light Bond filled sealant (Reliance), was applied to the etched teeth and light cured for 20 seconds.

The 30 teeth were divided into two groups. Hyaline brackets, not treated with the silane-coupling agent, were bonded to 15 teeth using Light Bond paste (Reliance), using 20 seconds of photoirradiation according to the manufacturer's instruction. To bond the remaining 15 teeth, Hyaline brackets were treated with a silane-coupling agent. A and B liquids of Porcelain Liner M were mixed thoroughly before application, and the mixed solution was placed directly onto Hyaline brackets. Silane-treated Hyaline brackets were bonded with Light Bond paste, using 20 seconds of photoirradiation according to the manufacturer's instruction.

Super Bond C&B group. A total of 30 teeth were etched with phosphoric acid gel from the Super Bond C&B kit for 30 seconds. After rinsing and drying, the 30 teeth were divided into two groups. Hyaline brackets, not treated with silane-coupling agent, were bonded to 15 teeth without the silane-coupling agent using Super bond C&B according to the manufacturer's instruction (brush-dip technique). To bond the remaining 15 teeth, Hyaline brackets were treated with the silane-coupling agent. A and B liquids of Porcelain Liner M were mixed thoroughly before application, and the mixed solution was placed directly onto the Hyaline brackets. Silane-treated Hyaline brackets were bonded with Super Bond C&B according to the manufacturer's instruction (brush-dip technique).

Transbond XT group. A total of 30 teeth were etched with Unitek TM etching gel (3M Unitek) from the Transbond XT kit for 30 seconds. After rinsing and drying, a thin coat of the Transbond XT primer was applied to the etched teeth, air thinned for 2 seconds, and light cured for 10 seconds.

The 30 teeth were divided into two groups. Hyaline brackets, not treated with the silane-coupling agent, were bonded to 15 teeth using Transbond XT paste, using 20 seconds of photoirradiation according to the manufacturer's instruction. To bond the remaining 15 teeth, Hyaline brackets were treated with the silane-coupling agent. A and B liquids of the Porcelain Liner M were mixed thoroughly before application, and the mixed solution was placed directly onto Hyaline brackets. The silane-treated Hyaline brackets were bonded with Transbond XT paste, using 20 seconds of photoirradiation according to the manufacturer's instruction.

Each bracket was subjected to a 300-g force, according to the report of Bishara et al,[11] and excess bonding resin was removed with a small scaler.

Bonding assessments

After bonding, the specimens were stored in deionized water at 37°C for 24 hours. Shear bond strength was measured according to the methods recommend-

TABLE 1. Shear Bond Strengths (MPa) Between Calcium Phosphate Ceramic Brackets and Teeth[a]

	Non			Porcelain Liner M		
	Mean	SD	Range	Mean	SD	Range
Kurasper F	$12.2^{A,a}$	5.6	4.5–23.0	$9.3^{A,c}$	4.0	4.9–16.9
Light Bond	$15.2^{B,a}$	7.2	6.9–28.6	$7.3^{C,c}$	1.8	5.9–11.5
Super Bond C&B	$12.8^{D,a}$	4.1	4.8–19.7	$8.9^{D,c}$	5.0	3.9–19.7
Transbond XT	$6.2^{E,b}$	1.7	3.5–7.4	$6.3^{E,c}$	1.4	3.7–9.1

[a] Mean values with same superscripts are not significantly different ($P > .05$).
Uppercase letters indicate the comparison of shear bond strength with and without Porcelain Liner M within the same adhesive resin, and lowercase letters indicate the comparison of shear bond strength among adhesive resin within the same pretreatment of teeth, that is, with or without Porcelain Liner M.
Significant differences in shear bond strength existed between with and without Porcelain Liner M when Light Bond was used ($P < .05$). No significant differences in shear bond strength were found between with and without Porcelain Liner M with Kurasper F, Super Bond C&B, and Transbond XT ($P > .05$).
Significant differences in shear bond strength existed between Transbond XT and other adhesive resins (Kurasper F, Light Bond, and Super Bond C&B) without porcelain Liner M ($P < .05$), and no significant differences in shear bond strength existed among four different adhesive resins using Porcelain Liner M ($P > .05$).

ed by Kawasaki et al[12] and International Organization for Standardization[13] using a testing machine (TG-5kN, Techno Graph, Minebea Co Ltd, Tokyo, Japan) at a crosshead speed of 2 mm/min. The shear bond strengths were expressed in megapascals.

After debonding, the porcelain teeth and brackets were examined with 10× magnifications. The debonding condition of each specimen was scored using the adhesive remnant index (ARI).[14] The ARI scores ranged from 0 to 3, ie, score 0 = no adhesive remained on the tooth surface; 1 = less than half of the adhesive remained on the tooth surface; 2 = more than half of the adhesive remained on the tooth; and 3 = all the adhesive remained on the tooth with a distinct impression of the bracket base.

TABLE 2. Frequency Distribution of the ARI[a,b]

Adhesive Resin	Silane	ARI 0	ARI 1	ARI 2	ARI 3
Kurasper F	Non	0	0	0	15
	Porcelain Liner M	0	0	0	15
Light Bond	Non	0	0	1	14
	Porcelain Liner M	0	0	0	15
Super Bond C&B	Non	0	0	2	13
	Porcelain Liner M	0	0	0	15
Transbond XT	Non	0	0	0	15
	Porcelain Liner M	0	0	0	15

[a] ARI indicates Adhesive Remnant Index.
[b] There were no significant differences in ARI scores among eight procedures ($\chi^2 = 10.598$, $P = .1571$). No enamel fracture was observed.

Statistical analysis

A total of 15 specimens were tested for each procedure. Two-way analysis of variance (ANOVA) and Fisher test for multiple comparisons were used to detect statistical differences in the mean measurements among the eight procedures. The chi-square (χ^2) test was used to analyze statistical differences in ARI scores among the eight protocols. Significance for all statistical tests was predetermined at $P < .05$.

RESULTS

Comparison of shear bond strengths

The results of shear bond strength measurements (in MPa) are shown in Table 1. Two-way ANOVA showed significant differences in bond strength among the types of adhesive resin ($P = .0028$) and with and without the silane-coupling agent ($P = .0005$). Two-way interactions were not found for type of adhesive resin and with and without the silane-coupling agent ($P = .0555$).

No significant differences in shear bond strength were found between with and without Porcelain Liner M in the case of Kurasper F, Super Bond C&B, and Transbond XT ($P > .05$). Significant differences in shear bond strength existed between with and without Porcelain Liner M when Light Bond was used ($P < .05$). Porcelain Liner M significantly decreased the shear bond strength using Light Bond adhesive ($P < .05$).

When Porcelain Liner M was not used, Transbond XT showed significantly decreased shear bond strength compared with other adhesive resins, Kurasper F, Light Bond, and Super Bond C&B ($P < .05$). No significant differences in shear bond strength existed among four different adhesive resins with the use of Porcelain Liner M ($P > .05$).

Comparison of ARI

The frequency distribution of ARI scores after debonding is shown in Table 2. Chi-square test showed no significant difference in ARI score among the eight conditions ($\chi^2 = 10.598$, $P = .1571$). No enamel fracture was observed after debonding in all eight conditions.

DISCUSSION

This study shows that the shear bond strength of Hyaline brackets to human enamel was influenced by the types of adhesive resin and that the application of silane-coupling agents did not improve the shear bond strength.

Reynolds[15] suggested minimum tensile bond strength of approximately 6–7 MPa for clinical orthodontic treatment. Retief[16] reported enamel fractures on debonding with bond strength of approximately 14 Mpa. The clinically acceptable shear bond strength is still not known, but three types of adhesive resins, Kurasper F, Light Bond, and Super Bond C&B, produced approximately 12–15 MPa of shear bond strength between Hyaline and human enamel without Porcelain Liner M. On the contrary, when used with Porcelain Liner M, all adhesives used in this study showed approximately 6–9 MPa of shear bond strength between Hyaline and human enamel. These values are compatible with the shear bond strength of metal brackets bonded with resin-modified glass ionomer cement.[17] Thus, the bond strength of Hyaline obtained in the present adhesive resin systems would be clinically acceptable. However, the relationship between bond strength values in vitro and bond failures values in vivo is complex. Pickett et al[18] demonstrated that mean bond strength recorded in vivo after comprehensive orthodontic treatment is significantly lower than bond strength recorded in vitro. The evaluation under simulated clinical condition should be further performed.

Although it is not clear why Transbond XT produced significantly lower bond strength, it should be noted that the types of adhesive resin would influence the bond strength of orthodontic bracket to enamel in orthodontic clinics. Vicente et al[19] compared the shear bond strength of Light Bond and Transbond XT to enamel and reported that the bond strength produced by Light Bond was significantly greater than that of Transbond XT, and the results of this study are in agreement.

The Porcelain Liner M consisted of two bottles, one containing the silane-coupling agent, γ-methacryloxypropyl trimethoxysilane, and the other carboxylic acid.[10] It is well known that acid is a catalyst for the activation of a silane-coupling agent, ie, hydrolysis of the organosilane to form organosilanol and the subsequent siloxane bond formation between the porcelain and the silane-coupling agent. It is suggested that the acid solution enhanced the formation of a siloxane bond between porcelain and the silane-coupling agent and facilitated the tight bonding of adhesive resin and porcelain.[10,20–23] However, in this study, the application of Porcelain Liner M to Hyaline did not produce any significant increase in bond strength, and Light Bond demonstrated significantly lower bond strength with the use of Porcelain Liner M. The main component of Hyaline was calcium phosphate. It is presumed that the lack of significant increase in the shear bond strength after the treatment of Porcelain Liner M was due to the different compositions of Hyaline bracket and dental porcelain.

After debonding, no fracture or crack formation was observed in the Hyaline bracket. The ARI scores are noteworthy: most of the resin remained on human enamel after debonding the bracket, indicating that the enamel may have a lower risk of enamel fracture or damages at the time of debonding. However, the clinical time necessary to remove the remaining adhesive resin from the enamel after debonding is possibly increased. Light Bond showed almost the same fracture mode (ARI = 3) with or without Porcelain Liner M. This means the use of Porcelain Liner M decreased the adherence of Light Bond resin to Hyaline brackets.

CONCLUSIONS

- Commercially available adhesive resins, such as Kurasper F, Light Bond, Super Bond C&B, and Transbond XT, are useful for bonding esthetic Hyaline brackets to human enamel in orthodontic clinics.

ACKNOWLEDGMENTS

Part of this study was supported by a grant from the Ministry of Education, Culture, Sports, Science and Technology to promote the 2001 Multidisciplinary Research Project (in 2001–2005)

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