

An *in vitro* investigation into the use of resin-modified glass poly(alkenoate) cements as orthodontic bonding agents

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SUMMARY This *in vitro* study was designed to determine the effect of time on the measured mean force to debond when brackets were bonded using resin-modified glass poly(alkenoate) cements and to compare them with a light-cured diacrylate. Changes in surface topography and composition of the cements were also investigated. Stainless steel orthodontic brackets were bonded to 160 upper premolar teeth in four test groups: Transbond, Fuji Ortho LC, and 3M Multi-Cure with and without enamel etching. Shear bond testing to failure was performed after 1 hour, 1 week, 1 month, and 1 year. The first three groups were then rebonded and stored for the same time periods before being shear tested again. Debond force was recorded in Newtons and the locus of bond failure was scored using the Adhesive Remnant Index (ARI). Surface topography and composition of the test materials were also studied at time periods of 1 day, and 1, 6, and 18 months, using scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX).

The mean force to debond (N) was observed to increase with time in all four test groups, with there being little significant difference between the groups. When the same brackets were rebonded, the mean force to debond reduced. Surface topography and compositional changes over time were only observed with the resin-modified glass poly(alkenoate) cements. Resin-modified glass poly(alkenoate) cements have a mean force to debond comparable with diacrylate bonding agents. However, unlike diacrylates they undergo surface changes with time, the significance of which is unknown.

Introduction

The latest materials developed for bonding of orthodontic brackets to the teeth are glass ionomer or, more correctly, glass poly(alkenoate) cements. Recently, resin-modified glass poly(alkenoate) cements have been introduced, which include the addition of hydroxyethyl methacrylate (HEMA). Glass poly(alkenoate) cements can adhere to base metal alloys, as well as to unetched enamel (Hotz *et al.*, 1977), making them attractive for use in orthodontic bonding. Resin-modified glass poly(alkenoate) cements such

as Fuji Ortho LC and 3M Multi-Cure have been specifically developed for such use. Poly(acrylic acid), which is less acidic than *o*-phosphoric acid, is supplied with some resin-modified glass poly(alkenoate) cements for enamel conditioning prior to bonding. However, whether any enamel pretreatment is required prior to their use is still unclear, since successful bonding in a wet environment without acid etching has been reported by Silverman *et al.* (1995) with only a 3.2 per cent bond failure rate over an 8-month period *in vivo*. Bonding without prior acid etching certainly reduces enamel damage and would be useful in

cases where etching is likely to be less efficient, e.g. fluorosis or amelogenesis imperfecta.

Fluoride has been shown to be released from glass poly(alkenoate) cement after initial curing and the set material is also known to absorb exogenous fluoride from saliva, fluoridated toothpaste, and topical fluoride gel (Ashcraft *et al.*, 1997). In this way, it acts as a rechargeable, slow-release fluoride device into the adjacent enamel (Hatibovic-Kofman and Koch, 1991). Decalcification under bands cemented with glass poly(alkenoate) cements is reported to be considerably less than when zinc phosphate cement is used (Copenhover, 1986). Similarly, no decalcification was seen on any teeth over the 8-month research period of Silverman *et al.* (1995), when resin-modified glass poly(alkenoate) cement was used for direct bonding. Some studies even verify the potential for glass poly(alkenoate) cements to initiate remineralisation of existing carious lesions (Donly *et al.*, 1995).

In vitro studies on glass poly(alkenoate) cements for direct bonding have generally found them to have a significantly lower shear bond strength than conventional composite resin based bonding agents (Fajen *et al.*, 1990; Øen *et al.*, 1991). In one study, Klockowski *et al.* (1989) found the shear bond strengths of glass poly(alkenoate) cement to be only just over one-third that of the composite resins under test. However, the *in vitro* tensile and shear bond strengths of the resin-modified glass poly(alkenoate) cement Fuji Ortho, has been reported to be greater than the conventional glass poly(alkenoate) Ketac-Cem, but still less than that of a no-mix autopolymerizing composite bonding resin (Rely-a-Bond; Komori and Ishikawa, 1997). In fact, in only one study (Jobalia *et al.*, 1997) was the visible light-cured glass poly(alkenoate) cement, Fuji Ortho LC found to approach the bond strength observed with diacrylate resins such as Rely-a-Bond and Phase II.

However, caution should be exercised when transferring such laboratory findings to clinical practice (Zachrisson, 1994). Lower bond strength does not necessarily imply increased bond failure rate *in vivo*. It is more appropriate to analyse such laboratory data using a survival analysis (Altman, 1991).

Another stated advantage of glass poly(alkenoate) cement use in orthodontic bonding is easier enamel clean-up at the end of treatment when compared with conventional composite resin bonding agents (Norevall *et al.*, 1996).

The aims of this current investigation were to compare two resin-modified glass poly(alkenoate) cements and a light-cured diacrylate as orthodontic bonding agents, and in particular to determine:

1. the effect of time on both the mean force to debond and locus of bond failure;
2. the effectiveness of the resin-modified materials for rebonding orthodontic brackets;
3. the effect of time on the surface topography and surface composition of the light-cured diacrylate Transbond, and the two resin-modified glass poly(alkenoate) cements, Fuji Ortho LC and 3M Multi-Cure, using scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX).

Materials and methods

One-hundred-and-sixty upper premolar teeth, extracted for orthodontic reasons, were used in this experiment. They were stored in a 70 per cent alcohol solution following extraction, and prior to use were immersed in water at room temperature for at least 1 day. This was carried out to reduce the effects of any dehydration as a result of the alcohol storage medium. Vacuum-formed polythene moulds were made using a Druformat thermopressure machine (Dreve, Germany) over a plaster of Paris block measuring 15 × 15 × 35 mm. Using these moulds, each premolar tooth was embedded in self-curing acrylic so that only its buccal surface was exposed. This exposed surface was then polished using a slow speed handpiece and a slurry of pumice and water in a rubber cup. The 160 teeth in their acrylic blocks were then subdivided into four groups of 40 teeth. The enamel in each group was then further prepared according to the material to be used to bond upper premolar orthodontic brackets (3M Unitek Dyna-Lock, Monrovia, USA) to the teeth. The enamel preparation and materials were as follows.

Group 1

The enamel was etched with 37 per cent *o*-phosphoric for 30 seconds followed by rinsing with copious amounts of water and then dried using oil-free compressed air, until frosty white in appearance. This etched surface was then primed using Transbond XT primer (3M Unitek™, Monrovia, USA). Filled Transbond resin, a light-cured diacrylate, was then placed onto the bracket base before placement on the tooth. Firm pressure was applied to the bracket using a Mitchell's trimmer and excess adhesive removed from around the margins using a probe. The adhesive was then light-cured for 10 seconds mesially and distally (20 seconds per tooth), as recommended by the manufacturers. The halogen light-curing unit (Ortholux™ XT curing lamp, 3M, St Paul, USA) produced a filtered blue light with a wavelength of 400–500nm and at an energy level of approximately 300 mW. The efficiency of the lamp was intermittently tested using the meter within the unit.

Group 2

The enamel was conditioned using GC Ortho Conditioner (GC Corporation, Tokyo, Japan) for 10–20 seconds, followed by washing with copious amounts of water. The enamel was then left moist. Fuji Ortho LC (GC Corporation), mixed according to the manufacturers' instructions, was then placed onto the bracket and the bracket positioned onto the tooth, as with group 1. Light curing was performed for 20 seconds mesially and distally (40 seconds per tooth) as recommended by the manufacturers.

Group 3

The enamel was etched for 30 seconds using 37 per cent *o*-phosphoric acid as recommended by the manufacturers. Following rinsing, the etched enamel was again left moist. 3M Multi-Cure was mixed according to the manufacturers' instructions, the brackets were bonded to the teeth, and any excess material removed as described previously. Each tooth was light-cured for 20 seconds mesially and distally (40 seconds per tooth).

Group 4

Brackets were again bonded to the teeth with 3M Multi-Cure as in group 3, but the enamel was not etched prior to use, and was left moist after pumicing and rinsing.

Following bracket placement, the specimens were allowed to bench cure for a further 10 minutes before being placed in tap water in a water bath at 37°C until bond testing. The water in the containers was changed weekly.

Shear bond testing to failure was performed using a custom-made testing jig (Figure 1) in a Lloyd Universal testing machine (Series 2000R, Lloyd Instruments, Southampton, UK) and with a crosshead speed of 2 mm/minute. Bond testing was performed after storage in the water bath at one of four time periods: 1 hour, 1 week, 1 month, and 1 year. Force to debond (N) and

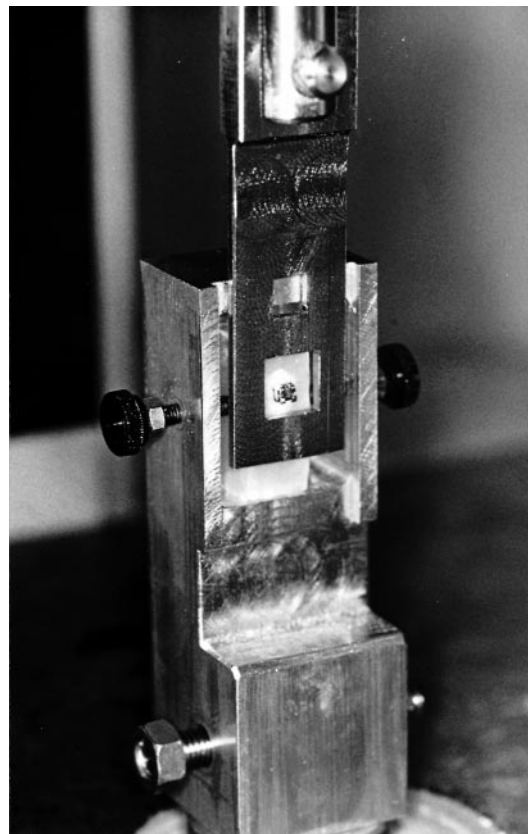


Figure 1 Custom made testing jig.

locus of bond failure (ARI score) were recorded for each case.

Brackets from the first three time periods were then rebonded using the original bonding agents and for the same time periods before again being shear tested to failure. The enamel in each case had any excess adhesive removed using a spiral fluted tungsten carbide bur in a slow speed handpiece. It was then re-treated as originally described for each of the test groups. Once the brackets had been rebonded they were then allowed to cure in the water bath over the same rebonding time intervals, namely 1 hour, 1 week, and 1 month before bond testing to failure, in order to determine the effectiveness of each of the materials for rebonding. Excess adhesive on the bracket base was removed using a green stone in a slow speed handpiece until the metal of the base was exposed as advocated by Wright and Powers (1985).

Determination of the locus of bond failure provides information on the adhesive bond as well as the cohesive strength of the resin, tooth and bracket (Gwinnett, 1982). This was scored using the ARI system to evaluate the amount of adhesive left on the tooth at debond (Årtun and Bergland, 1984):

Score 0 = No adhesive left on the tooth.

Score 1 = Less than half of the adhesive left on the tooth.

Score 2 = More than half of the adhesive left on the tooth.

Score 3 = All adhesive left on the tooth, with a distinct impression of the bracket mesh.

In order to study the effect of time on surface topography and composition, eight blocks, 5 mm thick and 10 mm in diameter, were made from each of the three materials, Transbond, Fuji Ortho LC and 3M Multi-Cure. They were fabricated in a plastic dappens pot to ensure that each had a smooth round base. After light-curing, the prepared blocks were left on the bench for 10 minutes before being placed in a water bath at 37°C for one of four time periods: 1 day, and 1, 6, and 18 months. Two specimens of

each material were stored for each of these time periods and the water was changed weekly. At the end of each time the specimens were carbon- or gold-coated before being examined using SEM (Hitachi S-520, Hitachi, Japan). This permitted a qualitative investigation of possible changes in surface topography. (EDAX;) Kevex Delta, Kevex Corp, San Carlos, USA was also performed in order to investigate compositional changes at the surface of each material with time.

Results

Data analysis was performed using Stata Version 5.0 (Stata Corporation, College Station, Texas, USA) and StatXact version 3.0 (Cytel Corporation, USA). The data were tested for normality using the Shapiro-Francia statistic (W') and were found to be normally distributed. Table 1 illustrates the mean and standard deviation for each of the test groups when initially tested to bond failure over the four time periods. For each material the mean force to debond (N) increased with time. Consideration of the survival plots and log rank tests (Figures 2–5) shows there to be little difference between the materials and the different enamel surface pre-treatments over the longer test periods of 1 month and 1 year. However, at 1 hour and 1 week there was a significant difference between 3M Multi-Cure where prior acid etching had not been performed compared with the other three test groups. With the rebond specimens the mean force to debond was much lower than with the initial specimens for each of the four test groups. Whilst mean force to debond generally increased with time, for Fuji Ortho LC it appeared to decrease over time.

The *in vitro* ARI scores were analysed using the Kruskal–Wallis non-parametric one-way analysis of variance (Table 2) with the null hypothesis of no difference in the ARI scores between the groups. The ARI scores between each of the four test groups were noted to be different when used for initial bonds, but not when used to rebond brackets.

Table 1 Summary statistics for the test groups.

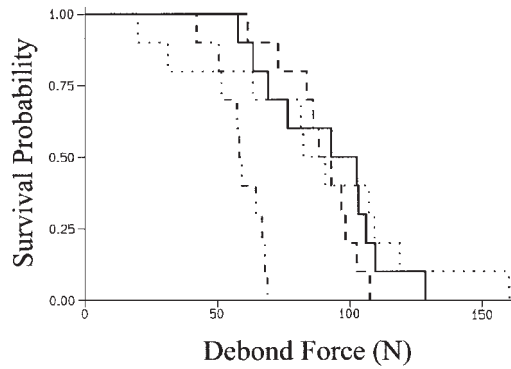
Material/time/bond	Observations	Mean	SD	W*	Pr > z
Fuji/1 hour	10	91.14	23.08	0.951	0.619
Fuji/1 hour rebond	10	72.06	14.12	0.789	0.013
3M No etch/1 hour	10	58.75	8.58	0.939	0.492
3M No etch/1 hour rebond	10	60.53	11.97	0.957	0.694
3M etch/1 hour	10	86.59	41.48	0.967	0.816
3M etch/1 hour rebond	10	55.49	21.96	0.963	0.771
Transbond/1 hour	10	89.21	13.86	0.953	0.653
Transbond/1 hour rebond	9	68.63	12.51	0.801	0.023
Fuji/1 week	10	100.07	18.76	0.949	0.605
Fuji/1 week rebond	10	71.76	17.88	0.938	0.482
3M No etch/1 week	10	91.99	14.77	0.919	0.308
3M No etch/1 week rebond	9	87.00	24.41	0.955	0.698
3M etch/1 week	10	119.79	15.06	0.961	0.745
3M etch/1 week rebond	10	65.96	17.72	0.763	0.007
Transbond/1 week	10	130.39	22.17	0.892	0.161
Transbond/1 week rebond	10	93.07	44.68	0.775	0.010
Fuji/1 month	10	121.37	34.12	0.934	0.438
Fuji/1 month rebond	10	54.32	17.33	0.935	0.444
3M No etch/1 month	10	115.43	48.19	0.904	0.213
3M No etch/1 month rebond	10	68.04	25.06	0.866	0.104
3M etch/1 month	10	129.46	60.38	0.643	0.000
3M etch/1 month rebond	10	73.12	15.60	0.914	0.277
Transbond/1 month	10	129.93	26.08	0.883	0.129
Transbond/1 month rebond	10	91.15	40.64	0.806	0.020
Fuji/1 year	10	143.66	48.38	0.877	0.111
3M No etch/1 year	10	114.45	36.41	0.926	0.361
3M etch/1 year	10	162.64	72.19	0.712	0.002
Transbond/1 year	10	128.97	50.09	0.660	0.000

Discussion

Considering each of the materials and their enamel surface pre-treatment, it can be seen that mean force to debond continues to increase with time in all cases (Table 1). With the light-cured diacrylate, Transbond, this is presumably a continuation of the free radical addition polymerisation process initiated following abstraction of electrons from the camphorquinone in the material during light curing. For the resin-modified glass poly(alkenoate) cements this increase in mean force to debond maybe due to the same process occurring within the resin component of the material. Alternatively, it may be a continuation of the acid base reaction between the glass and poly acid. In the case of 3M Multi-Cure, a dark cure reaction may also occur due to micro-encapsulated potassium persulphate and ascorbic acid reacting to produce free radicals also capable of initiating the

polymerisation of the resin. Comparison of the mean force to debond (Table 1) and Kaplan–Meier survival probability and log rank tests (Figures 2–5) shows that at each time interval there was little difference between any of the materials and their surface pre-treatments from the 1 week test period onwards. At 1 hour, the force to debond of 3M Multi-Cure without prior acid etching of the enamel was lower than the other three groups. This may be because the material in this instance does not rely on mechanical adhesion to the enamel surface and largely on chemical adhesion or the physical forces of adhesion, such as hydrogen bonding and van der Waals forces. Why there should be little difference between the various materials and their pre-treatments over the longer test periods of 1 week, 1 month, and 1 year is unclear.

With the rebond specimens there would seem to be little difference between the Transbond and 3M Multi-Cure with and without prior enamel







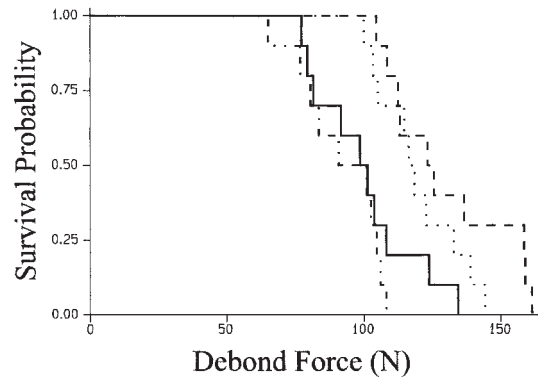
1 Hour	Material	Log rank	Pr> X^2
	Fuji Ortho	13.21	0.01
	Transbond	10.24	
	3M Etch	13.83	
	3M No Etch	2.72	

Figure 2 Kaplan–Meier survival probabilities and log rank test for the four test materials at 1 hour.







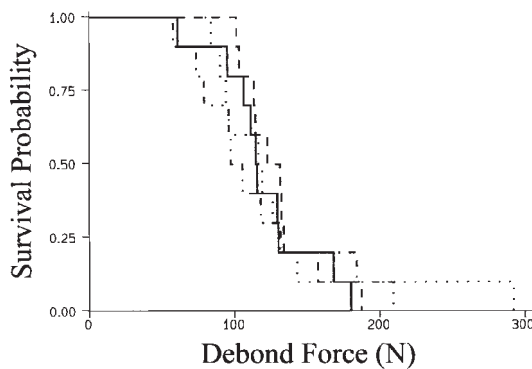
1 Week	Material	Log rank	Pr> X^2
	Fuji Ortho	5.81	0.01
	Transbond	18.48	
	3M Etch	12.27	
	3M No Etch	3.44	

Figure 3 Kaplan–Meier survival probabilities and log rank test for the four test materials at 1 week.







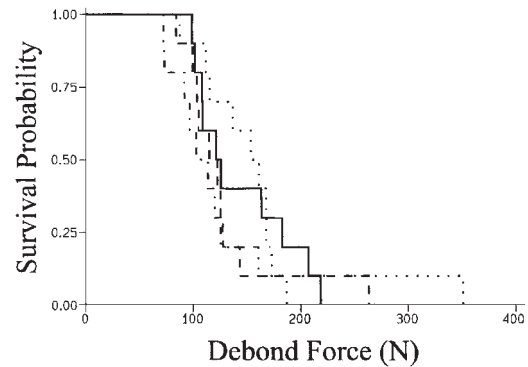
1 Month	Material	Log rank	Pr> X^2
	Fuji Ortho	8.85	0.91
	Transbond	11.70	
	3M Etch	10.42	
	3M No Etch	9.03	

Figure 4 Kaplan–Meier survival probabilities and log rank test for the four test materials at 1 month.







1 Year	Material	Log rank	Pr> X^2
	Fuji Ortho	11.50	0.22
	Transbond	8.36	
	3M Etch	14.06	
	3M No Etch	6.08	

Figure 5 Kaplan–Meier survival probabilities and log rank test for the four test materials at 1 year.

etching over all three test periods (Table 1). With Fuji Ortho LC however, the mean force to debond at 1 hour was higher than in the other three groups, where this then fell markedly between 1 hour and 1 week, with a lesser fall between 1 week and 1 month. In all cases the rebond mean force to debond was lower than the initial bonds, except for Fuji Ortho LC at 1 hour.

Analysis of the ARI scores using the Kruskal–Wallis one-way analysis of variance (Table 2) shows that within each time period the ARI scores were different between the groups at each of the four test periods. With Fuji Ortho LC and 3M Multi-Cure with etch, there was a mixed mode of failure, but predominantly involving the enamel surface, whilst with Transbond and 3M Multi-Cure with etch, there was also mixed mode, but predominantly at the bracket adhesive interface. With the rebonds the locus of bond failure was mainly at the bracket adhesive

interface in all cases. This is probably related to the method of reparation of the bracket base, namely the use of a green stone in a slow speed handpiece. Wright and Powers (1985) found this method of bracket recycling to produce bond strengths comparable with commercially recycled brackets, but with a wider scatter in the results. In this current investigation, commercially recycled brackets were not used, but the shift in the locus of bond failure towards the bracket adhesive interface would indicate that the bond between the recycled brackets and each of the adhesives was less efficient than when new brackets were used. It is not known whether the same effect would have been observed if new brackets had been bonded to the previously bonded teeth.

SEM and EDAX of the Transbond specimens shows the surface of the material to be relatively unchanged with time (Figures 6–9). Both Fuji Ortho LC and 3M Multi-Cure on the other hand demonstrated surface changes with time,

Table 2 Kruskal–Wallis non-parametric one-way analysis of variance of the *in vitro* Adhesive Remnant Index (ARI) scores.

Material/bond	Time	Observations	Observed statistic	$P > X^2$
Fuji	1 hour	10	19.94	0.01
Transbond	1 hour	10		
3M No etch	1 hour	10		
3M etch	1 hour	10		
Fuji	1 week	10	12.09	0.01
Transbond	1 week	10		
3M No etch	1 week	10		
3M etch	1 week	10		
Fuji	1 month	10	12.17	0.01
Transbond	1 month	10		
3M No etch	1 month	10		
3M etch	1 month	10		
Fuji	1 year	10	14.40	0.01
Transbond	1 year	10		
3M No etch	1 year	10		
3M etch	1 year	10		
Fuji rebond	1 hour	10	2.71	0.44
Transbond rebond	1 hour	9		
3M No etch rebond	1 hour	10		
3M etch rebond	1 hour	10		
Fuji rebond	1 week	10	6.05	0.05
Transbond rebond	1 week	10		
3M No etch rebond	1 week	10		
3M etch rebond	1 week	9		
Fuji rebond	1 month	10	2.35	0.50
Transbond rebond	1 month	10		
3M No etch rebond	1 month	10		
3M etch rebond	1 month	10		

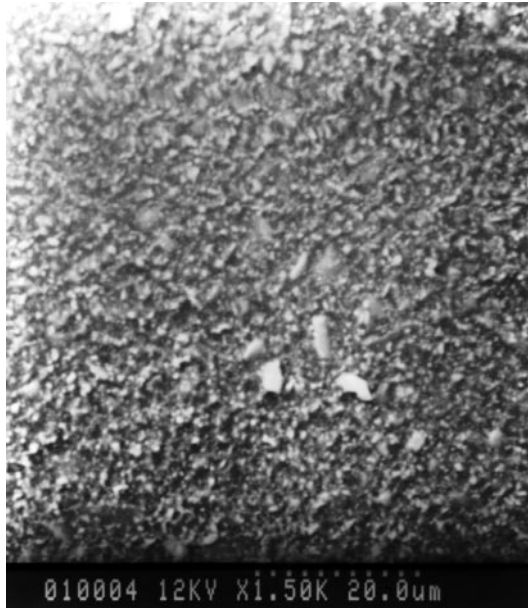


Figure 6 SEM ($\times 1.5k$) of Transbond at 1 day.

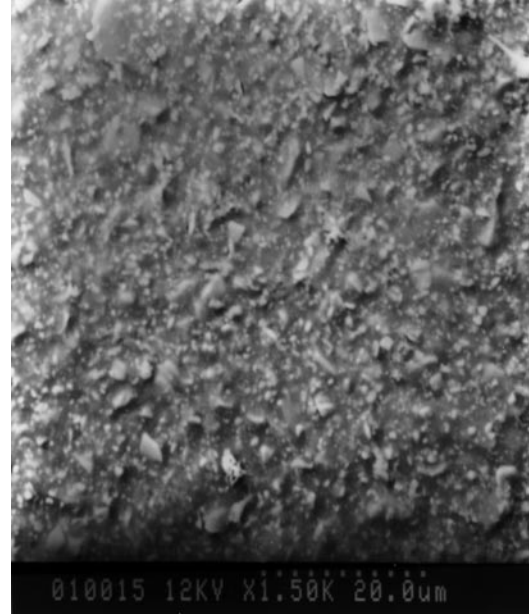


Figure 7 SEM ($\times 1.5k$) of Transbond at 1.5 years.

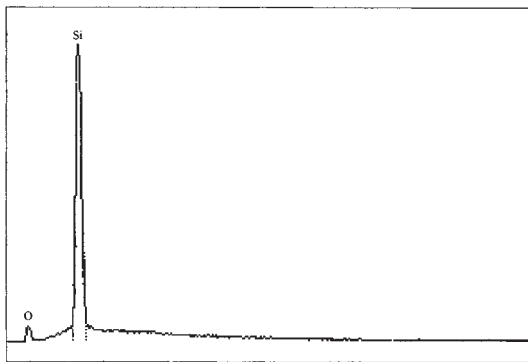


Figure 8 EDAX of Transbond at 1 day.

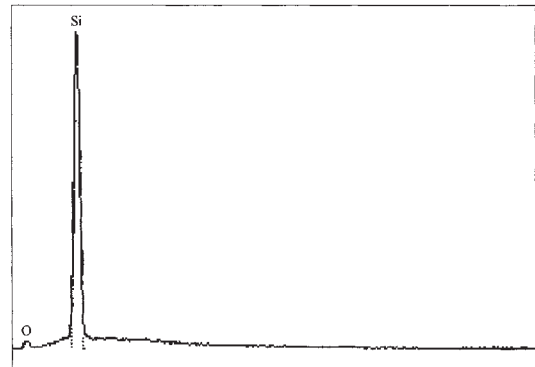


Figure 9 EDAX of Transbond at 18 months.

as illustrated by the 3M Multi-Cure specimens (Figures 10–13). EDAX for both the resin-modified glass poly(alkenoate) cements shows that the ratio of calcium, phosphorus, and copper in each case increases from 1 day to 18 months. This may be due to deposition onto the surface from the surrounding water, which had been changed at weekly intervals throughout the test period. Silicon levels on the other hand,

diminished with time. Why this occurs and how it might influence measured force to debond is unclear, but it may be due to loss of siliceous gel or silica particles embedded within the siliceous matrix of the cured resin-modified glass poly(alkenoate) cement. In orthodontic bonding the material is only exposed at the margins of the joint and so this effect may not be of any consequence.

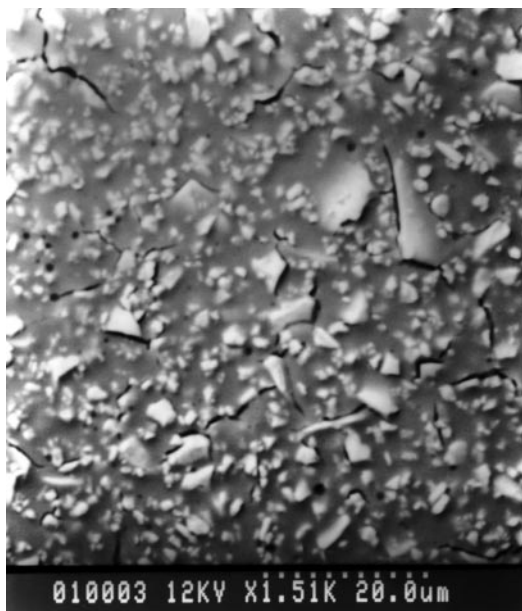


Figure 10 SEM ($\times 1.5k$) of 3M Multi-Cure at 1 day.

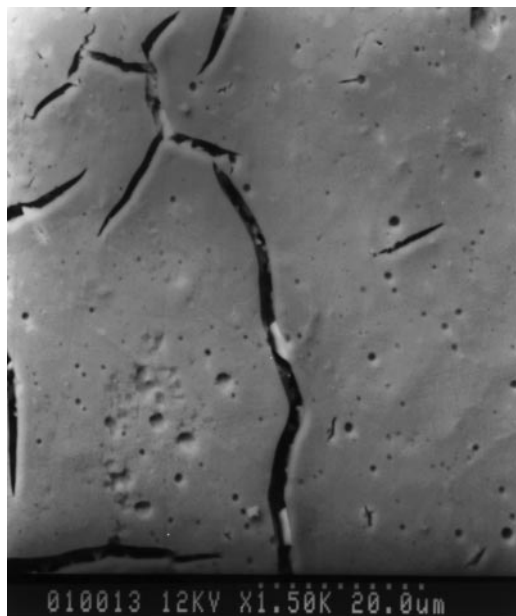


Figure 11 SEM ($\times 1.5k$) of 3M Multi-Cure at 1.5 years.

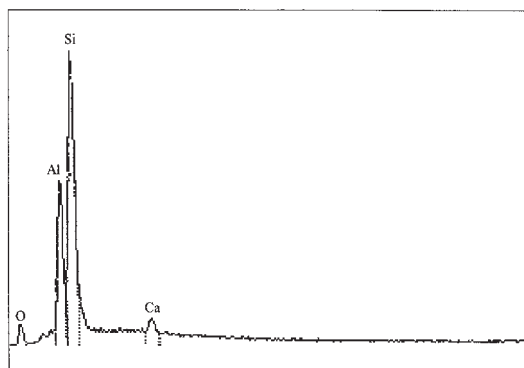


Figure 12 EDAX of 3M Multi-Cure at 1 day.

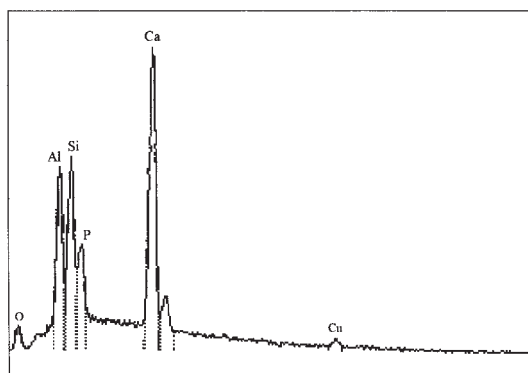


Figure 13 EDAX of 3M Multi-Cure at 18 months.

Conclusions

The following conclusions can be drawn from this investigation:

1. The *in vitro* mean force to debond (N) increased with time for all the tested materials.
2. Although there were differences in the measured *in vitro* mean force to debond (N) between the materials at 1 hour and 1 week,
3. there was little difference at 1 month and 1 year.
3. The locus of bond failure, as measured by the ARI scores, for the initial bonds was mixed mode in all cases, but for Fuji Ortho LC and 3M Multi-Cure without etch it predominantly involved the enamel surface. With Transbond and 3M Multi-Cure with etch it was predominantly at the bracket adhesive interface.

4. For the *in vitro* rebonds the locus of bond failure was, in all cases, predominantly at the bracket adhesive interface.
5. With Fuji Ortho LC the mean force to debond (N) for the rebonds had a tendency to fall with time, unlike the other materials under test.
6. Whilst Transbond appeared not to undergo surface changes with time when studied using SEM and EDAX over an 18-month time period, Fuji Ortho LC and 3M Multi-Cure underwent marked changes.

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Acknowledgements

The authors wish to thank Professor Alan Harrison, Bristol Dental School, for permission to use the Lloyd instrument, Mr Ken Brady of Electron Microscopy GKT, Guy's Campus for his help with the SEM and EDAX, and 3M Unitek and GC Corp. for providing the materials.

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