The influence of accelerating the setting rate by ultrasound or heat on the bond strength of glass ionomers used as orthodontic bracket cements

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SUMMARY Conventional glass ionomer cements (GICs) may be a viable option for bracket bonding when the major disadvantages of these materials, such as the slow setting reaction and the weak initial bond strength, are solved. The aim of this *in vitro* study was to investigate the influence of ultrasound and heat application on the setting reaction of GICs, and to determine the tensile force to debond the brackets from the enamel. A conventional fast-setting GIC, Fuji IX Fast, and two resin-modified glass ionomer cements (RMGICs), Fuji Ortho LC and Fuji Plus, were investigated. Three modes of curing were performed (n = 10): (1) according to the manufacturer's prescription, (2) with 60 seconds application of heat, or (3) with 60 seconds application of ultrasound. The tensile force required to debond the brackets was determined as the tension 15 minutes after the start of the bonding procedure. The mode of failure was scored according to the Adhesive Remnant Index (ARI) to establish the relative amount of cement remnants on the enamel surface.

Curing with heat and ultrasound shortened the setting reaction and significantly (P < 0.05) increased the bond strength to enamel. The ARI scores showed an increase for all materials after heat and ultrasound compared with the standard curing method, most notably after heat application.

Introduction

Since the introduction by Newman (1965) of the direct bonding technique for brackets, resin composite cements have been widely used as orthodontic adhesives (Wickwire and Rentz, 1973; Zachrisson and Brobakken, 1978; Trimpeneers and Dermaut, 1996). Due to the advantage of direct bracket fixation and its acceptable bonding under clinical conditions, this technique has become the one of choice. Nevertheless, resin composite cements also have some less desirable aspects, such as tooth tissue damage due to the etching procedure and the removal of remnants of adhesive after debonding, and occasionally the occurrence of enamel chipping during the debonding procedure (Fitzpatrick and Way, 1977; Brown and Way, 1978; Pus and Way, 1980; Diedrich, 1981; Lehman and Davidson, 1981; van Waes *et al.*, 1997).

The use of glass ionomer cements (GICs) as an alternative has the advantage that they achieve a chemical bond to enamel (Wilson *et al.*, 1983) without etching (van Dijken, 1990). The polyalkenoic acids slightly pit the enamel to enable the formation of a thin hybrid layer. Removal of this layer after debonding of the brackets shortens the total treatment time (Charles, 1998) and leads to less enamel damage compared with procedures involving resin composite cements. Another advantage of GICs is the ability to release fluoride over a long period of time. Hallgren *et al.* (1993; 1994) showed that deposition of fluoride in the plaque around the orthodontic bonding area led to a decrease in Mutans streptococcus and Lactobacillus bacteria. Furthermore, Marcusson et al. (1997) and Sadowsky et al. (1981) reported less demineralization and white spot formation when fluoride-releasing cements were used. With regard to saliva contamination, conventional GICs are less demanding to apply than resin-based cements. Besides these advantages, there are also disadvantages such as the slow curing reaction and the weak bond strength. However, ongoing developments have led to newer versions with improved curing rates and mechanical properties, and a new technique based on the application of ultrasonic energy has recently been introduced giving faster cure and improved material properties (Towler *et al.*, 2001; van Duinen *et al.*, 2001). The mechanism of the accelerated setting and increased mechanical properties is not yet clear, but preliminary results indicate that heat plays an important role in the ultrasound-mediated curing reaction. The application of heat to setting materials is not a new idea and has been described previously for resin-based materials (Vorster, 1979; Ward et al., 1998).

Another approach to overcome the disadvantages of GICs was the development of hybrid materials, which combine resin composite and conventional glass ionomer technologies. These so-called resin-modified GICs (RMGICs) contain resins that are auto-curing or light curable to improve the initial strength, and possess most of the

advantages of conventional GICs (Sidhu and Watson, 1995). Because of satisfactory bond strengths to enamel, RMGICs have been used with increasing success for bracket bonding (Voss, 1993; Charles, 1998). However, for these cements it is recommended by the manufacturer that the enamel surface should be conditioned with poly-acrylic acid. This step is not required for conventional GICs and should preferably not be part of the procedure to minimize damage of the enamel and the length of the procedure.

The purpose of this study was to evaluate the *in vitro* force required to debond orthodontic brackets bonded to enamel with one of the latest fast-setting conventional GICs, cured with heat or ultrasonic energy. Two RMGICs, one auto-curing and one light-curing, were included for comparison.

Materials and methods

Enamel from 90 freshly extracted bovine teeth, randomly collected from two-year-old cattle, was used as the substrate. The advantage of using bovine instead of human enamel is that the teeth can be easily collected and the specimens can be obtained from the same age group. According to Nakamichi *et al.* (1983) and Oesterle *et al.* (1998) bovine enamel can be used as a substitute for human enamel. Although scanning electron microscopy (SEM) studies have shown some differences between bovine and human enamel, these did not result in statistical differences in bond strength (Nakamichi *et al.*, 1983). The crowns of the teeth were cut from the roots, embedded in flat cylindrical PMMA moulds, and ground at the vestibular enamel surface on wet silicon carbide paper up to grit 1200 to create a flat surface.

Mesh-based brackets (Mini Twin, 'A' Company Orthodontics, San Diego, California, USA) intended for the central upper incisors with a curved bonding area size of 2.9×4.2 mm were used to cement to the enamel substrates.

The cements investigated for bonding the brackets are shown in Table 1. All cements were handled according to the manufacturers' prescriptions, while curing was performed either under standard, heat or ultrasound conditions. Heat was applied with a voltage modified (220–70V) soldering iron (Weller~220V/15W, Cooper Tools GmbH, Besigheim, Germany) producing a tip temperature of $70 \pm 2^{\circ}$ C, and ultrasonic energy with the Suprason P5 booster tooth scaler (Satelec, Merignac, France), with a vibration frequency of 25 000 to 30 000 Hz at stage 12. The H3 83780/1 tip was used. If light-curing was required, the Elipar Trilight curing unit (3M-espe Dental Products, Seefeld, Germany) was used in the standard mode at 750 mW/cm².

Specimen preparation

As recommended by the manufacturer of Fuji Ortho LC, the enamel was conditioned with a polyacrylic acid-gel (GC Dentin Conditioner, GC Corp., Tokyo, Japan) for 20 seconds. No conditioning of the enamel was performed for Fuji Plus and Fuji IX Fast.

Following bracket placement, the cements were either heat cured (HC), ultrasound cured (UC), or cured in the standard way (SC). For each curing condition 10 specimens were prepared. HC and UC were applied for 60 seconds; HC by placing the tip of the soldering iron directly on the wings of the bracket, and UC by holding the tip of the ultrasonic unit slightly above the wings (no direct contact), with a liquid medium (Scotchbond Multipurpose, 3M-espe Dental Products) between the tip and bracket (Figure 1). The light curing cement, Fuji Ortho LC, was additionally light cured for 40 seconds with the Elipar Trilight after the HC or UC treatment. For SC, light curing for 60 seconds after bracket placement was used initially to keep the light curing similar to HC and UC conditions. The specimens were stored in water at $22 \pm 1^{\circ}$ C and tested after 15 minutes.

Debonding force determination

A round stainless steel wire (diameter 1 mm) was tied with a harness ligature to the bracket with the ends of the wire bent perpendicular to the enamel surface. This method of fixing created an axis for the bracket, allowing rotational movement around the wire. The free ends of the wire were clamped to the connecting piece of the tensilometer (Instron 6022, High Wycombe, Bucks, U.K.). The rotation axis in this connecting piece and the axis formed by the wire, which were perpendicular to each other, excluded torsion forces on the bracket–adhesive–enamel system during tensile loading (Figure 2).

Fifteen minutes after the start of the bonding procedure the specimens were loaded in tension with a crosshead speed of 0.1 mm/minute until fracture. The loads at fracture were recorded in Newtons. After testing, the type of fracture was scored using the Adhesive Remnant Index (ARI; Årtun and Bergland, 1984) to identify the weakest point in the bracket–adhesive–enamel system. A score of 0 indicates

 Table 1
 Glass ionomer cements used in this study.

Material	Manufacturer	Cement type	Batch number	Expiry date
Fuji IX Fast	GC Corporation	Conventional glass ionomer	9911125	September 2003
Fuji Plus	GC Corporation	Resin-modified glass ionomer	0107051	July 2003
Fuji Ortho LC	GC Corporation	Resin-modified glass ionomer	0105115	May 2003



Figure 1 Application of ultrasound (a) and heat (b) to the bracket. Ultrasonic energy was applied by holding the tip slightly above the wings (no direct contact) with a liquid medium between the tip and bracket. Heat was applied by placing the tip of the soldering iron directly on the wings of the bracket.



Figure 2 Schematic representation of the set-up for tensile loading.

that no adhesive is left on the enamel, 1 that less than half of the adhesive remains, 2 that more than half of it remains, and 3 that all the adhesive remains on the enamel surface. The scores were determined with a stereomicroscope at a magnification of $\times 25$.

Temperature measurement in the pulp chamber

To measure the temperature rise in the pulp chamber during application of heat or ultrasound, a thermocouple (K-type) was inserted retrograde and fixed in the pulp chamber of a human incisor, which was filled with a hydrocolloid impression material (Combiloid, Cavex Nederland B.V., Haarlem, The Netherlands) to mimic pulpal tissue. The experiments were carried out with the root section placed in water at 37°C. The temperature in the incisor was registered with a calibrated TC-08 data logger (Pico Technology Limited, St Neots, U.K.) during the 60 seconds that ultrasound or heat was applied.

Statistical analysis

Two-way analysis of variance (ANOVA) was used to test the effect of the curing method and the cements on the debonding force. Furthermore, one-way ANOVA was used to determine differences in debonding force between SC, HC and UC within the materials (Bonferroni *post hoc*). P < 0.05 was considered significant. The software used was SPSS 10.0 (SPSS Inc., Chicago, Illinois, USA).

Results

From visual observation, HC and UC accelerated the setting of both the conventional and RMGICs.

Two-way ANOVA analysis showed that the curing method (F = 17.2; P < 0.001) and the cement (F = 55.0; P < 0.001) had a significant effect on the debonding force, while the interaction effect was not significant (F = 1.4; P = 0.199). Pairwise comparision demonstrated that both UC and HC significantly (P < 0.001) improved the debonding force with respect to SC, but UC and HC were not significantly different (P = 0.156). One-way ANOVA analysis showed a significant increase in tensile force required to debond the brackets (P < 0.05) for the RMGICs, Fuji Ortho LC and Fuji Plus after HC and UC compared with SC. For the conventional GIC, Fuji IX Fast, only HC had a beneficial effect (Figure 3). ARI scores for each group are shown in Figure 4.

The temperature measurements in the pulp chamber showed a maximum rise in temperature of 2.5°C after 60 seconds of heat application and of 1.0°C after ultrasound application. These values are only meant for direct comparison of the temperature rise caused by the two methods. It should be noted that, as the thermal conductivity of the experimental pulp tissue is unknown, the results cannot be extrapolated to the clinical analogue.



Figure 3 Mean tensile fracture force and standard deviation determined 15 minutes after the start of the bonding procedure. HC = heat cure, UC = ultrasonic cure and SC = standard cure. Values within a group that are not significantly different from each other are marked with an asterisk (P > 0.05).



Figure 4 Mean Adhesive Remnant Index (ARI) scores for each group. SC = standard cure; UC = ultrasonic cure; HC = heat cure.

Discussion

During HC or UC an increased setting rate was observed for both the conventional and RMGICs compared with SC. In addition, the two treatments had a beneficial effect on the bond in the early stages – the first 15 minutes (Figure 3). The tensile forces required to debond the brackets cemented with the RMGICs, Fuji Ortho LC and Fuji Plus, were significantly higher than those with SC. For the conventional GIC, Fuji IX Fast, a stronger bond was only observed for HC. Heat is also generated by ultrasound, from its kinetic energy, but the temperature rise was lower than that with the soldering iron, as shown by the results of temperature measurements in the pulp chamber. Beside the generation of heat, ultrasound may also contribute to acceleration of the reaction by de-clustering glass particles and enhancing the diffusion of the reaction components. Mechanical properties could be improved as well. The material can be condensed by a reduction of porosity, which is vibrated out of the mix. However, SEM evaluation did not show declustering of the glass particles or a reduction of porosity, so it seems that heat played the major role in bond strength improvement with UC.

After mixing the liquid and powder components of the RMGICs, the setting proceeds along two different chemical reactions. The first, which starts at mixing and progresses relatively slowly (Williams and Billington, 1989, 1991; Pearson and Atkinson, 1991; Soltész and Leupolz, 1993; Cattani-Lorente et al., 1994; de Gee et al., 1996), is an acidbase reaction between a polyalkenoic acid and the basic glass powder. The second reaction is a polymerization initiated by a catalyst, as for Fuji Plus, or by light irradiation. Fuji Ortho LC is initiated both by a catalyst and by light. It has been shown previously (de Gee et al., 1998) that under normal setting conditions the acidbase reaction is severely hindered in RMGICs by the presence of the polymer structure. As heat will generally accelerate chemical reactions, the application of heat is also expected to increase the acid-base reaction rate as well. So for both materials, Fuji

Plus and Fuji Ortho LC, the polymerization reaction together with the acid-base reaction could benefit from HC and UC. HC increased the tensile debonding force for Fuji Ortho LC and Fuji Plus by 27 and 52 per cent, respectively. For UC the increase was 32 and 30 per cent, respectively. In the case of Fuji IX Fast, which does not contain resins, only the acidbase reaction was accelerated. This could be the reason why HC had an effect on the debonding force (27 per cent), but not UC (3 per cent), as UC added less heat to the reacting medium.

The evaluation of the mode of failure after debonding showed an increase of cement remnants on the enamel surface in all groups after HC or UC compared with SC. The results, expressed as ARI scores, were most notable after HC, but for Fuji Plus and Fuji IX Fast, both HC and UC also had a clear effect (Figure 3). This is certainly remarkable, as no conditioning of the enamel was performed prior to application of these materials. Apparently, ultrasonic energy or heat can improve the bond strength to enamel. The high ARI score of 2.9 found for Fuji IX Fast after HC, indicates that the bracket-adhesive bond forms the weakest point. This gives the opportunity of improving the bond strength at the bracket-adhesive interface, which may be easier to accomplish than at the enamel-adhesive interface. Not only were the ARI scores raised, but the standard deviations of the results became lower after HC, which indicates that a more predictable bond can be achieved not only in the test set-up, but possibly also in the clinical situation.

Normally, RMGICs and conventional GICs require several days to reach full strength (Williams and Billington, 1989, 1991; Pearson and Atkinson, 1991; Soltész and Leupolz, 1993; Cattani-Lorente *et al.*, 1994; de Gee *et al.*, 1996). During this period the cements are still weak and the conventional GICs, in particular, are susceptible to dissolution. A faster setting with HC and UC may change this situation, as the ultimate strength can be reached sooner. However, at this stage with current materials such as Fuji IX Fast, the bond strength obtained with HC and UC is still low compared with the regular cement, Fuji Ortho LC. Fuji Ortho LC has shown its clinical usefulness, and therefore forces of around 40 Newtons (Figure 3) to debond brackets can be used as a baseline value.

A difficulty with the UC method is steady fixation of the bracket during application. Because of vibration, the bracket tends to move during curing, which can negatively affect the bond strength. The viscosity of the liquid used as a medium for transferring the ultrasonic vibration from the tip to the bracket may play an important role in modifying the intensity of waves, and bracket material may influence performance. The HC method is easier to apply, although alternative ways of applying heat, which are easier to handle inside the oral cavity, should be researched. Also new material formulations especially designed to absorb heat could help to accelerate the procedure.

Conclusions

- Accelerated setting by means of heat or ultrasound results in significantly higher tensile forces being required to debond brackets from enamel for Fuji Plus and Fuji Ortho LC, while for Fuji IX Fast only heat is effective.
- The debonding force obtained after heat treatment of current conventional GICs such as Fuji IX Fast cannot yet compete with that of contemporary materials such as the RMGIC Fuji Ortho LC.
- Heat application results in higher ARI scores compared with the standard method of curing, while ultrasound curing raises the scores only for Fuji Plus and Fuji IX Fast.

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References

- Årtun J, Bergland S 1984 Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. American Journal of Orthodontics 85: 333–340
- Brown C R, Way D C 1978 Enamel loss during orthodontic bonding and subsequent loss during removal of filled and unfilled adhesives. American Journal of Orthodontics 74: 663–671
- Cattani-Lorente M A, Godin C, Meyer J M 1994 Mechanical behavior of glass ionomer cements affected by long-term storage in water. Dental Materials 10: 37–44
- Charles C 1998 Bonding orthodontic brackets with glass ionomer cement. Biomaterials 19: 589–591
- de Gee A J, Leloup G, Werner A, Vreven J, Davidson C L 1998 Structural integrity of resin-modified glass ionomers as affected by the delay or omission of light activation. Journal of Dental Research 77: 1658–1663
- de Gee A J, van Duinen R N, Werner A, Davidson C L 1996 Early and long-term wear of conventional and resin-modified glass ionomers. Journal of Dental Research 75: 1613–1619
- Diedrich P 1981 Enamel alterations from bracket bonding and debonding: a study with the scanning electron microscope. American Journal of Orthodontics 79: 500–522
- Fitzpatrick D A, Way D C 1977 The effects of wear, acid etching, and bond removal on human enamel. American Journal of Orthodontics 72: 671–681
- Hallgren A, Oliveby A, Twetman S 1993 Fluoride concentration in plaque adjacent to orthodontic appliances retained with glass ionomer cement. Caries Research 27: 51–54
- Hallgren A, Oliveby A, Twetman S 1994 L(+)-lactic acid production in plaque from orthodontic appliances retained with glass ionomer cement. British Journal of Orthodontics 21: 23–26
- Lehman R, Davidson C L 1981 Loss of surface enamel after acid etching procedures and its relation to fluoride content. American Journal of Orthodontics 80: 73–82

- Marcusson A, Norevall L I, Persson M 1997 White spot reduction when using glass ionomer cement for bonding in orthodontics: a longitudinal and comparative study. European Journal of Orthodontics 19: 233–242
- Nakamichi I, Iwaku M, Fusayama T 1983 Bovine teeth as possible substitutes in the adhesion test. Journal of Dental Research 62: 1076–1081
- Newman G V 1965 Epoxy adhesives for orthodontic attachments: progress report. American Journal of Orthodontics 51: 901–912
- Oesterle L J, Shellhart W C, Belanger G K 1998 The use of bovine enamel in bonding studies. American Journal of Orthodontics and Dentofacial Orthopedics 114: 514–519
- Pearson G J, Atkinson A S 1991 Long-term flexural strength of glass ionomer cements. Biomaterials 12: 658–660
- Pus M D, Way D C 1980 Enamel loss due to orthodontic bonding with filled and unfilled resins using various clean-up techniques. American Journal of Orthodontics 77: 269–283
- Sadowsky P L, Retief D H, Bradley Jr E L 1981 Enamel fluoride uptake from orthodontic cements and its effect on demineralization. American Journal of Orthodontics 79: 523–534
- Sidhu S K, Watson T F 1995 Resin-modified glass ionomer materials. A status report for the American Journal of Dentistry. American Journal of Dentistry 8: 59–67
- Soltész U, Leupolz M 1993 Härte- und Festigkeitsverhalten von Glasionomerzementen. Deutsche Zahnärztliche Zeitschrift 48: 237–241
- Towler M R, Bushby A J, Billington R W, Hill R G 2001 A preliminary comparison of the mechanical properties of chemically cured and ultrasonically cured glass ionomer cements, using nano-indentation techniques. Biomaterials 22: 1401–1406
- Trimpeneers L M, Dermaut L R 1996 A clinical evaluation of the effectiveness of a fluoride-releasing visible light-activated bonding system to reduce demineralization around orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 110: 218–222
- van Dijken J W 1990 The effect of cavity pretreatment procedures on dentin bonding: a four-year clinical evaluation. Journal of Prosthetic Dentistry 64: 148–152
- van Duinen R N B, de Gee A J, Davidson C L 2001 Advantageous effects of ultrasound on the setting and mechanical properties of glass ionomers. Journal of Dental Research 80: 1203 (Abstract)
- van Waes H, Matter T, Krejci I 1997 Three-dimensional measurement of enamel loss caused by bonding and debonding of orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 112: 666–669
- Vorster S W 1979 Electrothermic bonding. Journal of Clinical Orthodontics 13: 816–819
- Voss A 1993 *In vivo* bonding of orthodontic brackets with glass ionomer cement. Angle Orthodontist 63: 149–153
- Ward G J, Mizrahi E, Cleaton-Jones P E 1998 Electrothermal bonding: shear bond strength of orthodontic brackets after two weeks. British Journal of Orthodontics 25: 123–126
- Wickwire N A, Rentz D 1973 Enamel pretreatment: a critical variable in direct bonding systems. American Journal of Orthodontics 64: 499–512
- Williams J A, Billington R W 1989 Increase in compressive strength of glass ionomer restorative materials with respect to time: a guide to their suitability for use in posterior primary dentition. Journal of Oral Rehabilitation 16: 475–479
- Williams J A, Billington R W 1991 Changes in compressive strength of glass ionomer restorative materials with respect to time periods of 24 h to 4 months. Journal of Oral Rehabilitation 18: 163–168
- Wilson A D, Prosser H J, Powis D M 1983 Mechanism of adhesion of polyelectrolyte cements to hydroxyapatite. Journal of Dental Research 62: 590–592
- Zachrisson B U, Brobakken B O 1978 Clinical comparison of direct versus indirect bonding with different bracket types and adhesives. American Journal of Orthodontics 74: 62–78

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