A New One-Step Dental Flowable Composite for Orthodontic Use: An *In Vitro* Bond Strength Study

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Abstract: A new flowable composite, Denfil Flow®, has shown an acceptable shear bond strength for bonding orthodontic brackets, when used with an intermediate, unfilled, low-viscosity resin. According to the manufacturer, it also shows a good viscosity for use with no preliminary adhesive. This could reduce the total time of bonding procedure while maintaining clinically useful bond strength. The aim of the current research was to assess this property. Eighty extracted human premolars were randomly divided into four equal groups. Stainless steel brackets were bonded to etched enamel using (1) Denfil Flow, (2) a traditional flowable composite (Dyract Flow®), (3) Denfil Flow composite resin and an intermediate liquid resin, and (4) Transbond XT® adhesive. Debonding was performed with a shearing force. The residual adhesive on the enamel surface was evaluated using the adhesive remnant index. The bond strength of Denfil Flow (34.8 MPa) showed no significant difference with the other control groups and was clinically acceptable. Denfil Flow and Dyract Flow tended to display cohesive failure within the adhesive. Denfil Flow can be used without liquid resin to reduce the bonding procedure time while maintaining acceptable bond strength. Further studies are required to evaluate the enamel surface of the teeth after the same polishing procedure in the four groups. (*Angle Orthod* 2005;75:574–579.)

Key Words: Shear bond strength; Flowable composite; Orthodontic brackets

INTRODUCTION

Since the advent of bonding brackets,[1] clinicians and researchers have worked to improve the qualities of bonding agents. The qualities that have been of most interest include bond strength, adequate working time, shorter cure time, and improved ease of use. Among the composite resins that could be used in orthodontics as bonding agents today, flowable composite merits great attention because of its clinical handling characteristics.[2] Flowable composites show two desirable clinical handling characteristics that have not existed for composites until very recently: (1) nonstickiness, so that materials could be packed or condensed, and (2) fluid injectability.

These characteristics are associated with the low viscosity of this type of composite resin. Generally, all mechanical properties of a composite resin improve with filler loading. Traditional dental composite resins are densely loaded with reinforcing filler particles for strength and wear resistance. Wear resistance increases when small, highly packed filler particles protect the polymer matrix in the composite from food bolus wear.[3] Flowable composites were created by retaining the same small particle sizes of traditional hybrid composites but by reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture. The authors have recently been involved in the testing of a new composite resin named Denfil Flow (Vericom Laboratories Ltd, Anyang, Korea), which guarantees a clinical acceptable shear bond strength (SBS), compared with Transbond XT, when used for bonding orthodontic brackets.[4] Denfil Flow is

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an enamel-bonding agent that belongs to a new generation of flowable composites. It is composed of Bis/GMA/TEGDMA, with barium glass and silica. The content of inorganic filler (mean particle size is 0.01–2.5 μm) is 60% by weight.

According to the manufacturer, Denfil Flow provides a low viscosity and a high SBS and film thickness and can be used for bonding orthodontic brackets without the use of a preliminary liquid resin on the etched enamel surface.

The objective of this study was to test the compatibility of Denfil Flow for bonding orthodontic brackets without liquid resin. Consequently, the SBS of the composite resin in direct bonding of orthodontic metal brackets to enamel was evaluated, as was the mode of failure after the debonding of the brackets. The findings were compared with those of other representative commercial adhesives (Transbond XT, Dyract Flow, and Denfil Flow with the preliminary low-viscosity resin).

MATERIALS AND METHODS

Teeth

Eighty human premolar teeth were collected and stored in distilled water at room temperature, with thymol crystals added to inhibit bacterial growth (0.1%). Approximately six months elapsed between extraction of the teeth and experimentation. Exclusion criteria included previously restored teeth and teeth with enamel defects or cracking and delamination of the enamel. The teeth were examined with a magnifier (10×).

Bonding

The teeth were randomly divided into four equal groups. The buccal crown surface of each tooth was rinsed and dried after a 15-second polish with fluoride-free pumice slurry. Stainless steel metal premolar Standard Edgewise brackets (Apollo™ class G&H, Greenwood, Ind) were bonded to the teeth with a different adhesive used for each group. All brackets were bonded by the same operator (TS) who was blind to the groupings of the teeth. The bonding adhesives were all light cured with a curing light (XL300; 3M/Unitek Dental Products, Monrovia, Calif), which was calibrated every 10 minutes to ensure consistent light intensity. At no time did the curing light intensity fail to measure at least 400 mW/cm².
Group 1: composite resin, Transbond XT (control). The buccal enamel surface was etched with 37% phosphoric acid for 30 seconds, rinsed for 15 seconds, and dried with oil-free and moisture-free air until the enamel had a faintly white appearance. Transbond XT primer was applied to the etched surface in a thin film

and light cured for 10 seconds. Transbond XT adhesive paste was applied to the bracket base, and the bracket was positioned on the tooth and pressed firmly with a Hollenback carver to expel the excess adhesive. In both the groups, each bracket was subjected to a 300-g compressive force using a force gauge (Correx Co, Berne, Switzerland) for 10 seconds, after which excess bonding resin was removed using a sharp scaler. Then, the adhesive was light cured for 20 seconds from the incisal edge and 20 seconds from the gingival bracket edge. Although the manufacturer recommends 20 seconds of light curing, Wang and Meng[5] found that brackets bonded with Transbond XT and cured for 40 seconds had a stronger bond than did those cured for only 20 seconds.

Group 2: flowable composite resin, Denfil Flow with intermediate unfilled liquid resin. The Denfil Flow was obtained from manufacturers in A-2 shades. Etching, rinsing, and drying were done according to the Transbond XT protocol. An intermediate unfilled low-viscosity liquid resin (Vericom Laboratories Ltd, Anyang, Korea) was applied on the air-dried and etched enamel. It was left for 10 seconds, lightly dried, and then light cured for 10 seconds. Denfil Flow was applied to the bracket base, the base was positioned, and the adhesive was light cured according to the Transbond XT protocol.

Group 3: flowable composite resin, Denfil Flow. Etching, rinsing, and drying were done according to the Transbond XT protocol. No intermediate low-viscosity liquid resin was applied on the etched enamel surface. Denfil Flow was applied to the bracket base, the base was positioned, and the adhesive was light cured according to the Transbond XT protocol.

Group 4: flowable composite resin, Dyract Flow (DeTrey Dentsply, Konstanz, Germany). Dyract Flow was obtained from manufacturers in A-2 shades. Etching, rinsing, and drying were done according to the Transbond XT protocol. Dyract Flow was applied to the bracket base, the base was positioned, and the adhesive was light cured according to the Transbond XT protocol.

Storage after bonding

The bracketed teeth were immersed in sealed containers of deionized water and placed in an incubator at 37°C for 72 hours to permit adequate water absorption and equilibration.[6]

Debonding

A 0.021 × 0.025-inch stainless steel wire was ligated into each bracket slot to minimize deformation of bracket during debonding (Figure 1). Each specimen was then mounted in a standardized 20 × 23-mm

FIGURE 1. Bonded teeth set in acrylic block; a 0.021 × 0.025-inch stainless steel wire was ligated into each bracket slot to minimize deformation of bracket during debonding; a 0.020-inch loop was made from a 0.012-inch stainless steel ligature wire and placed under the gingival wings of the twin bracket.

acrylic block (Figure 1) (autopolymerizing polymethyl methacrylate, PMMA, Esschem Co, Portland, Oregon). Each of the 160 samples was assigned a four-digit sample number so the examiner (MD'A) was blinded to the sample group. For shear testing, each bonded bracket was positioned in a testing machine (Lloyd 30K, Lloyd Instruments Ltd, Segensworth, UK), with a computerized method of measurements (Nexigen, v. 4.0), parallel to the direction of load application (Figure 2). A 0.020-inch loop was made from a 0.012-inch stainless steel ligature wire and placed under the gingival wings of the twin bracket (Figures 1 and 2). The loop was then moved gingivo-occlusally at a crosshead speed of 1 mm/minute. The load range was 50 kg.

To minimize variation in the direction of the debonding force, each block was secured in a bench vice with the pad of the bracket positioned parallel to the plunger of the testing machine (Figure 1). The load applied at failure was recorded in Newtons (N), and the stress was calculated in megapascals (1 MPa = 1 N/mm^2) by dividing the force in N by the bracket base area of 9 mm^2. The surface area of the base was determined by measuring length and width and by computing the mean area.

Bond failure assessment

The debonded enamel surfaces were examined under 16× magnifications fiber-optic transillumination.

The residual adhesive remaining on the teeth was assessed by using the adhesive remnant index (ARI), as described by Årtun and Bergland[7] and modified by Lalani et al[8] to include a score for enamel fracture (EF). The remaining adhesive was scored with respect

FIGURE 2. Bonded teeth set in acrylic block and positioned in a testing machine.

to the amount of resin material that remained on the surface of the tooth: 0, no adhesive remained on the tooth; 1, less than 50% of the adhesive remained on the tooth; 2, more than 50% of the adhesive remained on the tooth; and 3, all adhesive remained on the tooth. The ARI scores were used to assess the sites of bond failure on the enamel-adhesive interface and the adhesive-bracket interface.

Statistical analysis

Descriptive statistics included for the study and the control group included the mean, standard deviation (SD), range, variance, minimum and maximum of SBS (MPa) (Table 1), as well as frequency distribution of the ARI scores (%) (Table 2). A one-way analysis of variance (ANOVA) and Tukey's multiple comparison tests were used to determine the statistical significance of any difference in mean SBSs between the four groups. The ARI was analyzed for percentage and frequency of fracture type, and a chi-square test

TABLE 1. Mean Shear Bond Strengths (MPa) and Descriptive Statistics

	N	Mean	SD	Variance	Range	Minimum	Maximum	Significance
Denfil Flow	20	34.80	19.70	387.79	91.92	16.78	108.70	NS[a]
Dyract Flow	20	28.80	16.24	263.84	71.10	12.93	84.03	NS
Denfil Flow/Denfil Primer	20	25.52	7.12	50.65	32.61	14.75	47.36	NS
Transbond XT	20	23.23	5.20	27.0	20.43	14.72	35.14	NS

[a] NS indicates not significant; * $P < .05$.

TABLE 2. Frequency Distribution of the Adhesive Remnant Index (ARI) Scores (%) for the Two Groups[a]

	ARI					
Group	0	1	2	3	EF	χ^2 test
Denfil Flow	5	35	40	15	5	
Dyract Flow	0	30	55	14	1	
Denfil Flow/Denfil Primer	5	12	18	58	7	($\chi^2 = 6.510$)
Transbond XT NS	5	5	15	65	10	

[a] 0 indicates that no adhesive remained on the tooth; 1, less than 50% of the adhesive remained on the tooth; 2, more than 50% of the adhesive remained on the tooth; 3, all adhesive remained on the tooth; EF, enamel fractured; NS, not significant.

was used as the statistical test. Significance for all statistical tests was predetermined at $P < .05$.

RESULTS

Shear bond strengths

Descriptive statistics are provided in Table 1. The ANOVA indicated no significant differences in bond strength among the four groups. Denfil Flow displayed the higher mean SBS (34.8 MPa), SD (19.7), and range (91.92 MPa).

Adhesive remnant index

The chi-square analysis comparing the ARI scores indicated no significant differences in the ARI scores among the four groups (Table 2). In both the groups bonded with flowable composite (group 1 and group 2), the greatest frequency was observed at ARI scores of 2 and 3 (75% and 85%, respectively), whereas group 4 and group 3 displayed the greatest frequency at an ARI score of 3 (65% and 58%). Dyract Flow showed the lower frequency of EF after debonding (Table 2).

DISCUSSION

Traditionally, the use of a primer was an essential part of the bonding procedure of composite adhesives to allow good wetting and penetration of the sealant into the enamel surface.[9] Recently, the use of self-etching primers for orthodontic purposes was believed

to simplify the clinical handling of adhesives systems by combining the etchant and the primer in one application.[10] However, the earlier generations of acidic primers were selectively compatible with different adhesives and, thus, they produced significantly lower bond strength or needed significantly more working time.[10]

The goal of current orthodontic research is to improve the bonding procedure by minimizing the time of working during bonding and debonding without jeopardizing the ability to maintain a clinically useful bond strength. This investigation revealed that a flowable composite can be used for bonding orthodontic brackets without the intermediate low-viscosity resin while, concomitantly, increasing bond strength and reducing the working time. The in vivo performance of the fixed appliances bonded with Denfil Flow will be assessed in a future clinical trial. Nonetheless, the early in vitro SBSs seem promising. By reducing the number of steps during bonding, clinicians are able to save time and reduce the potential for error through contamination during the bonding procedure.

Shear bond strengths

The bond strength was not measured under oral conditions, where mechanical impact and biochemical changes may result in adhesive material fatigue and inadvertent debonding forces. Nevertheless, in vitro shear debonding forces are an acceptable methodology to determine future in vivo comparative conditions.[11]

The bond strengths of the four adhesives tested were greater than the 5.9 to 7.8 MPa considered by Reynolds[12] to be adequate for routine clinical use. The bond strength for the control composite Transbond XT, at 23.23 MPa, was greater than that observed in some previous studies,[8,13–15] although similar to that found by Rock and Abdullah,[16] Sinha et al,[17] Tang et al,[18] and Rix et al.[19] Denfil Flow with no intermediate low-viscosity liquid resin showed the largest SD (SD: 19.70) and the greater range (91.92) for bond strength of the four adhesives, suggesting that the bond strength for this material may be more technique-sensitive than the others. However, because of the observed high bond strength mean values, we can expect a decrease in unexpected debonding during treatment. Our six

months of clinical experience with Denfil Flow with no preliminary liquid resin have confirmed this. Future clinical investigation will be performed to confirm this data.

Enamel fractures

The four groups displayed a low frequency of EF on debonding. The frequency of EF for Transbond XT in this study was 7%, compared with 16.2% for similarly treated samples by Lalani et al[8] and 57.5% by Rix et al.[19] Denfil Flow and Dyract Flow displayed lower frequencies of EF (5% and 1%, respectively) on debonding than did Transbond XT. This means that flowable composites, with no intermediate low-viscosity resin, seem to be sufficient to obtain clinically acceptable bond strength, with the added advantage of causing no damage to the enamel surface. In this study, the higher frequency of EF occurred with the materials that were applied with the use of an intermediate liquid resin (Transbond XT and Denfil Flow with Denfil Primer). However, the high level of EF noted in these groups does not occur clinically and may be due to the extensive cracking from extraction process and the generally higher bond strengths obtainable in ideal benchtop conditions.

Adhesive remnant index

The chi-square analysis comparing the ARI scores indicated no significant differences between the four groups in the type of bond failure (Table 2). In both the groups bonded with flowable composite (group 1 and group 2), the greatest frequency was observed at ARI scores of 2 and 3 (75% and 85%, respectively), whereas group 4 and group 3 displayed the greatest frequency at an ARI score of 3 (65% and 58%, respectively). The ARI score of 3 indicates that the entire adhesive remained on the tooth after debonding and, consequently, that the bond failure occurred at the adhesive-bracket interface.

The ARI of zero indicates that no adhesive remained on the tooth after debonding and, consequently, that the bond failure occurred at the enamel-adhesive interface. The ARI scores of 1 and 2 indicate, respectively, that less than 50% or more than 50% of the adhesive remained on the tooth after debonding, which is defined as a cohesive failure within the adhesive. Examination for ARI indicates that there was less adhesive remnant left on the teeth when flowable composites were used with no intermediate low-viscosity resin. This was probably because wetting and penetration of the composite into the enamel surface was reduced as compared with when a lower viscosity resin was used. However, this reduced penetration into the enamel surface did not reduce the SBS that

was clinically acceptable for both Denfil Flow and Dyract Flow. In addition, examination of the ARI score showed that flowable composites with no intermediate low-viscosity resin tended to display adhesive cohesive failure within the adhesive. On the other hand, Transbond XT and Denfil Flow with use of low-viscosity resin tended to display adhesive failure at the adhesive/bracket interface.

CONCLUSIONS

- There was no significant difference in mean SBSs between the four adhesives.
- The bond strengths for the four adhesives were clinically acceptable.
- Transbond XT and Denfil Flow with the adjunction of low-viscosity liquid resin tended to display adhesive failure at the adhesive/bracket interface, whereas Denfil Flow and Dyract Flow tended to display cohesive failure within the adhesive.
- Dyract Flow displayed the lower frequency of EF after debonding (1%).

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