Inhibitory Effect and Physical Properties of Dental Glass-ionomer Cements

Kunio Wakasa, Hideaki Shintani*, Kazumi Kimura*, Kousei Tanaka*, Hidenori Urabe*, Naoki Satou* and Yasuhiro Yoshida

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ABSTRACT

An inhibitory effect of bacteria against the surface of dental glass-ionomer cements (GICs) was examined using six types of bacteria strains. Also, fluoride release, water uptake and solubility were measured in order to clarify their physical properties related to the surface of hardened GICs. The released amount of fluoride from the cements for 24 hours was higher in light-activated lining cement (VB) and luting cement (HY) than the other GICs. For 48 and 72 hours (immersion period), their released amounts were still detected. An inhibitory (antibacterial) effect estimated by the inhibitory zone size was observed in the hardened GIC (VB) for all bacteria strains, or one bacteria strain for GICs FL and HY. Examining the liquid and powder components in light-activated GICs, all liquid components showed an inhibitory effect, but the powder component of VB showed the effect and the powder component of FL no effect. The bacterial inhibility on the surface of hardened GICs was observed for light-activated GICs and also the light-activated one with greater released fluoride had the bacterial inhibility for all bacteria strains.

INTRODUCTION

Dental glass-ionomer cement (GIC) restorative materials have been used clinically in dental field, and the dental use has increased as compared with composite resins which promoted bacterial growth[1,2]. Their cements were classified into light-activated lining cement, chemically-cured lining cement and luting cement for restorations[3-5]. The GIC showed the decreased physical property and rougher surface[6,7], and it is expected that dental light-activated GICs by visible light have the improved adhesion (chelate linkages to dentine) and also the antibacterial effect[8-13]. Dental light-activated GIC hardened initially with light activation, and continuously resulted in setting reaction by HEMA (hydroxyethyl methacrylate; liquid component) and pendant methacrylate groups of polymer component[14]. Conventionally, the acid/base setting reaction occurred with polyacrylic acid (liquid component) and aluminosilicate cement (powder component)[12,13]. The cement powder was composed of the mixture of silica and alumina (melting temperature range; 1100 to 1500°C, which was melted by adding such fluxes as CaF_2 (1360°C), Na_3AlF_6 (1020°C), NaF (992°C) and $AlPO_4$ (1000°C)[15]. The elements of F, Al, Ca, and Na were identified by the solubility test, but the amount of the compounds added to the powder in commercial GICs was not analysed[15-21]. It is thus important to measure eluted fluoride from GICs in order to confirm the difference of composition in the GICs. This study was to examine the inhibitory effect against the surface of dental GICs which were exposured to six types of bacteria strains, and to measure such physical properties as released fluoride amount, water uptake and solubility in distilled water from the surface of GICs.

MATERIALS AND METHODS

1. Dental glass-ionomer cements

Six dental GICs (VB, FL, LC, KB, FI, HY) examined in

Department of Dental Materials, School of Dentistry Hiroshima University, Kasumi 1-chome, Minamiku, Hiroshima City, 734-8553 Japan (Chairman: Professor M. Yamaki).

* Department of Operative Dentistry, School of Dentistry Hiroshima University, Kasumi 1-chome, Minamiku, Hiroshima City, 734-8553 Japan (Chairman: Professor H. Shintani).

Dr Kunio Wakasa to whom all correspondence should be addressed.

Table I Dental glass-ionomer cements tested. Three types of cements were light-activated lining cement, conventional chemically-cured lining cement and luting cement for restorations.

Code	Material	Batch No. powder/liquid	Manufacturer
VB	Vitrabond	028Y0265	3M Co.
FL	Fuji Lining LC	210902/040901	GC Co.
LC	GC Lining Cement	310794/180791	GC CO.
KB	Ketac Bond	61BY96	ESPE Co.
FI	Fuji Bond	240112/140611	GC Co.
HY	HY-Bond Glassionomer-C	038854/038853	Shofu Inc.

this study are listed in Table I. They were classified into three types: light-activated lining cements, VB and FL; conventional chemically-cured lining cements, LC and KB; and luting cement for restoratives FI and HY. NaF (1 wt% concentration) aqueous solution (Katayama Kagaku Kougyou, Osaka) and Cephacrol (Bacteria sensitive agent; Showa Disc, Showa Yakuhin Kakou, Tokyo) were used, to compare them with the mixing liquid component of light-activated GICs.

2. Inhibitory effect and physical properties

(1) Inhibitory effect

The light-activated GIC sample (0.75 mm diameter × 0.20 mm height) was filled into a polypropylen tube with 0.75 mm in a diameter × 0.20 mm long, and then cured by visible light after loading at 4.9 N on a glass slab. Conventional cement powders were mixed with distilled water and filled into the tube and loaded. After hardening, the samples were kept in a dessicator at 25° C, because a chemical setting reaction continued for up to 24 hours[14]. These GICs were used under the following test conditions. The inhibition test was done using six types of bacteria strains: *Streptococcus mutans (S. mutans) Ingbritt (IB) 1600, Streptococcus mutans (S. mutans)* OMZ175, *Streptococcus sobrinus (S. sobrinus)* OMZ176, *Streptococcus sobrinus (S. sobrinus)* 6715–13, *Streptococcus sanguis (S. sanguis)* ATCC10556, *Streptococcus salivarius (S. salivarius)* ATCC9222. The bacteria strains were routinedly cultivated in Brain Heart Infusion (BHI) matrix (Difco Laboratories, Tokyo) for 48 hours. The bacteria test was carried out using a disc diffusion method[10,11]. Disc-shaped matrix was 85 mm in diameter and 4 mm thick. Their test GIC samples (0.75 mm diameter × 0.20 mm height) which were placed on the BHI matrix were incubated at 37° C for 24 hours after removal of the water on the sample surface, where the bacteria was cultivated with O.D. 600 (logarithmic forming period) = 0.7 to 0.8. The difference between inhibitory zone diameter and cement diameter (0.75 mm) was determined as the corrected inhibitory zone size for nine samples at each test.

(2) Physical properties

The release from the hardened GICs (0.75 mm diameter × 0.20 mm height) was measured with a fluoride ion meter (Orion Research Inc, Boston, NY, USA) and composite fluoride ion electrode (96–09, Orion Research Inc). To investigate the presence or absence of interference by ions such as Al^{3+} and Fe^{3+}, a solution of known fluoride concentration was added to the eluate from the GICs to determine the true concentration of released fluoride.

Water uptake and solubility were measured using the following samples. The GICs which were kept in a dessicator at 25° C were weighed repeatedly until the weight reached to a constant value (Wo). One sample was immersed into the bottle for 3, 10, 30, and 60 days, and sample weight was measured after removing the water on the sample surface by allowing to dry in air. Water uptake in the cements (0.75 mm diameter × 0.20 mm height) was calculated by the equation.

$$\text{Water uptake (wt\%)} = ((Wa - Wo) / Wo) \times 100$$

where Wa is the weight (mg) after immersion for 3, 10, 30 and 60 days in distilled water (25 mL; 37° C).

After immersing a test sample into distilled water, it was dried at 100° C in air for 24 hours and the weight was measured.

Table II Bacterial inhibitory zone size of glass-ionomer cements and the control solutions (NaF and Cephacrol). The values (mm) were measured as described in the text. Symbols mean as follows: –, not detected; +, very little detected; ±, not detected or very little detected. Mean ± standard deviation of the values.

Code	*S. mutans* Ingbritt 1600	*S. mutans* OMZ 175	*S. sobrinus* OMZ 176	*S. sobrinus* 6715–13	*S. sanguis* ATCC 10556	*S. salivarius* ATCC 9222
VB	1.33 ± 0.54	2.88 ± 0.60	2.13 ± 1.44	0.96 ± 0.67	2.67 ± 1.09	1.63 ± 0.41
FL	–	+	+	–	0.12 ± 0.21	–
LC	–	–	–	–	–	–
KB	–	–	–	–	–	–
FI	–	±	±	–	–	–
HY	0.19 ± 0.33	±	–	–	±	–
NaF 1%	9.94 ± 2.06	10.27 ± 2.90	3.81 ± 0.54	5.59 ± 2.41	6.00 ± 12.0	3.38 ± 0.32
Cephacrol	19.20 ± 4.99	20.96 ± 1.33	14.69 ± 1.24	25.44 ± 3.04	24.28 ± 1.07	13.76 ± 0.97

$$\text{Solubility (wt\%)} = ((Wo - Ws)/Wo) \times 100$$

where Ws is the weight (mg) after drying the sample which was immersed at an immersion period (3 to 60 days) and Wo the initial weight (mg) when used in water uptake test. The amount of solubility was measured for the same sample as water uptake test. Five samples at each immersion period were used for each GIC sample.

RESULTS

Table II indicates the inhibitory effect against the bacterias (antibacterial effect) on the set surface of GICs as a linear dimension (mm). The solutions NaF (1% concentration) and Chephacrol as control samples showed the antibacterial effect for all bacteria strains tested. Conventional lining GICs (LC and KB) showed no antibacterial effect, but light-activated GIC (VB) showed the antibacterial effect for all bacteria strains and FL showed it for one bacteria strain, *S. sanguis* ATCC10556. HY showed the antibacterial effect only for *S. mutans* IB1600. In the case of light-activated GICs composed of liquid and powder components, their components in VB had antibacterial effect for all bacteria strains,but only liquid component in FL had antibacterial effect for all bacteria strains although the powder component had no antibacterial effect. The GIC VB significantly showed the antibacterial effect between VB and the other GICs ($p < 0.05$). Fig. 1 shows the released fluoride from the cements, indicating that VB had the highest amount and HY the second highest after 24 hours' immersion (first day) and the other

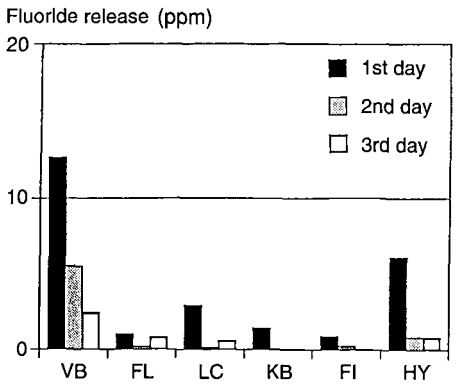

Figure 1 Fluoride amounts released from glass-ionomer cements at first, second and third day, as described in text. Unit = ppm.

GICs had a very small amount at each immersion day. VB showed greater amount after 48 hours (second day), as compared with the other GICs. There appeared a significance difference of released fluoride at first day between VB and the other GICs (FL, LC, KB, FI), or between VB and HY ($p < 0.05$).

Table III indicates water uptake in the GICs tested. Water uptake in LC and HY was the largest at 3 days, and also KB at 10 days and VB, FL and FI at 30 days had respectively the largest amount. In Table IV (solubility), each of six GICs had respectively the constant solubility amount (0.60 to 1.17%) at each immersion day (3 to 60 days). Of dental GICs tested light-activated GIC FL had the smallest amount (0.60%), and VB the second smallest amount (0.81%).

Table III Water uptake (wt%) of the cements. Mean ± standard deviation.

Code	Water uptake (%)			
	3 days	10 days	30 days	60 days
VB	0.17 ± 0.03	0.11 ± 0.03	0.28 ± 0.08	0.22 ± 0.03
FL	0.35 ± 0.04	0.32 ± 0.03	0.59 ± 0.14	0.52 ± 0.08
LC	0.57 ± 0.23	0.21 ± 0.12	0.29 ± 0.05	0.29 ± 0.05
KB	0.21 ± 0.13	0.75 ± 0.30	0.14 ± 0.15	0.02 ± 0.06
FI	0.15 ± 0.03	0.14 ± 0.04	0.25 ± 0.10	0.21 ± 0.04
HY	0.34 ± 0.18	0.25 ± 0.16	0.28 ± 0.13	0.27 ± 0.08

Table IV Solubility (wt%) of the cements. Mean ± standard deviation.

Code	Solubility (%)			
	3 days	10 days	30 days	60 days
VB	0.81 ± 0.03	0.81 ± 0.04	0.81 ± 0.04	0.81 ± 0.04
FL	0.60 ± 0.06	0.60 ± 0.06	0.60 ± 0.06	0.60 ± 0.06
LC	1.17 ± 0.45	1.17 ± 0.45	1.17 ± 0.45	1.17 ± 0.45
KB	0.85 ± 0.22	0.85 ± 0.22	0.85 ± 0.22	0.85 ± 0.22
FI	1.13 ± 0.10	1.13 ± 0.10	1.13 ± 0.10	1.13 ± 0.11
HY	1.05 ± 0.20	1.05 ± 0.22	1.15 ± 0.23	1.05 ± 0.23

DISCUSSION

Chemical setting reaction in a light-activated GIC continued for up to 1 day while light activation produced a rapid initial setting[14]. In a case of the light-activated GICs which were composed of the liquid (including HEMA) and powder components, a dual setting reaction at light activation resulted in the setting due to a polymerisation of HEMA molecules and the methacrylate groups of the polymer molecules[14,16]. The release of fluoride was detected for the light-activated lining GIC similar to a chemically-cured one and the antibacterial effect might be expected to occur in the GICs[10,17]. Regarding to the antibacterial effect on the surface of the GIC, we measured the released fluoride and bacterial inhibility using six types of bacteria strains. The highest released fluoride amount was achieved with the smallest amount of solubility from the cement[18]. Conventional chemical-cured lining GICs (LC, KB) showed no inhibility (Table II), and a small amount of fluoride was detected as compared with the other GICs (Fig. 1). A light-activated GIC (VB) had the antibacterial effect to bacteria strains, associated with greater amounts of released fluoride (Table II, Fig. 1). A luting GIC (HY) had also the antibacterial effect with second greater release of fluoride.

HEMA in aqueous solution had an antibacterial agent and was activated clearly by an addition of sodium salicylate to it[19]. This suggests that HEMA works as a bactericidal agent. The liquid component in light-activated GICs showed the antibacterial effect, because the mixture containing HEMA was used as one solution. The fluoride compounds were contained within the powders in light-activated GICs, supposing that fluoride was released from the cement powder. The liquid components of GIC tested (VB) were composed of polyacrylic acid (35 wt%), HEMA (31 wt%) and water (34 wt%)[20,21]. Imazato et al found that HEMA release was observed for the light-activated GIC (VB)[20]. The related property to antibacterial effect is not clear, but they do describe that the HEMA as the liquid component is effective as a bacterial inhibition and the possible factor might be the re-

lated release of fluoride. In our study, the release test of fluoride found the released amount to be significantly greater in GIC (VB) with antibacterial effect than in the other GICs. Generally, fluorite (CaF_2), aluminum trifluoride (AlF_3) and sodium fluoride (NaF) are present in commercial GICs[11-22], but it is not known whether HEMA solution interferes with the release of fluoride.

CONCLUSION

This study summarized that the an antibacterial effect was not observed in chemically-cured lining GICs, but clearly in light-activated GICs using six types of bacteria strains, similar to one of luting GICs. One of light-activated GICs, which showed the inhibility for all bacteria strains tested, had a greater released fluoride at lower levels of solubility during solubility test in distilled water.

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