

An *in vitro* comparison of the shear bond strength of a resin-reinforced glass ionomer cement and a composite adhesive for bonding orthodontic brackets

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SUMMARY The shear bond strength (SBS) of a light-cured, resin-reinforced glass ionomer and a composite adhesive in combination with a self-etching primer was compared after different setting times to evaluate when orthodontic wires could be placed. Additionally, the fracture site after debonding was assessed using the Adhesive Remnant Index (ARI). Eighty freshly extracted human premolars were used. Twenty teeth were randomly assigned to each of four groups: (1) brackets bonded with Transbond XT with a Transbond Plus etching primer and debonded within 5 minutes; (2) brackets bonded with Fuji Ortho LC and debonded within 5 minutes; (3) brackets bonded as for group 1 and debonded within 15 minutes; (4) brackets bonded as for group 2 and debonded within 15 minutes. The SBS of each sample was determined with an Instron machine.

The mean SBS were, respectively: (1) 8.8 ± 2 MPa; (2) 6.6 ± 2.5 MPa; (3) 11 ± 1.6 MPa and (4) 9.6 ± 1.6 MPa. Interpolating the cumulative fracture probability by means of a Weibull analysis, the 10 per cent probabilities of fracture for the groups were found to be attained for shear stresses of 6.1, 3.1, 8.3 and 7.1 MPa, respectively. Chi-square testing of the ARI scores revealed that the nature of the remnant did not vary significantly with time, but the type of bonding material could generally be distinguished in leaving more or less than 10 per cent of bonding material on the tooth. After debonding, the Transbond system was likely to leave adhesive on at least 10 per cent of the bonded area of the tooth.

The present findings indicate that brackets bonded with either Transbond XT in combination with Transbond Plus etching primer and Fuji Ortho LC had adequate bond strength at 5 minutes and were even stronger 15 minutes after initial bonding.

Introduction

Direct bonding of brackets and other attachments with composite resins have become a routine technique in fixed orthodontic treatment (Newman, 1965; Retief and Dreyer, 1967; Silverman *et al.*, 1972). However, bonding orthodontic attachments with composite resins requires conditioning of the enamel surface with phosphoric acid, and a substantial amount of enamel is lost by etching (Gwinnett and Matsui, 1967; Fitzpatrick and Way, 1977; Brown and Way, 1978; Pus and Way, 1980; Diedrich, 1981; Øgaard *et al.*, 2004). Another disadvantage of composite resins involves enamel damage caused by post debonding clean-up procedures. Grinding off the adhesive from the tooth surface may lead to enamel alterations (Zachrisson and Årtun, 1979). Several variables that may influence bond strength have been investigated. These include concentration of acid (Retief, 1973; Gorelick, 1977), etching time (Gorelick, 1977; Nordenvall *et al.*, 1980; Mardaga and Shannon, 1982; Barkmeier *et al.*, 1985; Carstensen, 1986; Oilver, 1987; Kinch *et al.*, 1988), type of adhesive (Johnson *et al.*, 1976; Faust *et al.*, 1978), and type of bracket backing (Reynolds and von Fraunhofer, 1976; Lopez, 1980).

Recently, a new acidic primer to be used for orthodontic purposes was introduced (Transbond Plus, 3M Unitek, Monrovia, California, USA). Combining conditioning and priming into a single treatment step results in improvement in both time- and cost-effectiveness for clinicians as well as for patients (White, 2001; Asgari *et al.*, 2002; Buyukyilmaz *et al.*, 2003; Bishara *et al.*, 2004). One important advantage of simultaneous etching and priming is that the primer penetrates the entire depth of the etch, ensuring an excellent mechanical interlock (Buyukyilmaz *et al.*, 2003).

Glass ionomer cements (GICs) were introduced as dental cements by Wilson and Kent (1972). GICs possess many unique properties such as the ability to form chemical bonds with enamel, dentine and metal. Enamel decalcification adjacent to orthodontic attachments is a common phenomenon (Gorelick *et al.*, 1982; Øgaard 1989). GICs contain significant amounts of fluoride, which may protect against enamel decalcification (Kvam *et al.*, 1983; Rezk-Lega *et al.*, 1991). They also possess the advantage of easier debond with less potential for damage to the enamel.

The introduction of resin-reinforced GICs (RRGICs) combines the advantages of conventional GICs with the physical properties of composite resins (Antonucci and

Stansbury, 1989; Mathis and Ferracane, 1989). *In vitro* studies on the bond strength of RRGICs have shown low initial bond strengths which indicated that they were unsuitable for clinical use (Ashcraft *et al.*, 1997; Cook *et al.*, 1996; Øen *et al.*, 1991). Others (Cacciafesta *et al.*, 1998; Compton *et al.*, 1992; Komori and Ishikawa, 1997; McCarthy and Hondrum, 1994; Rezk-Lega and Øgaard, 1991) concluded that these materials probably possess sufficient strength to be recommended for clinical use.

White (1986), Øen *et al.* (1991) and Bishara *et al.* (1999b) reported that GICs had a low initial bond strength, which increased within 24 hours. In comparison, the composite adhesive had a significantly greater initial bond strength that doubled within 24 hours (Bishara *et al.*, 1999b). However, many clinicians use these GICs routinely and claim low failure rates. The purpose of this investigation was therefore two-fold:

1. To compare the bond strength of orthodontic brackets bonded with a light-cured resin RRGIC and a composite adhesive each used in combination with a self-etching primer at different time intervals, and to evaluate when archwires could be placed after bonding.
2. To examine the fracture site at debond with the two different bonding systems.

Material and methods

Eighty extracted human premolars were collected and stored in distilled water in a refrigerator at 4°C. The buccal surfaces were examined for cracks and other defects using a Wild Photomicroscope M-400 (Wildheerbrugg Ltd, Heerbrugg, Switzerland). The roots of the teeth were cut off with a cooled diamond disk and the pulp chamber enlarged in order to enhance mechanical retention of the crowns in the embedding medium. The crowns were embedded in plastic moulds filled with Epofix (resin and catalyst ratio: 15 and 2 ml, respectively; Struers, Copenhagen, Denmark) up to their facial surface, which was left intact. Before testing, all buccal surfaces were polished with a rubber cup and a fluoride-free pumice, sprayed with water and dried with a compressed oil free air stream. Universal premolar brackets (Ormco edgewise wide twin slot, ref no. 340-1404, Ormco Corporation, Glendora, California, USA) were bonded to all teeth. The surface area of the bracket base was 16.1 mm².

Two orthodontic adhesives were used: a light-cured RRGIC, Fuji Ortho LC (GC Corporation, Tokyo Japan) and a composite adhesive, Transbond XT (3M Unitek), in combination with a self-etching primer (3M Unitek). For both adhesive systems, the bonding approach followed the manufacturers' instructions. The procedure for Fuji Ortho LC included 10 per cent polyacrylic acid enamel conditioner for 20 seconds, rinsing with water and wiping the surfaces of the tooth with a moist cotton roll immediately prior to bracket bonding. The capsule containing Fuji Ortho LC was activated and mixed with an amalgamator

for 10 seconds. The brackets, with adhesive, were placed on the tooth surface and light cured for 10 seconds each at the occlusal, gingival, mesial and distal sides.

The procedure for Transbond XT included application of a self-etching primer on the labial surface for 3 seconds followed by a gentle burst of oil- and moisture-free air. The brackets, with adhesive, were placed on the labial surface and light cured for 10 seconds at a time from the mesial and distal sides. The light source came from a curing light XL 3000 (3M Dental Products, Model 5530-134). The original bulb was used with a minimum output light intensity of 580 mW/cm². Before light curing, the brackets were pressed on the tooth with the bracket base perpendicular to the long axis of the crown. Excess adhesive was removed with a sharp scaler.

The teeth were randomly allocated into four groups of 20 teeth as follows:

Group 1: Transbond XT debonded within 5 minutes of initial bonding.

Group 2: Fuji Ortho LC debonded within 5 minutes of initial bonding.

Group 3: Transbond XT debonded within 15 minutes of initial bonding.

Group 4: Fuji Ortho LC debonded within 15 minutes of initial bonding.

The teeth in groups 3 and 4 were kept wet to avoid dehydration during debonding.

The specimens were mounted in a special holding device, which was suspended in an Instron testing machine (Model 1121, Serial No. H1875, Instron Ltd, High Wycombe, Bucks, UK). An occlusal gingival load was applied to the bracket, producing a shear force at the bracket base. The shear load was applied with a crosshead speed of 1mm/minute and the force required to dislodge the bracket was measured in Newtons (N). The bond strength in MPa (N/mm²) was then calculated on the basis of a bracket area of 16.1 mm². The handling of the materials and debonding was performed at 22.8°C at 55 per cent humidity. The analyses were carried out according to standard procedures at NIOM – Scandinavian Institute of Dental Materials, Norway, and therefore no reproducibility test was performed.

The Adhesive Remnant Index (ARI) system was used to evaluate the amount of adhesive left on the labial surface after the brackets were dislodged from the prepared premolar (Oilver, 1988) using the Photomicroscope M-400 connected to a Wild Photoautomat MPS 45.

The criteria for evaluation were:

Score 1: All the adhesive remained on the tooth.

Score 2: More than 90 per cent of the adhesive remained on the tooth.

Score 3: More than 10 per cent but less than 90 per cent of the adhesive remained on the tooth.

Score 4: Less than 10 per cent of the adhesive remained on the tooth.

Score 5: No adhesive remained on the tooth.

Statistical analysis

Descriptive statistics including the mean, standard deviation and ranges were calculated for each of the four groups tested.

Data on fracture strength can be summarized by the Weibull (1951) distribution for the probability of fracture (p_F):

$$p_F = 1 - \exp\{-(\sigma/S_0)^m\}$$

where S_0 represents the load for which 63 per cent (i.e. $1 - p_F = \exp\{-1\}$) of a sample survival analysis. The Weibull modulus, m , is a measure of the range of loads causing fracture.

The Weibull distribution has no predictive power but can be employed to interpolate between measured data, e.g. to obtain survival probabilities justified by the number of samples tested. Estimation of the load for 10 per cent probability of failure, for example, requires a minimum of 15 specimens to be tested (ISO/TS 11405:2003).

Other statistics are available for comparing cumulative distributions without computing Weibull parameters. The Kolmogorov–Smirnov (K–S) statistic enables a straightforward test based on the largest difference between distributions represented as cumulative probabilities (as in Figure 1). The significance to be attributed to the

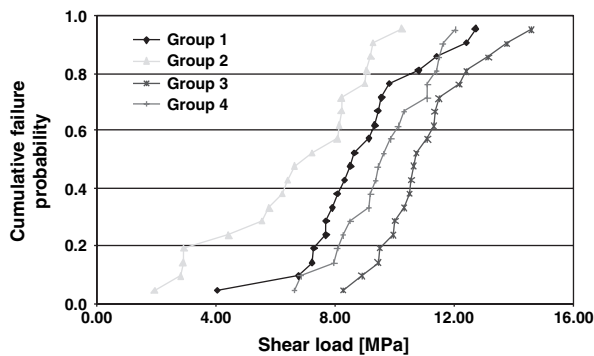


Figure 1 Cumulative failure probabilities versus shear load for Transbond XT and Fuji Ortho LC at 5 and 15 minutes. Group 1: Transbond XT debonded within 5 minutes of initial bonding. Group 2: Fuji Ortho LC debonded within 5 minutes of initial bonding. Group 3: Transbond XT debonded within 15 minutes of initial bonding. Group 4: Fuji Ortho LC debonded within 15 minutes of initial bonding.

Table 1 Descriptive statistics of the shear bond strengths for the four different groups.

Test group	Sample size	Mean (MPa)	SD (MPa)	Range	m	S_0 (MPa)	P_{10} (MPa)
1. Transbond XT (5 minutes)	20	8.8	2.0	4.0–12.7	5.1	9.5	6.1
2. Fuji Ortho LC (5 minutes)	20	6.6	2.5	1.9–10.2	2.5	7.7	3.1
3. Transbond XT (15 minutes)	20	11.0	1.6	8.3–14.6	7.2	11.4	8.3
4. Fuji Ortho LC (15 minutes)	20	9.6	1.6	6.6–12.0	5.8	10.4	7.1

SD, standard deviation.

Weibull parameters m and S_0 were obtained from cumulative failure probabilities by unweighted least-squares fitting to the data shown in Figure 1. Loads for 10 per cent probability of failure are estimated by $P_{10} = S_0 [0.1054]^{1/m}$.

maximum absolute difference in probability D between the distributions is given by:

$$P(D > \text{observed}) = Q_{KS}(D\sqrt{N})$$

where N , represents the number of measurements to be compared with a parameterized distribution such as the Weibull, or $N = N_1 N_2 / (N_1 + N_2)$ where N_1 and N_2 are the numbers of measurements in each of two measured distributions. The function $Q_{KS}(\lambda)$ is evaluated numerically (Press *et al.*, 1986) by summing terms until the sum

$$Q_{KS}(\lambda) = 2 \sum_{j=1}^M (-1)^{j-1} \exp\{-2 \cdot j^2 \lambda^2\}$$

does not change. The value of $P(D > \text{observed})$ is then the significance level at which one can reject the null hypothesis that the two distributions are from the same population.

A chi-square test of the ARI scores was used to assess the degree of association between the different groups.

Results

Shear bond strength

The descriptive statistics for the SBS are presented in Table 1. Applying the K–S statistic to the cumulative distributions confirms the increase in SBS with setting time, apparent from the mean values of both products, at the 100 per cent and 98 per cent levels for Transbond XT and Fuji Ortho LC, respectively. Applying the K–S statistic to a comparison of the products shows them to differ at a 95 per cent significance level after 5 minutes, and an 89 per cent level after 15 minutes setting time. Bonding with Transbond XT showed a higher mean SBS than Fuji Ortho LC after both setting times.

Previous experience (McCabe and Carrick, 1986; Fox *et al.*, 1991; Durning *et al.*, 1994; Mitchell *et al.*, 1995; Czochrowska *et al.*, 1999) suggested that the data might be distributed according to the Weibull distribution. The K–S statistic indicates that the Weibull distributions with the parameters in Table 1 do not differ significantly from the measured data. This provides the basis for interpolating between the data to estimate the 10 per cent probabilities of failure quoted in Table 1.

Bond failure site

Table 2 shows the fracture site at debond assessed according to the ARI system. Chi-square testing revealed an association between the type of bonding material and the setting time, but no association was apparent between the types of adhesive system.

Figure 2 shows both the tooth surface and bracket base of the specimens in the two different adhesive groups.

Discussion

A review of the literature identified a large variety of methods used to measure bond strength of orthodontic attachments. Therefore in the present study, the suggestions made by Fox *et al.* (1994) for *in vitro* bond strength testing in orthodontics were followed.

Table 2 Frequency distribution of Adhesive Remnant Index (ARI) score.

Group tested (<i>n</i> = 20)	ARI score				
	1	2	3	4	5
1	0	1	13	4	2
2	0	1	1	15	3
3	1	0	16	3	0
4	0	0	0	15	5

In the oral cavity, bonded brackets are subject to either shear, tensile or torsion forces, or a combination of these. These forces are difficult to measure and hence to quantify. According to Newman (1965) and Wheeler and Ackerman (1983) orthodontic forces do not generally surpass 4.45 N per tooth. In another study by Reynolds and von Fraunhofer (1976), a minimum bond strength of 5.9 to 7.8 MPa was found adequate for most clinical orthodontic needs. Furthermore, the SBS recommended for successful clinical bonding was estimated to be 7 MPa by Lopez (1980). The maximum load per tooth occurring in a clinical situation is probably 17.8 N (Wheeler and Ackerman, 1983). On the other hand, several other studies have suggested maximum bond forces of 35.6 N (Keizer *et al.*, 1976) and 97.88 N (Maijer and Smith, 1979). This wide range of values is probably the result of variations in testing devices. There are no specific *in vitro* or *in vivo* tests that can be valid for all of the various clinical applications of adhesive materials.

The present findings indicate that both Fuji Ortho LC and Transbond XT in combination with a self-etching primer are adequate adhesives for bonding of orthodontic brackets. In the initial five minutes, the mean SBS of Fuji Ortho LC was 6.6 ± 2.5 MPa, which is adequate according Reynolds and von Fraunhofer (1976), but slightly below the bond strength recommended by Lopez (1980). It does, however, support the findings of a clinical study by Silverman *et al.* (1995).

On the other hand, the present findings regarding the SBS of Fuji Ortho LC disagree with those of Bishara *et al.*

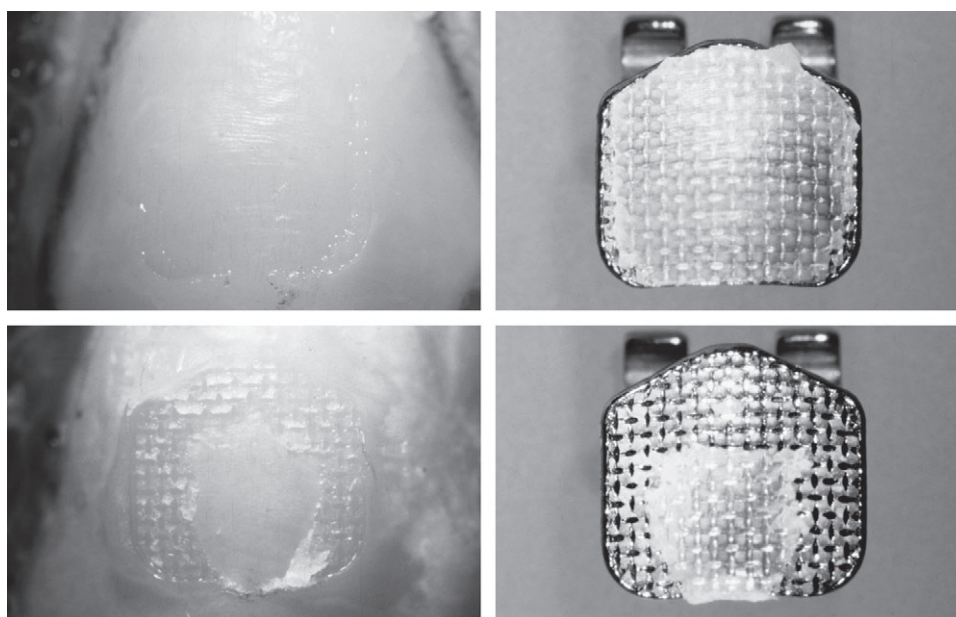


Figure 2 Upper left and right: buccal tooth surface and bracket base after debonding bracket bonded with Fuji Ortho LC. Note: most of the bonding material on bracket base. Lower left and right: buccal tooth surface and bracket base after debonding bracket bonded with Transbond XT. Note: a significant amount of the bonding material on the tooth surface.

(1999b). They found the SBS of Fuji Ortho LC to be 0.4 ± 1.0 MPa within 30 minutes of initial bonding, compared with 6.6 ± 2.5 MPa (5 minutes) and 9.6 ± 1.5 MPa (15 minutes) in the present investigation. One possible explanation could be that the enamel surfaces were not sufficiently moist. An overly dry or desiccated enamel surface will adversely affect the bond strength. The mean SBS of Fuji Ortho LC was 9.6 ± 1.6 MPa within 15 minutes. Transbond in combination with self-etching primer had a SBS of 8.8 ± 2 MPa within 5 minutes and 11 ± 1.6 MPa within 15 minutes. These values were above those recommended by Lopez (1980).

The use of self-etching primers simplifies the clinical handling of adhesive systems by simultaneously combining the conditioning and priming steps (Chigira *et al.*, 1989; Nishida *et al.*, 1993; Nakabayashi, 1991). Asgari *et al.* (2002) concluded, in a clinical study, that self-etching primers such as Transbond Plus can result in fewer bond failures compared with the traditional acid-etch technique, when used for orthodontic bracket bonding. In addition, self-etching primers may save time because they combine the etching and primer steps and eliminate the need for rinsing. Arnold *et al.* (2002) found that a stainless steel bracket could be bonded to enamel with a new self-etching primer (Transbond Plus self-etching primer) as an alternative to separate etching and priming. A delay of up to 10 minutes after priming and before bonding appears to have no significant effect on SBS.

Recently, Buyukyilmaz *et al.* (2003) concluded, in an *in vitro* study, that conditioning with Transbond Plus self-etching primer before bonding orthodontic brackets with Transbond XT to the enamel surface resulted in sufficient bond strength to be recommended for clinical use. They found a higher bond strength (16 ± 4.5 MPa) compared with the present study. This difference may be due to different debonding times.

The SBS of Transbond XT with Transbond Plus self-etching primer was higher in the present study compared with Bishara *et al.* (1999a) of 2.8 ± 1.9 MPa. The differences could be a result of the fact that they used a different acidic primer (Clearfil Liner Bond 2). However, Bishara *et al.* (1998), indicated that the use of acidic primers to bond orthodontic brackets to enamel surfaces can provide a clinically acceptable SBS (10.4 ± 4.4 MPa) when used with a highly (77 per cent) filled adhesive (Panavia 21). This was also confirmed recently by Bishara *et al.* (2004) comparing two self-etching primers: one a no-mix and the other a two component system.

In the composite group (Transbond XT), bond failure was equally present on the tooth surface and bracket. In the GIC group (Fuji Ortho LC) bond failure was mainly at the enamel–adhesive interface (Table 2, Figure 2). This results in easier clean-up after debonding with the Fuji Ortho LC compared with the Transbond XT system.

Conclusion

The results of the present investigation indicate that both Transbond XT with Transbond Plus etching primer and Fuji Ortho LC reached adequate bond strength within 5 and 15 minutes of initial bonding. After debonding, the Transbond system was likely to leave adhesive on at least 10 per cent of the bonded area of the tooth. However, as with any *in vitro* study, caution must be exercised when attempting to extrapolate the results to the clinical setting.

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