

An approach to enhance the interface adhesion between an orthodontic plastic bracket and adhesive

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SUMMARY For the purpose of improving the degree of success of plastic bracket bonding, based on the analysis of the chemical components of plastic brackets, a systematic method for the treatment of the adhesive surface of plastic brackets was introduced in this study. After sandblasting the adhesive surfaces of two commercially available plastic brackets (Spirit and Clear Bracket), a favourable surface treatment was obtained with the application of a silane coupling agent, γ -methacryloxy propyl trimethoxy silane. The findings showed that (i) the fillers added to the plastic brackets were glass fillers with Si-OH groups distributed on their surfaces; (ii) sandblasting of the bracket surface resulted in exposure of the glass fillers; (iii) combined with sandblasting, silane coupling treatment significantly increased the bond strength ($P < 0.05$), which was adequate to withstand the forces generated during orthodontic therapy; and (iv) treatment with sandblasting and silane coupling 24 hours before direct bonding did not cause a significant reduction in bond strength. It is concluded that sandblasting and silane coupling treatment offers the benefit of increasing the *in vitro* bond strength of plastic brackets for orthodontic application.

Introduction

Plastic brackets have been used in orthodontics for approximately three decades in response to the demand for improved aesthetics. Orthodontic plastic attachments (Newman, 1964; Miura and Nakagawa, 1969) have alleviated many of the problems encountered with stainless steel bands and metal brackets, such as poor aesthetics, possible loosening of the bands, and irritation to the gingiva. However, these attachments show certain disadvantages such as torque deformation (Dobrin *et al.*, 1975; Alkire *et al.*, 1997) and lower bond strengths (Moser *et al.*, 1979; de Pulido and Powers, 1983). Therefore, research efforts have focused on improvement of the characteristics of the attachment material, as the plastic attachments are normally made of pure polycarbonate. Several attempts have been made to develop new orthodontic plastic attachments by the insertion of precision-stamped stainless steel slots and ceramic fillers (Feldner *et al.*, 1994; Crow, 1995). However, the chemically inert bases of the

brackets still exhibit failure to form good bonding with acrylic and diacrylic adhesives. Even though manufacturers have introduced several methods for retention, including the application of a primer to the bracket base surface during direct bonding and the modification of the bracket base morphology, such as formation of a mechanical union via grooves or undercuts, these brackets still show lower bond strengths than conventional metal and ceramic brackets (Crow, 1995; Akin-Nergiz *et al.*, 1996).

Sandblasting is generally used for cleaning the bonding surfaces and for achieving both microretentive topography and increased surface area (Kern and Thompson, 1993; Zachrisson and Buyukyilmaz, 1993; Zachrisson, 1994), whilst silanes, as coupling agents, increased the adhesion of organic materials to ceramic materials (Newburg and Pameijer, 1978; Nishiyama, 1994; Soderholm, 1996). No studies have reported the application of these techniques to the bonding of orthodontic plastic brackets.

The purpose of the present investigation was to evaluate the effect of the sandblasting and silane coupling technique on the bond strength of orthodontic plastic brackets and adhesive.

Materials and methods

Component analysis of plastic brackets

Two commercial orthodontic plastic brackets, Spirit (SP; Ormco Co., Glendora, CA, USA) and Clear Bracket (CB; Sankin Co., Tokyo, Japan), were used.

One gram of the pulverized pieces of each plastic bracket was immersed in chloroform for 2 hours at room temperature to dissolve the organic materials. After filtration, the fillers were rinsed carefully with chloroform and dried with a desiccator for content measurement. The separated fillers were further subjected to energy-dispersive X-ray spectroscopy, infra-red spectroscopy, and X-ray diffraction.

Energy dispersive X-ray spectroscopic analysis

The composition of the surface layer of the fillers were confirmed by energy-dispersive X-ray spectroscopy (Noran Voyager III M3100; Noran Instruments Inc., Middleton, USA) attached to a scanning electron microscope (SEM; DS-720; Topcon, Tokyo, Japan). After carbon coating, the specimens were analysed with an accelerating voltage of 10 kV, spot size 100 nm, and a counting time of 100 seconds.

Infra-red spectroscopic analysis

The absorption spectra of the fillers were determined with an infra-red spectrophotometer (IR-810; Japan Spectroscopic Co., Tokyo, Japan). The testing plate, made of 0.5 per cent pulverized fillers in potassium bromide, was scanned from wave number 4000 to 400 per cm in continuous mode on a strip-chart recorder.

X-ray diffraction analysis

The X-ray diffraction patterns were recorded with a diffractometer system (Rint-1300; Rigaku

Denki Co., Tokyo, Japan) at 40 kV and 30 mA. The specimens of the fillers were scanned, respectively, from 10 to 60 degrees in 2θ (where θ is the Bragg angle) in continuous mode (1.0 degree 2θ /minute, time constant 2 seconds) on a strip-chart recorder.

Treatment of plastic bracket base

The base surface of the plastic brackets was sandblasted with 50 μ m grained alumina (PL-169; Paasche Airbrush Co., Harwood, USA) at a fixed distance of 5 mm from the bracket base, with a 0.39 MPa compressive pressure for 20 seconds. After sandblasting, the specimens were subjected to 5-minute ultrasonic washing followed by silane-coupling carried out with 2 per cent γ -methacryloxy propyl trimethoxy silane (MPTS; KBM-503, Shinetsu Chemical Co., Niigata, Japan) solution in 70 per cent ethanol. After treatment of the bracket base with silane at $23 \pm 1^\circ\text{C}$ for 20 seconds, the specimens were dried with an oil- and water-free air syringe for 15 seconds.

SEM observation of the plastic bracket base

To observe any morphological change in the bracket base before and after sandblasting, SP and CB brackets were immersed in a gold evaporator for conventional gold coating. The exact morphology of the bracket base was then observed by SEM.

Effects of treatment of bracket base surface on shear/peel bond strengths

Both SP and CB were used for the shear/peel debonding test. The plastic brackets without base surface treatment (Non), with sandblasting only (Sa), and with a combination of Sa and silane-coupling (SS) were subjected to bond testing with two different orthodontic adhesives, Orthomite Superbond (OS; Sun Medical, Tokyo, Japan) and Kurasper-F (KF; Kuraray, Osaka, Japan). The shear/peel debonding test was also performed for the specimens with SS treatment 24 hours prior to direct bonding (SS24) to evaluate the stability of SS treatment. All brackets were those for the upper central incisor, as used in

the standard edgewise technique. A conventional metal bracket with a foil-mesh base (Metal; Tomy Co., Tokyo, Japan) was used as the control.

A total of 540 sound bovine incisors were cleansed of tissue debris. After separating the crown from the root, the pulps were extirpated and the crowns were stored in distilled water at 4°C until testing. The teeth, which were randomly divided into four equal groups and used as the specimens to test the combinations of bracket and adhesive, were mounted individually in self-curing epoxy resin (Struers, Copenhagen, Denmark) in a mould (1-inch Ring Forms; Buehler, Lake Bluff, USA) with the labial enamel surface exposed. After the enamel surface had been polished with prophylaxis paste (Young Dental Mfg. Co., Earth, USA) for 15 seconds, the middle region of the labial enamel surface was covered with a piece of masking tape with a hole 5 mm in diameter to define the bonding region. The enamel surface was treated according to the manufacturers' instructions. After placing the prepared adhesive on the bracket base and positioning the bracket at the centre of the treated enamel surface, the brackets were seated using a uniform 200 g force measured by a gauge. The excess adhesive was then removed with a dental probe without disturbing the bracket. After the adhesive had set for 20 minutes at room temperature, the specimens were immersed in 37°C distilled water for 24 hours. Half of the specimens were then subjected to the debonding test, and the other half to 10,000 thermocycles between 4 and 60°C water baths with a 1 minute dwell time in each bath. For comparison, the conventional metal bracket with a foil-mesh base (A-436, Tomy Co.) and non-treated plastic brackets were also tested according to the manufacturers' instructions.

The specimens for shear/peel debonding were mounted in a universal testing machine (DCS-2000; Shimadzu, Kyoto, Japan) according to the standard method of ISO/TR 11405, with the enamel surface adjusted parallel to the shearing rod. After the shearing rod had been fitted to the base of the bracket, a shear/peel force was applied to the bracket by lowering the shearing rod perpendicularly in a gingival direction at a crosshead speed of 2 mm/minute. The maximum

shear/peel load needed to debond the bracket was recorded. The shear/peel bond strength was obtained by dividing the maximum shear/peel load by the bonding area of the bracket base. Fifteen specimens of each bracket-adhesive combination were tested. Mean values of bond strengths and standard deviations were computed, and the data were statistically analysed by a non-parametric test (Mann-Whitney *U*-test). After the shear/peel debonding test, the debonded enamel surfaces and bracket bases were examined under a magnifier (8-DN; Nikon Co., Tokyo, Japan) to evaluate fracture patterns.

Results

The two plastic brackets had different filler contents, i.e. 14.2 per cent (wt) for SP and 9.2 per cent (wt) for CB. Figure 1 shows the base morphology of plastic bracket before and after sandblasting. The SP base was a smooth and flat type, whereas the CB base was a mechanical bonding type with square process-like unions, but no undercut. Before sandblasting, little filler was exposed (Figure 1, CB-1 and SP-1), whereas after sandblasting, filler particles, having a uniform fibre of 10 µm in diameter for both CB and SP, were exposed on the adhesive surface. CB showed a relatively small amount of filler exposure compared with SP (Figure 1, CB-2 and SP-2). Figure 2 depicts the energy-dispersive X-ray spectrum of the fillers. The five significant peaks revealed the presence of carbon (added during carbon coating) at 0.282 keV, oxygen at 0.523 keV, aluminium at 1.487 keV, silicon at 1.740 keV, and calcium at 3.691 keV, indicating that the filler was most probably E-glass. The infra-red spectroscopic analysis for the fillers displayed a typical absorption at 800 to 900 per cm (Figure 3), demonstrating the existence of active Si-OH. When the X-ray diffraction analysis was conducted, the filler showed a spectral pattern with a broad peak at $2\theta = 25$ to 30 degrees, indicating that the filler is amorphous material, i.e. glass phase. The fillers in both CB and SP had the same chemical and structural features.

Table 1 indicates the shear/peel bond strengths of all of the combinations between bracket and

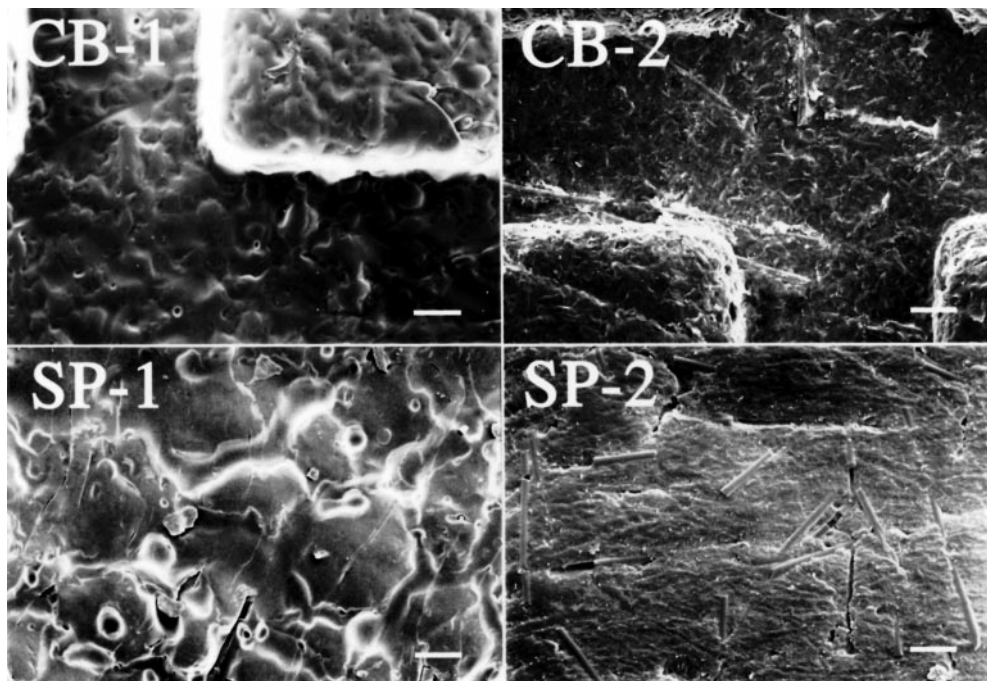


Figure 1 SEM photographs of the base morphology of plastic brackets before and after sandblasting ($\times 200$). CB-1 and SP-1, before; CB-2 and SP-2, after. Bar = 50 μm .

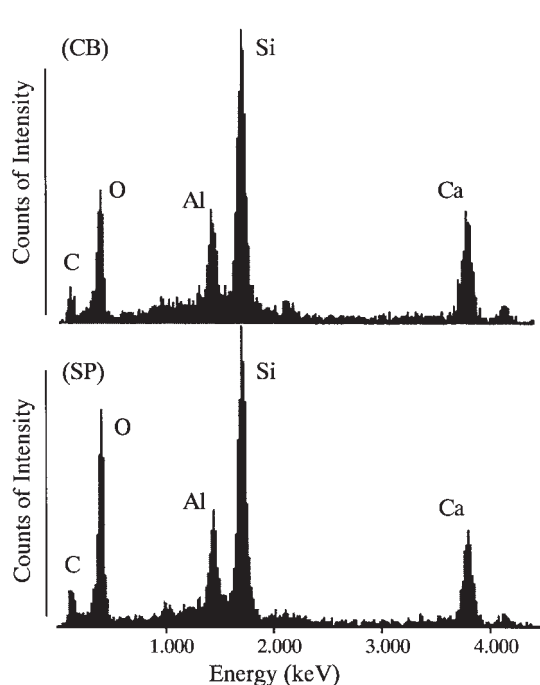


Figure 2 Energy-dispersive X-ray spectra of the fillers.

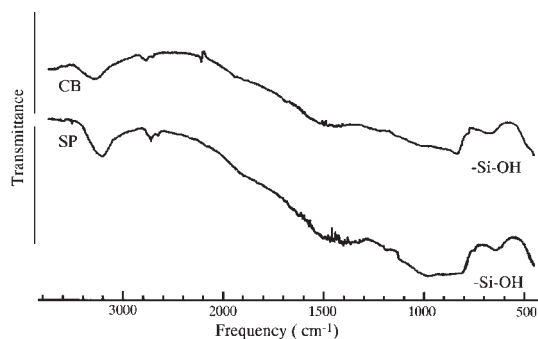


Figure 3 Infra-red absorption spectra of the fillers.

adhesive with different bracket base treatment. Bonding plastic brackets without base surface treatment (Non) such as sandblasting and silane coupling resulted in bond strengths ranging from only 1.2 to 8.0 MPa. These values were significantly lower than that of the metal bracket ($P < 0.05$). Although sandblasting (Sa) led to relatively higher values than those of Non after 24 hours direct bonding, the shear/peel bond strength decreased after thermocycling, resulting

Table 1 Shear/peel bond strengths (MPa, $n = 15$).

System	Treatment	After 24 hours		After thermocycling	
		Mean \pm SD	Range	Mean \pm SD	Range
OS + SP	Non	55 \pm 1.1	4.0–8.0	5.0 \pm 1.4	3.0–6.7
OS + SP	Sa	8.8 \pm 3.3	4.6–14.0	6.8 \pm 1.8	4.5–9.7
OS + SP	SS	15.1 \pm 4.5***	6.3–22.6	11.2 \pm 3.5****	5.9–15.5
OS + SP	SS24	12.3 \pm 4.5*	5.6–18.7	9.4 \pm 2.9***	5.5–14.0
KF + SP	Non	3.9 \pm 1.0	2.6–5.9	3.7 \pm 1.3	1.2–5.8
KF + SP	Sa	7.6 \pm 2.1*	3.8–10.1	5.7 \pm 1.7	3.5–8.1
KF + SP	SS	10.1 \pm 3.4*	5.6–15.0	8.4 \pm 1.9****	5.4–10.8
KF + SP	SS24	9.5 \pm 2.8*	5.6–14.4	8.2 \pm 1.8***	5.4–10.5
OS + CB	Non	5.1 \pm 1.1	3.8–7.7	4.7 \pm 1.1	2.7–7.8
OS + CB	Sa	6.4 \pm 1.1	4.4–8.4	5.6 \pm 1.3	3.7–8.7
OS + CB	SS	11.7 \pm 2.9***	6.8–15.6	8.0 \pm 1.8***	5.7–11.3
OS + CB	SS24	9.8 \pm 1.5*	6.1–12.6	7.0 \pm 1.2***	5.4–9.9
KF + CB	Non	4.3 \pm 1.3	2.8–6.6	3.6 \pm 1.3	2.0–5.7
KF + CB	Sa	5.9 \pm 1.5	4.1–9.5	5.5 \pm 1.2	3.8–7.5
KF + CB	SS	8.6 \pm 1.5*	5.8–11.0	6.7 \pm 1.2***	5.8–8.8
KF + CB	SS24	7.7 \pm 1.5*	5.0–10.2	6.5 \pm 1.3***	5.0–8.6
OS + Metal	Non	10.5 \pm 3.3*	6.1–15.4	8.1 \pm 2.3***	5.5–12.8
KF + Metal	Non	8.1 \pm 1.8*	5.5–11.5	7.1 \pm 1.4***	5.1–9.4

*Significant difference compared with Non in the same bracket–adhesive combination after direct bonding for 24 hours; in the case of metal bracket compared with Non of CB or SP ($P < 0.05$). **Significant difference compared with Sa treatment ($P < 0.05$). ***Significant difference compared with Non after thermocycling ($P < 0.05$). Non = plastic brackets without base surface treatment; Sa = plastic brackets with sandblasting only; SS = plastic brackets with a combination of sandblasting and silane-coupling; SS24 = specimens with SS treatment 24 hours prior to direct bonding.

in no significant difference from Non. When comparing Sa and silane-coupling (SS), there was a further increase of bond strength and even after thermocycling, a favourable bond strength was found that was significantly higher than that of Non ($P < 0.05$). Similarly, bond strength for the specimens SS24, where SS treatment was completed 24 hours prior to direct bonding, showed significantly higher values than those of Non ($P < 0.05$). The SS method actually enhanced the bonding between the base surface and adhesive. In comparison with the bond strengths of SP and CB, SP showed relatively higher values under the conditions Sa, SS and SS24.

Examination of the fracture patterns after debonding showed that when un-treated plastic brackets (Non) or treated plastic brackets (Sa, SS, SS24) were bonded with OS adhesive, most of the specimens showed bond failure at the enamel/adhesive interface (E/A). For KF, in the condition of Non, most of the specimens showed bond failure at the bracket/adhesive interface

(B/A); whereas in the case of Sa, SS and SS24, many of the bond failures occurred at E/A or both E/A and B/A (Table 2).

Discussion

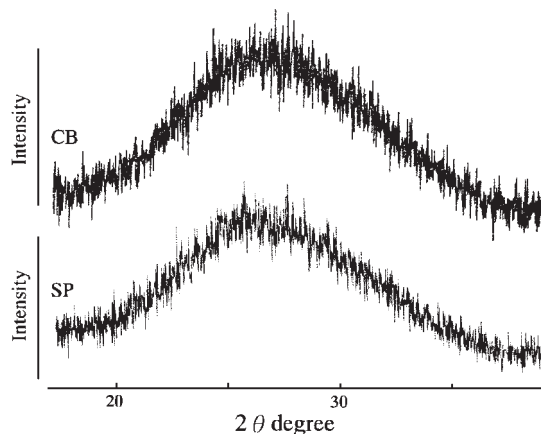
Reinforced plastic brackets ensure a favourable hardness, toughness and a smooth surface polishing of the bracket body when exposed to the oral environment (Gomi *et al.*, 1993), but the base surface has not yet been improved in terms of bond strength. Previous studies concerning plastic brackets have shown their bonding deficiency. However, few reports have documented the characteristics of the matrix and the filler of plastic brackets but rather observed only the morphology of the fillers (de Pulido and Powers, 1983; Crow, 1995).

With the inert polycarbonate matrix of plastic brackets, adequate adhesion is difficult to achieve even when applying primer (Crow, 1995). In the present study, the component analysis of the

Table 2 The number of bond failures at each location.

Adhesive-bracket combination	Bracket		B/A interface		Mixed		E/A interface		Enamel	
	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)
OS + SP	0	0	0	0	1	1	14	14	0	0
OS + SP-Sa	0	0	0	0	0	0	15	15	0	0
OS + SP-SS	0	0	0	0	0	0	15	15	0	0
OS + SP-SS24	0	0	0	0	0	0	15	15	0	0
KF + SP	0	0	12	13	3	2	0	0	0	0
KF + SP-Sa	0	0	8	10	7	5	0	0	0	0
KF + SP-SS	0	0	6	7	6	5	3	3	0	0
KF + SP-SS24	0	0	6	8	7	7	2	0	0	0
OS + CB	0	0	0	0	2	3	13	12	0	0
OS + CB-Sa	0	0	0	0	0	2	15	13	0	0
OS + CB-SS	0	0	0	0	0	1	15	14	0	0
OS + CB-SS24	0	0	0	0	0	1	15	14	0	0
KF + CB	0	0	14	13	1	2	0	0	0	0
KF + CB-Sa	0	0	10	11	5	4	0	0	0	0
KF + CB-SS	0	0	7	8	4	6	4	1	0	0
KF + CB-SS24	0	0	8	8	3	5	4	2	0	0
OS + Metal	0	0	3	2	4	7	8	6	0	0
KF + Metal	0	0	4	2	7	6	4	7	0	0

Bracket = bracket fracture; B/A = bracket/adhesive interface; E/A = enamel/adhesive interface; mixed = both B/A and E/A. Enamel fracture: (1) 24 hours after direct bonding; (2) after thermocycling. $n = 15$. Sa = plastic brackets with sandblasting only; SS = plastic brackets with a combination of sandblasting and silane-coupling; SS24 = specimens with SS treatment 24 hours prior to direct bonding.

**Figure 4** X-ray diffraction spectra of the fillers.

plastic brackets SP and CB revealed a filler of glass fibres with active Si-OH (Figures 3 and 4). Since the manufacturing method is injection moulding, where the dispersion of fillers in a polymer is forced into the mould at controlled temperatures, the fillers added to each plastic

bracket are fairly well distributed, with a limited part of the filler exposed on the bracket surface. In view of this component information, a sandblasting-silane coupling method to improve the plastic bracket bond strength was investigated. First, sandblasting exposed more filler on the adhesive surface of the bracket base. Secondly, silane-coupling with the application of γ -MPTS resulted in a reaction between silicon and silanol groups, while on the opposite side of the silane molecule co-polymerization occurred between the silane molecule and adhesive in either the MMA or Bis-GMA system (Soderholm, 1996). Therefore, by combining the mechanical and the chemical mechanisms, this method could enhance the plastic bracket bond strength (Table 1).

Sandblasting treatment has been extensively applied in orthodontics for increasing mechanical interlock of metal, porcelain and amalgam during bonding (Millett *et al.*, 1993; Zachrisson and Buyukyilmaz, 1993; Zachrisson, 1994). In the present study, sandblasting of the adhesive surface of the plastic bracket was a primary

treatment. Although sandblasting caused a relatively higher bond strength 24 hours after direct bonding, the increased bond strengths were reduced after thermocycling resulting in no significant difference from the non-treatment, especially in the case of CB. Adhesives might form only a simple mechanical bonding with the fillers exposed on the bracket surface. Silane coupling treatment has also been extensively applied in the bonding of organic materials to ceramic materials in dentistry, as has sandblasting (Nishiyama, 1994). As a representative agent of silanes, γ -MPTS has been used for the surface treatment of the inorganic filler in composite resins (Soderholm, 1996). As the fillers are embedded in the matrix with only a small part exposed, increasing their exposure area by sandblasting might simultaneously remove the silane agents. Therefore, re-treatment with γ -MPTS in the present study further increased the bonding of plastic brackets and adhesives. Even when subjected to thermocycling, the bond strength still showed a favourable endurance property ($P < 0.05$). An additional chemical bond might have formed when the adhesive came into contact with the SS-treated base.

The two plastic brackets, SP and CB, had different filler contents; SP had the higher content (14.2 per cent) and CB the lower (9.2 per cent). When the bond strengths of the SS groups were compared, SP showed a relatively larger increase than CB, which might have resulted from the difference in filler content. The amount of filler might affect the elasticity of the bracket material and the surface microstructure of the bracket base. Furthermore, with a higher filler content, SP might offer more opportunities for chemical interlocking.

Orthodontic attachments should obviously be removed completely after active treatment. In the present study, many of the fracture sites of the SS-treated plastic brackets bonded with KF occurred at the E/A interface, whereas with surface treatment, fracture sites occurred at the B/A interface. There is a tendency for enamel to fracture as the bonding between the bracket and adhesive interface increases (Bishara and Trulove, 1990; Winchester, 1991). Plastic brackets exhibit a mechanical feature that ensures bracket

fracture before high force levels are achieved (Gomi *et al.*, 1993). Therefore, the mechanical feature of the plastic bracket can help prevent enamel from fracturing and thus protect tooth integrity.

Whilst SS treatment performed at chairside may be time-consuming, the present study also demonstrated that pre-treatment with sandblasting-silane coupling 24 hours before direct bonding was effective. The durability evaluation of silane coupling treatment to glass plate has shown durable bonding even with a small increase in the first month after silane treatment (Nishiyama *et al.*, 1991, 1995). Therefore, the sandblasting-silane coupling method may be suitable as a pre-treatment procedure before bonding. Furthermore, this application is possible not only for plastic brackets but also for some new ceramic products with plastic bases (Olsen *et al.*, 1997).

Conclusion

The fillers added to the plastic brackets were glass filler with Si-OH groups distributed on their surface.

Sandblasting of the bracket surface resulted in exposure of the glass filler.

Combined with sandblasting, silane-coupling treatment significantly increased the bond strength to withstand the forces generated during orthodontic treatment.

Completing the sandblasting-silane-coupling treatment 24 hours before direct bonding did not decrease the bond strength.

It is concluded that the method of combined sandblasting and silane-coupling treatment offers the benefit of increased *in vitro* bond strength of plastic brackets for orthodontic application.

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