Original Article

Shear Bond Strength, Bond Failure, and Scanning Electron Microscopy Analysis of a New Flowable Composite for Orthodontic Use

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Abstract: A new dental flowable composite, Denfil Flow, was evaluated for the bonding of orthodontic brackets by determining its shear bond strength (SBS) and the mode of bond failure after debonding. Eighty extracted human premolars were divided into two equal groups. Metal brackets were bonded to etched enamel using a composite resin control (Transbond XT) or Denfil Flow. After 72 hours of incubation in saline solution at 37°C, debonding was performed with a shearing force. The SBS and the mode of bond failure were examined. In addition, representative samples from each group were examined by scanning electron microscopy (SEM). No significant difference was observed in the SBS between the groups, and a clinically acceptable SBS was found for the two adhesives. Bond failures occurred mostly in the bracket–adhesive interface, without significant differences between the groups. At SEM analysis, Denfil Flow showed a greater frequency of air bubbles within the resin than did Transbond XT. In conclusion, Denfil Flow displayed the same SBS as traditional composite resins and similar bond failures. Further clinical studies are required. (*Angle Orthod* 2005;75:410–415.)

Key Words: Composite; Flowable; Brackets; Shear bond strength; Bond failure

INTRODUCTION

The use of acid etch techniques in the direct bonding of orthodontic brackets was reported in 1965 by Newman,[1] and the direct bonding of orthodontic brackets is perhaps the most significant development in orthodontics during the past three decades. Bonding orthodontic brackets to tooth surfaces improves the esthetic aspect of orthodontic appliances, minimizes treatment time, and allows a better standard of oral hygiene to be achieved.

Since the bis-phenol A glycidyl methacrylate (BIS-GMA) resins were first applied in clinical orthodontic practice as adhesives,[2] the acid etched/composite technique has become the most widely adopted bonding system in contemporary orthodontic practice. However, such a system still has a number of shortcomings such as loss of enamel after acid etching,[3] enamel damage caused by postdebonding cleanup procedures,[4] and enamel fracture (EF), which may take place during debonding.

To address these problems, practitioners have searched for an adhesive that could overcome these shortcomings and simplify the procedures involved in bonding. In this quest, flowable composite resins merit great attention because of two of their clinical handling characteristics, which have not existed for composites until very recently, ie (1) nonstickiness, so that materials could be packed or condensed, and (2) fluid injectability.[5] These characteristics are associated with the low viscosity of the mixture. 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 filler particles are highly packed to protect the polymer matrix in the composite.[6]

Flowable composites were created by retaining the same small particle size of traditional hybrid composites but reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture. They were originally considered for restorative procedures.[7] However, because flowable composites were not as robust as conventional

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composites in any category of in vitro mechanical testing,[7] the clinicians limited their use to applications that benefited from better flow and were not associated with high stress. Therefore, the bond strength characteristics of flowable composite resins for use as orthodontic adhesives were never investigated in previous studies.

However, recently, a new composite resin named Denfil Flow (Vericom Laboratories Ltd, Anyang, Korea) has been introduced. It belongs to a new generation of flowable composites and 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. Because of the very small size of particles, the filler particles can be more highly packed than other flowable composites, even maintaining the same low viscosity. This will increase their wear resistance. The hypothesis of this investigation is that Denfil Flow, because of its characteristics, could provide an adequate shear bond strength (SBS) and a good viscosity and, therefore, also could be used for bonding orthodontic brackets. The objective of this study was to test the use of Denfil Flow for orthodontic purposes.

MATERIAL AND METHODS

Teeth

Eighty human premolar teeth were 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. Previously restored teeth or teeth with enamel defects or cracking (observed at a magnification of 10×) were excluded.

Bonding

The 80 teeth were divided randomly into two 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 in each group. All brackets were bonded by the same operator who was blind to the aim of the study. The bonding adhesives were all light cured with a curing light (XL300; 3M/Unitek Dental Products, Monrovia, Calif), calibrated every 10 minutes to ensure consistent light intensity.

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 and moisture-free air until the enamel had a faintly white appearance. Transbond XT primer was applied in a thin film to the etched surface 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 an instrument 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, Bern, 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 edge of the bracket.[8]

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 0.012-inch stainless steel ligature wire and placed under the gingival wings of the twin bracket.

Group 2: flowable composite resin, Denfil Flow. Etching, rinsing, and drying were done following the Transbond XT protocol. An intermediate, unfilled, low-viscosity liquid resin (Vericom) was applied on the air-dried and etched enamel to maximize the bond strength, left for 10 seconds, dried lightly, and light cured for 10 seconds. Then, Denfil Flow was applied following 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.[9]

Debonding. Each specimen was mounted in a standardized 20 × 23–mm acrylic block (Figure 1) and was assigned a four-digit sample number, so the examiner was blind to the sample group. A 0.021 × 0.025–inch stainless steel wire was ligated into each bracket slot to minimize bracket deformation during debonding (Figure 1). A 0.020-inch loop was made from 0.012-inch stainless steel ligature wire and placed under the wings of the twin bracket (Figures 1 and 2). The loading of the bracket on the wings, rather than close to the base, was more representative of in vivo loading and ensured a more consistent application of debonding force.[10] For a shearing test, each specimen was positioned in an Instron machine (Lloyd 30K, Lloyd Instruments Ltd, Segensworth, UK) with a computerized method of measurements (Nexigen version 4.0), parallel to the direction of load application (Figure 2). To minimize variation in the direction of the debonding force, each block was secured in a bench vice with the pad of the bracket

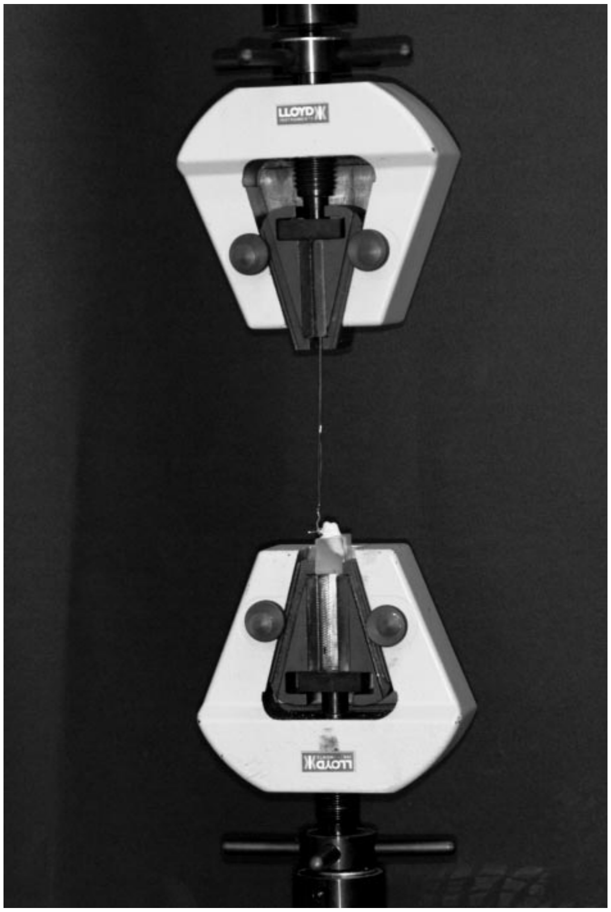

FIGURE 2. Bonded teeth set in acrylic block and positioned in Instron testing machine.

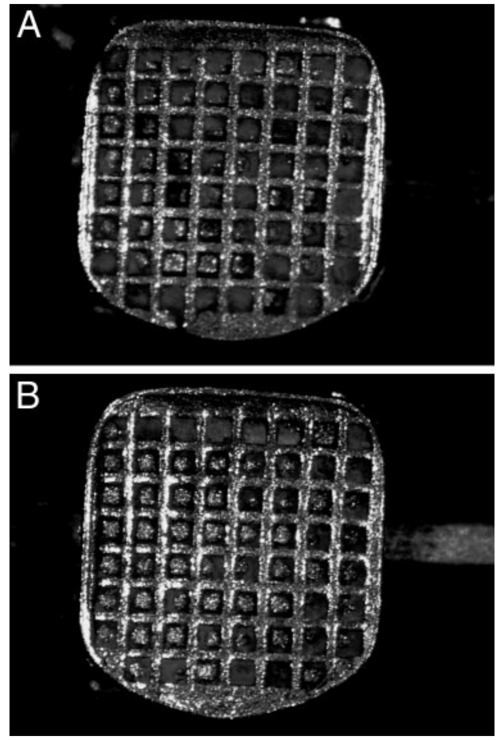

FIGURE 3. Stainless steel bracket base after debonding (16× magnifications): (a) Transbond XT and (b) Denfil Flow.

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

Group	Mean	SD	Min	Max	N	Significance
Transbond XT	23.47	4.86	14.72	35.14	40	NS[a]
Denfil Flow	24.98	7.33	14.75	47.36	40	

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

positioned parallel to the plunger of the testing machine (Figure 1). The ligature of the tooth was then moved gingivo-occlusally at a crosshead speed of one mm/min. The load range was 50 Kg. The load applied at failure was recorded in newtons and divided by the bracket-base area of nine mm^2 to be recorded in megapascals (one Mpa = one N/mm^2).

Bond failure assessment. The debonded enamel surfaces were examined with 16× magnification using fiber-optic transillumination (Figure 3). The residual adhesive remaining on the teeth was assessed using the adhesive remnant index (ARI),[11] modified to include a score of EF.[12] The ARI scores were also used as a more complex method of defining the site of bond failure between the enamel, the adhesive, and the bracket.

Scanning electron microscopy

Two representative bracket bases from the two groups were selected under a microscope at 16× magnification and mounted on scanning electron microscopy (SEM) studs (Figures 3 through 6). The specimens were stored for two days in absolute alcohol, air dried for two hours, mounted on SEM stubs so that the relevant area of interest could be seen, sputter coated with 10 nm of platinum in a Polaron E5100 SEM coating unit (Polaron Equipment Ltd, Hertfordshire, England), and examined in a Hitachi-S-2500 SEM (Hitachi Ltd, Tokyo, Japan) at an operating voltage of 10 kV. Scanning electron micrographs were used to analyze bracket surfaces qualitatively.

Statistical analysis

Descriptive statistic included mean, standard deviation (SD), range of SBS (in MPa) (Table 1), and frequency distribution of ARI scores for the two groups (Table 2). A Student's *t*-test was used to evaluate significant differences in mean SBS between the groups. The chi-square test was used to evaluate statistically significant differences in the frequencies of ARI scores between the groups. Statistical significance for all tests was set at $P < .05$.

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

	ARI[b]					
Group	0	1	2	3	EF	χ^2 test
Denfil Flow	5	5	15	65	10	6.714
Transbond XT	5	12	18	58	7	NS

[a] ARI indicates adhesive remnant index; EF, enamel fracture; and NS, not significant.

[b] 0 indicates 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.

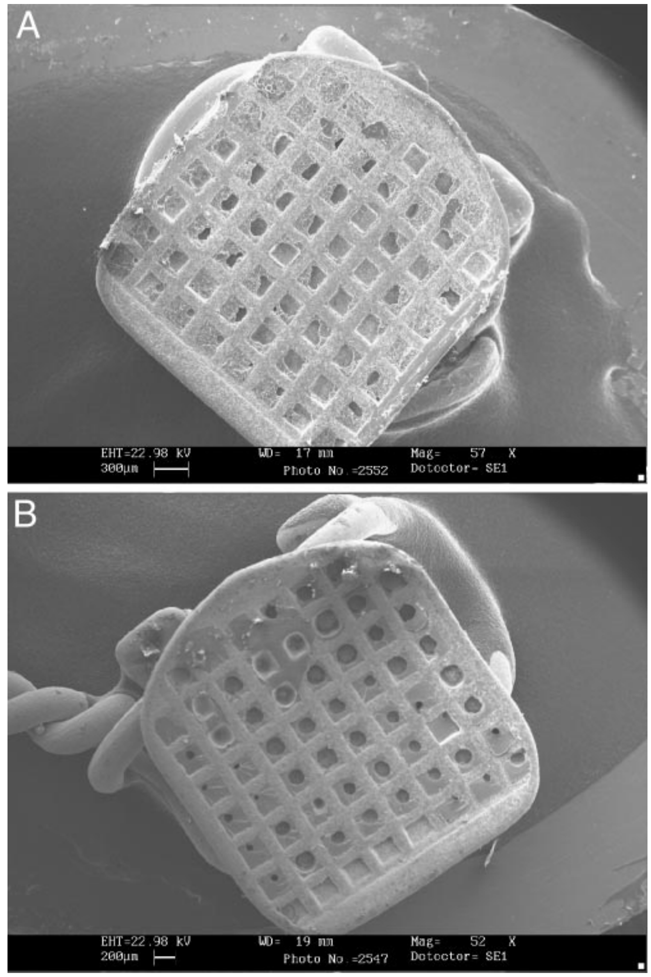

FIGURE 4. Scanning electron micrographs of the bracket base after debonding: (a) Transbond XT (57×, working distance (WD) = 17 mm, 22.98 kV, scale bar = 300 μm) and (b) Denfil Flow (52×, WD = 19 mm, 22.98 kV, scale bar = 200 μm). Very little adhesive was retained on the two bracket bases. Denfil Flow (b) showed bubbles within the adhesive.

RESULTS

Shear bond strength

The summary statistics of SBS are provided in Table 1. The analysis of variance showed no significant differences in mean SBS between the two groups.

Adhesive remnant index

The chi-square analysis showed no significant differences between the two groups in the ARI scores (Table 2). In both the groups, the highest score of 3 occurred more frequently than the other scores. This finding shows a greater trend for the two adhesives to remain on the tooth surface after debonding, with a distinct impression of the bracket mesh on the adhesive remaining on the tooth surface. The site of bond failure was the adhesive-bracket interface in about 60% of the specimens. The lowest score of 0 occurred less frequently than the other scores in both the groups (5%). This confirmed the trend for the two adhesives to remain on the enamel surface after debonding. In addition, both groups of teeth with EF displayed ARI score of 0 or 1. Finally, approximately 35% of the total sample displayed a bond failure within the adhesive and displayed ARI scores of 1 or 2.

SEM analysis

In each of the two representative bracket bases, the mesh-like pattern is visible (because the bond failure occurred mostly at the bracket-adhesive interface), although it is not well defined because of the residual bonding material. A notable difference between the two groups was observed in the greater incidence of air bubbles on the bracket bases bonded with Denfil Flow (Figures 4 through 6) rather than Transbond XT. Transbond XT displayed a well-defined resin penetration into the areas of the base bracket.

DISCUSSION

Shear bond strength

The SBS was not measured under oral conditions. Nevertheless, the in vitro SBS was found to be an acceptable methodology to determine future in vivo comparative conditions.[13] For the two adhesives, the mean SBS that resulted was greater than the 5.9-Mpa limit, considered to be adequate for routine clinical use.[14] The mean SBS of Transbond XT was greater than that observed in some previous studies,[12,15–17] although similar to that found in other investigations.[18–21] This points to the importance of other variables, such as the retention of the bracket base and the enamel pretreatment in determining the SBS. Denfil Flow showed the largest SD and a greater range of SBS values than did Transbond XT (Table 1), suggesting that the SBS for this material may be more technique sensitive than the other.

Enamel fracture

The two groups displayed a very low frequency of EF on debonding (Table 2). For the Transbond XT specimens, it was 7% compared with 16.2%[12] and 57.5%[21] for similarly

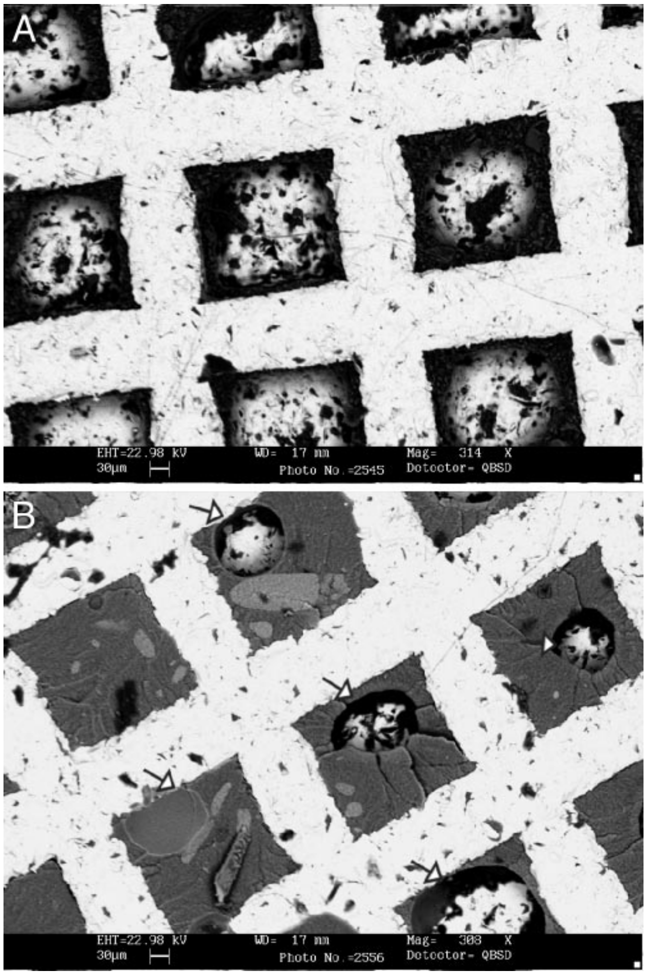

FIGURE 5. Scanning electron micrographs of the bracket base after debonding (a) Transbond XT (760×, working distance (WD) = 17 mm, 22.98 kV, scale bar = 20 μm) and (b) Denfil Flow (498×, WD = 17 mm, 22.98 kV, scale bar = 30 μm). Denfil Flow (b) showed bubbles within the adhesive (labeled in white).

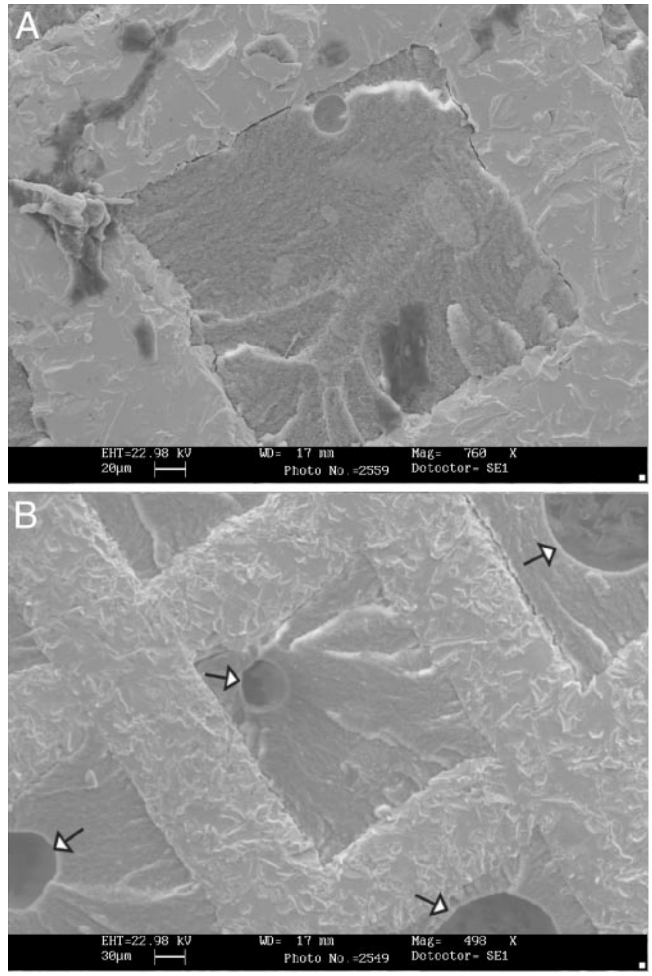

FIGURE 6. Scanning electron micrographs of the bracket base after debonding (a) Transbond XT (308×, working distance (WD) = 17 mm, 22.98 kV, scale bar = 30 μm) and (b) Denfil Flow (314×, WD = 17 mm, 22.98 kV, scale bar = 30 μm). Denfil Flow (b) showed very little adhesive retained on the bracket base (labeled in white).

treated samples. In both the groups, teeth with EFs always displayed an ARI score of 0 or 1. This seems to show that the weak point of each of the two systems is represented by the enamel-adhesive interface. Furthermore, the two adhesives showed the highest frequency (approximately 60% of the total group) of ARI at a score of 3 (bonding failure at the bracket-adhesive interface) and, consequently, a low frequency of EF. Considering that the clinical search for optimal bond strength means (1) minimizing unexpected debonding during treatment[14] and (2) obtaining an undamaged enamel surface after debonding,[22] both Transbond XT and Denfil Flow seem to provide optimal in vitro performances.

Adhesive remnant index

No significant differences between the two groups were observed in the ARI scores (Table 2). In both groups, the greatest frequency of ARI score observed was 3 with bond failure occurring mostly at the adhesive-bracket interface. For Transbond XT, the percentage of 58% does not agree with that of 2.5% of a previous investigation,[21] where 95% of the specimens displayed ARI scores of 2 or 3, suggesting a trend for Transbond XT to display a cohesive failure within the adhesive, as confirmed successively by Tang et al.[20] This points to the influence of other variables in determining the type of bond failure, such as the bracket retention mechanism.[23]

In fact, although metal micromesh brackets were used in the two cited studies, G&H Apollo™ class brackets used in this investigation present a presandblasted, simple foil-mesh bonding pad.

It has been suggested that adherence of the adhesive to the bracket shows surface enamel removal during the debonding process, whereas adherence to the tooth assures an intact enamel surface.[24] Although final polishing of the teeth after debonding would appear to be the same after both types of resin fracture, it also can be suggested that if the majority of the brackets debond at the enamel-adhesive interface, the fluoride-rich surface enamel can be compromised and an intensive topical fluoride regimen after debonding is recommended. According to these observations, the bond failure at the bracket-adhesive interface is desirable when the adhesive is a composite resin that does not release fluoride.

SEM analysis

The two specimens considered in Figures 4 through 6, with the same ARI score, were selected to observe the morphological aspect of the bracket base after debonding. Two characteristics are notable: (1) Denfil Flow specimen displays a lot of air bubbles, probably associated with the low viscosity of the moisture (Figures 4b, 5b, 6b); however, the presence of these air bubbles seems to not decrease the SBS to under clinically acceptable values[14] and (2) mechanical retention of the bracket bases did not seem very appreciable because of the adhesive loss from the mesh in both the specimens (Figures 4 through 6).

CONCLUSIONS

This investigation revealed that Denfil Flow can be used for bonding orthodontic brackets that reduces working time while concomitantly maintaining adequate SBS as compared with a traditional composite resin. Our one year of clinical experience with Denfil Flow confirms a decrease in unexpected debonding during treatment. However, the in vivo performance of flowable composites will be better investigated in a future study. SEM analysis revealed that Denfil Flow showed a greater frequency of air within the adhesive, probably caused by the low viscosity of the moisture.

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