INTRODUCTION

Indirect bonding systems (IDBSs) are now used worldwide for bonding lingual and buccal orthodontic brackets as a group, and fixed retainers. An IDBS involves a two-stage procedure. First, before bonding, the brackets are positioned on a study model of the patient’s teeth with water soluble glue or filled resin. The brackets are then transferred from the model to the patient’s mouth via a transfer tray. An IDBS offers advantages such as increased precision and accuracy of bracket placement, reduced chair time and need for rebonding, improved bracket bonding to posterior teeth, and less operator stress. The drawbacks of this system are technique sensitivity, the need for an additional set of impressions, and increased laboratory preparation time.

It has been reported that the shear bond strength (SBS) in an IDBS is comparable to, or lower than, that in a direct bonding system (DBS). Several studies of IDBSs have reported the effects of different adhesive systems, enamel surface treatment, adhesion promoters, on the SBS. Some studies have reported that an adhesion promoter significantly increased the SBS in IDBSs and DBSs, whereas others have reported that this promoter did not significantly increase the SBS in a DBS.

Some DBS studies have shown that the SBSs achieved with high viscosity adhesives with a high amount of filler were significantly higher than those obtained with low viscosity adhesives with a small amount of filler. However, other studies found no significant differences among the SBSs achieved with adhesives of different viscosities.

No literature was found on the effects of adhesive viscosity and an adhesion promoter on the SBS of brackets in an IDBS. The aim of this study was to assess the effects of adhesives with three different viscosities and an adhesion promoter on the shear bond strength (SBS) of orthodontic brackets bonded to human premolars with an indirect bonding system (IDBS). High, medium and low viscosity IDBSs with and without application of the adhesion promoter were used. The mean SBSs of the high and low viscosity IDBSs were significantly higher than that of the medium viscosity IDBS. Application of the adhesion promoter significantly increased the SBSs. The adhesion promoter significantly increased the surface roughness and free-energy of enamel. Irrespective of application or nonapplication of the adhesion promoter, the high and low viscosity IDBSs are effective for bracket bonding. Use of the medium viscosity IDBS in combination with the adhesion promoter is recommended for obtaining a clinically acceptable SBS.

The aim of this study was to assess the effects of three adhesives with different viscosities and an adhesion promoter on the shear bond strength (SBS) of orthodontic brackets bonded to human premolars with an indirect bonding system (IDBS). High, medium and low viscosity IDBSs with and without application of the adhesion promoter were used. The mean SBSs of the high and low viscosity IDBSs were significantly higher than that of the medium viscosity IDBS. Application of the adhesion promoter significantly increased the SBSs. The adhesion promoter significantly increased the surface roughness and free-energy of enamel. Irrespective of application or nonapplication of the adhesion promoter, the high and low viscosity IDBSs are effective for bracket bonding. Use of the medium viscosity IDBS in combination with the adhesion promoter is recommended for obtaining a clinically acceptable SBS.

Keywords: Indirect bonding, Adhesion promoter, Viscosity, Shear bond strength, Adhesive remnant index

MATERIALS AND METHODS

The Ethics Committee of our institution approved this study (ECNG-R-330). A priori power analysis showed that 17 teeth would be required in each group to determine a significance with a power of 0.95, an alpha error probability of 0.05, an effect size of 0.4 and six groups.

Tooth preparation

One hundred and thirty-two extracted premolars were collected and stored in a 0.1% thymol solution at 4°C prior to use. The selection criteria for teeth included enamel without any chemical pretreatment, hypoplastic, cracks or caries. Buccal enamel of each tooth was cleaned and polished using a mixture of water and pumice without fluoride in a rubber cup for 10 s, washed with a water for 10 s, and then air dried.

Brackets used

Metal premolar standard edgewise brackets (Victory series; 3M Unitek, Monrovia, CA, USA) with a mean bracket base area of 9.94 mm² were used in this study.

Transfer tray fabrication

Transfer trays were made for the IDBS. A water-soluble glue (Yamato Nori, Yamato, Tokyo, Japan) was applied to the bracket base. The bracket with glue was put on buccal enamel and pressed firmly into place to squeeze...
glue from the rim of the bracket base. An explorer was used to remove excess glue, and the glue was allowed to dry for 20 min. Paraffin wax (Rinkai, Tokyo, Japan) was built up at the cervical side of the bracket placed on buccal enamel to provide a spillway. The transfer trays were made of a vinyl polysiloxane impression material (Regofix Transparent, Dreve, Unna, Germany). After the transfer tray material had set, the transfer trays with the embedded the brackets were removed from the enamel surface. The glue on the bracket base was scrubbed using a toothbrush under running water and the bracket base was blown dry. Buccal enamel was washed to remove the glue.

**Bonding protocols**

The 102 teeth and their corresponding transfer trays were divided equally into six groups of 17. The brackets were indirectly bonded to buccal enamel according to one of the following six protocols. The orthodontic adhesives, adhesion promoter and light-curing unit used are listed in Table 1.

**IDBSs without application of adhesion promoter**

1. High viscosity (Transbond XT adhesive) IDBS

Excess water on buccal enamel was blotted with a cotton pellet to leave slightly the surface moist. Transbond Plus self-etching primer (SEP; 3M Unitek) was rubbed on buccal enamel for 5 s and then blown off lightly using a drier for 2 s. Transbond XT (TXT) adhesive (3M Unitek) was applied to the bracket base embedded in the transfer tray. The transfer tray with the bracket was placed on the tooth. The excess adhesive that overflowed into the spillway was removed from buccal enamel using an explorer. The adhesive was light-cured using an Ortholux Luminous Curing Light Unit (3M Unitek) for 6 s.

2. Medium viscosity (BeautyOrtho Bond Paste Viscos) IDBS

Buccal enamel of each tooth was thoroughly dried using the air drier. BeautyOrtho Bond (BOB) SEP (Shofu, Kyoto, Japan) was rubbed onto buccal enamel for 10 s and was blown off lightly for 5 s. BOB Paste Viscos (Shofu) was applied to the bracket base. The placement of the transfer tray, removal of excess adhesive, and light-curing of the adhesive were performed in the same way as in the high viscosity IDBS.

3. Low viscosity (BOB Flowable Paste) IDBS

Each tooth was dried, and BOB SEP was applied the same way as in the medium viscosity IDBS. BOB Flowable Paste was put on the bracket base. The placement of the transfer tray, removal of excess adhesive, and light-curing of the adhesive were performed in the same way as in the high and medium viscosity IDBSs.

**IDBSs with application of adhesion promoter**

Each tooth crown was thoroughly dried, an adhesion promoter (Enamel Conditioner, Shofu) was applied to buccal enamel for 10 s, and then the enamel was rinsed. Buccal enamel was blotted and thoroughly dried in the same way as in the high, medium, and low viscosity IDBSs, respectively. The brackets were bonded indirectly in the same way as in the high, medium, and low viscosity IDBSs.

**Embedding and SBS measurements**

After polymerization of the adhesive, the transfer tray was removed in each group. The crown and root of each tooth were separated using a diamond disk. The tooth crowns were embedded in a metal cylinder using a self-curing acrylic resin. The buccal enamel surface was projected above, and parallel to, the brim of the

### Table 1 Orthodontic adhesives, adhesion promoter and light-curing unit used in this study

| Material                  | Components             | Composition                                                                 | Lot no.  | Manufacturer            |
|---------------------------|------------------------|----------------------------------------------------------------------------|----------|-------------------------|
| Transbond                 | Plus Self-etching primer | Water, Methacrylated phosphoric acid, Esters, Amino benzoate, Camphorquinone, etc. | 71030A   | 3M Unitek, Monrovia, CA, USA |
|                           | XT adhesive            | Bis-GMA, TEGDMA, Silane treated quartz, Amorphous silica, Camphorquinone, etc. | N780925  |                        |
| BeautyOrtho Bond          | Self-etching primer    | Acetone, Water, Bis-GMA, Carboxylic acid monomer, Phosphoric acid monomer, etc. | 011806   |                        |
|                           | Paste Viscos           | Bis-GMA, TEGDMA, S-PRG filler, etc.                                       | 101908   | Shofu, Kyoto, Japan     |
|                           | Flowable Paste         | Bis-GMA, TEGDMA, S-PRG filler, etc.                                       | 091912   |                        |
| Adhesion Promoter         | Enamel Conditioner     | Organic acid, Thickener, etc.                                              | 71913    |                        |
| Ortholux Luminous Curing Light Unit | Light-cure | Light source: LED, Light intensity: 1,600 mW/cm², Wavelength: 430–480 nm (a peak of 455±10 nm), Light guide tip diameter: 8 mm | 658844   | 3M Unitek               |

Bis-GMA indicates bisphenol A-glycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; S-PRG filler, surface pre-reacted glass ionomer filler; LED, light-emitting diode.
metal cylinders. All the metal cylinders with embedded teeth were immersed in distilled water at 37°C for 24 h. A universal testing machine (EZ Test; Shimadzu, Kyoto, Japan) was used to measure the SBS. The metal cylinders were arranged in this machine so that a load was applied to the bracket base with a force in the occlusogingival direction parallel to the buccal enamel surface. The force required to shear off the bracket was recorded in newtons at a cross-head speed of 1.0 mm per min. The SBS (MPa) was then calculated by dividing the shear force by the bracket base area.

**ARI score measurements**

After each SBS was measured, buccal enamel was photographed with a stereomicroscope at 10× magnification. The photographs were coded by a person unrelated to this study. The first measurement of ARI scores was made by an investigator (KI), who also performed the second measurement two months later, as well as another investigator (KT), who conducted the measurement independently. To avoid any examination bias, the investigators examined the coded materials blindly to group. Intra- and Inter-examiner Kappa values was 0.93 and 0.89, respectively, thus demonstrating almost perfect intra- and inter-examiner agreement.

**Surface roughness measurement**

Of the 30 remaining premolars, 10 were assigned equally into five groups of two, based on the following tooth enamel treatments: untreated enamel, enamel treated with the Transbond Plus and BOB SEPs, and with an adhesion promoter followed by these primers. The teeth in each group were washed with acetone and distilled water, dehydrated and sputter-coated with gold-palladium and examined by scanning electron microscopy (SEM; JSM-IT300LA, JEOL, Tokyo, Japan) at ×800 magnification.

**Flowability assessment**

The flowability for the TXT adhesive, the BOB Paste Viscos and BOB Flowable Paste was determined in accordance with the American Dental Association guidelines for evaluating the flowability of the endodontic sealing materials. A total of 66 sets of two glass plates (76×52×1.3 mm; Matsunami, Osaka, Japan) were divided equally into 3 groups of 22 set according to 3 different adhesives. A 0.1 mL aliquot of adhesive was placed at the center of one glass plate and another plate was placed on the first plate so that the two plates sandwiched the adhesive. A constant weight of 300 g was applied vertically to the center of the stacked plates for 30 s at room temperature (22°C) and 27% humidity, thus forming a nearly circular disk of the adhesive. The adhesive disks were then subjected to light curing for 30 s using the Ortholux Luminous Curing Light Unit. The maximum diameter and the minimum diameter (in millimeters) of the resulting nearly circular disk was measured using a digital caliper (Mitutoyo, Tokyo, Japan), and an average of these two diameters was calculated and defined as the diameter of the disk. For each adhesive, the mean diameter of 22 disks was taken as the flowability of the adhesive.

**Surface free-energy measurement**

The remaining 20 premolars were equally divided into two groups, one group consisting of 10 teeth with untreated enamel; and another group, 10 teeth with enamel treated using the adhesion promoter. A profilometer (Surfcom 470A, Tokyo Seimitsu, Tokyo, Japan) was used to measure the average surface roughness (Ra) of buccal enamel. Three vertical and three horizontal lines of 3.5 mm and 3.0 mm long, respectively were drawn at equal intervals (1 mm) on buccal enamel that correspond to the bonding area of the bracket base, and then nine intersections were marked. The Raes (cutoff of 0.25 mm) were measured with a length of 0.25 mm (0.125 mm in both directions from the center of the intersection) on vertical and horizontal lines. A total of 18 measurements were made and the mean enamel surface roughness (ESR) was calculated for buccal enamel.

**Statistical analysis**

The Excel software BellCurve (version 2.12, SSRI, Tokyo, Japan) was used for statistical analyses. The means, standard deviations, and ranges of the SBSs were calculated for each group. Two-way ANOVA and Tukey’s test were used to test the significant differences in the distribution of ARI scores between IDBS and the adhesion promoter on the SBS. Mann-Whitney U test and Kruskal-Wallis test were used to test significant differences in the distribution of ARI scores between nonapplication and application of the adhesion promoter in each IDBS and among 3 IDBSs in nonapplication and application groups, respectively. One-way ANOVA and Tukey’s test were used to test the significant differences in ESR and SFE measurements between the untreated and treated enamel surfaces with adhesion promoter. These parametric tests were performed after testing the normality of the distribution and homogeneity of the variance. All statistical tests were performed at the **p**<0.05 level of significance.
Table 2  Shear Bond Strength (MPa)

| Adhesion promoter | Non application (−) | Application (+) |
|-------------------|---------------------|-----------------|
|                   | Mean    | SD     | Range    | Mean    | SD     | Range    |
| High viscosity IDBS | 10.33   | 1.71   | 8.14–13.80 | 10.91   | 1.48   | 8.36–12.74 |
| Medium viscosity IDBS | 7.38    | 1.33   | 5.73–10.90 | 9.17    | 1.77   | 6.13–11.46 |
| Low viscosity IDBS  | 8.94    | 1.23   | 7.43–12.23 | 10.70   | 1.70   | 8.61–14.58 |

IDBS indicates indirect bonding system; SD, standard deviation.

Table 3  Distribution of Adhesive Remnant Index (ARI) Scores

| Adhesion promoter | Non application (−) | Application (+) |
|-------------------|---------------------|-----------------|
|                   | 0        | 1 | 2 | 3 | 4<sup>a,b</sup> | 0 | 1 | 2 | 3 |
| High viscosity IDBS | 0       | 4 | 9 | 4<sup>a,b</sup> | 0 | 1 | 14 | 2<sup>a</sup> |
| Medium viscosity IDBS | 1       | 8 | 6 | 2<sup>a</sup> | 0 | 0 | 17 | 0<sup>a</sup> |
| Low viscosity IDBS  | 0       | 10 | 6 | 1<sup>a</sup> | 0 | 5 | 6 | 6<sup>a</sup> |

IDBS indicates indirect bonding system. ARI score: 0, no adhesive remaining on the tooth surface; 1, less than half the adhesive remaining on the tooth surface; 2, more than half the adhesive remaining on the tooth surface; 3, all adhesive remnants on the tooth surface with a distinct impression of the bracket base.

RESULTS

Kolmogorov-Smirnov and Leven tests confirmed the normality of the distribution and the homogeneity of the variance for the SBS, the disk diameter, the ESR and the SFE, respectively.

The two-way ANOVA indicated significant differences among the mean SBSs for the three IDBSs (p<0.001), and between application and nonapplication of the adhesion promoter (p<0.001), and no significant interaction between these two variables (p=0.1911, Table 2). Tukey’s test showed that the high and low viscosity IDBSs gave significantly higher mean SBSs than the medium viscosity IDBS (p<0.001), and no significant differences were found between the high and low viscosity IDBSs (p=0.0917, Table 2). The overall mean SBSs of all six IDBSs reached 6 MPa, which is the minimum requirement for clinical use (Table 2). The SBSs for two specimens out of 17 (11.8%) were less than 6 MPa in the medium viscosity IDBS without application of the adhesion promoter, whereas those of all specimens in the other IDBSs reached 6 MPa (Table 2).

The Kruskal-Wallis test showed no significant differences in the distribution of ARI scores among IDBSs with (p=0.9086) or without (p=0.0678) application of the adhesion promoter (Table 3). The Mann-Whitney U test showed that in the medium and low viscosity IDBSs, there were significant differences in the distribution of ARI scores between application and nonapplication of the adhesion promoter (p<0.05, Table 3). This test also showed no significant difference between application and nonapplication of the adhesion promoter in the high viscosity IDBS (p=0.7864, Table 3).

Typical SEM images are shown in Fig. 1. The untreated enamel surface showed a typical perikymata pattern (Fig. 1a). Irrespective of whether the adhesion promoter was applied or not, deeper demineralization on the enamel surface was observed with the Transbond Plus SEP (Figs. 1b and d) than with the BOB SEP (Figs. 1c and e). Combined use of the Transbond Plus and BOB SEPs and the adhesion promoter (Figs. 1d and e) produced a rougher enamel than did use of the primers alone (Figs. 1b and c).

As shown in Table 4, one-way ANOVA and Tukey’s
Table 4  Disk diameter for flowability (mm)

|                | Mean | SD  | Range      |
|----------------|------|-----|------------|
| TXT adhesive   | 12.6 | 0.5 | 12.1–13.1  |
| BOB Paste Viscos | 15.2 | 0.7 | 14.5–15.9  |
| BOB Flowable Paste | 21.6 | 0.7 | 20.9–22.3  |

TXT indicates Transbond XT; BOB, BeautyOrtho Bond; SD, standard deviation.

Table 5  Surface roughness (µm) of untreated and treated enamel surfaces with adhesion promoter

|                | Untreated | Treated |
|----------------|-----------|---------|
| Mean           | 0.086     | 0.195   |
| SD             | 0.008     | 0.007   |
| Range          | 0.071–0.099 | 0.184–0.204 |

Table 6  Surface free-energy (mN/m) of untreated and treated enamel surfaces with adhesion promoter

|                | Untreated | Treated |
|----------------|-----------|---------|
| Mean           | 62.90     | 76.90   |
| SD             | 3.49      | 2.66    |
| Range          | 55.35–68.58 | 72.30–80.40 |

SD indicates standard deviation.

test identified the largest mean disk diameter in the BOB Flowable Paste, followed by the BOB Paste Viscos and TXT adhesive in that order (BOB Flowable Paste>BOB Paste Viscos>TXT adhesive). This table also showed that the differences among the three adhesives were significant (p<0.001).

Tables 5 and 6 showed that the treated enamel surface with the adhesion promoter exhibited significantly higher ESR and SFE than the untreated enamel surface (p<0.001).

DISCUSSION

The null hypothesis, that the adhesive viscosity and adhesion promoter would have no effect on the SBS and ARI scores was rejected. The inverse of viscosity is called flowability, thus demonstrating that an adhesive with a high viscosity has a low flowability. In this study, the adhesive with the largest mean disk diameter was the BOB Flowable Paste, followed by the BOB Paste Viscos and the TXT adhesive in that order. These results were supported by Scougall Vilchis et al. and Li et al., who reported that a significantly higher SBS was achieved with the TXT DBS than with the BOB DBS without application of an adhesion promoter. The reason for the high SBS in the high viscosity IDBS is the difference between the pHs...
of the SEPs. The pH of the Transbond Plus SEP was 1.2 and increased little within 10 min (pH 1.2 to 1.3); that of the BOB SEP was 3.2 and did not increase at all. These results are in agreement with those reported by Yuasa et al.\textsuperscript{20}. The difference between the pHs of these primers resulted in deeper demineralization on the enamel surfaces with the Transbond Plus SEP (Figs. 1b and d) than with the BOB SEP (Figs. 1c and e). The Transbond Plus SEP, with a low pH, creates a rough enamel surface and enables sufficient penetration of the adhesive resins into the enamel surface. The SBS in the high viscosity IDBS was therefore higher than that in the medium viscosity IDBS. Another possible reason is the difference between the filler contents of the adhesives. Faltermeier et al.\textsuperscript{10} reported that the SBSs of orthodontic adhesives increased with increasing adhesive filler content. The filler content was higher in the TXT adhesive (75.9%) than in the BOB adhesive (67.2%)\textsuperscript{22}, therefore the high viscosity IDBS gave a higher SBS than did the medium viscosity IDBS, as evidenced by this study.

In this study, regardless of whether or not an adhesion promoter was applied, the low viscosity IDBS gave a significantly higher SBS than the medium viscosity IDBS did. This can be explained by the effect of the filler content on the flowability. Park et al.\textsuperscript{12} showed that the flowability of a flowable resin is achieved mainly by lowering the filler loading. This suggests that the flowability of the BOB Flowable Paste, which has a low filler content, is high and the paste could flow easily onto a bracket base and enhance the SBS to the level of the high viscosity IDBS.

In this study, in all three IDBSs, application of an adhesion promoter containing polyacrylic acid gave a significantly higher SBS than in the case of nonapplication of this promoter. Some studies have shown that adhesion promoters significantly increase SBSs,\textsuperscript{7,8,20} as evidenced by this study, while others showed that any adhesion promoters did not significantly increase the SBS\textsuperscript{20}. Es-souini et al.\textsuperscript{26} reported that polyacrylic acid leached calcium and phosphorus from hydroxyapatite, and formed a fine polymeric film on the enamel surface. The formation of a polymeric film containing calcium and phosphorus is considered to enhance the level of chemical bonding between the enamel surface and the SEPs. The pH of the adhesion promoter was 2.4 and did not increase within 10 min. This is higher than the pH of phosphoric acid (0.4)\textsuperscript{27}. Some studies reported that enamel etching with the polyacrylic acid and phosphoric acid increased the ESR\textsuperscript{28,29} and SFE\textsuperscript{30}, leading to improved bond strength. In this study, the adhesion promoter increased significantly ESR and SFE, as shown in Tables 5 and 6, thus causing the fact that the treated enamel surface exhibited significantly high SBS. Iijima et al.\textsuperscript{31} reported that increasing the application times of SEPs with high pH increased the etching efficacy. However, Hamdani et al.\textsuperscript{32} reported that an increase in the application time of phosphoric acid, which has a low pH, impaired the integrity of honeycombed prismatic structures on the enamel, which negatively affected the SBS. It was considered that combined use of a slightly acidic adhesion promoter and SEPs increased the etching time, which increased the etching efficacy and SBSs compared with those obtained with single these primers alone. This was verified by our SEM observations, which showed that the Transbond Plus and BOB SEPs with application of the slightly acidic adhesion promoter (Figs. 1d and e) created rougher enamel than did these SEPs alone (Figs. 1b and c).

In this study, the mean SBSs and the SBSs of all the specimens reached 6 MPa in five bonding systems, but not in the medium viscosity IDBS, without application of the adhesion promoter. This shows that irrespective of application or nonapplication of this promoter, the high and low viscosity IDBSs were effective for bracket bonding. In the medium viscosity IDBS without the adhesion promoter, the SBSs for two (11.8%) of 17 specimens were less than 6 MPa, and the mean SBS reached 6 MPa. These results are consistent with the findings of Goto et al.\textsuperscript{11} that the SBSs for two (8.7%) to three (13.0%) specimens out of 23 were less than 6 MPa in the BOB Paste Viscos DBS. To obtain a clinically adequate level of SBS in the medium viscosity IDBS, this system needs to be used in combination with an adhesion promoter.

In this study, medium and low viscosity IDBSs with the adhesion promoter had lower frequencies of bond failure at the enamel-adhesive interface than those without this promoter, as shown in a previous study\textsuperscript{24}. These results may be caused by the speculation that the combination of adhesion promoter and the BOB SEP moderately demineralizes enamel and enhances mechanical bonding between the adhesive and enamel surface, as previously mentioned. Scougall Vilchis et al.\textsuperscript{21} found a positive correlation between SBS and adhesive remnant. Pithon et al.\textsuperscript{30} reported that higher SBS remained larger amount of adhesive. Their findings may collaborate with our results that adhesion promoter containing polyacrylic acid enhanced the SBSs of BOB Paste Viscos and Flowable Paste, and increased the remaining amount of adhesive on the enamel surface.

The adhesion promoter in the high viscosity IDBS significantly increased SBS, but there was no significant difference in the distribution of ARI scores between application and nonapplication of this promoter in this study. This finding was not supported by our speculation that higher SBS exhibited more adhesive remnants, as demonstrated in the medium and low viscosity IDBS. Our result of the high viscosity IDBS may be supported by Naidu et al.\textsuperscript{34} who showed that preconditioning of enamel with the adhesion promoter increased the SBS, but the amount of residual resin remained unchanged in the TXT DBS. It may be considered that the discrepancy in association of ARI and SBS between the high, medium and low viscosity IDBSs in this study might be caused by different viscosities of the adhesives.

**CONCLUSIONS**

1. Irrespective of application or nonapplication of an adhesion promoter, the high and low viscosity
IDBSs is effective for bracket bonding.

2. The medium viscosity IDBS needs to be used in combination with an adhesion promoter to obtain a clinically acceptable SBS.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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