J Osaka Dent Univ 2015 (April) ; **49 (1)** : 115–121.

Study on adhesion of orthodontic brackets on enamel with resin cements

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We evaluated the mechanical properties and bond strength of commercially available adhesives used with orthodontic brackets on enamel. Debonding of the brackets must be done gently so as not to cause pain or injury to the enamel. Three commercially available adhesive resin cements, Super-Bond (SB), Beauty Ortho Bond (BO) and Rely X™ Unicem 2 Automix (UN), were used. Two self-etching primer adhesive systems, Beauty Ortho Bond (BO) and Rely X™ Unicem 2 Automix (UN), were used. The bond strength to enamel of SB placed in 37 °C water was 18.8 Mpa after one day, 16.6 Mpa after 6 months, and 19.2 Mpa after one year. The bond strength to enamel of BO in 37°C water was 11.6 Mpa after one day, 8.9 Mpa after six months, and 13.6 Mpa after one year. The bond strength to enamel of UN in 37°C water was 5.8 Mpa after one day, 4.0 Mpa after six months, and 13.6 Mpa after one year.

The adhesives showed excellent bond strength of approximately 6～15 Mpa with both precious alloy brackets after 10,000 thermocycles consisting of alternately cycling the specimens in 5°C and 55°C water for one minute each. We concluded that the application of adhesives greatly improved the bond strength of the resin cements. (J Osaka Dent Univ 2015 ; 49 (1) : 115–121)

Key words : Enamel ; Direct bonding ; Etching

INTRODUCTION

The edgewise method is the most frequently used treatment to control tooth alignment in orthodontics. Buonocore described bonding between acrylic resin and enamel in 1955.[1] In orthodontic dentistry, bonding is typically performed on healthy enamel. This bonding method was applied to orthodontic dentistry, and a direct bonding method in which brackets are directly bonded to teeth was subsequently developed in 1965.[2, 3] Manipulation of the materials and esthetics have been improved by the development of the direct bonding system. Due to improvements in bonding technology, enamel and brackets are solidly bonded during the dynamic treatment period.

Previous studies on the direct bonding method mainly focused on improving the bond strength, and obtaining satisfactory results. Disadvantages of this technique include pain and damage to the enamel during bracket removal due to the increased bond strength. These issues need attention. The adhesive should bond directly to the tooth surface set quickly in the moist oral environment, retain a solid bond against complex oral and orthodontic forces, and allow for easy removal of the brackets without pain or damage to the enamel.[4] We used three resin adhesives with different surface treatments. Bond strength of the bracket to the enamel and damage during bracket removal were compared for the three adhesives on extracted bovine teeth. Special attention was paid to chronological changes in the bond strength in water and injury to the enamel on removal. We established an easy and durable bonding method and determined the bond strength of the orthodontic brackets to the enamel.

MATERIALS AND METHODS

Materials

The adhesive resin cements used were Super-Bond (SB) (Sun Medical, Shiga, Japan), Beauty Ortho Bond

Table 1 Materials

Super-Bond (Sun medical)	Etchant Gel : 20% Phosphoric acid Monomer : MMA, 4META Polymer powder : PMMA Catalyst : Tri-n-butylborane
Beauty Ortho Bond (Shofu)	Primer A : Water, Acetone, Others Primer B : Phosphonate monomer, Anhydrous Ethanol, Coloring agent, Others Paste : Bis-GMA, TEGDMA, Glass filler, Others
RelyX™ Unicem 2 Automix (3M ESPE)	Base Paste : Glass filler, MDP, TEGDMA, SiO2, Catalyst Paste : Glass filler,

MMA : 4-[2-(Methacryloyloxy) ethoxycarbonyl] phthalicanhydride, Bis-GMA : 2, 2-bis[4-(3-methacryloxy-2-hydroxypropoxy) phenyl] propane, TEGDMA : Triethyleneglycol dimethacrylate, MOP : 10-ethacryloxydecyl dihydrogen phosphate.

(BO) (Shofu, Kyoto, Japan), and RelyX™ Unicem 2 Automix (UN) (3M ESPE, Tokyo, Japan). Table 1 shows the composition of each bonding system. Stainless steel metal brackets for the maxillary central incisors (Tomy International, Tokyo, Japan) were used.

Fabrication of a bonding sample

The experiments were done on frozen extracted bovine anterior teeth that had been defrosted. The roots were removed near the cervical area under irrigation, the contents of the pulp cavity were extirpated, and the sample was embedded in epoxy with the labial surface exposed. The adhesive surface was prepared to make it flat by trimming the sample under running water using a model trimmer, and polishing it with # 600 water-resistant polishing paper. Light-curing resin was applied on the bracket base surface according to each manufacturer's instructions as shown in Table 1, after surface treatment, and 200 gm of force were applied. Surplus resin was removed with an explorer, and light irradiation was performed for 10 seconds at 45 degrees from the mesial and distal sides of the bracket using a visible light curing unit (Curing Light XL 3,000 ; 3M ESPE). The thermal cycle test, which is an accelerated deterioration test that simulates the oral environment, was used to test the enamel bonding of 10 samples fabricated for each group.

Measurement methods

Long-term water immersion test

Test samples were immersed in water at 37°C for 24 hours, 6 months, and 1 year after fabrication to test the tensile and bonding strengths with the enamel. A universal tester (IM-20 ; INTESCO, Chiba, Japan) was used at a crosshead speed of 0.3 mm/min for these measurements. Ten samples were prepared for each study, and the mean and standard deviation (SD) were calculated. A one-way analysis of variance and Tukey's test were performed at the 5% significance level for statistical analyses (n = 10).

Thermal cycle test

Fabricated samples were immersed in water at 37°C for 24 hours, and each sample was then immersed in constant temperature water tanks at 5°C and 55°C (Thermal cycling tank K 178 ; Tokyo Giken, Tokyo, Japan) for cycles of 1 minute each. The thermal cycle was applied 10,000 times, and measurements were taken using the IM-20 universal testing machine at a crosshead speed of 0.3 mm/min. Ten samples were prepared for each test, and the mean and SD were calculated (n = 10).

Observation of the destruction of enamel after the tensile test

Fracture cross-sections in the samples were vacuum-dried after the long-term water immersion and thermal cycle tests. Gold evaporation treatment was done using a JFC-1500 ion sputtering device, and observed with a JSM-5610 LV scanning electron microscope (both from JEOL, Tokyo, Japan).

Measurement of the fluoride eluted

Disk shaped samples of adhesive 4 mm in diameter and 2 mm thick were allowed to set in a mold according to the manufacturers' instructions. The samples were immersed in 10 mL of water immediately after fabrication, stored in a constant temperature chamber at 37°C for 24 hours, and the eluate was collected. A total of 0.5 mL of TISAB III (Merck, Tokyo, Japan) was added to each sample, and the amount of fluoride in the eluate was measured using a Model 720 A pH ion meter (Orion Research, Boston, MA, US) and a Orion 9609 BNWP (Thermo Fisher Scientific, Waltham, MA, US). The fluoride electrode was checked four times at 0.02, 0.1, 1, and 20 ppm. The samples were transferred into 10 mL of distilled water, and the same procedure was repeated with fluoride measurements at 3 days, 5 days, 1 week, 2 weeks, 3 weeks and 1 month.

RESULTS

Bond strength to enamel after long-term water immersion

Figure 1 shows the bond strength of the three resin adhesives with different bonding systems to the enamel surface after immersion for 24 hours, 6 months and 1 year in water at 37°C. The bond strength for SB was 20.4 ± 3.6 MPa at 24 hours, 15.2 ± 5.7 MPa at 6 months, and 19.3 ± 10.4 MPa at 1 year. There were no significant differences among the three. The bond strength for BO to enamel was 15.4 ± 4.3 MPa at 24 hours and 8.9 ± 3.1 MPa at 6 months, and 7.3 ± 3.8 MPa at 1 year. These valves were significantly lower after 6 months and 1 year of water immersion compared with after 24 hours. The bond strength of UN was 7.8 ± 1.7 MPa at 24 hours, 4.0 ± 1.9 MPa at 6 months, and 13.6 ± 6.6 MPa at 1 year. Although no significant difference was observed between 24 hours and 6 months, the bond strength to enamel was significantly greater after 1 year compared with after 24 hours and 6 months.

Thermal cycle test

Figure 2 shows the bond strength of each sample after water immersion at 37°C for 24 hours and 10,000 thermal cycles. The bond strength of SB was 20.4 ± 3.6 MPa after 37°C water immersion for 24 hours, and 20.7 ± 5.7 MPa after 10,000 thermal cycles. The bond strength of BO was 15.4 ± 4.3 MPa after 37°C water immersion for 24 hours, and 15.8 ± 2.9 MPa after 10,000 thermal cycles. These values for UN under the same conditions were 7.8 ± 1.7 MPa, and 6.2 ± 2.4 MPa, respectively. No significant differences were observed in the bond strength to enamel after 37°C water immersion for 24 hours and 10,000 thermal cycles.

Observation of the destruction of enamel after the tensile test using scanning electron microscopy (SEM)

Figure 3 shows SEM images of the enamel surface of each resin cement sample after the tensile test over time. The enamel surface of SB, which was treated

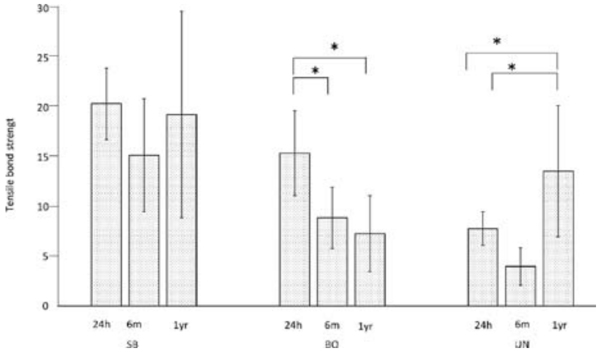

Fig. 1 Influence of long-term immersion test on the tensile bond strength of each material (*$p < 0.05$ by Tukey's test).

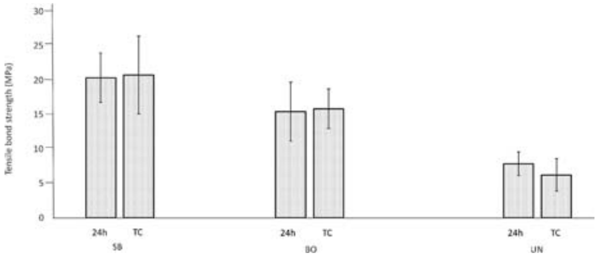

Fig. 2 Influence of thermal cycling on the tensile bond strength of each material.

There were significant differences among the results labeled with the same letters.

24 h : After bonding, the specimens were stored in water at 37 ℃ for 24 hours, TC : Thermal cycling with immersion in constant temperature water tanks at 5℃ and 55℃ for 10,000 cycles of 1 minute each, SB : Super-Bond, BO : Beauty Ortho Bond, UN : Rely X^{TM} Unicem 2 Automix.

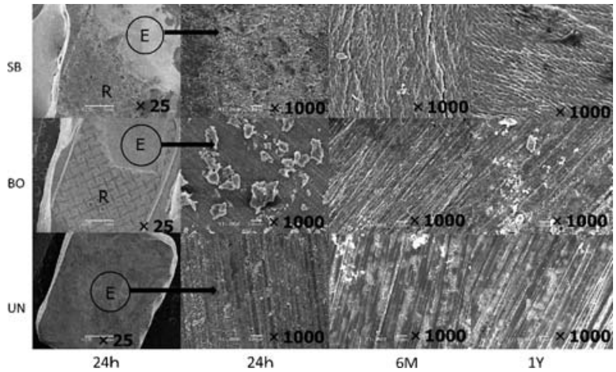

Fig. 3 SEM image of enamel surfaces after the tensile bond strength test at 24 hours (24 h),
6 months (6 M) and 1 year (1 Y).
E : Enamel, R : Resin.

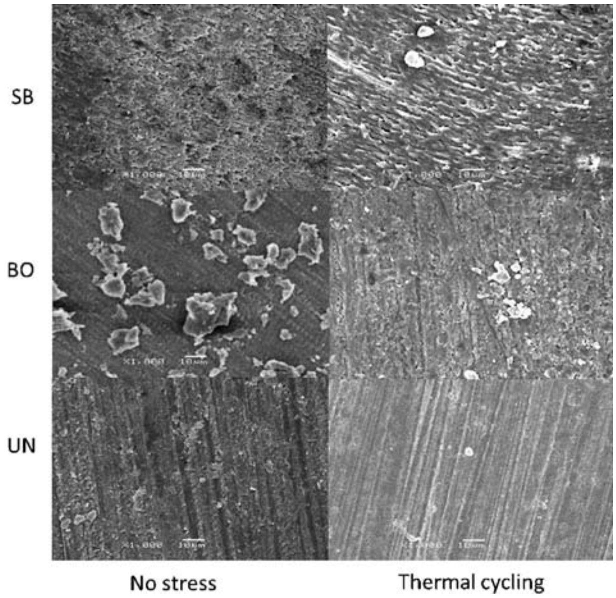

Fig. 4 SEM image of the enamel surfaces after the tensile bond tests (×1000).

with phosphoric acid, was markedly decalcified, and a clear enamel prism structure was observed, suggesting cohesive failure with the enamel. Destruction of adhesion between the resin and enamel surface was observed with BO, which involved a self-etching primer. A smooth enamel surface was observed in UN, which is a self-adhesive resin cement. However, no marked changes were observed in the resin layer that was detached from the enamel surface (Fig. 4).

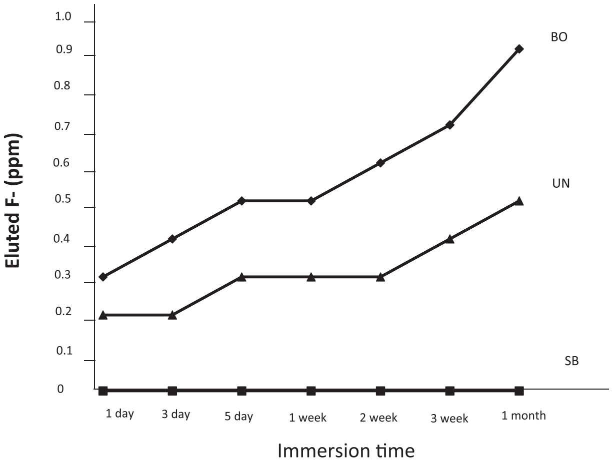

Fig. 5 Amount of fluoride eluted after immersion for 1 day, 3 days, 5 days, 1 week, 2 weeks, 3 weeks and 1 month.

Amount of fluoride eluted

Figure 5 shows the amount of fluoride eluted from the enamel after 24 hours, 3 days, 5 days, 1 week, 2 weeks, 3 weeks and 1 month of immersion. The elution of fluoride was not detected in SB. The elution of fluoride peaked at 24 hours in BO, and gradually decreased at 3 days, 5 days, and 1 week. However, these results suggest that approximately the same amount of fluoride ion was eluted between 3 days and 2 weeks, as between 3 weeks and 1 month. The elution of fluoride also peaked at 24 hours in UN, while approximately the same amount of fluoride was eluted at 3 days, 5 days, 1 week, 2 weeks, 3 weeks and 1 month.

DISCUSSION

Because compression and tension are applied in multiple directions to brackets used in orthodontic treatment, it is difficult to evaluate the bond strength required in clinical practice using only a one-directional bond test. Various methods have been investigated in which the brackets were directly bonded to the teeth for dynamic orthodontic treatment.[4–10] The direct bonding method is frequently used in treatments involving resin adhesive, especially light-curing resin, due to its easy manipulation and short chair-time. Previous bonding systems included etching, water irrigation, and drying before bonding with resin cement. However, a 2-step bonding system using a self-etching primer and light-curing resin was developed to further simplify this procedure. Self-etching primer is less invasive to enamel than phosphoric acid, resulting in less enamel damage during bracket removal. However, the decalcification ability of the acid-resistant enamel surface is poor, and there is a risk of bracket detachment during dynamic treatments due to poor bonding.[11, 12]

Bracket bonding in clinical practice is performed in a more difficult environment than in our *in vitro* study using extracted teeth. Bracket bonding is especially difficult in the molar region where isolation is difficult due to saliva and high moisture levels. We used a long period of water immersion and a cold thermal load cycle test to examine bonding between brackets and enamel. According to ISO guidelines regarding bonding,[13] a thermal cycle test of 500 times in water between 5°C and 55°C is recommended to test bonding durability. Miyazaki *et al.*[14] and Gale *et al.*[15] reported that loading 5,000 times corresponded to one year of loading in an intraoral environment, suggesting that this number can be used as an index for durability.

Optimal pretreatment method

Previous studies have reported that pretreatment of the tooth surface with acid increases the bond strength. Buonocore treated enamel with 85% phosphoric acid to improve resin bonding to enamel.[1, 16] Acid treatment decalcified the enamel surface, resulting in the formation of a reticular structure. An irregular surface is known to increase the bonding area, allowing the monomer to infiltrate the enamel surface and create mechanical interlocking.[17] The mechanism underlying decalcification of the enamel surface by the etching involves dissolution of highly calcified enamel prisms containing a large amount of calcium, leaving a weakly calcified prism sheath.[17–19] However, Kirino *et al.*[20] showed that decalcification started in the prism sheath when enamel was treated with acid for a short time. Acid pretreatment is also thought to create an irregular reticular surface on the organic-rich enamel surface. A thorough infiltration and curing of the resin into the decalcified surface is important for mechanical interlocking.

Among the three resin adhesives examined in the present study, only SB used phosphoric acid etching. BO used a self-etching primer treatment, while UN is a self-adhesive cement. The bond strength of BO was lower than that of SB. SEM images of BO revealed destruction of the resin layer and enamel surface interface. These results suggest that resin tags were formed by the polymerization setting of the infiltrated resin cement monomer into the irregular enamel surface formed by the acid etching. However, since the decalcification capacity of the self-etching primer is lower than that of phosphoric acid, and because the monomer components in the pastes, Bis-GMA and TEGDMA (Triethyleneglycol dimetacrylate), are highly hydrophobic and have low affinities for water, the monomer did not infiltrate or diffuse deeply into the decalcified area.

Apart from SB, the other two resins contain bifunctional methacrylate with a high molecular weight as well as a large amount of nonorganic filler. Therefore, wettability and infiltration into the enamel are poor. A monomer cannot thoroughly infiltrate when resin cement bonds with the pretreated enamel. MMA liquid is used in SB, which is a monomer with a relatively low molecular weight. It more easily penetrates the tooth than the other two resin cements. MMA polymerization is accelerated by the polymerization initiator tri-n-butyl borane. Since polymerization starts from the enamel interface, the bond strength is high. Enamel prisms were observed in SEM images, suggesting cohesive failure with enamel.

In the self-adhesive cement UN, enamel damage was reduced because the enamel was not pretreated. Since UN is hydrophilic before setting and becomes hydrophobic after setting, it is good for use in a moist environment. However, the bond strength of UN was lower than that of the other two cements. Furthermore, the bond strength after 6 months was decreased, whereas that after 1 year was greater than the initial strength. This was attributed to the phosphate ester monomer, which is an adhesive monomer contained in base cement and is highly hydrophilic. It infiltrates into and diffuses around the enamel surface, and is bonded by polymerization setting. Since UN becomes hydrophobic after TEGMA sets, it presents a high bond strength in a moist environment. A polished enamel surface was observed in SEM images, and it was confirmed that the cement detached from the enamel interface, suggesting that there was no decalcification of the enamel. The bond strength of UN was lower than that of the two other cements due to the low permeability of the monomer components.

Bracket adhesiveness

As already described, more factors influence the tooth, bracket and adhesive materials in a clinical setting than in basic extraoral studies. Various forces are imparted during orthodontic treatment such as the force of the arch wire and rubber rings, occlusal force, immersion in saliva, and intraoral thermal changes. Other factors that influence bonding strength in a clinical setting include thick bonding layers between the tooth and bracket, and difficulty in drying the tooth surface. According to Jarabak, a light force is considered to be 1～4 ounces (25～100 g), and a heavy force 16～23 ounces (400～575 g), for example, a Bull loop.[21] The maximum force generated by the rectangular wire used in the edgewise method is 3 pounds (1,350 g).

Newman[6, 8] reported that the maximum stress on a bracket directly bonded to a tooth was 2.9 MPa in a clinical setting. However, this value did not consider accidental impact forces, creep deformation of an arch wire, long-term immersion in saliva, and intraoral thermal changes. Reynolds[22] reported that a 5.9～7.9 MPa bond strength was the minimum required for bracket bonding. Retief[23] observed enamel destruction when a force of 13.5 MPa was applied during bracket removal. Miura *et al.*[24] showed that a force greater than 3.9 MPa was necessary in a clinical setting. We found that the bond strength of SB and BO exceeded these values even after immersion in water and thermal cycling. The bond strength of UN, a self-adhesive resin cement, was lower than 3.9 MPa at 6 months. This was attributed to the low number of resin tags between the cement and enamel surface due to the absence of a phosphoric acid pretreatment and self-etching primer, as well as inadequate polymerization. Furthermore, there was failure to polymerize during the change from the hydrophilic state to the hydrophobic state due to the low permeability of the bonding monomer. However, the resin monomer thoroughly infiltrated the enamel, and setting was accelerated over time.

Phosphoric acid etching, which is done as pretreatment in SB, destroys hydroxyapatite structures, and may result in damage to the enamel surface. Difficulties with brushing due to the presence of orthodontic appliances may cause accumulation of plaque, white spots and decalcification.[25, 26] Remineralization to strengthen the quality of the teeth may be necessary when self-etching primer is used without phosphoric acid treatment and self-adhesive cement. Fluoride is often applied to strengthen the tooth. BO and UN used in the present study contain fillers. Long-term tooth fortification can be expected by the fluoride release.

Saito *et al.*[27] reported that the bond strength of chemically cured resin-modified glass ionomer cement was similar to that of resin adhesive as an orthodontic adhesive, and that pain and residual cement during debonding were minimal. Koga[28] demonstrated the absence of enamel damage during debonding with conventional and light-cured resin-modified glass ionomer cement. However, completion of the glass ionomer curing reaction requires time. The water absorption rate of the cement after setting is slightly high, resulting in inferior physical properties to those of composite resin due to water absorption swelling and color changes. BO used in the present study is a composite resin (S-PRG) containing fluoride-releasing aluminosilicate glass, which is a major component of glass ionomer cement. Acid-reactive fluoride containing glass and polyacid was sufficiently reacted under water, and the glass ionomer phase only formed on the filler surface. Nakatsuka *et al.*[29] reported that the fluoride-releasing and -recharging abilities of S-PRG resin were similar to those of glass ionomer cement. These findings suggest that fluoride-releasing materials are effective in orthodontic treatments.

A self-adhesive resin cement has not yet been developed for orthodontic treatment. Bond strength to the tooth has been found to be slightly lower in the one-step bonding system than in the two-step bonding system.[30, 31] Improvements in the bonding of self-adhesive resin cement to enamel should be investigated multilaterally in order to achieve bracket bonding without enamel damage with the two-step bonding system.

CONCLUSIONS

We compared the bonding ability of three resin adhesives with different bonding systems and found that although SB, which uses phosphoric acid treatment, showed the highest bond strength, enamel damage was observed after debonding. The bond strength of BO, which requires a self-etching primer treatment, and UN, a self-adhesive resin cement, were lower than that of SB. Also, slight enamel damage after debonding was observed with fluoride-releasing BO and UN. The results of the present study suggest that to be ideal as an orthodontic direct bonding material, a cement should have fluoride-releasing properties that fortify tooth quality around the bracket, and have adequate bond strength without damaging the enamel.

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