J Mater Sci: Mater Med (2007) 18:845–849
DOI 10.1007/s10856-006-0085-y

Shear bond strengths of glass-ionomer cements to sound and to prepared carious dentine

Beata Czarnecka · Patricia Deręgowska-Nosowicz · Honorata Limanowska-Shaw · John W. Nicholson

Received: 30 August 2005 / Accepted: 22 February 2006 / Published online: 4 January 2007

Summary The aim of this study was determine whether bonding of glass-ionomer cements to non-carious dentine differed from that to carious dentine. Five commercial cements were used, namely Fuji IX GP, Fuji IX capsulated, Fuji IX Fast capsulated (all GC, Japan), Ketac-Molar and Ketac-Molar Aplicap (both 3M-ESPE, Germany). Following conditioning of the substrate with 10% poly (acrylic acid) for 10 s, sets of 10 samples of the cements were bonded to prepared teeth that had been removed for orthodontic reasons. The teeth used had either sound dentine or sclerotic dentine. Shear bond strengths were determined following 24 h storage. For the auto-mixed cements, shear bond strength to sound dentine was found not to differ statistically from shear bond strength to sclerotic dentine whereas for hand-mixed cements, shear bond to sound dentine was found to be higher than to carious dentine (to at least $p < 0.05$). This shows that the chemical effects arising from interactions of glass-ionomer cements with the mineral phase of the tooth are the most important in developing strong bonds, at least in the shorter term.

B. Czarnecka · P. Deręgowska-Nosowicz ·
H. Limanowska-Shaw
Department of Biomaterials and Experimental Dentistry, Poznań University of Medical Sciences, Poznań, Poland

J. W. Nicholson (✉)
Biomaterials Group, School of Science, University of Greenwich, Chatham, Kent ME4 4TB, UK
e-mail: J.W.Nicholson@gre.ac.uk

1 Introduction

Glass-ionomer dental cements are aesthetic materials suitable for use as direct permanent fillings [1]. Their particular advantages are that they are naturally adhesive to dentine and enamel [2], have adequate strength, are biocompatible towards the tooth [3], and are able to release fluoride in a sustained way for several years [4]. Recent improvements have led to materials with sufficient strength to resist biting forces in class I and class II cavities in both deciduous and permanent teeth [5, 6]. They are the materials of choice for use with the Atraumatic Restorative Treatment (ART) technique [7] and for use in class V cavities [1].

Over the years, many studies have been carried out to demonstrate that glass-ionomers bond naturally to the tooth surface [1, 8]. Bond strengths are lower than for composite resins bonded with appropriate bonding agents, but the durability of the bond appears greater [9]. This may be attributed to the bioactive nature of the interface between the cement and the tooth [1, 10], which leads over time to a strong, durable bond formed by an ion-diffusion process [11]. Laboratory studies typically use sound teeth in bonding studies, and this may give results that are misleading.

Under clinical conditions, the most frequent reason for repairing a tooth with glass-ionomer is that it has been damaged by caries. When a tooth has been subject to caries, it undergoes changes that manifest themselves as morphological and biochemical changes within the dentine [12, 13]. These changes may permeate the dentine well beyond the region that can be diagnosed with a dental probe, and hence even after removal of the most severely damaged region of the dentine, the substrate left behind is different from the dentine in a non-carious tooth. This remaining dentine can be assumed to be sclerotic and hypermineralised [12], and is softer than normal dentine [14, 15]. The dentinal tubules are typically blocked with hydroxyapatite crystals originating from the peritubular dentine [16] and the walls of these tubules may still contain carious acids, mainly lactic but also acetic and propionic acids [17]. Glass-ionomer cements have been shown to be able to react rapidly with such acids in vitro [18] and to be capable of reducing the pH of thin films of such acids from approximately 4.5, corresponding to active caries, to pH 5.5, corresponding to arrested caries, in less than 30 s [19].

These differences in morphology and of biochemistry are likely to affect the bond strength of materials attached to these substrates. This has been confirmed for bonding systems for composite resins. For example, Yoshiyama et al. [16] showed that bond strengths of both a total-etch adhesive and an experimental self-etching adhesive were significantly higher on sound dentine compared to either caries-infected or caries-affected dentine, a finding which was attributed to the lower tensile strength of the caries-affected dentine. In addition, blocking of the tubules would have adversely affected resin tag formation, which would also contribute to the reduction in bond strength.

To date, however, the effect of bonding glass-ionomers to carious dentine has received less attention. One report has shown that microtensile bond strengths of conventional glass-ionomers were lower to caries-affected dentine than that of a resin-modified glass-ionomer [20]. However, no comparison was made with non-carious dentine.

The present study aims to rectify this omission. In aiming to determine whether bond strengths to previously carious dentine differ from those to non-carious dentine, we have determined shear bond strengths of a number of restorative grade glass-ionomer cements to both types of dentine and compared values. The results are important given the growing use of glass-ionomers clinically. They also give an insight into the interactions between glass-ionomers and the tooth surfaces they encounter under practical service conditions.

2 Materials and methods

Fifty human 3rd molars with primary caries that had been extracted for orthodontic reasons were obtained, and used in these experiments. Because they had been extracted for clinical reasons, and as there was no means of identifying the patients from whom they came, it was not necessary to obtain ethical permission to use them. These 50 teeth were stored for up to 5 days in water, then divided randomly into five groups. They were cut through the carious lesion perpendicular to the long axis using a low-speed diamond saw slice by slice until hard but discoloured dentine was reached. Slices were 0.7 mm thick.

All cut surfaces were prepared identically, ie conditioned with commercial poly (acrylic acid) conditioning agent (10% Fuji conditioner applied for 10 s), as recommended by both manufacturers, after which cylindrical specimens (4 mm diameter × 4 mm height) of glass-ionomer cement were attached. This was achieved by packing cement into cylindrical moulds placed on the cut surface of the tooth, and packing them until they were full. They were left to harden for 10 min, then the mould carefully removed. Bonding was to regions that occurred naturally, which for the teeth which had been carious, meant to surfaces which were partly or mainly discoloured. Five different cements were used, with 10 specimens of each being prepared. Two hand-mixed cements and three capsulated cements, designed for mixing in a standard reciprocating automixer, were used. Details of these materials are found in Table 1. Specimens were sealed with petroleum jelly and stored in high humidity conditions at 37°C for 24 h.

A further set of 50 human molars, also extracted for clinical reasons, but which were caries-free, were obtained and prepared in the same way. They were also conditioned, and cylindrical specimens of glass-ionomer cement attached as for the carious teeth. Shear bond strengths were determined using a Hounsfield Universal Testing machine, at a loading rate of 1 mm/min, using a knife edge placed 1 mm away from the interface. Loads at failure were converted to bond strengths by dividing by the contact areas of the cylinders.

Data were analysed by one-way ANOVA, and pairs of results for each individual cement were analysed using Student's *t*-test.

Table 1 Glass-ionomer cements employed in the study

Brand	Manufacturer	Type
Fuji IX GP	GC (Japan)	Hand-mixed
Ketac-Molar	3M-ESPE (Germany)	Hand-mixed
Fuji IX caps	GC (Japan)	Capsulated, automixed
Fuji IX FAST caps	GC (Japan)	Capsulated, automixed
Ketac-molar Aplicap	3M-ESPE (Germany)	Capsulated, automixed

3 Results

Results are shown in Table 2. For bonding to sound dentine, there were no statistically significant differences between any of the cements. For bonding to carious dentine, there were no differences except for Ketac-Molar, which showed a significantly lower bond strength ($p < 0.01$).

Considering the pairs of results for the individual cements, the bond strength to sound dentine did not differ significantly for the three capsulated cements. By contrast, for the two hand-mixed cements, bond strengths to sound dentine were higher and the differences were significant ($p < 0.05$ for Fuji IX; $p < 0.001$ for Ketac-Molar).

4 Discussion

Previous studies have shown that typical shear bond strengths of glass-ionomer cements to dentine lie in the range 1–3 MPa, and rarely exceed 5 MPa [21–23]. Microtensile bond strength values tend to be higher [24], because of the differences in stress distribution compared with shear testing, though to date results have been obtained only for sound dentine surfaces. Failure of bonded glass-ionomers has been found to be a mixture of adhesive and cohesive, as was confirmed in the present study using a low-powered optical microscope. However, because of the known chemical interactions between the tooth surface and the material [11], the tooth surface may be expected influence the observed load at failure in certain circumstances.

In the present study, bond strengths of capsulated materials to carious and to sound dentine do not differ in a statistically significantly way, but that they were found to be differ for the hand-mixed cements, with lower values being found for bonding to carious dentine than to sound dentine. Both types of cement are applied to the tooth when unset, and hence still essentially liquid. It is not clear why the hand-mixed cements should show a difference from the automixed (capsulated) ones, but it is of interest that bond strengths observed experimentally were lower to carious dentine.

Table 2 Shear bond strengths (MPa) (standard deviations in parentheses)

Material	Bond strength, carious dentine	Bond strength, sound dentine
Fuji IX GP	1.3 (0.5)	2.3 (1.2)
Ketac-Molar	0.6 (0.4)	1.9 (0.5)
Fuji IX caps	3.1 (2.1)	3.2 (1.6)
Fuji IX FAST caps	4.2 (2.2)	3.3 (0.6)
Ketac-Molar Aplicap	2.3 (1.9)	1.9 (1.4)

The surface of the dentine to which the bond is formed has been shown to be different in teeth that had caries from those in teeth with no caries [25]. Under clinical conditions, caries is removed as far as the hard underlying sclerotic dentine. However, this is not the same as non-carious tooth surface. It has been shown to be porous, and thus to have the capability of retaining traces of lactic acid [25], and also to stain differently from non-carious tooth substance [26], even after storage for periods of time of a few days. Its overall structure and chemical composition is therefore known to be different from that of non-carious dentine.

Previous studies have shown that these cements react rapidly with the acids involved in dental caries [19] and that bonding of glass-ionomers involves the formation of a diffusion bonding layer with the tooth [11]. This may be the reason that, in certain cements, the bond to carious teeth differs from that to sound teeth. However, such differences were not observed for the capsulated cements, and in the case of the hand-mixed cements, the bonds were lower to sclerotic dentine, despite the greater chemical reactivity. It is not clear why hand-mixed cements behaved differently, though in these materials, the setting reaction is relatively less advanced when they are placed on the tooth surface than it is for capsulated materials.

In previous studies, individual brands of glass-ionomer cement have been shown to interact differently with the tooth surface. For example, Fuji IX Caps gave a significantly better microtensile bond strength to healthy dentine than Ketac-Molar Aplicap [24]. The intermediate layer below the Fuji IX Caps included denatured collagen from the original smear layer, but the collagen fibrils from the underlying dentine were intact and exhibited characteristic crossbanding. By contrast Ketac-Molar Aplicap had been used in association with a much more aggressive conditioning regime that had removed more of the smear layer and opened the dentinal tubules to an extent, allowing cement tags to form. However, these tags were mechanically weak, and made only a limited contribution to retention. Other authors have reported the absence of a hybrid layer for Ketac-Molar [27], and also have not reported the occurrence of cement tags.

In the present study, all conditioning was done with the same process, so that by contrast with some of the work previously reported, all surfaces should have been the same in terms of the nature and thickness of the remaining smear layer. Any differences found must be attributable to fundamental differences between the surfaces that arise as a result of the presence or absence of caries.

Conditioning the surface of sclerotic dentine would not be expected to alter the fact that the surface typically consists of sclerotic dentine with tubules blocked by hydroxyapatite crystals. The lower or equal bond strengths suggest that micromechanical attachment may play some role in the bonding of glass-ionomer cements to dentine. Such bonding would be inhibited by the blocking of the dentinal tubules, thus resulting in lower recoded bond strengths, as was found experimentally. There are other factors that would be expected to affect the bond strengths. Sclerotic dentine is relatively demineralised, and thus the anticipated bonding, arising from the affinity for carboxylic acid groups in the polymer for hydroxyapatite surfaces, would be less. An earlier study concluded that the reason for lower bond strengths to primary dentine than to permanent dentine is that primary dentine is less mineralised [28].

The depth to which the dentine is cut has also been shown to affect bond strength [29]. Bonding to dentine close to the pulp was of lower strength than bonding to dentine close to the enamel, a result that was attributed to the greater water content of the deep dentine. In both this case, and the case of permanent versus primary dentine, the unifying factor is that reduced mineral content leads to lower bond strengths.

The results of the present study show the same effect. Bond strengths of glass-ionomer to sclerotic dentine are statistically indistinguishable from, or lower than, bond strengths to sound dentine. This suggests that the major factor in bonding glass-ionomers to dentine is the interaction with the mineral phase. For sclerotic dentine, this is either equivalent to or less than that which develops in sound dentine. Possible enhanced interactions due to residual amount of carious acids appear insufficient to promote improved bonding. Hence, although the detailed mechanisms of bonding are different from the bonding of composite resins with tailored bonding agents, the overall results are the same: bond strengths of glass-ionomers tend to be greater to sound dentine than to carious dentine [16].

5 Conclusions

Shear bond strengths of auto-mixed restorative glass-ionomer cements to prepared, sclerotic dentine have been found not to differ statistically from shear bond strengths to sound dentine whereas for hand mixed restorative glass-ionomers, shear bond strengths were lower to sclerotic dentine than to sound dentine. This suggests that these substrates are very similar despite the fact that sclerotic dentine is structurally and chemically different from non-carious dentine, even at depths that remain hard and are left in place during clinical treatment. That these substrates are not absolutely identical is shown by the behaviour of the hand-mixed cements, which showed statistically significant differences between the two groups of dentine, and lower shear bond strengths to the carious dentine.

Known aspects of the chemistry of glass-ionomers, such as the possible formation of a diffusion-based interaction layer with the tooth [30], or the potentially rapid reaction with the acids responsible for caries [19], which might be expected to be enhanced in the sclerotic dentine, did not enhance the bond strength. Hence may it may be concluded that they do not make a significant contribution to bond development in carious teeth, at least in the shorter term.

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