European Journal of Orthodontics 29 (2007) 550–554
doi:10.1093/ejo/cjm046
Advance Access publication 4 September 2007

A method for producing controlled fluoride release from an orthodontic bracket

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SUMMARY The aim of this study was to manufacture and test, *in vitro*, a novel modification to provide fluoride-releasing orthodontic brackets. Thirty-two orthodontic brackets were drilled to produce a recess (approximately 1.3 mm in diameter and 0.7 mm in depth) at the centre of the bracket base. Four materials, with and without the addition of sodium fluoride, a glass ionomer cement (Ketac Cem μ), a resin-modified glass ionomer cement (RMGIC; GC Fuji Ortho LC), a zinc phosphate (Zinc Cement Improved), and a resin (Transbond XT) were used to fill the recess in the bracket base. Fluoride release was measured daily during the first week and then weekly for 10 weeks. An ion chromatograph with suppressed conductivity was used for free fluoride ion determination. Statistical analysis to determine the amount of flouride release was undertaken using analysis of variance and Tukey's test.

During the first 2 weeks, the resin group, with the addition of 38 per cent sodium fluoride added, released significantly more free fluoride ($P < 0.05$), but after 2 weeks the fluoride release markedly decreased. After 5 weeks, the RMGIC group, with 15 per cent added sodium fluoride, had significantly higher ($P < 0.05$) daily fluoride release than the other groups. The findings demonstrated that an appropriate fluoridated material can be used as a fluoride-releasing reservoir in a modified orthodontic bracket to enable it to release fluoride over the period of fixed appliance treatment.

Introduction

Enamel demineralization and white spot lesions are common side effects occurring during the period of orthodontic therapy with fixed appliances (Zachrisson, 1977; Årtun and Brobakken, 1986; O'Reilly and Featherstone, 1987). The occurrence of enamel demineralization after the use of a fixed orthodontic appliance can occur in as many as 50 per cent of patients (Gorelick *et al.*, 1982; Årtun and Brobakken, 1986).

It has been shown that fluoride in low concentrations can inhibit the formation of, and enhance the remineralization of, such lesions (Geiger *et al.*, 1988; Ögaard *et al.*, 1998). The self-administration methods of applying fluoride, such as fluoride toothpastes and mouth rinses, are efficient for reducing white spot lesions, but the patient's co-operation is essential (Gorelick *et al.*, 1982; Geiger *et al.*, 1988; Jordan, 1998). Compliance rates as low as 13 per cent have been reported for patients asked to decrease their caries risk with a daily fluoride mouth rinse (Geiger *et al.*, 1992). Professional means of fluoride application have included fluoride-releasing bonding agents, fluoridated elastomeric ligature ties and fluoride varnish (Sonis and Snell, 1989; Ögaard *et al.*, 1992; Frazier *et al.*, 1996; Todd *et al.*, 1999; Wiltshire, 1999; Gillgrass *et al.*, 2001; Mattick *et al.*, 2001; Pascotto *et al.*, 2004).

Glass ionomer cements (GICs) have been shown to release fluoride over a long period of time and have the ability to absorb fluoride during exposure to fluoride rinses or fluoride-containing toothpaste, and of subsequently maintaining a high fluoride release level by re-release (Takahash *et al.*, 1993). When used for orthodontic bonding, GICs have been shown to inhibit enamel demineralization (Marcusson *et al.*, 1997). Unfortunately, because of their low bond strengths, it has been suggested that they are not suitable for routine use in bonding orthodontic brackets (Millett and McCabe, 1996). The recently introduced polyacid-modified composite and resin-modified glass ionomer cements (RMGICs) have a stronger bond strength compared with GICs while maintaining some ability to release fluoride and be recharged (Cohen *et al.*, 2003). However, their fluoride-releasing ability varies between products which may be combined with a high bracket failure rate. Also similar rates of demineralization have been reported with a RMGIC compared with a composite resin (Gaworski *et al.*, 1999).

Professional application of fluoride varnish to the labial enamel surface (Frazier *et al.*, 1996) has been reported to produce a 50 per cent reduction in white spot appearance, although the use of fluoride varnish increases chairside time and requires regular repeated applications (Todd *et al.*, 1999; Schmit *et al.*, 2002).

Fluoridated elastomeric modules were considered to be an ideal solution to supplement fluoride around orthodontic brackets because they do not interfere with routine clinical practice and could ensure a 'fresh' delivery of fluoride at each visit (Wiltshire, 1999; Mattick *et al.*, 2001). Unfortunately, due to their poor mechanical performance, and swelling as a result of water absorption, fluoridated elastomers have not provided an acceptable solution to the problem. Ideally, a method which produces a sustained fluoride release without intervention by either the operator or the patient is required.

The methods described above have some disadvantages. However, if the orthodontic bracket itself had the ability to release fluoride during the period of orthodontic treatment, it would offer a potential solution to these problems. Therefore, the aim of this research was to manufacture and test a novel modification to provide a fluoride-releasing orthodontic bracket.

Materials and methods

Bracket preparation

Thirty-two metal incisor brackets (GAC, Omni Arch, Bohemia, New York, USA) were used. A recess (nominally 1.3 mm in diameter and 0.7 mm in depth) was drilled at the centre on the labial side of the base of each metal bracket. The recess did not penetrate the bracket base.

Four materials, with or without the addition of sodium fluoride (Table 1), were used to fill the recess in the bracket base. The 32 brackets were divided into eight equal groups. The amount of sodium fluoride to be added to the materials was determined by a pilot experiment. Table 1 shows the groups of brackets with details of their modification with the added cement. The brackets were weighed before and after being filled. Figure 1 shows a bracket after being modified and filled.

The materials were mixed (if required) and used to fill the prepared bracket recess and cured as recommended by the manufacturers. They were allowed to mature for 24 hours in a moist environment, following which each filled bracket was stored in individual plastic containers with 2.5 ml deionized water at 37°C and left for 24 hours. The brackets were then removed, blotted dry, and immersed in 2.5 ml of fresh deionized water. This procedure was repeated daily for 1 week and then weekly for 10 weeks.

Measurement of fluoride release

The free fluoride ion was measured using an ion chromatograph (DX 100; Dionex, Camberley, Surrey, UK) with suppressed conductivity, fitted with an ION PAC AS14 analytical column (Dionex) and ION PAC AG14 guard column (Dionex). The determination of fluoride ions was made using 0.5 ml of each storage solution which was injected into the injection loop of the instrument. The loop was designed such that 250 μl was fed to the column for analysis. A flow rate of 1.0 ml/minute was used. Free fluoride ions have a well-defined retention time and the peak corresponding to fluoride could readily be determined from the chromatogram. The peak area was used to determine fluoride concentration by linear interpolation between standard solutions of concentration slightly higher and lower than the test solution. The determination of each solution was made three times and fluoride concentration measured to an accuracy of 0.001 ppm. The free fluoride ion concentration in each solution was converted to the amount of fluoride release per unit weight of fillings (μg/mg).

Statistical analysis

For comparison of the amount of fluoride released by the materials, analysis of variance and Tukey's test were used to determine significance at a level of 0.05.

Results

The mean daily free fluoride ion release (logarithmic scale) from six test groups for 70 days is shown in Figure 2. Because no detectable fluoride release was observed for Transbond XT (group 6) after day 3 and for zinc phosphate (group 8) after day 5, these two groups were excluded from Figure 2. There was a greater fluoride release at the initial stage but the rate of fluoride release decreased quickly. Group 7, the resin composite with 38 per cent sodium fluoride added, had a significantly higher mean daily fluoride release during the first 2 weeks ($P < 0.05$). However, after 4 weeks, group 3, the RMGIC with 15 per cent sodium fluoride added, released more fluoride than the other groups ($P < 0.05$). During weeks 3 and 4, groups 3 and 7 released significantly more fluoride than the other groups ($P < 0.05$).

Figure 3 shows the cumulative fluoride release from four different materials with and without sodium fluoride added,

Table 1 The groups of brackets with different fillings.

	Fillings		
	Powder/liquid ratio	Percentage of sodium fluoride	Base material
Group 1 (G1)	2:1	7.7	Ketac Cem μ, 3M ESPE, Seefeld, Germany (glass ionomer)
Group 2 (G2)	2:1	None	
Group 3 (G3)	Capsule	15	GC Fuji Ortho LC, Tokyo, Japan (resin-modified glass ionomer)
Group 4 (G4)	Capsule	None	
Group 5 (G5)	5:1	10	Zinc Cement Improved, SS White, Gloucester, UK (zinc phosphate)
Group 6 (G6)	5:1	None	
Group 7 (G7)	Capsule	38	Transbond XT, 3M Unitek, Monrovia, California, USA (resin)
Group 8 (G8)	Capsule	None	

Figure 1 An orthodontic bracket after filling with fluoride-releasing material.

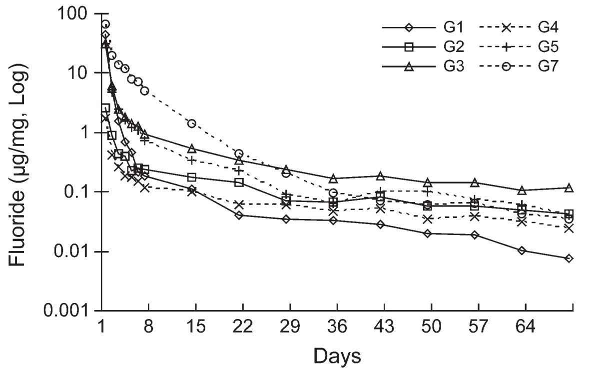

Figure 2 The mean daily free fluoride ions (log) released from six groups over a period of 70 days. See Table 1for key to groups.

where the mean fluoride release of the specimens of each group is plotted against square root of time (day). There were two stages of fluoride release after the addition of sodium fluoride. The initial stage that lasted from 3 days to 2 weeks gave a higher fluoride release. After that time, fluoride release continued at a lower but stable rate for the RMGIC and zinc phosphate with added fluoride, while for the GIC and resin, with added fluoride, very little fluoride was released after the initial period.

Discussion

The release of fluoride in the oral environment has been widely investigated in relation to its therapeutic effects, in particular the demineralization of dental hard tissues (ten Cate and Featherstone, 1991). To reduce white spot lesions which may occur by demineralization during orthodontic treatment, devices such as elastomeric ligatures (O'Dwyer *et al.*, 2005), wires (Lee *et al.*, 2005), and adhesive materials (Staley *et al.*, 2004), with fluoride release have been designed.

The purpose of the present research was to manufacture and test a novel modification to provide a fluoride-releasing orthodontic bracket as a novel way of approaching the prevention of decalcification. The fluoride release may be achieved using certain fluoride-releasing materials incorporated within the bracket. Orthodontic brackets with fluoride-releasing materials incorporated have the potential to provide a reliable and sustained fluoride release during the period of orthodontic treatment.

A recess was produced in the base of a bracket while attempting to avoid any damaging stress concentration. High stress levels have been observed at areas of abrupt change in geometry and shape (Ghosh *et al.*, 1995). Although this modification is unlikely to influence the performance of the orthodontic brackets, further research on this modification needs to be undertaken.

Ion chromatography was used to monitor fluoride release in this study. This has the advantage of being accurate at very low concentrations and gives a direct measurement of the effective free fluoride ion concentration which may not be possible using other methods (McCabe *et al.*, 2002).

The fluoride release of a fluoride-releasing orthodontic bracket depends upon the materials that are placed into the prepared bracket. In this regard, fluoride release has been considered as one advantage of GICs and related materials which have been already widely used in clinical dentistry. The pattern of fluoride release for glass ionomers indicates a high initial release rate followed by a rapid reduction in the rate of release over time (Shaw *et al.*, 1998). A material based on a GIC matrix but modified to optimize fluoride content may achieve long-term-sustained fluoride release. Four different materials, conventional GICs, RMGICs, zinc phosphate, and composite, were used as base materials with the addition of different amounts of sodium fluoride. Although in the groups in which GICs, composite and zinc phosphate were used as base materials released significantly greater fluoride than the RMGICs group in the initial stage, the RMGICs group had a more stable fluoride release over a period of 10 weeks.

In order to uniformly mix the sodium fluoride into the GICs and zinc phosphate cement, a low powder/liquid ratio (2:1) was used which may increase the solubility of the tested materials. Therefore, fluoride release from the tested materials may be affected by both matrix erosion and water diffusion throughout the matrix.

Because of the amount of added sodium fluoride (38 per cent), the resin material released the highest amount of fluoride in the first 2 weeks but then the fluoride release decreased sharply. The resin itself does not have the ability to release fluoride. The long-term fluoride release of this material was not greater than those materials that have an inherent ability to release fluoride.

The results of this research showed that, overall, RMGIC was the most effective material for incorporation into a bracket because it demonstrated more stable fluoride release ability over the longer term. Unlike the resin, it has some inherent

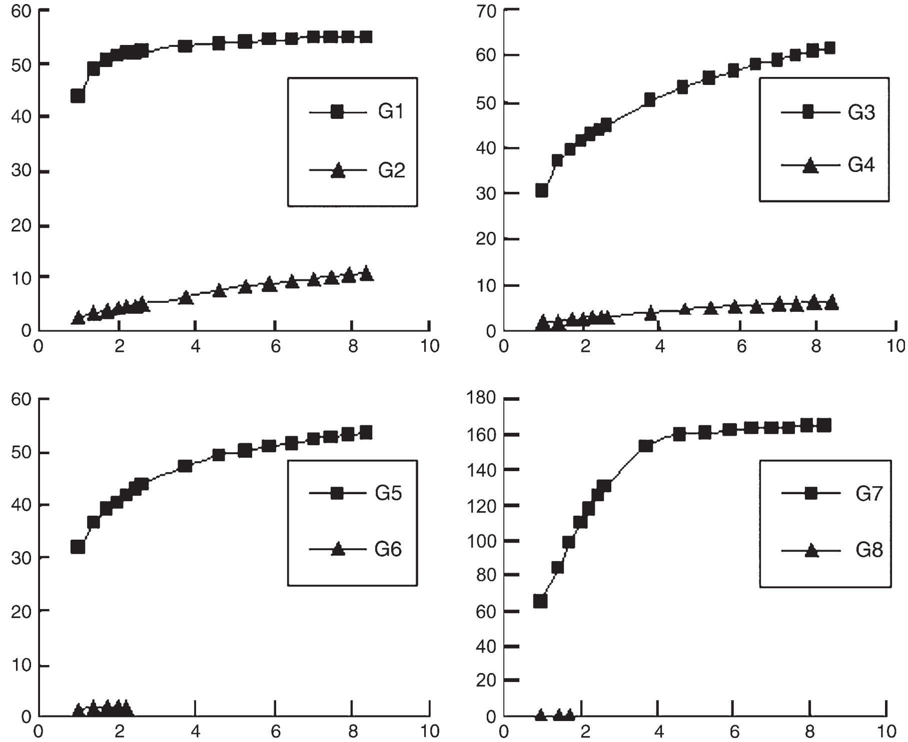

Figure 3 The cumulative fluoride release (mg/g; *y*-axis) against the square root of time $(\text{days})^{1/2}$ (*x*-axis). See Table 1 for key to groups.

fluoride release and can provide a longer period of fluoride release after most of the extrinsic fluoride has dissolved into water. Because the average period of orthodontic treatment lasts 1.5–2 years, long-term stable fluoride release may be preferable to a short-concentrated 'burst'.

Information on the level of fluoride release required to prevent demineralization around orthodontic brackets has not been known until now. It may be assumed that within a safe limit, the fluoride level should be as high as possible and these fluoride-releasing orthodontic brackets could provide a locally higher level of fluoride than can be achieved using other methods. Table 2 shows the levels of fluoride released from fluoride-releasing orthodontic brackets prepared by adding 15 per cent sodium fluoride to a RMGIC and this can be compared or contrasted with values of 2.45 on day 1 down to 0.01 on day 28 for samples of an unmodified cement as used in the study by Rix *et al.* (2001). Although the data are from two different investigations, a large difference in the amount of fluoride release can be observed.

Another important issue related to fluoride-releasing orthodontic brackets is that of safety. The amount of filling material per bracket was approximately 2 mg. The maximum amount of sodium fluoride added was 0.8 mg (if 38 per cent of sodium fluoride was added). The maximum amount of sodium fluoride that a patient could theoretically ingest would be 16 mg if 20 brackets were placed in the mouth and the fluoride was released rapidly. The probably toxic dose (PTD), 5.0 mg F/kg, is defined as the dose of ingested fluoride that should trigger immediate therapeutic intervention and hospitalization because of the likelihood of serious toxic consequences (Whitford, 1987). For a child weighing 40 kg, the PTD of fluoride would be 200 mg. Therefore, the maximum ingestible amount of 16 mg, which is much lower than PTD, from these brackets is safe.

Besides the ability to provide a long-term, high level of fluoride release, fluoride-releasing orthodontic brackets have other advantages compared with alternative fluoride therapy regimes. However, further clinical trials are needed to study the performance of these brackets. This method does not interfere with routine clinical practice, there is no influence on the mechanical performance of the brackets and it is independent of patient compliance. The further development of this method may produce an ideal fluoride-controlled release device for orthodontic treatment.

Conclusions

The novel modification to an orthodontic bracket using a fluoride-releasing material provides a significant fluoride release that may prevent demineralization associated with orthodontic treatment, although further work is required to optimize the bracket modification and fluoride-releasing material before a clinical trial can be undertaken. However, this research has demonstrated the potential for an important advance that will be of benefit to patients.

Table 2 Mean daily fluoride release (μg per bracket per day) from fluoride-releasing orthodontic brackets and Fuji Ortho LC.

Day	Fluoride-releasing brackets*		Fuji Ortho LC†	
	Mean	SD	Mean	SD
1	71.03	4.63	2.45	0.89
2	14.48	1.954	1.79	0.67
3	5.9	1.44	0.42	0.15
4	4.32	1.59	0.46	0.24
5	3.38	1.02	0.38	0.17
6	2.95	0.92	0.37	0.15
7	2.24	0.81	0.32	0.11
14	1.27	0.49	0.03	0.01
21	0.81	0.22	0.02	0.01
28	0.55	0.14	0.01	0.01

SD, standard deviation.*Data from group 3.†Data from research using a bracketed tooth model (Rix *et al.*, 2001).

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References

Årtun J, Brobakken B O 1986 Prevalence of carious white spots after orthodontic treatment with multibonded appliances. European Journal of Orthodontics 8: 229–234

Cohen W J, Wiltshire W A, Dawes C, Lavelle C L B 2003 Long-term *in vitro* fluoride release and rerelease from orthodontic bonding materials containing fluoride. American Journal of Orthodontics and Dentofacial Orthopedics 124: 571–576

Frazier M C, Southard T C, Doster P M 1996 Prevention of enamel demineralization during orthodontic treatment: an *in vitro* study using pit and fissure sealants. American Journal of Orthodontics and Dentofacial Orthopedics 110: 459–465

Gaworski M, Weinstein M, Borislow A J, Braitman L E 1999 Decalcification and bond failure: a comparison of a glass ionomer and a composite resin bonding system *in vivo*. American Journal of Orthodontics and Dentofacial Orthopedics 116: 518–521

Geiger A, Gorelick L, Gwinnett A J, Benson B J 1992 Reducing white spot lesions in orthodontic populations with fluoride rinsing. American Journal of Orthodontics and Dentofacial Orthopedics 101: 403–407

Geiger A M, Gorelick L, Gwinnett A J, Griswold P G 1988 The effect of a fluoride program on white spot formation during orthodontic treatment. American Journal of Orthodontics and Dentofacial Orthopedics 93: 29–37

Ghosh J, Nanda R S, Duncanson Jr M G, Currier G F 1995 Ceramic bracket design: an analysis using the finite element method. American Journal of Orthodontics and Dentofacial Orthopedics 108: 575–582

Gillgrass T J, Creanor S L, Foye R H, Millett D T 2001 Varnish or polymeric coating for the prevention of demineralization? An *ex vivo* study. Journal of Orthodontics 28: 291–295

Gorelick L, Geiger A M, Gwinnett A J 1982 Incidence of white spot formation after bonding and banding. American Journal of Orthodontics 81: 93–98

Jordan C 1998 Prevention of white spot enamel formation during orthodontic treatment. General Dentistry 46: 498–502

Lee S H, Kim H W, Kong Y M, Kim H E, Lee S H, Chang Y I 2005 Fluoride coating on orthodontic wire for controlled release of fluoride ion. Journal of Biomedical Materials Research Part B: Applied Biomaterials 75: 200–204

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

Mattick C R, Mitchell L, Chadwick S M, Wright J 2001 Fluoride-releasing elastomeric modules reduce decalcification: a randomized controlled trial. Journal of Orthodontics 28: 217–219

McCabe J F, Carrick T E, Sidhu S K 2002 Determining low levels of fluoride released from resin based dental materials. European Journal of Oral Sciences 110: 380–384

Millett D T, McCabe J F 1996 Orthodontic bonding with glass ionomer cement—a review. European Journal of Orthodontics 18: 385–399

O'Dwyer J J, Tinsley D, Benson P E 2005 The effect of stretching on the release of fluoride from fluoridated elastomeric ligatures. American Journal of Orthodontics and Dentofacial Orthopedics 128: 471–476

Øgaard B, Rezk-Lega F, Ruben J, Arends J 1992 Cariostatic effect of fluoride release from a visible light-curing adhesive for bonding of orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 101: 303–307

Øgaard B, Rölla G, Arends J 1998 Orthodontic appliances and enamel demineralization. Part 1. Lesion development. American Journal of Orthodontics and Dentofacial Orthopedics 94: 68–73

O'Reilly M M, Featherstone J D B 1987 Decalcification and remineralisation around orthodontic appliances: an *in vivo* study. American Journal of Orthodontics and Dentofacial Orthopedics 92: 33–40

Pascotto R C, Navarro M F, Capelozza Filho L, Cury J A 2004 *In vivo* effect of a resin-modified glass ionomer cement on enamel demineralization around orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 125: 36–41

Rix D, Foley T F, Banting D, Mamandras A 2001 A comparison of fluoride release by resin modified GIC and polyacid-modified composite resin. American Journal of Orthodontics and Dentofacial Orthopedics 120: 398–405

Schmit J L, Staley R N, Wefel J S, Kanellis M, Jakobsen J R, Keenan P J 2002 Effect of fluoride varnish on demineralization adjacent to brackets bonded with RMGI cement. American Journal of Orthodontics and Dentofacial Orthopedics 122: 125–134

Shaw A J, Carrick T, McCabe J F 1998 Fluoride release from glass-ionomer and compomer restorative materials: 6 months data. Journal of Dentistry 26: 355–359

Sonis A L, Snell W 1989 An evaluation of a fluoride-releasing visible light-activated bonding system for orthodontic bracket placement. American Journal of Orthodontics and Dentofacial Orthopedics 95: 306–311

Staley R N, Mack S J, Wefel J S, Vargas M A, Jakobsen J R 2004 Effect of brushing in fluoride release from 3 bracket adhesives. American Journal of Orthodontics and Dentofacial Orthopedics 126: 331–336

Takahash K, Emilson C, Birkhed D 1993 Fluoride release *in vitro* from various glass ionomer cements and resin composites after exposure to NaF solutions. Dental Materials 9: 350–354

ten Cate J M, Featherstone J D B 1991 Mechanistic aspects of the interactions between fluoride and dental enamel. Critical Reviews in Oral Biology 2: 283–296

Todd M A, Staley R N, Kanellis M J, Donly K J, Wefel J S 1999 Effect of a fluoride varnish on demineralization adjacent to orthodontic treatment. American Journal of Orthodontics and Dentofacial Orthopedics 116: 159–167

Whitford G 1987 Fluoride in dental products: safety considerations. Journal of Dental Research 66: 1056–1060

Wiltshire W A 1999 *In vitro* and *in vivo* fluoride release from orthodontic elastomeric ligature ties. American Journal of Orthodontics and Dentofacial Orthopedics 115: 288–292

Zachrisson B 1977 A posttreatment evaluation of direct bonding in orthodontics. American Journal of Orthodontics 71: 173–189