Effects of different primers on indirect orthodontic bonding: Shear bond strength, color change, and enamel roughness

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Objective: We aimed to perform in-vitro evaluation to compare 1) shear bond strength (SBS), adhesive remnant index (ARI), and color change between self-etched and acid-etched primers; 2) the SBS, ARI and color change between direct and indirect bonding; and 3) the enamel roughness (ER) between 12-blade bur and aluminum oxide polisher debonding methods. Methods: Seventy bovine incisors were distributed in seven groups: control (no bonding), direct (DTBX), and 5 indirect bonding (ITBX, IZ350, ISONDHI, ISEP, and ITBXp). Transbond XT Primer was used in the DTX, ITBX, and ITBXp groups, flow resin Z350 in the IZ350 group, Sondhi in the ISONDHI group, and SEP primer in the ISEP group. SBS, ARI, and ER were evaluated. The adhesive remnant was removed using a low-speed tungsten bur in all groups except the ITBXp, in which an aluminum oxide polisher was used. After coffee staining, color evaluations were performed using a spectrophotometer immediately after staining and prior to bonding. Results: ISONDHI and ISEP showed significantly lower SBS (p < 0.01). DTX had a greater number of teeth with all the adhesive on the enamel (70%), compared with the indirect bonding groups (0–30%). The ER in the ITBX and ITBXp groups was found to be greater because of both clean-up techniques used. Conclusions: Direct and indirect bonding have similar results and all the primers used show satisfactory adhesion strength. Use of burs and polishers increases the ER, but polishers ensure greater integrity of the initial roughness. Resin tags do not change the color of the teeth.

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INTRODUCTION

The use of adhesive systems has provided benefits in orthodontic bonding procedures, including longer working time, accuracy in positioning of the brackets, and better adhesion. These adhesives allow orthodontic attachments to be bonded directly onto the tooth surface. Despite advantages such as the reduced need for bands, less soft tissue irritation, and improvement in esthetics, direct bonding still requires substantial amounts of clinical time, causes discomfort to the patient, and poses difficulties in the accurate positioning of the attachments.¹

Indirect bonding was proposed to solve some of these problems. In this technique, the brackets were first positioned in the dental casts and then bonded onto the dentition using a transfer device. Accordingly, the clinical time was reduced and transferred to the laboratory stage. The advantages of this technique include efficiency in the positioning of attachments; simultaneous bonding of brackets, resulting in a shorter clinical time; and reduction in both patient discomfort and bonding failure;² although some authors found no difference in the failure rates between direct and indirect bonding.³ On the other hand, indirect bonding has some disadvantages, such as the extended laboratorial time due to the greater number of steps; higher cost; problems at the interface between the bracket and tooth; and the possibility of compromised adhesion.⁴

At the end of the orthodontic treatment, following bracket debonding and protocols for enamel clean-up, the surface of the teeth must be smoothed and polished. Clinical reports indicate that the resin tags can remain after bracket debonding and cause chromatic changes in the enamel even after the use of enamel clean-up methods, thereby compromising the long-term esthetics. In addition, the rotary instruments used to remove the adhesive remnant increase the unevenness of the enamel surface, causing greater likelihood of dye pigmentation.⁵

The aims of this study were to perform an in-vitro evaluation to compare: 1) the shear bond strength (SBS), adhesive remnant index (ARI), and color change between self-etched and acid-etched primers; 2) the SBS, ARI and color change between direct and indirect bonding; and 3) the enamel roughness after the 12-blade bur debonding and aluminum oxide polisher debonding methods.

MATERIALS AND METHODS

A total of 70 bovine incisors were used. The teeth were stored in 0.1% thymol at 5°C. To allow adequate and standardized positioning during the methodological tests, the teeth were prepared. The mesio-distal diameters of the teeth were reduced to 8 mm, simulating a human maxillary central incisor. The vestibular faces were sanded with sandpaper (400 granulation) on a machine (Ecomet II; Buehler, Lake Buff, IL, USA) under irrigation with water. The incisal edges were also cut in the same machine to obtain flat faces and perpendicular to the vestibular face. The root portion was placed in polyvinyl chloride cylinders, with the teeth positioned in a parabolic format and filled with stone.

| Table 1. Distribution of the groups according to the method used for brackets bonding, the adhesive system used, and the adhesive removal method |
|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
| **Group** | **Bonding technique** | **Primer** | **Adhesive** | **Removal adhesive methods** |
| Control | No bonding | - | - | - |
| DTBX | Direct | Transbond Primer | Transbond XT | 12-blade bur at low speed |
| ITBX | Indirect | Transbond Primer | Transbond XT | 12-blade bur at low speed |
| IZ350 | Indirect | Z350 (color A2) | Transbond XT | 12-blade bur at low speed |
| ISONDHI | Indirect | Sondhi Rapid-Set | Transbond XT | 12-blade bur at low speed |
| ISEP | Indirect | Transbond SEP | Transbond XT | 12-blade bur at low speed |
| ITBXp | Indirect | Transbond Primer | Transbond XT | Polisher of aluminum oxide |

Control. No bonding was performed; DTBX, Transbond XT Primer and Adhesive (3M Unitek, Monrovia, CA, USA) were used to direct bonding, and 12-blade bur at low speed to remove the adhesive remnant; ITBX, Transbond XT Primer and Adhesive (3M Unitek) were used to indirect bonding, and 12-blade bur at low speed to remove the adhesive remnant; IZ350, Transbond XT Primer and Z350 flow resin (3M Unitek) were used to indirect bonding, and 12-blade bur at low speed to remove the adhesive remnant; ISONDHI, Sondhi Rapid-set and Transbond XT Adhesive (3M Unitek) were used to indirect bonding, and 12-blade bur at low speed to remove the adhesive remnant; ISEP, Transbond Plus Self-Etching Primer (SEP, 3M Unitek) and Transbond XT Adhesive (3M Unitek) were used to indirect bonding, and 12-blade bur at low speed to remove the adhesive remnant; ITBXp, Transbond XT Primer and Adhesive (3M Unitek) were used to indirect bonding, and polisher of aluminum oxide at low speed to remove the adhesive remnant.
The sample was randomly divided into seven groups of ten teeth \((n = 10)\), according to Table 1.

**Direct bonding**

Metal brackets (10.30.201; Morelli, Sorocaba, Brazil) were used in all groups.

**Control group:** No bonding was performed.

**DTBX group:** After prophylaxis, 37% phosphoric acid was applied on the teeth for 30 seconds, and washed and dried with the triple syringe. Transbond XT Primer (3M Unitek, Monrovia, CA, USA) was applied, followed by air jet and curing for 20 seconds. The brackets were positioned 5 mm from the incisal edge with Transbond XT Adhesive (3M Unitek). The excess adhesive was removed and curing was performed for 40 seconds per tooth, 20 seconds on each proximal face, with a curing light (Optilight Max LED; Gnatus, Ribeirão Preto, Brazil).

**Indirect bonding**

Alginate impressions were taken and working models were made in stone. Guidelines were drawn on the casts, one horizontal line at 5 mm from the incisal edge and one vertical line on the center of the teeth. Cel-lac (SS White, Rio de Janeiro, Brazil) was applied, after diluting it in water in a 1:1 ratio. Transbond XT Adhesive was used for all indirect bonding groups. The brackets were positioned according to the guidelines, and the excess resin was removed and light-cured.

Vacu-formed indirect bonding trays were prepared using the double tray technique, as recommended by Nojima et al. The author used a flexible tray (Soft [EVA]; Bio-Art, São Carlos, Brazil) unit to vacu-form a 1.0-mm-thick layer, overlayed with a 1.0-mm-thick rigid layer (Cristal [PET-G]; Bio-Art). The machine used was PlastVac P7 (Bio-Art). Before the second tray, the spray (liquid silicone, Bucas Spray; Biojet Quimica, Umuarama, Brazil) was applied to permit easier separation of the two trays.

The trays were cut and segmented, and extended beyond three or four teeth. The resin base of the brackets was sandblasted with 50-μm aluminum oxide for 2 seconds. Immediately after finishing to customize the transfer trays, the teeth underwent prophylactic procedures and the subsequent bonding procedures varied among the groups. The enamel was etched with 37% phosphoric acid for 30 seconds in all the groups, except ISEP.

**ISEP:** The enamel was etched with Transbond Plus Self-Etching Primer (3M Unitek). The microbrush was wiped on the tooth for 3 seconds and applied onto the bracket bases, followed by tray adaptation and curing. The curing time in all indirect bonding groups was 40 seconds per tooth, 20 seconds on each proximal face, with a curing light (Optilight Max LED). The specimens were stored in distilled water at 37°C for 72 hours until bracket debonding.

**Shear bond strength testing**

The maximum SBS for bracket debonding was determined by a universal testing machine (EMIC DL 2000; EMIC, São José dos Pinhais, Brazil), with a shear load of 50 gf, and at a speed of 1 mm/min. The force was applied between the bracket base and wing. The teeth were inspected under a stereomicroscope (110AL2X; Olympus, Tokyo, Japan), and scored according to ARI: 0 = no adhesive adhered to the enamel; 1 = less than half of the adhesive adhered; 2 = more than half of the adhesive adhered; 3 = all adhesive material adhered.

**Removal of adhesive remnant**

The adhesives in all groups, except the ITBXp group, were removed with a 12-blade tungsten bur (H23R.21.012; Brasseler, Savannah, GA, USA) at a low speed. In the ITBXp group, an aluminum oxide polisher (DU10CA Ortho; Dhpco, Curitiba, Brazil) was used.

**Three-dimensional (3D) evaluation of enamel roughness**

Surface morphology (3D) and enamel roughness were evaluated with a Zygo NewView 7100 (Zygo, Middlefield, CA, USA) optical rugosimeter, using the interferometry technique with 20× magnification. The medium roughness (Ra) and medium depth roughness (Rz) parameters were analyzed. The area scanned was 469 × 352 μm.

Enamel roughness was evaluated only in the ITBX and ITBXp groups at the following timepoints: initial roughness (IR) prior to bonding and final roughness (FR) after resin removal. The efficiency of the adhesive removal was determined by calculating the roughness variation (ΔR), where \(ΔR = (FR − IR)/IR\), for Ra and Rz.

**Color change**

The samples were exposed to staining with a coffee solution maintained at 37°C in an incubator during the protocol; the staining procedure was performed for 72 hours, because a 24-hour staining period has been reported to be insufficient.

Color evaluations were performed before bonding...
(T0) and after staining (T1) using a Vita Easyspace spectrophotometer (Model DEASYC220; VITA Zahnfabrik, Bad Sackingen, Germany). Color changes were characterized using the Commission Internationale de l’Eclairage (CIE) LAB criteria, which separates color into components: “L,” which represents the color values of black and white; “a,” which measures the color from green to red; and “b,” which evaluates the color from yellow to blue.

Prophylaxis was done in all samples before analysis of color changes. Measurements of color were performed with the spectrophotometer tip perpendicular to the buccal face, using an adapter made of 0.7-mm wire. Thus, color could always be measured in the same position. The color change (ΔE) was calculated by the equation

\[ \Delta E = \left[ (\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2 \right]^{1/2} \]

where ΔL, Δa, Δb are the differences between values of “L”, “a” and “b” at T0 and T1.

Statistical analysis
The results were analyzed by SPSS software version 23.0 (IBM, Chicago, IL, USA). The level of significance was 5% and the normality of the samples was verified by the Shapiro-Wilk test.

One-way analysis of variance (ANOVA) and Tukey’s post-tests were used to assess the SBS and ΔE (color change). The paired t-test was used to evaluate the Ra and Rz parameters. The t-test of independent samples was applied for ΔRa and ΔRz. The Kruskal-Wallis test was used to assess the ARI findings.

RESULTS
The results of the SBS evaluations are presented in Table 2. There were no differences between the DTBX, ITBX, and IZ350 groups. The ISONDHI and ISEP groups had significantly lower mean values. The distribution of ARI values is shown in Table 3. The Kruskal-Wallis test showed no difference in the values (p < 0.17).

The results for enamel roughness are summarized in Table 4. With regard to Rz values, there were no differences between IR and FR in the ITBXp group, but differences were detected in the ITBX group. As for the Ra values, differences were observed in both the ITBXp and ITBX groups, which showed a higher FR. The surface morphologies are indicated in Figure 1.

The results for ΔRa and ΔRz are shown in Table 5. No significant difference was detected between the groups for ΔRa and ΔRz. The results of the color change are presented in Table 6. There was no difference among the groups including the control group in which no bonding was performed.

DISCUSSION
In this study, bovine incisors were used because of

| Table 2. The shear bond strengths (MPa) |
|----------------------------------------|
| Group | Mean ± SD | Median (range) | p-value |
| DTBX  | 13.60 ± 0.63 | 13.59 (12.56–14.84) | < 0.187 |
| ITBX  | 12.92 ± 0.44 | 12.95 (12.11–13.55) | < 0.187 |
| IZ350 | 13.10 ± 0.79 | 13.12 (12.12–14.67) | < 0.480 |
| ISONDHI | 5.72 ± 0.42 | 5.83 (4.99–6.34) | < 0.001 |
| ISEP  | 7.05 ± 0.95 | 6.83 (6.04–8.85) | < 0.001 |

SD, Standard deviation. ANOVA and Tukey’s post-hoc test were performed. A,B,C Same letters indicate no statistically significant difference among groups (p > 0.05).

For the definition of each group, see the footnote in Table 1.

| Table 3. Number of subjects for each adhesive remnant index (ARI) |
|---------------------------------------------------------------|
| Group | ARI scores |
|       | 0 | 1 | 2 | 3 |
| DTBX  | 0 | 2 | 1 | 7 |
| ITBX  | 0 | 3 | 4 | 3 |
| IZ350 | 1 | 5 | 1 | 3 |
| ISONDHI | 2 | 2 | 3 | 3 |
| ISEP  | 0 | 3 | 7 | 0 |

The Kruskal-Wallis test showed that there were no statistically significant difference among the 5 groups (p > 0.05).

For the definition of each group, see the footnote in Table 1.

| Table 4. Roughness parameter analyses according to the paired t-test |
|---------------------------------------------------------------|
| Parameter | Group | Mean ± SD (μm) | p-value | Mean ± SD (μm) | p-value |
| Ra       | ITBX  | 0.288 ± 0.188 | < 0.004* | 0.256 ± 0.080 | < 0.001* |
|          | ITBXp | 0.579 ± 0.182 | 0.501 ± 0.091 |
| Rz       | ITBX  | 5.843 ± 6.281 | < 0.042* | 5.845 ± 1.900 | < 0.604 |
|          | ITBXp | 10.744 ± 8.485 | 6.657 ± 4.395 |

ITBX, Transbond XT Primer and Adhesive with 12-blade bur were used; ITBXp, Transbond XT Primer and Adhesive with polishing of aluminum oxide were used; SD, standard deviation; Ra, medium roughness; Rz, medium depth roughness.

*Statistically significant differences between initial and final values (p < 0.05).
the easy access and their similarities with the human dental structure, although the use of bovine incisors is associated with lower values of SBS.\textsuperscript{10,11}

The bonding of an orthodontic appliance must be strong enough to withstand masticatory and orthodontic forces, and allow easy removal of the brackets without damaging the enamel. The minimum SBS value of the adhesive should be 5.88 to 7.84 MPa for it to be considered adequate for clinical needs.\textsuperscript{12}

In this study, the average shear strength ranged from 5.72 (± 0.42) to 13.60 (± 0.63) MPa among the groups. Since direct bonding with the Transbond XT system is considered the gold standard in the literature today, we made comparisons with the DTBX group.\textsuperscript{13}

No differences were found between the DTBX and ITBX groups when the same bonding material was used (Table

### Table 5. Comparisons of $\Delta$Ra and $\Delta$Rz for ITBX and ITBXp groups

| Group       | $\Delta$Ra (μm) | $\Delta$Rz (μm) | p-value  |
|-------------|-----------------|-----------------|----------|
| ITBX        | 1.397 ± 1.098   | 1.399 ± 2.299   | < 0.359  |
| ITBXp       | 1.032 ± 0.509   | 0.249 ± 0.823   | < 0.154  |

Values are presented as mean ± standard deviation. Independent t-tests were performed. ITBX, Transbond XT Primer and Adhesive with 12-blade bur were used; ITBXp, Transbond XT Primer and Adhesive with polisher of aluminum oxide were used; Ra, medium roughness; Rz, medium depth roughness.

### Table 6. Color changes ($\Delta E$ values) before bonding and after clean-up

| Group            | Mean ± SD |
|------------------|-----------|
| Control          | 21.05 ± 6.95 |
| DTBX             | 23.84 ± 12.56 |
| ITBX             | 19.20 ± 11.03 |
| IZ350            | 28.12 ± 10.63 |
| ISONDHI          | 29.22 ± 13.14 |
| ISEP             | 26.69 ± 10.08 |
| ITBXp            | 17.41 ± 7.79 |

SD, Standard deviation. ANOVA and Tukey’s post-hoc test were performed. There was no statistically significant difference among groups (p > 0.05). For the definition of each group, see the footnote in Table 1.
2). Therefore, there was no difference between the direct and indirect techniques, corroborating the results of several authors. In indirect bonding, in which different bonding materials were used, the differences were detected only between the ISONDHI and ISEP groups, with mean values of 5.72 and 7.05 MPa, respectively. However, both of these values were within the range for determining whether the materials are suitable for clinical use. These results agree with those of some authors, and disagree with those of others, who found no differences between these materials.

Flow resin has been used for orthodontic bonding, but is not used specifically for this purpose. In our study, there was no difference between the IZ350 and DTBX groups, suggesting that flow resin may be an alternative resource for the indirect bonding.

The results of the ARI assessments showed no difference among the groups, although the direct bonding group contained a larger number of samples with all the bonding material adhered to the enamel, compared with the indirect bonding group (Table 3), as corroborated by previous studies. This difference shows that there was less adhesive on the enamel surface in indirect bonding, especially when using self-etching primers, as observed in the ISEP group, which had no samples with all the adhesive material adhered to the tooth (Table 3). Clinically, this is an interesting finding because it indicates that less material must be removed from the tooth surface. In direct bonding, the resin is cured with the bracket in the final position, and it is assumed that adhesion of the material to the surface of the enamel occurs more frequently. However, in indirect bonding, the resin is cured on the surface of the dental casts. This custom copy of the enamel is taken with the mouth in the same position as the source, and, in this case, the bonding is performed by a primer or a more fluid resin. Since adhesion and debonding occur with the primer between the adhesive base and the surface of the enamel, less leftover material can be visualized on the tooth, as shown in Table 3. Unlike other studies that use unit transfer trays for each tooth, our study used a tray that extended beyond a couple of teeth, thus being more representative of indirect bonding in the mouth.

The variation in the surface flatness of the materials is determined by measuring roughness. Several studies have used different methods to compare the dental surface after bracket debonding, including ARI assessments and scanning electron microscopy (SEM).

However, evaluation of enamel roughness by SEM is only qualitative and cannot be used for this comparison. To obtain quantitative results, studies have used the contact rugosimeter, a profilometer. This device quantifies roughness by measuring parameters along a line, and can often provide non-representative values, depending on the area analyzed. Therefore, analyzing the roughness of the surface area is the ideal method. In our study, enamel roughness was measured with an optical rugosimeter based on the interferometry technique, which allows measurement of an infinite number of lines in the same area. No study that used this method was found in the literature. There are studies that have used confocal microscopy and atomic force microscopy, which analyze a smaller area than that assessed in the present study.

The Ra assessments performed in our study suggest that the two adhesive removal methods significantly increased the surface roughness of the enamel (Table 4), corroborating the findings of some previous studies that used the profilometer, although there is disagreement among authors. Many studies used the Ra parameter only to analyze the surface. This parameter has limitations, because it does not define the shape of the profile irregularities, or distinguish between peaks and valleys. In this study, the Rz parameter was used, similar to other studies. Assessments using the Rz values showed a significant increase in the enamel roughness in the ITBX group, better represented by the creation of valleys, in blue (Figure 1D). The use of a low-speed bur, which may have scratched the surface, is a method that has also been employed in studies. However, some authors disagree that the bur can increase enamel roughness. This discordance can be associated with the different methodologies used and the pressure placed on the rotary instrument to remove the adhesive, especially when rubber polishers are used following use of the bur, thus reducing enamel roughness and making the procedure clinically imperceptible. The present study found that polishers also create irregularities on the enamel, but cause less roughness than burs.

Although Ra assessments showed an increase in enamel roughness in both groups, the Rz measurements did not indicate any difference between the initial and FR in the ITBXp group. This variance could have occurred because of the presence of atypical peaks or valleys, which are not considered in calculating the Ra. In the IBTX group, valleys were created, as evidenced by the blue color (Figure 1D); on the other hand, this did not occur in the ITBXp group (Figure 1B). Thus, our study corroborates previous findings showing that polishers induce minor changes in the enamel roughness. However, when comparing ∆Ra and ∆Rz among the groups, no differences were found between the two methods.

The high variety of colorants in contemporary food can affect the color stability of dental materials. The coffee staining protocol has proved to be an efficient method, since coffee is widely consumed by the popu-
The materials used for bonding may exhibit color instability due to the presence of remnant resin tags even after removal of the brackets, and the cleaning and polishing of the dental surface. Color changes may also be more greatly influenced by the greater surface enamel roughness attributed to the use of rotatory instruments, like high- or low-speed burs, during the removal of the adhesive remnant.

The methodology employed enabled observation of the color change with the naked eye in all the groups. However, compared with the control group, where no bonding was used, there were no differences in any of the primers tested (Table 6). Therefore, resin tags alone could not cause color changes in the teeth.

CONCLUSION

Comparisons of the direct and indirect bonding techniques using different primers and two methods of adhesive removal yielded the following conclusions:

- Direct and indirect bonding presented similar results for SBS. The primers used are well-suited to clinical needs in regard to adhesion strength, although the ISONDHI and ISEP groups required less force to remove the brackets.
- Indirect bonding tended to result in less adhesive on the dental surface after bracket debonding.
- The use of low-speed burs to remove the adhesive increases the enamel roughness. The same is true of aluminum oxide polishers. However, the polishers guarantee less variation of the initial roughness, based on the Rz values.
- After bracket debonding and adhesive removal, the resin tags do not change the color of the teeth.

CONFLICTS OF INTEREST

No potential conflict of interest relevant to this article was reported.

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