# Shear bond strength of nanofilled flowable resins used for indirect bracket bonding

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## Abstract

Aim: To evaluate the bond strength of brackets fixed with different materials (two light-cured nanofilled resins - Transbond Supreme LV and Flow Tain LV, a light-cured resin - Transbond XT (control) and two chemically cured resins for indirect bonding - Sondhi Rapid- Set and Custom I.Q.) using the indirect bonding technique after 10 min and 24 h, and evaluate the type of failure. **Methods:** One hundred premolars were selected and randomly divided into groups (n=10) according to the material and fixation period. The brackets were bonded through the indirect technique following the manufacturer's instructions and stored in deionized water at 37°C for 10 min or 24 h. After, the specimens were submitted to a shear bond strength (SBS) test (Instron) at 0.5 mm/min and evaluated for adhesive remnant index (ARI). The data were submitted to ANOVA and Tukey's test (p<0.05) and the ARI scores were submitted to the chi-square test. Results: It could be observed a significant difference among the materials (Flow Tain LV = Transbond Supreme LV = Transbond XT> Sondhi Rapid-Set > Custom I.Q.). There was no significant difference in resistance values between 10 min and 24 h, regardless of the materials. Most groups showed adhesive remaining adhered to the enamel (scores 2 and 3) without statistically significant difference (p>0.05). Conclusions: It was concluded that the light-cured nanofilled materials used in indirect bonding showed greater resistance than the chemically cured materials. The period of fixation had no influence on the resistance for different materials.

Keywords: orthodontics, indirect bonding, orthodontic brackets.

## Introduction

The success of orthodontic treatment with fixed appliances depends, within other factors, on an accurate bracket positioning and long-term retention of these accessories.<sup>1</sup> The time spent during the bracket bonding is an important factor in the treatment cost and the necessity of rebonding brackets can retard the progress of treatment.

To avoid errors during bracket positioning and facilitate bracket bonding, the indirect bond technique was introduced by Silverman et al.  $(1972)^2$  and progressed with Thomas  $(1979)^3$ . This technique has been practiced for many years in various forms<sup>4-7</sup> and consists of the pre-positioning of appliances on a working model and the use of a transfer tray to capture the appliances and convey them to the patient's mouth<sup>4</sup>. The indirect bonding is essential in lingual

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Gisele Maria Correr Program in Clinical Dentistry, Positivo University Rua Pedro Viriato Parigot de Souza, 5300, Campo Comprido, CEP: 81280-330 Curitiba, PR, Brasil Phone: +55 41 33173403 Fax: +55 41 33173082 E-mail: giselenolasco@up.com.br orthodontics because of the difficulty of positioning the brackets directly to tooth and also by the variation of the anatomy of the lingual anterior teeth<sup>6</sup>.

Bracket positioning is facilitated by indirect bonding because it provides a direct and better view of the model teeth in all planes<sup>6</sup>. The indirect bonding has some advantages such as shorter clinical time, greater patient comfort, greater accuracy in positioning of brackets<sup>6,8</sup> and reduced plaque accumulation around braces<sup>9</sup>. However, this technique also has disadvantages such as laboratory work time<sup>8</sup> higher cost, more phases (laboratorial and clinical) and presence of remaining adhesive in the bracket base, which may impair adhesion<sup>10</sup>.

Bracket bonding to etched teeth using chemical- or lightcure adhesives can be considered a standard clinical practice<sup>11</sup>. Advances in the adhesive procedures had occurred over the years, especially regarding the bonding materials. These materials have evolved in their composition and manipulation technique, aiming at sharing the efficient adhesion to the individual needs of the orthodontic patients. Several materials are available in the market for bracket bonding, such as resin-modified glass ionomer cements, resin composites, orthodontic adhesives, flowable resins and more recently introduced nanofilled materials<sup>12-13</sup>.

Flowable resins have some advantages compared with traditional resin composites such as no stickiness and fluid injectability<sup>14</sup>. These materials can have reduced filler content, increased diluents monomers or altered rheology to reduce the viscosity of the mixture. However, this may eventually weaken the mechanical properties of the flowable resins, which are influenced by filler loading within the resin matrix<sup>15</sup>. Flowable resins have been applied for orthodontic use by many clinicians<sup>11,13,16</sup>. However, there are still few studies available that evaluate the bonding properties of these materials<sup>11,13,17</sup> with contradictory reports on the shear bond strength<sup>14,16-17</sup>.

Based on the interest in flowable restoratives for orthodontic use, especially for indirect bonding, a nanofilled low-viscosity light cure indirect bonding adhesive was introduced by 3M Unitek, Monrovia, CA, USA - Transbond Supreme LV. The adhesive contains a dimethacrylate polymer that modifies the rheology, allowing the material to flow under pressure, yet hold its shape after placement until light cured<sup>4</sup>. This characteristic is beneficial for indirect bonding, since the material will not slump, run, or drift from the bracket base prior to placement in the patient's mouth. Also, this material is a nanofilled resin that shows a reduction on filler size and increase on filler content (nanoclusters), allowing better mechanical properties<sup>18</sup>. However, little is known about the bonding characteristics of this material, especially for indirect bonding.

As observed, there are various materials used for bracket bonding, however, most studies evaluated the bond strength of these new systems by direct bonding technique. Another important factor is the post-fixation time that can influence the brackets bond strength and is important for the installation of the arches or accessories in the oral cavity. Thus, the aim of the present study was to evaluate the shear bond strength (SBS) of brackets fixed with different materials (two lightcured nanofilled low viscosity resins - Transbond Supreme LV and Flow Tain LV, a light-cured resin - Transbond XT (control) and two chemically cured resins for indirect bonding - Sondhi Rapid- Set and Custom I.Q.) using the indirect bonding technique after 10 min and 24 h. The hypothesis of this study is that the SBS values of nanofilled resins will be higher than those of the other materials and there will be no influence of post-fixation time on SBS values.

## Material and methods

One hundred sound extracted human premolars were stored in a 0.5 Chloramine T solution at 4°C for a maximum of 6 months after extraction. Exclusion criteria included previously restored premolars and premolars with enamel defects or cracking and delamination of the enamel. This study was carried out after approval of the Institutional Review Board (protocol # 168/09).

Teeth were randomly assigned into 10 groups (n=10) according to the material (Transbond XT – 3M/Unitek, Transbond Supreme LV – 3M/Unitek, Sondhi Rapid - Set – 3M/Unitek, Custom I.Q. – Relience and Flow Tain LV - Relience) and the post-fixation time (10 min or 24 h).

For the indirect bonding technique, the teeth were mounted in wax (Orto Central, Poá, SP, Brazil) to simulate a dental arch, totaling 10 teeth *per* arch. After, working models in orthodontic stone were obtained from accurate alginate impressions. The working models were prepared with careful trimming, removal of bubbles and filling of small voids, to avoid any problem in fitting of the bonding tray. The models were numbered according to the respective group. Reference lines (long axis of the tooth) were drawn on the models to facilitate placement of the brackets. A thin layer of separating medium (Cel-lac, SSWhite, Rio de Janeiro, RJ, Brazil) was applied to the models and allowed to dry for approximately 1h.

Transbond XT Light Cure Adhesive was placed on the mesh pad of individual metallic brackets (Mini Dyna-Lock "Roth" .022 - 3M/Unitek, Monrovia, CA, USA) that were positioned on the model and pressed firmly with a Hollenback carver to expel the excess adhesive. Each bracket was subjected to a 300-g compressive force using a force gauge (Correx Co, Berne, Switzerland) for 10 s, after which excess bonding resin was removed using a sharp scaler. The position of the bracket was carefully checked with a bracket gauge. The adhesive was light cured for 20 s from the occlusal edge and 20 s from the gingival bracket edge. The bonding adhesive was light cured with a light curing unit (XL300, 3M/Unitek), with a light intensity of 1000 mW/cm<sup>2</sup> measured with a built-in radiometer, which was calibrated every 10 min to ensure consistent light intensity.

After bracket bonding, the bonding trays were made using a vacuum unit (Plastivac, Bioart, São Carlos, SP, Brazil) to vacuum-form a 0.9-mm-thick flexible silicone layer (Soft tray sheets, Ultradent, Indaiatuba, SP, Brazil), overlayered with a 1 mm thick rigid PVC crystal layer (Bioart). A jet of an oil-free silicone spray (3M, Sumaré, SP, Brazil) was applied on models before lamination of the flexible layer, and applied again before the lamination of the rigid layer. This set was submerged in water for 1 h to facilitate the separation of materials. The trays were outlined and excess material was trimmed with crown and bridge scissors and scalpel. The bonding tray's hard outer shell was trimmed away from all heights of contour for patient comfort and closer fit because it only permits firm seating of the soft tray. Then, they were cleaned using bicarbonate/water jet and rinsed in water.

Following, the brackets basis was sandblasted with aluminum oxide (45 µm, Polidental, Cotia, SP, Brazil), for about 1 min on each tray, in order to increase retention, but without removing the resin Transbond XT, and cleaned with acetone.

The buccal enamel surface of each premolar was cleaned with fluoride-free pumice slurry, etched with 37% phosphoric acid gel (Etch-37, Bisco, Schaumburg, IL, USA) for 30 s, rinsed for 15 s, and dried with oil-free and moisturefree air for 20 s until the enamel had a faintly white appearance. After, the different materials (Transbond XT, Transbond Supreme LV, Flow Tain LV, Sondhi Rapid-Set and Custom I.Q.) were applied following the manufacturer's instructions and the trays were positioned over the teeth. For Transbond XT, Transbond Supreme LV and Flow Tain LV a small amount of the adhesive was applied to the bracket basis on the bonding tray that was brought into position and firmly hold. Each tooth was was light cured for 20 s from the occlusal edge and 20 s from the gingival bracket edge. Then, the trays were removed. For Sondhi Rapid-Set a thin layer of resin A was applied to each tooth surface and a thin layer of resin B was applied at each bracket basis. The bonding tray was brought into position and pressed for 30 s, after two min the trays were removed. For Custom I.Q. a thin layer of part A was applied to each tooth surface and a thin layer of part B was applied at each bracket basis. The set was placed in position and pressed for 30 s, after four min the trays were removed.

The bonding adhesives (Transbond XT, Transbond Supreme LV and Flow Tain LV) were light cured with a lightcuring unit (XL300, 3M/Unitek) with light intensity of 1000 mW/cm<sup>2</sup> measured with a built-in radiometer, which was calibrated every 10 min to ensure consistent light intensity.

After bracket bonding the trays were carefully removed with the aid of a scaler. The teeth were removed from the wax and the roots were embedded in self-cured acrylic resin (Vipi Flash, Pirassununga, SP, Brazil) using PVC cylinders (Tigre S.A. Tubos e Conexões, Castro, PR, Brazil) as moulds. The specimens were then immersed in deionized water and maintained in an oven at 37°C during 10 min or 24 h, and the shear bond strength (SBS) test was performed.

The specimens were secured in a jig attached to the base plate of a universal testing machine (Model 4411, Instron Corp, Canton, MA, USA). A chisel-edge plunger was mounted in the movable crosshead of the testing machine and positioned so that the leading edge was aimed at the enamelcomposite interface before being brought into contact. A crosshead speed of 0.5 mm/min was used. After debonding,

each specimen was examined under a stereoscopic zoom microscope (Olimpus SZX9, São Paulo, SP, Brazil) to identify the location of the bond failure. The residual composite remaining on the premolar was assessed by using the adhesive remnant index (ARI), where each specimen was scored according to the amount of material remaining on the enamel surface as follows: 0 - no adhesive remaining, 1- less than 50% of the adhesive remaining, 2 - more than 50% of the adhesive remaining, and 3 - all adhesive remaining with a distinct impression of the bracket base.

Descriptive statistics including means (MPa) and standard deviations (SD) were calculated for the SBS analysis. A two-way analysis of variance (ANOVA) and Tukey's multiple-comparison tests were used to determine the statistical significance of any difference in mean SBSs among groups. The ARI was analyzed for percentage and frequency of fracture type and submitted to a chi-square test. Significance for all statistical tests was predetermined at p > 0.05. All statistical analyses were performed using BioEstat 5.0.

## Results

### Shear Bond Strengths

Mean bond strengths and standard deviations for each group are given in Table 1. There was a statistically significant difference among the materials regardless of the post-fixation time (Flow Tain LV = Transbond Supreme LV = Transbond XT > Sondhi Rapid-Set > Custom I.Q.) (p<0.05). The lightcured materials (Transbond XT, Transbond Supreme LV and Flow Tain LV) showed higher values compared with the chemically cured materials (Sondhi Rapid-Set and Custom I.Q.).

There was no statistically significant difference between 10 min and 24 h regardless of the materials (p > 0.05).

Table 1 - Mean (MPa) and standard deviation of shear bond strength (SBS) at 10 min and 24 h for the different materials.

Material	SBS at 10 min	SBS at 24 h
Transbond XT	4.12 (0.51) Aa	4.57 (1.00) Aa
Transbond Supreme LV	5.61 (2.09) Aa	4.24 (2.25) Aa
Flow Tain LV	5.44 (1.29) Aa	5.09 (0.99) Aa
Sondhi Rapid-Set	2.40 (1.30) Ba	2.86 (0.95) Ba
Custom I.Q.	1.22 (1.07) Ca	1.53 (0.86) Ca

Different capital letters in columns and small letters in rows indicate statistically significant difference (Tukey's test - p<0.05).

#### ARI

The distribution of failure modes, as expressed by ARI scores (%), is given in Figure 1. According to statistical analysis (chi-square analysis) of the ARI scores, all of the test groups exhibited similar bracket failure modes (p > 0.05). Regardless of the bond material, bond failure occurred partly at the bracket-adhesive (resin) interface but mainly within the adhesive (resin) (scores 1, 2 and 3). Enamel fractures were not observed in any of the tested specimens.

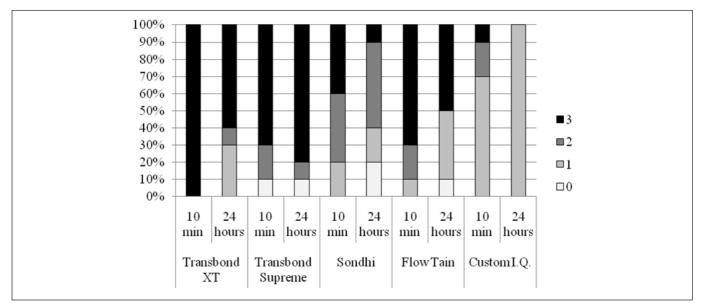


Fig.1 - Distribution of failure modes, ARI scores (%), found after the SBS test.

# Discussion

The material and technique of bracket bonding should promote sufficient adhesion between the brackets and teeth, supporting the application of forces during orthodontic treatment. With the introduction of the indirect bonding technique, several materials have been developed and investigated<sup>1,4,19</sup>, but the effectiveness of the increase in the bond strength of these materials at the tooth/bracket interface remains unclear.

The minimum shear bond strength of an adhesive should be between 5.9 and 7.9 MPa to be considered adequate for clinical needs<sup>20</sup>. When the results of this study were compared with these reference values, it was found that all the adhesives showed lower resistance values, but it could be noticed that these reference values were recommended based on the direct bonding technique.

In the present study, three light-cured resins and two chemically cured resins for indirect bonding were selected and applied according to the manufacturer's instructions. It could be observed that the light-cured materials showed higher bond strengths than the chemically cured ones. This finding was similar to other studies that also found lower bond strength values for chemically cured materials compared with light-cured ones<sup>19,21</sup>.

Nanofilled resins that present smaller and uniformly distributed filler particles, might present greater cohesive strength to penetrate into the etched enamel and also in the bracket base resin, increasing the bond strength<sup>18,21</sup>. However, the results of this study showed no significant difference among the SBS values of the nanofilled resins and Transbond XT resin. Thus, the hypothesis that the nanofilled resins would have a better performance than the traditional resins could not be validated. This could have occurred because the materials were applied in the resin previously attached

to the bracket base, which would have hindered a complete penetration of the material on this region. Uysal et al.  $(2010)^{12}$  did not found lower values for nanofilled materials (resin and ionomer) compared to a conventional orthodontic composite (Transbond XT).

For the chemically cured resins, which showed lower SBS values, it can be suggested that because these materials have no manipulation prior to its use, the catalyst and the base pastes are mixed only by the pressure at the time of bracket placement. This procedure could lead to incomplete polymerization of some portions of the material, which compromises their resistance<sup>21</sup>.

Constant changes are being made in the bonding materials and also on brackets bases in order to increase the bond strength, aiming at an improvement of clinical results. However, increased adherence difficult removal of the accessories in the end of treatment, which may cause cracks and fractures on the enamel surface<sup>22</sup>. Thus, fractures at the adhesive/bracket interface or within the adhesive, leaving the material adhered to the tooth surface, are favorable because these failures avoid enamel loss and the remaining adhesive can be safely removed with appropriate rotary instruments<sup>23</sup>.

Regarding the adhesive remnant index, it was observed that the failure of light-cured resins after 10 min and 24 h occurred more frequently at the adhesive/bracket interface and all the material remained adhered to the enamel (score 3). For Sondhi Rapid – Set group, after 10 min to 24 h, the score was predominantly 2, where half of adhesive remained adhered to the tooth. This result indicates that there was a poor adhesion between the material and the resin on the bracket base, which could be related to the indirect bonding technique used in the study. For the Custom I.Q. group, failure was predominantly score 1 (less than half of the adhesive adhered to the enamel), confirming the results found in other study<sup>24</sup>.

Regarding the post-fixation time, there was no significant

difference between 10 min and 24 h tests, regardless of the materials, as observed in other studies<sup>25-26</sup>. Thus, the hypothesis that it will be no influence of post-fixation time on SBS values could be validated. However, other studies have found higher SBS values for groups tested after 24  $h^{27-28}$ . This could be related to the materials selected in other studies, i.e., glass ionomer cements and resin-modified glass ionomer cements that reach their final resistance after 24 h. In this study, only resin based materials (light-cured or chemically cured) were used, demonstrating that after 10 min the polymerization of the materials allowed the material to reach an adequate resistance to be tested.

For orthodontists, it is important to know the properties of resins used in bracket bonding procedures because the resin must have the ability to maintain orthodontic accessories firmly adhered to the teeth during the treatment, resisting to masticatory forces and the forces generated by orthodontic mechanics.

As shown in this study, many materials of different properties and characteristics can be used for indirect bonding technique. However, light-cured resins were more effective. The indirect bonding technique was found to be simple, and with little ability, any professional can run it safely. This technique is very efficient, provides less wear to the orthodontist at the time of bonding, shortening the chairtime for the complete assembly of the appliance, and also offers the patient greater comfort.

Based on the results of this study, it could be concluded that light-cured adhesives showed the higher shear bond strength values compared with chemically cured adhesives; the storage period had no influence on shear bond strength values, and the failures were predominantly at the bracket/ resin interface, where most of the material staid on the enamel (scores 1, 2 and 3).

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