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Resin-modified glass ionomer, modified composite or conventional glass ionomer for band cementation?—an *in vitro* evaluation

D. T. Millett*, A. Cummings**, S. Letters*, E. Roger* and J. Love***
*Orthodontic Unit and **Dental Materials Science, Glasgow Dental Hospital and School, *** Robertson Centre for Biostatistics, University of Glasgow, UK

SUMMARY The aims of this study were to compare the mean shear-peel bond strength and predominant site of bond failure of micro-etched orthodontic bands cemented with resin-modified glass ionomer cement (RMGIC; Fuji Ortho LC or 3M Multi-Cure), a modified composite or a conventional GIC. The survival time of bands was also assessed following simulated mechanical stress in a ball mill.

One hundred and twenty molar bands were cemented to extracted human third molars. Eighty bands (20 cemented with each cement) were used to assess the debonding force and 40 bands (10 cemented with each cement) were used to determine survival time. The specimens were prepared in accordance with the manufacturers' instructions for each cement. After storage in a humidor at 37°C for 24 hours, the shear debonding force was assessed for each specimen using a Nene M3000 testing machine with a crosshead speed of 1 mm/minute. The predominant site of band failure was recorded visually for all specimens as either at the band/cement or cement/enamel interface. Survival time was assessed following application of mechanical stress in a ball mill.

There was no significant difference in mean shear-peel bond strength between the cement groups ($P = 0.816$). The proportion of specimens failing at each interface differed significantly between cement groups ($P < 0.001$). The predominant site of bond failure for bands cemented with the RMGIC (Fuji Ortho LC) or the modified composite was at the enamel/cement interface, whereas bands cemented with 3M Multi-Cure failed predominantly at the cement/band interface. Conventional GIC specimens failed mostly at the enamel/cement interface. The mean survival time of bands cemented with either of the RMGICs or with the modified composite was significantly longer than for those cemented with the conventional GIC. The findings indicate that although there appears to be equivalence in the mean shear-peel bond strength of the band cements assessed, the fatigue properties of the conventional GIC when subjected to simulated mechanical stress seem inferior to those of the other cements for band cementation.

Introduction

While bonded attachments are now used routinely as part of fixed appliance treatment, bands are still widely favoured for molar teeth (Gottlieb *et al.*, 1986). Close adaptation of the band to the enamel surface confers mechanical retention which is aided by the cement lute (Williams *et al.*, 1965). In the past two decades, glass ionomer cements (GICs) have become popular for band cementation. These adhere to enamel and metal (Hotz *et al.*, 1977), release and uptake fluoride (Creanor *et al.*, 1994) and inhibit microbial activity (De Schepper *et al.*, 1989). They are, however, susceptible to moisture contamination during the setting reaction and maximum strength is only attained after 24 hours (Wilson *et al.*, 1979).

Resin addition to the cement formulation has facilitated light curing, allowing a snap-set and rapid strength development (Mount, 1994). One-paste, two-paste or encapsulated formulations are currently available, affording a more reproducible cement mix as powder–liquid proportioning is eliminated. Due to the variation in chemical composition and setting reaction which exists among these resin–ionomer hybrids, products have been categorized as resin-modified glass ionomers or modified composites (McCabe, 1998). Resin-modified GICs (RMGICs) are a hybrid of their resin composite and glass ionomer parent groups. These cements are often marketed in capsular form and setting includes an acid–base reaction. Modified composites, however, are fundamentally resin-matrix composites with an ion-leachable glass replacing the filler. Setting is via free radical polymerization of methacrylate groups which is often light activated; an acid–base reaction is not involved (Nicholson, 1998). These newer cements have demonstrated significantly greater bond strengths, *in vitro*, relative to their predecessors (Millett *et al.*, 1998;

Mennemeyer *et al.*, 1999; Aggarwal *et al.*, 2000) particularly when used with micro-etched bands (Mennemeyer *et al.*, 1999; Aggarwal *et al.*, 2000).

Two of the newer RMGICs (Fuji Ortho LC, GC America Inc., Chicago, IL, USA, and 3M Multi-Cure, 3M Monrovia, CA, USA) have each been compared, *in vitro*, with modified composites for band cementation (Aggarwal *et al.*, 2000). No laboratory study, however, appears to have compared these RMGICs with each other for this purpose. Furthermore, no *in vitro* study has compared Fuji Ortho LC with the conventional GIC (Ketac-Cem, Espe, Seefeld Oberbay, Germany) for band cementation. As currently there are no orthodontic clinical trials reported which have compared these three band cements, laboratory studies should attempt to predict their possible clinical performance by subjecting banded specimens to simulated mechanical stress.

The aims of this study were to compare the mean shear-peel bond strength and predominant site of bond failure of micro-etched orthodontic bands cemented with a RMGIC (Fuji Ortho LC or 3M Multi-Cure), a modified composite (Ultra Band-Lok, Reliance Orthodontic Products Inc., Itasca, IL, USA) or a conventional GIC (Ketac-Cem). The survival time of bands was also assessed following simulated mechanical stress in a ball mill.

Materials and methods

Eighty caries-free extracted human third molars were collected and stored in distilled water in a refrigerator following decontamination in 0.5 per cent chloramine. The teeth were randomly divided into four groups of 20 teeth, each group comprising 10 maxillary and 10 mandibular third molars. Each tooth was then notched in the apical third with a diamond bur before being mounted to below the amelocementum junction in a block of self-curing acrylic resin with the long axis vertical.

The teeth were then cleaned with a pumice slurry, rinsed in distilled water and dried thoroughly in a stream of air. As bands do not exist for third molars, first permanent molar bands with micro-etched fitting surfaces (3M Unitek) were used. A band was selected and adapted optimally to the crown of each tooth. Twenty bands were cemented with each of the four cements. Summary characteristics of each cement are given in Table 1. Fuji Ortho LC is an encapsulated RMGIC containing a powder and liquid. Following trituration for 10 seconds, the capsule is placed in a loading gun from which the cement is dispensed to the band fitting surface. A tri-cure reaction is responsible for setting the cement. This comprises an acid–base reaction of the glass ionomer components, a free radical addition polymerization reaction initiated by visible blue light and finally self-curing of the resin ionomer.

Table 1 Summary characteristics of each cement.

Brand name	Cement type	Curing mechanism
Fuji Ortho LC	RMGIC	Tri-cured (acid–base reaction, light cure and self-cure)
3M Multi-Cure	RMGIC	Tri-cured
Ultra Band-Lok	Modified composite	Light cured
Ketac-Cem	Conventional GIC	Chemically cured

GIC, glass ionomer cement; RMGIC, resin-modified GIC

3M Multi-Cure is also an RMGIC but is supplied as separate powder and liquid components, rather than in an encapsulated form. Following mixing, an acid–base reaction is initiated but setting is completed by light polymerization and also by a self-curing mechanism. Ultra Band-Lok is a single-paste modified composite formed by combining a composite resin with glass ionomer particles. It is supplied in a sealed tube and hardens only through photopolymerization. Ketac-Cem is a powder–liquid-based conventional GIC which is chemically cured. It sets initially via an acid–base reaction followed later by a cross-linking reaction.

The manufacturers' instructions were followed in relation to each cement. To standardize specimen preparation, band selection and cementation were undertaken by one operator (DM). Two bands were cemented from each mix of Fuji Ortho LC, 3M Multi-Cure or Ketac-Cem while Ultra Band-Lok was applied directly to the fitting surface of each band. Following band placement, excess cement was removed with dry cotton wool rolls.

Fuji Ortho LC, 3M Multi-Cure and Ultra Band-Lok were light cured with a dental curing light (3M Unitek) for 20 seconds from the occlusal aspect of each band, in line with the manufacturers' guidelines. Ketac-Cem was allowed to bench cure for 5 minutes following band cementation. The specimens were then transferred to a humidor at 37°C for 24 hours prior to assessing the shear-peel debonding force using a Nene M3000 testing machine (Nene Instruments Ltd, Wellingborough, Northants, UK) with a crosshead speed of 1 mm/minute. The specimens were loaded into the jig by two 0.9 mm stainless steel loops which engaged fully under the buccal tube and the lingual cleat of each band. For each specimen, testing proceeded until the band was removed from the tooth. The maximum force (N) recorded during shear-peel debanding was recorded for each specimen and then converted to bond strength values (MPa) using band surface area data provided by the manufacturers. As the bands were micro-etched, a true measurement of surface area was not possible. The manufacturers' data, therefore, represent nominal rather than exact surface area measurements. The

Table 2 Bond strength values for 20 molar bands cemented with Fuji Ortho LC, 3M Multi-Cure, Ultra Band-Lok or Ketac-Cem.

Cement	Mean bond strength (MPa)	Standard deviation	Characteristic strength	Weibull modulus	Correlation coefficient
Fuji Ortho LC	1.54	0.33	1.67	4.34	0.993
3M Multi-Cure	1.63	0.41	1.82	3.75	0.994
Ultra Band-Lok	1.58	0.46	1.72	3.51	0.996
Ketac-Cem	1.65	0.37	1.80	4.94	0.996

predominant site of failure at band removal was assessed visually by one operator (DM) and classified as occurring at either the enamel/cement or cement/band interface.

To assess survival time, a further 40 extracted human third molars were collected and treated in a manner identical to that used for bond strength testing. They were divided into four groups, each with five maxillary and five mandibular molar teeth. The root surface of each tooth was grooved with a diamond bur in the mid-third to aid later identification. The bands were cemented with each cement following the same procedure as described previously. The specimens were then transferred to a humidor at 37°C for 24 hours prior to being subjected to mechanical stress in a ball mill. This contained 470 g of ceramic spheres and 250 ml of distilled water at 37°C. Operating at 100 revolutions/minute, the contents of the mill were assessed on an hourly basis. Specimens with loose bands were deemed to have failed and were removed. A fresh sample of distilled water at 37°C was then placed in the ball mill and testing recommenced. This process continued until all specimens had failed.

After checking the validity of the necessary statistical assumptions (normality, and equality of standard deviations in the four groups), one-way analysis of variance (ANOVA) was used to compare mean shear-peel bond strengths of the evaluated cements. Weibull analysis (Weibull, 1951; McCabe and Carrick, 1986) was used to calculate the probability of failure (Pf) at given values of applied force. This form of analysis has been used previously to assess fracture processes in chemically and light-cured composites (McCabe and Carrick, 1986) and in the evaluation of the bond strength of bracket bonding agents (Fox *et al.*, 1991) and orthodontic band cements (Durning *et al.*, 1994; Millett *et al.*, 1995, 1998). Pf is related to applied stress (δ) as follows: $\mathrm{Pf} = 1 - \exp\{-(\delta - \delta u/\delta o)^m\}$, where δu and δo are constants. δu is the lowest level of stress at which Pf approaches zero and it is usually assumed that δu = 0. δo is the characteristic strength, replacing mean bond strength for this analysis. A Weibull modulus, *m*, is generated which indicates the dependability of the cement. Where *m* is high, close grouping of the bond strength data is demonstrated which indicates good bond reliability (Fox *et al.*, 1991). A comparison of the site of failure between cement groups was made using a χ^2 test. Due to some expected frequencies being small, all follow-up analyses were based on Fisher's exact test.

For the ball mill experiment, comparisons of survival time distributions of bands in each cement group were based on Kaplan–Meier estimates of the survivor functions and the log rank test. Follow-up analyses were adjusted for the number of comparisons made using Bonferroni correction.

Results

Shear-peel bond strength data for each band group are given in Table 2. There were no statistically significant differences between the groups (P = 0.816). Weibull data are also shown in Figure 1. The Weibull moduli varied from 3.51 for Ultra Band-Lok to 4.94 for Ketac-Cem, indicating a slightly greater bond reliability with the latter cement.

There were significant differences between the four cements with regard to the proportion of specimens failing at each of the sites recorded ($P < 0.001$; Table 3). The patterns of site of failure differed between 3M

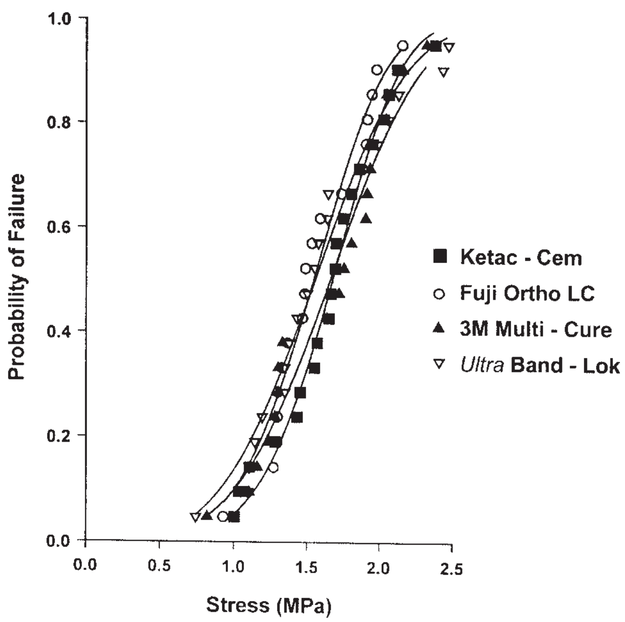

Figure 1 Weibull curves for 20 molar bands cemented with each cement.

Table 3 Predominant site of band failure for 20 molar bands cemented with each cement.

	Enamel/cement interface	Cement/band interface
Fuji Ortho LC	20	0
3M Multi-Cure	8	12
Ultra Band-Lok	20	0
Ketac-Cem	15	5

Multi-Cure and Fuji Ortho LC specimens ($P < 0.001$) and also between 3M Multi-Cure and Ultra Band-Lok specimens ($P < 0.001$). 3M Multi-Cure specimens failed predominantly at the cement/band interface, whereas Ketac-Cem specimens failed mostly at the enamel/cement interface. The predominant site of failure of bands cemented with Fuji Ortho LC or Ultra Band Lok was at the enamel/cement interface.

The survival time distribution for bands cemented with each cement is shown in Figure 2. The mean survival time of bands cemented with Fuji Ortho LC (11.2 hours), 3M Multi-Cure (8.8 hours) or Ultra Band-Lok (10.8 hours) was significantly longer than for those cemented with Ketac-Cem (3.4 hours).

Discussion

The mean shear-peel bond strength of micro-etched orthodontic bands cemented with RMGIC, modified composite or conventional GIC has been assessed. Micro-etched bands were chosen as these exhibit superior bond strength and a five- to eight-fold lower clinical failure rate compared with untreated bands (Millett *et al.*, 1995; Mennemeyer *et al.*, 1999; Hodges *et al.*, 2001).

The regime used for the storage of the extracted teeth and the sample size of each cement group comply with recommendations for studies on bond strength testing (Fox *et al.*, 1994). In relation to the evaluation of orthodontic band cements, rather than bracket bonding agents, a similar sample size, tooth type and specimen storage protocol to those in the present study have been used in previous investigations (Millett *et al.*, 1995, 1998; Aggarwal *et al.*, 2000).

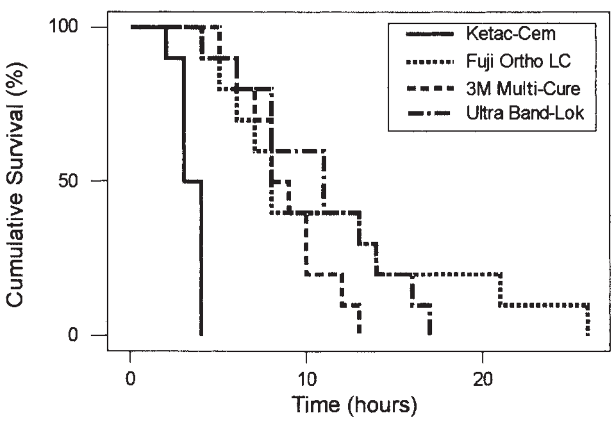

Figure 2 Survival time distribution for 10 molar bands cemented with each cement.

The cements evaluated in this study have not been compared previously, but similar products have been assessed (Mennemeyer *et al.*, 1999; Aggarwal *et al.*, 2000). The study by Mennemeyer *et al.* (1999) incorporated three of the products used in the present investigation, namely Fuji Ortho LC, Ultra Band-Lok and Ketac-Cem. In that study, 10 specimens of each cement were placed in 3 mm diameter moulds at the bonding interface and bonded to 6×6 mm stainless steel band specimens mounted to acrylic blocks. The tensile, rather than shear, debonding force was then recorded. Due to the different bonding substrate and testing mode employed in that study versus those used in the present investigation, it would be invalid to draw comparisons between the results obtained for either investigation.

In the present study, the mean shear-peel bond strength did not differ significantly between cement groups. Aggarwal *et al.* (2000) also found no significant differences in shear-peel bond strength of bands cemented with RMGIC (3M Multi-Cure or Optiband) or modified composite (Ultra Band-Lok) confirming the findings of the present study. Bond strength values also did not differ significantly between the two RMGICs assessed. This lends support to the findings for the products tested from this cement group. The present results show that the conventional GIC demonstrated a comparable shear-peel bond strength to the other cements assessed. It appears that no previous laboratory study has compared the newer cements with a conventional GIC for band cementation using banded molar teeth. Aggarwal *et al.* (2000) made a comparison with zinc phosphate cement, rather than a conventional GIC, and found bands cemented with zinc phosphate had a significantly lower shear-peel bond strength.

Weibull analysis, applied to bond strength data, allows information in relation to bond reliability to be ascertained. The Weibull modulus, an indicator of bond reliability, was greatest with the conventional GIC and least with the modified composite. For the RMGICs, Fuji Ortho LC exhibited a higher Weibull modulus than 3M Multi-Cure.

The predominant mode of bond failure was evaluated by only one assessor, who was familiar with the appearance of the various cement types. It is acknowledged that this may have introduced some bias, and ideally at least two assessors blind to the cement types should have been used for this purpose.

Bond failure for bands cemented with Fuji Ortho LC or Ultra Band-Lok occurred predominantly at the enamel/cement interface, whereas failure at both the cement/band and enamel/cement interfaces was observed for 3M Multi-Cure and Ketac-Cem specimens. With the exception of bands cemented with 3M Multi-Cure, the predominant site of bond failure for all specimens was at the enamel/cement interface, a finding reported previously for micro-etched bands (Millett *et al.*, 1995). Although cement remnant scores following debanding were not recorded, minimal cement remained on the enamel for Fuji Ortho LC and Ultra Band-Lok specimens. This would imply that the clean-up time following debanding may be faster clinically where bands are cemented with these luting agents than with the other cements. In this investigation, the enamel surface was dried prior to band cementation to mirror customary clinical practice. The results, however, with respect to the site of bond failure may have been different had the enamel remained moist during the banding procedure, which may unintentionally occur in the clinical situation.

Previous studies have subjected banded specimens to either thermocycling (Norris *et al.*, 1986; Aggarwal *et al.*, 2000) or mechanical insult in a ball mill (Durning *et al.*, 1994; Millett *et al.*, 1995, 1998) in an attempt to simulate the environmental insults likely to be encountered clinically. Ten banded specimens for each cement type were used for this purpose, as this sample size has been shown to be adequate to detect statistically significant differences between samples of this type (Millett *et al.*, 1995, 1998). The survival time of bands cemented with either of the RMGICs or with the modified composite in the present study tended to be longer than for those cemented with the conventional GIC. The findings with regard to the modified composite and conventional GIC reflect those of a previous study (Millett *et al.*, 1998) where untreated bands were cemented with the modified composite Band-Lok and the conventional GIC, Ketac-Cem.

Although bands cemented with Ketac-Cem had the highest mean shear-peel bond strength and the greatest Weibull modulus, specimens cemented with this cement had the shortest mean survival time in the ball mill experiment. This apparent conflict between the outcome of bond strength tests and that exhibited following simulated mechanical stress in a ball mill has been noted previously for bands cemented with either GIC or zinc phosphate cement (Durning *et al.*, 1994). It is interesting to observe that for the RMGICs and the modified composite, the results of the two experiments (shear-peel bond strength testing and survival time) appear to lend support to each other. In contrast, the findings with respect to the conventional GIC indicate a difference in response to each mode of testing. This requires further investigation. The ball mill has been shown to be a useful predictor of clinical performance for banded specimens (Millett *et al.*, 1995). Based on that, one would expect the clinical failure rate of bands cemented with either of the RMGICs or the modified composite used in this study to be similar. Randomized clinical trials comparing these products are required to assess this further.

Conclusions

There was no significant difference in the mean shear-peel bond strength of micro-etched molar bands cemented with any of the cements assessed. The predominant site of band failure for bands cemented with Fuji Ortho LC or Ultra Band-Lok was at the enamel/cement interface whereas bands cemented with 3M Multi-Cure failed predominantly at the cement/band interface. Ketac-Cem specimens failed mostly at the enamel/cement interface. Survival times of bands cemented with either of the RMGICs or with the modified composite were significantly longer than for bands cemented with the conventional GIC.

Address for correspondence

D. T. Millett
Unit of Orthodontics
Glasgow Dental Hospital and School
378 Sauchiehall Street
Glasgow G2 3JZ
UK

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