Use of glass-ionomers for bracket bonding—an *ex vivo* study evaluating a testing device for *in vivo* purposes

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SUMMARY Seven glass-ionomer cements were tested and compared with a composite resin in order to find the glass-ionomer cement with the highest bond strength to enamel with normal anatomy and composition (not ground). Five types of surface treatment were used. The results show that Aqua Cem® and Ketac Cem®, both water-hardening, present the highest bond strength, followed by the conventional glass-ionomer cement Fuji IIF®. Surface treatment according to the manufacturers' instructions and surface treatment with polyacrylic acid were found to give rise to the highest bond strength for all of the seven cements. However, none of the glass-ionomer cements reached the values for the composite resin Concise®. The testing instrument designed for *ex vivo* purposes measured with high precision and accuracy and with a low methodological error.

Introduction

The problem of caries lesions during and after orthodontic treatment is well known (Mitchell, 1992). In the absence of fluoride the caries process underneath an orthodontic band is fast and caries can develop cavities within 4 weeks (Øgaard *et al.*, 1988). The frequency of mutans streptococci has also been reported to increase during orthodontic treatment (Svanberg *et al.*, 1984; Rosenbloom and Tinanoff, 1991) and is hard to control if patients do not fully co-operate with oral hygiene (Lundström and Krasse, 1987).

Composite resin, frequently used for the fixation of fixed orthodontic appliances, has been reported to increase the accumulation of plaque (Skjörland, 1973), as well as the proportion of *mutans streptococci* in plaque (Skjörland and Sønju, 1982; Svanberg *et al.*, 1984, 1990). Glass ionomer cements (GICs), on the other hand, will reduce or prevent decalcification of dental enamel (Valk and Davidson, 1987) and also give rise to an increase of fluoride uptake in plaque (Hallgren *et al.*, 1993). Svanberg *et al.* (1990) reported significantly lower viable counts of mutans streptococci on GIC than on amalgam,

while Hallgren *et al.* (1993) compared frequency of counts of mutans streptococci on composite and GIC (Aqua-Cem), with the significantly lowest counts for the GIC.

In general, GIC has been recommended for caries risk patients (Norevall *et al.*, 1990), but it should also be possible to use in several groups of handicapped patients where the caries situation is difficult to control, but where the need for orthodontic treatment is high.

The use of GIC for cementation of bands in orthodontic therapy has shown 75 per cent fewer band failures than zinc-phosphate cement (White, 1986). The use of GIC for bonding of brackets, however, is significantly weaker than with composite (Cook and Youngson, 1988; Fajen et al., 1990; Rezk-Lega et al., 1991). Pretreatment with pumice polishing creates the strongest bond (Cook and Youngson, 1988; Fajen et al., 1990). Fischer-Brandies et al. (1992), in a preliminary report, found approximately 30 per cent higher bond strength using benzoic acid. Powis et al. (1982) compared polyacrylic acid (PAA) with citric acid, and found a more etched surface with citric acid and lower bond strength, but still a significantly higher bond strength than on the untreated control. Kullman (1986), on the other

hand, found no increase in bond strength after etching the enamel surface.

So far, different opinions have been presented concerning the possibility of using GIC for bracket bonding as well as to how the enamel surface should be treated prior to bonding. Evaluation of testing devices used in these studies, with respect to measurement accuracy and calibration, is seldom presented and is mostly restricted to information about the name of the device.

The purpose of the present study was to analyse a number of GICs and surface treatments in order to find materials with acceptable adhesive and cohesive properties, making their use possible for bracket bonding.

Furthermore, *in vitro* testing of bond strength has mostly been performed with very heavy devices. Therefore, the second purpose of this study was to design a device with a measurement reliability and accuracy comparable to the ones normally used, which is easily calibrated and can be used clinically for force registration.

Materials and methods

Bonding materials and test specimens

Seven different GICs and one composite resin were used (Table 1). The procedures recommended by the manufacturers were strictly followed. Human extracted premolars were used. Immediately after extraction they were placed in

0.9 per cent NaCl and then stored for 1–2 weeks at +8°C. Prior to bonding the teeth were allowed to normalize for 24 hours at room temperature and the enamel surfaces were examined with light microscopy (magnification: ×50) to exclude teeth with enamel fractures.

For the seven groups of GICs, 50 premolars were randomly used, and for the composite 10. All of the teeth were polished with pumice mixed with water, rinsed with water for 20 seconds, and dried with oil-free compressed air. The enamel surfaces were not ground.

Enamel treatment

Five different procedures were used for each of the seven GICs, including two acids as described in Table 2. Polyacrylic acid includes free groups of COOH⁻ which form covalent and hydrogen bonds to Ca²⁺ in the enamel and ensure good wetting of the enamel surface, while etching of the enamel creates a rough surface. The substances were applied with a small brush and after application, the enamel was rinsed with water for 30 seconds and dried with oil-free compressed air.

Bonding procedure

Stainless steel cylinders with an inner diameter of 3 mm were bonded to the surface of the extracted teeth (Figure 1). The flattest part of

 Table 1
 Materials used and number of test specimens: glass-ionomer cement.

Material	Manufacturer	Туре	Premolars $n = 360$	
Filling cement Fuji IIF®	GC Dental Industrial Corp, Tokyo, Japan	Conventional	50	
Ketac Fil®	Espe, GmbH, Seefeld, Germany	Water-hardening*	50	
Luting cement Fuji I [®]	GC Dental Industrial Corp, Tokyo, Japan	Conventional	50	
Ketac Cem®	Espe, GmbH, Seefeld, Germany	Water-hardening	50	
Aqua Cem®	Dentsply, De Trey Div, Weybridge, UK	Water-hardening	50	
Fascinate	Ortho Organizer, Calif, USA	Conventional	50	
Fissure blocking cement Fuji III®	GC Dental Industrial Corp, Tokyo, Japan	Conventional	50	
Composite resin Concise®	3M Dental Products, St Paul, Minn, USA	Two-paste mix	10	

^{*}Encapsulated.

 Table 2
 Enamel treatment.

Substances	Application time (seconds)		
Treated as recommended by manufacturer	_		
2. 25 per cent polyacrylic acid (PAA) (Dentin Conditioner			
GC Dental Industrial Corp, Tokyo, Japan)	30		
3. 37 per cent phosphoric acid	30		
4. As number 3 followed by PAA			
5. As number 3 followed by resin (Enamel Bond System,			
Dental Products, St Paul, Minn, USA) with a setting time of 5 minutes			

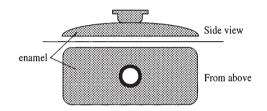


Figure 1 Steel test cylinder with 3-mm inner diameter with the attachment for the tensile bond testing instrument at the top.

the buccal surface of each tooth was chosen. The cylinders were prepared in 96 per cent ethanol in an ultrasonic bath for 10 minutes and then rinsed in distilled water. They were dried with oil-free compressed air. The GICs were mixed on a chilled glass plate, except for the encapsulated ones.

The cylinders were filled with the materials to be tested and excess material outside the cylinder mantle on the enamel surface was carefully removed with a scalpel before setting of the material. The GIC surfaces were covered with waterproof varnish. The cement was allowed to set for a period of 1 week at 37°C, in 100 per cent humidity before testing. The top of the test cylinders served as an attachment to the tensile bond instrument (Figure 2).

Testing device

The instrument [Portable Wheatstone Force Measurement (PWFM) device] (Figure 2) was constructed with four ohmic and inductive transducers in a Wheatstone full-bridge (C), connected to a carrier wave measuring bridge (Philips, PR 9307, Tarasconw. 2, NL-5627 GB, Eindhoven,

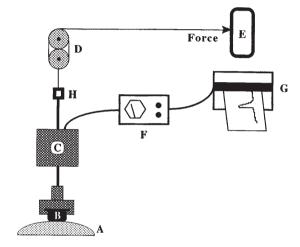


Figure 2 The design of the test device (PWFM device). A = enamel; B = test cylinder; C = Wheatstone-bridge; D = tackle; E = handpiece; for *in vivo* use or crosshead mechanics for *in vitro* use; F = bridge amplifier; G = chart recorder; H = vertical cord. The chart recorder can, in clinical use, be replaced by a voltmeter.

Holland) (F), which produced a bridge voltage, proportional to the load, which was registered on a chart recorder (G). The tensile bond testing instrument was calibrated over an interval of 1–15 MPa before testing and between every third measurement. The Wheatstone bridge was constructed for weights in the range of 1–500 N. The instrument was made portable, but still with high precision and accuracy. The device was constructed in four separate parts to ensure that all parts were optimal for the purpose and easily transportable.

For the debonding procedure, a vertical cord (H) attached to the PWFM instrument indicated

an exact direction perpendicular to the enamel surface and parallel with the cylinder axis. The force was distributed with a wire from the handpiece over a double tackle (D) situated exactly above the test cylinder. The dislodging force was applied with a speed of 10 mm/minute.

After debonding, the surfaces of the tooth were observed using a light microscope (magnification ×50) in order to determine if the fracture occurred within the cement (cohesive) or at the enamel-cement interface (adhesive). No fractures occurred between the inner mantle surfaces of the test cylinder and the different materials.

Statistical analysis and error of the method

The statistical methods comprised a one- and two-way analysis of variance with interaction with the Neuman–Keul test for multiple comparisons.

The reliability of the device was assessed by determinations of five, to the operator unknown, weights in the range 1–15 MPa three consecutive times. The standard deviation was 0.4 per cent with weight independence.

Results

The different surface treatments were pooled for each material used and the results are presented in Table 3. The tensile bond strength for Concise® had the highest value and Fascinate the lowest. The others are in descending order. None of the GICs reached the level of tensile

Table 3 Tensile bond strength (MPa) of glassionomer cements to enamel. The different surface treatments are pooled for each material used. Materials connected with a vertical bar are not significantly different at the 5 per cent level. The value for Concise® was 7.75 MPa.

Bonding materials	Number of specimens	Mean		
Aqua Cem® Fuji IIF®	50 50	4.42 4.00		
Ketac Cem®	50	3.36		
Fuji III®	50	3.09		
Fuji I®	50	2.83		
Ketac Fil®	50	2.71		
Fascinate	50	2.06		
Fascinate	50	2.		

Table 4 Tensile bond strength (MPa) of glass-ionomer cement to enamel. The different glass-ionomer cements are pooled for each surface treatment given in Table 2. The mean value for the composite resin is not included as the enamel was treated according to the manufacturers' instructions. Surface treatments connected with a vertical bar are not significantly different at the 5 per cent level.

3.91
3.90
3.03
2.90
2.57

bond strength (7.75 MPa) for the composite resin (Concise[®]). Aqua Cem[®] was the GIC with the highest values, reaching 60 per cent of the strength of Concise[®].

With the GICs pooled for each surface treatment, the debonding force is presented in Table 4. The highest bond strength (3.83 MPa) was observed when following the manufacturers' recommendation (only pumice), as well as with application of PAA (3.83 MPa).

The value for difference between methods was P = 0.0001, between materials P = 0.0001, and for the interaction between methods and materials P = 0.0329.

The mean values for the different materials, treated according to the manufacturers' instructions in relation to frequency of cohesive fractures, are presented in Table 5. When more than two-thirds of the enamel surface at the site for cementation was covered by GIC, the fractures were designated as cohesive. For the other surface treatments the frequency of adhesive fractures was generally higher. The bond strength for Aqua Cem® and Ketac Cem® reached almost 70 per cent of the strength of Concise®. The results also indicate that the adhesion of the materials is good, while the cohesive properties of the materials are responsible for the weaker bond strength than for Concise®.

In Table 6 a two-way analysis shows the complete relationship between methods and materials with *P*-values.

Table 5	Tensile	bond	strength	(MPa)	of	GIC.	Two-way	table	of	means	comparison	between	materials
and meth							•				•		

Methods	Manufacturer (1)	25% PAA (2)	37% Phosphorus (3)	37% Phosphorus + 25% PAA (4)	37% Phosphorus + resin	Significance <i>P</i> value (4)
Materials						
Aqua Cem®	5.34 ± 1.972	5.15 ± 1.981	4.34 ± 2.306	3.53 + 0.984	3.76 + 1.232	P = 0.0571
Fuji IIF®	4.60 + 1.457	4.44 + 1.091	3.72 + 1.200	3.59 + 1.002	3.65 + 1.179	P = 0.1891
Ketac Cem®	5.03 + 1.784	3.93 + 1.300	2.74 + 0.809	3.07 + 1.596	2.06 + 1.053	P = 0.0002
Fuji III®	2.44 + 1.310	3.92 + 1.333	3.21 + 1.670	2.98 + 0.906	2.90 + 0.957	P = 0.1428
Fuji I®	3.00 + 1.056	3.55 + 1.029	2.18 + 0.937	3.22 + 1.124	2.19 + 1.221	P = 0.0183
Ketac Fil®	4.32 + 2.415	3.39 + 1.660	2.28 + 1.506	2.07 + 0.970	1.50 + 1.046	P = 0.0021
Fascinate	2.08 + 0.989	2.41 + 1.069	1.56 + 0.975	2.20 + 1.285	2.03 + 1.055	P = 0.4989

Table 6 Tensile bond strength (MPa) of GIC and frequency of cohesive fractures to enamel. Only values for surface treatment according to manufacturers' instructions are included.

Material	Surface treatment	Number of specimens	No. of specimens with cohesive fracture	Mean
Aqua Cem®	1	10	10	5.34
Aqua Cem [®] Ketac Cem [®]	1	10	10	5.03
Fuji IIF®	1	10	9	4.60
Ketac Fil®	1	10	7	4.32
Fuji I®	1	10	10	2.99
Fuji III®	1	10	10	2.44
Fascinate	1	10	6	2.08

Discussion

The present study has shown that all the GIC materials have a bond strength inferior to composite resin, but that a water-hardening GIC can be used in orthodontic bonding procedures. Furthermore, a force measurement device arrangement such as the Portable Wheatstone Force Measurement device (PWFM device) is suitable for studies with special interest in gaining increased knowledge of orthodontic forces.

The present investigation was performed in order to find materials with acceptable adhesive and cohesive properties for direct bonding of orthodontic brackets on enamel. This was achieved by analysing a variety of GICs used for cementing and restorative dentistry under conditions recommended by their manufacturers (Fox *et al.*, 1994). The testing technique used in this study has earlier been presented by Jemt

et al. (1986) who reported good accuracy with the technique. They used a technique suitable for in vivo testing which has been further developed in this study under the name PWFM device. Rezk-Lega et al. (1991) also decided to use tensile bond strength testing as it is more exactly definable than shear/peel forces. The error of the method in the present study indicates a high precision in relation to the force applied. In comparison with other types of devices, for example the series of Instron® devices, the PWFM device was found to be easy to handle and had a high precision and accuracy. Due to its small size it can be used for different kinds of force registration in vitro, as well as in vivo. Registrations of loads more than 500 N and cyclic loading can not, however, be performed. In studies using, for example, the Instron® device, calibration is almost never described and very seldom the error of the method. The PWFM device can be calibrated by the user continuously, which increases the reliability in relation to the normal heavy duty devices and at a much lower cost

This indicates that the method used, as well as the testing device, in this study seem to produce results with sufficient accuracy. The choice of testing method therefore was regarded as adequate as the question of the choice of bracket base (Cook and Youngson, 1988) and cement thickness was eliminated using the cylinder system, earlier described by Jemt *et al.* (1986).

The bond strengths of the tested materials in the present study were somewhat higher than Jemt *et al.* (1986), which can probably be explained by their *in vivo* environment. Aboush and Jenkins (1986) reported higher values when using a completely flat enamel surface, while Kullman (1986), after only slight enamel grinding, reported values which correspond well with the results in this study. However, the tensile forces and to some extent the torsional forces obtained by Fajen *et al.* (1990) using brackets cemented with Fuji I[®] and Ketac Cem[®], to a very high extent resemble the present values.

As a consequence of correct prebonding treatment procedures (e.g. post-extraction storage), undamaged extracted teeth were used and grinding of the enamel surfaces avoided. Several earlier studies comparing the adhesion of different GICs have used ground enamel surfaces which alter the enamel structure. The ion composition in the enamel surface (Chabala et al., 1988) also changes dramatically when the surface is ground. As the polyacrylic acid (PAA) has free COOH- groups it is essential that the ion composition in the surface is as realistic as possible. Powis et al. (1982) have reported that adhesion to an enamel surface with normal anatomical roughness will be diminished, while Aboush and Jenkins (1986) found that bond strength is reduced by increasing surface roughness. Therefore, it was interesting to use a PAA which improves the adhesion of the material, but allows the effect of the PAA on an unground surface to be studied.

The surface treatments compared in Table 4 indicate that an enamel surface treated as recommended by the manufacturer or a surface treated

with PAA creates the highest bond strengths when the materials are pooled, which is in line with Cook and Youngson (1988), and Fajen *et al.* (1990). The increased bond strength when using PAA was less than expected from the results of Powis *et al.* (1982). This could be due to the difference in structure, as they used ground enamel. The enamel structure therefore seems to be less important. In the present study it was also evident that PAA created a bond strength equal only to pumice.

Etching of the enamel did not increase the bond strength, which was found by Kullman (1986), and Cook and Youngson (1988), though in a different experimental set up, and the combination etching-PAA also did not improve the adhesion.

The use of a resin was chosen as GIC is often used as a cavity liner underneath composite resins. However, no studies have explained any possible bonding mechanism when GIC is used on top of a resin. It was still included to clearly verify that the use of resin as enamel treatment before the use of a GIC may reduce the bond strength, as it is often used in the opposite order or as a mixture in materials such as Fuji Ortho LC® (Silverman *et al.*, 1995).

The results are presented in a two-way table of means (Table 5). The GICs with the strongest bond strength were approximately 30 per cent weaker than the composite resin. The results in this study for 'the best' GIC in relation to a composite resin are more favourable than described by Evans and Oliver (1991). They reported that their best GIC (KetacFilencapsulated[®]) only reached 36 per cent of the strength of Concise®. One could expect that an alteration in base shape would break the GIC adhesion more easily and thereby create a weaker bond. In the present study this effect was completely eliminated. Based on the results and the number of specimens in this study, it is difficult to select one material having a significantly higher bond strength than others. However, it is possible to select two GICs which have a weaker bond strength than the others in this specific study.

The high frequency of cohesive fractures (Table 6), for the GICs with the strongest bonding

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strength, indicate that further attempts to increase the adhesion of GIC to enamel will not give rise to a significant increase in the bond strength. First, the cohesive properties need to be improved and second the adhesive which may be achieved by mixing with resin materials. The water-hardening materials were in general found to have higher bond strength than the conventional ones, Fuji IIF® being an exception. Evans and Oliver (1991) also stated that increasing the powder liquid ratio in general creates a higher bonding strength.

Setchell *et al.* (1985) compared the solubility of Ketac Cem® and Aqua Cem®, and found that Aqua Cem® was less soluble than Ketac Cem®. Prosser *et al.* (1984) compared Ketac Fil, Fuji IIF®, Ketac Cem®, Fuji I®, and Aqua Cem®, and found Aqua Cem® to be the most resistant GIC against early water contamination and concluded that the development of water-hardening cements has resulted in an improvement of the handling qualities. However, for orthodontic use, the degree of solubility probably plays a minor role as the appliance normally is used for less than 2–3 years. Ketac Cem® is more soluble than Aqua Cem® and, therefore, more fluoride will be released.

Conclusion

The choice of GIC for orthodontic use should be a water-hardening GIC and the use of different surface treatments can be restricted to the recommendations of the manufacturer. When an increased wetting or cleansing is desired, PAA can be used. It also seems that 4 MPa in this study corresponds to the threshold for clinical success.

This study has also indicated that a few types of GICs can be used not only for cementing, but also for other purposes.

Finally, the testing device used can be recommended for institutions or larger clinics, and especially for those with a research interest in orthodontic forces. It can also be used by postgraduate students, as well as for orthodontists, in order to test new materials from a more critical standpoint.

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