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Short Communication

FLUORIDE RELEASE FROM NEWLY DEVELOPED DENTAL ADHESIVES

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Abstract

This study compared fluoride release from three fluoride-releasing dental adhesives (Trial KBF Bond, One-up Bond F, Reactmer Bond) and one conventional dental adhesive (Clearfil SE Bond) for 70 days *in vitro*. From each material, five disk-shaped specimens (diameter: 9.0 mm, height: 1.5 mm) were prepared and immersed in 5 ml of 10 mM phosphate buffered solution (pH 7.0). After 1, 3, 7, 14, 21, 28, 35, 42, 49, 56, 63 and 70 days, the samples were transferred into new solutions. The fluoride content was determined with a combined fluoride sensitive electrode attached to an ion analyzer. Data were statistically analyzed with ANOVA, followed by Scheffé's test. Reactmer Bond showed the greatest fluoride release over 70 days ($280.2 \pm 10.1 \mu g/cm^2$) among the materials tested. The values for One-up Bond F and Trial KBF Bond were $83.4 \pm 5.3 \mu g/cm^2$ and $58.6 \pm 1.5 \mu g/cm^2$, respectively. The values were significantly different among the four groups ($p < 0.0001$). Clearfil SE Bond showed almost no fluoride release over 70 days.

Key words: Fluoride release—Dental adhesive

INTRODUCTION

Fluoride-releasing restorative materials, also known as glass-ionomer cements (GICs), are well known to prevent secondary caries formation due to fluoride uptake by the tooth substrate after it is released from the material[4,13]. A concept of minimal intervention (MI) has recently been proposed[16], suggesting the application of fluoride-releasing materials for caries treatment. For effective caries prevention, only extremely small concentrations of fluorides in the immediate vicinity of restorative materials seem to be necessary to inhibit demineralization. Our previous study showed that conventional GICs release higher amounts of fluoride than resin-modified GICs or compomer and that the amount of fluoride release from fluoride-releasing adhesive resin was the smallest among the fluoride-releasing restorative materials[9].

The current development of resin bonding to tooth structures has proven reliable, and it is also essential for esthetic treatment of dental caries[3]. Newly developed resin bonding systems more strongly bond to both enamel and dentin, are more durable after bonding, involve simpler procedures, and are more preventive of secondary caries. In this study, we investigated the fluoride release of three newly developed fluoride-releasing adhesive resins and one conventional adhesive resin as a control.

MATERIALS AND METHODS

As shown in Table 1, three fluoride-releasing dental adhesives (KBF, OB, RB) and one non-fluoride-releasing dental adhesive (SE) were used in this study. Each material was used to fill an acrylic ring (inner diameter: 9.0 mm, height: 1.5 mm) placed on a slide glass, covered with another slide glass, and light cured for 30 sec on both the upper and lower sides, respectively, with New Light VL-2 (GC, Tokyo, Japan). After the acrylic ring was removed from the cured specimen, the result was a disk-shaped specimen of polymerized adhesive (diameter: 9.0 mm, height: 1.5 mm). The disk was then immersed in 5 ml of 10 mM phosphate buffered solution (pH 7.0). After 1, 3, 7, 14, 21, 28, 35, 42, 49, 56, 63 and 70 days, the samples were transferred to new solutions. The fluoride concentration was determined with a combined fluoride sensitive electrode (Model 96-09, Orion Research, Beverly, MA, USA) attached to an ion analyzer (Model 290A, Orion Research) following the addition of TISAB III (Orion Research) at 10 vol% of determined solution. There were five samples of each material, and the data were statistically analyzed by one-way ANOVA, followed by Scheffé's test.

RESULTS AND DISCUSSION

The cumulative amounts of fluoride release from each material are shown in Fig. 1. RB showed the greatest fluoride release over 70 days ($280.2 \pm 10.1 \mu g/cm^2$), significantly more than any of the other three materials ($p < 0.0001$, respectively). Cumulative fluoride releases from OB and KBF were 83.4 ± 5.3 $\mu g/cm^2$ and $58.6 \pm 1.5 \mu g/cm^2$, respectively; these values were also significantly different ($p < 0.0001$). The differences in the amount of fluoride release were probably due to the mechanism involved. Generally, GICs release fluoride from fluoridated alumino-silicate glass, which dissolves into the water, while almost conventional compomers or fluoride-releasing resin composites are contained in the filler. The fluoride in filler is ion-exchanged to OH^- in the immersed solution: therefore the fluoride is released in smaller quantities than that from GICs due to the insufficient supply of H^+ ions under a tightly polymerized hydrophobic resin[10]. Our previous study demonstrated that the cumulative fluoride release from Imperva Fluorobond dental adhesive (Shofu, Kyoto, Japan) was only 11.9 ± 1.3 $\mu g/cm^2$ over 70 days[9] and was smaller than that of the three fluoride-releasing dental adhesive investigated in this study. KBF contains sodium fluoride (NaF) fillers in the base resin. It has been reported that fluoride release from NaF fillers is possibly easier than from fluoridated polymer containing materials, because sodium dissolves quite easily when it comes into contact with water[2,5]. RB has been developed using a new concept; pre-reacted glass-ionomer (PRG) technology, which involves an acid-base reaction of fluoride-containing glass with polyalkenoic acids[8]. The new chemistry underlying these materials might increase the fluoride release. Control SE showed almost no fluoride release over 70 days.

The changes in fluoride release from each material as a function of time are shown in Fig. 2. The fluoride release on the first day reflected the cumulative amounts. RB yielded the largest fluoride release in one day ($37.3 \pm 2.8 \mu g/cm^2$), significantly more than any of the other three materials ($p < 0.0001$). Fluoride releases by OB and KBF in one day were $8.2 \pm 0.5 \mu g/cm^2$ and $6.4 \pm 0.8 \mu g/cm^2$, respectively, and did not significantly differ. All the materials except for SE released fluoride over 70 days. However, the amounts decreased daily.

Table 1 Adhesives investigated in this study

Adhesive	Manufacturer/Composition[1]	Lot No.	Code
Trial ABF	Kuraray, Osaka, Japan	991130	
KBF	MDP Bis-GMA HEMA hydrophilic dimethacrylate photoinitiator microfiller		KBF
Reactmer Bond	Shofu, Kyoto, Japan	040002	RB
Bond A	F-PRG fluoro-alumino sillicate glass initiator water solvent		
Bond B	4-AET 4-AETA HEMA UDMA photoinitiator		
One-up Bond F	Tokuyama Dental, Tokuyama, Japan	450290	OB
bonding agent A	acidic phosphate monomer MAC-10 monomer photoinitiator		
bonding agent B	monomer water fluoro-alumino sillicate glass photoinitiator		
Clearfil SE Bond*	Kuraray, Osaka, Japan	011160	
Bond	MDP Bis-GMA HEMA hydrophilic dimethacrylate photoinitiator microfiller NaF		SE

* also known as Clearfil Megabond in Japan

[1] Composition as provided by respective manufacturer:
Bis-GMA = Bisphenol-glycidyl methacrylate;
MDP = 10-Methacryloyloxydecyl dihydrogen phosphate;
HEMA = 2-hydroxyethylmethacrylate; NaF = Sodium fluoride;
F-PRG = full-reaction type pre-reacted glass-ionomer;
4-AET = 4-acryloxyethyl trimellitic acid;
4-AETA = 4-acryloxyethyl trimellitic anhydride;
UDMA = urethane dimethacrylate;
MAC-10 = 10-methacryloyloxydecamethlene malonic acid

Considering the report that inhibition of fluoride uptake and demineralization at 60 days were larger than those at seven days[5)], this gradual decrease of fluoride may have little impact from a clinical perspective[12)].

Han *et al.* also investigated the fluoride

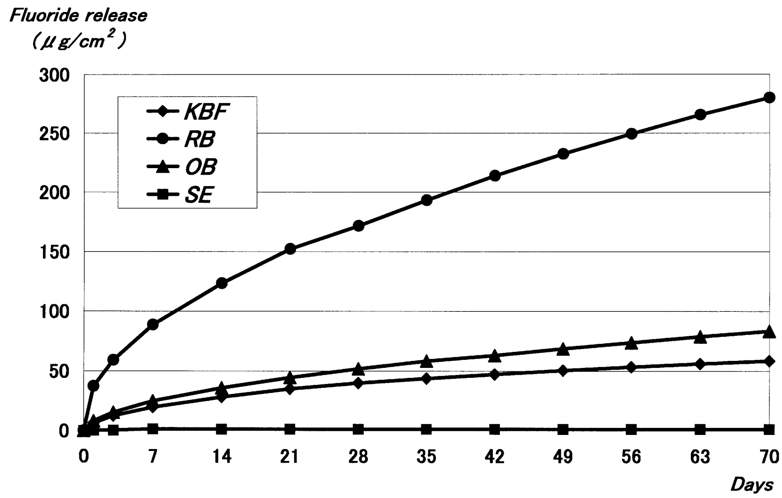

Fig. 1 Cumulative amounts of fluoride release from each material over 70 days.

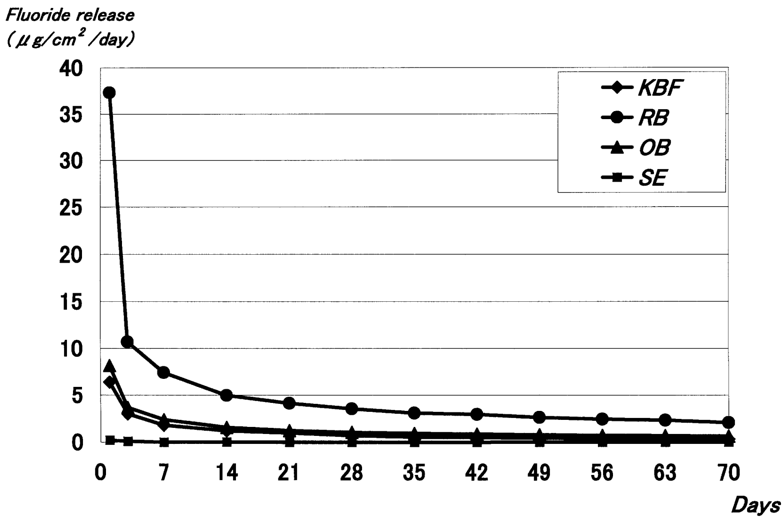

Fig 2 Changes in fluoride release from each material as a function of time.

release of KBF and OB[6]. However, the fluoride releases in their data was significantly smaller than those in our experiments. Their mean cumulative-fluoride releases from KBF and OB during 56 days were 10.5μg/cm^2 and 7.5μg/cm^2, respectively. On the other hand, those values during 56 days in this study were 53.4 $\pm$ 1.4μg/cm^2 and 73.8 $\pm$ 4.9μg/cm^2, respectively. The differences between their and our results may be due to the difference of the immersing solution[1]. They immersed the specimens into deionized water; in contrast, we used phosphate buffer solution. The use of buffer solution for immersion probably does not affect the gradient of ion concentration between the immersing solution and the specimen. Thus the slower fluoride release in their investigation may reflect inhibition.

Recently, caries treatment has been a major issue in two different restorative concepts;

hybridization of tooth structure with resin composite[3] and MI (minimal intervention) with GIC-based restoration[16]. It has been suggested that a resin-unprotected demineralized area exists immediately beneath the hybrid layer in almost all cases, and dentin bonding may be reduced by hydrolysis of acid-damaged dentin protein, thereby reducing the bond durability[11,15]. However, GIC-based restorative materials may degrade in a wet environment and their mechanical properties may deteriorate. Considering some reports that a fluoride-releasing dentin adhesive can result in long-term bond durability[7,14], these materials may genuinely inhibit secondary caries.

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REFERENCES

1) Carvalho, A.S. and Cury, J.A. (1999). Fluoride release from some dental materials in different solutions. *Oper Dent* **24**, 14–19.
2) Donly, K.J. and Gomez, C. (1994). *In vitro* demineralization of enamel caries at restorative margins utilizing fluoride-releasing composite resin. *Quintessence Int* **25**, 291–296.
3) Eick, J.D., Gwinnett, A.J., Pashley, D.H. and Robinson, S.J. (1997). Current concepts on adhesion to dentin. *Crit Rev Oral Biol Med* **8**, 306–335.
4) Erickson, R.L. and Glasspoole, E.A. (1995). Model investigations of caries inhibition by fluoride releasing dental materials. *Adv Dent Res* **9**, 315–323.
5) Han, L., Abu-Bakr, N., Okamoto, A. and Iwaku, M. (2001). Study of the fluoridated adhesive resin cement; Fluoride release, fluoride uptake and acid resistance of tooth structures. *Dent Mater J* **20**, 114–122.
6) Han, L., Edward, C., Okamoto, A. and Iwaku, M. (2002). A comparative study of fluoride-releasing adhesive resin materials. *Dent Mater J* **21**, 9–19.
7) Ikemura, K., Kouro, T. and Endo, T. (1998). A new fluoride-releasing dental adhesive and its bonding durability to teeth under long-term water immersion. *J Adhes Soc Jpn* **34**, 85–97.
8) Ikemura, K., Nishino, M., Tomita, S. and Endo, T. (1998). A SIMS study on fluoride ion uptake into dentin substrate derived from a new pre-reacted glass-polyalkenoate filler in a dental adhesive. *J Adhes Soc Jpn* **34**, 334–343.
9) Kameyama, A., Tsukamoto, R., Haruyama, C., Nakazawa, Y., Hirai, Y., Koga, H., Tomori, T., Ishihara, H., Matsukubo, T. and Takaesu, Y. (1999). A study on fluoride release and uptake from various restorative materials *in vitro*. *The Shikwa Gakuho* **99**, 383–392. (in Japanese)
10) Kawai, K., Tantbirojn, D., Kamalawat, A.S., Hasegawa, T. and Retief, D.H. (1998). *In vitro* enamel and cementum fluoride uptake from three fluoride-containing composites. *Caries Res* **32**, 463–469.
11) Kiyomura, M. (1987). Bonding strength to bovine dentin with 4-META/MMA-TBB resin. Long-term stability and influence of water. *J Jpn Dent Mater* **6**, 860–872. (in Japanese)
12) Muller, U., Kielbassa, A.M., Schulte-Monting, J. and Hellwig, E. (2000). Fluoride release from light-curing restorative materials. *Am J Dent* **13**, 301–304.
13) Nagai, Y., Inaba, D., Minami, K. and Matsuda, K. (2000). The effects of fluoride-releasing restorative materials on inhibition of secondary caries *in vitro*. *Cariology Today* **1**, 16–19.
14) Saito, A. (1996). Effect of fluoride in adhesion to dentin. *J Jpn Dent Mater* **15**, 78–88. (in Japanese)
15) Spencer, P. and Swafford, J.R. (1999). Unprotected protein at dentin-adhesive interface. *Quintessence Int* **30**, 501–507.
16) Tyas, M.J., Anusavice, K.J., Frencken, J.E. and Mount, G.J. (2000). Minimal intervention dentistry—a review. *Int Dent J* **50**, 1–12.

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