ORIGINAL ARTICLE

Porous Hydroxyapatite Can Improve Strength and Bioactive Functions of Glass Ionomer Cement

Takako NISHIMURA[1], Yukari SHINONAGA[2], Yoko ABE[2], Saki KAWAI[2], and Kenji ARITA[2]

[1]*Graduate School of Dentistry (Department of Pediatric Dentistry), Osaka Dental University,*
[2]*Department of Pediatric Dentistry, Osaka Dental University, Osaka, Japan*

Synopsis
The aim of this study is to investigate the role of hydroxyapatite (HAp) in novel apatite ionomer cement (AIC), an improved glass ionomer cement (GIC) that contains porous HAp. AIC samples were prepared using the latest conventional GIC, Fuji IX GP Extra™, with porous and spherical HAp (HApS), and were subjected to mechanical strength measurements, fluoride release test, scanning electron microscopy observations and multi-mineral release test, and were compared with IX-Ex, Fuji IX GP™ (IX-GP) and S-PRG filler containing composite resin, Beautifil II™ (GIOMER). The flexural strength and fluoride release properties of AIC were significantly higher than those of IX-Ex. The release of fluoride, aluminum, silicon and phosphorus from AIC specimens was greater when compared with IX-Ex and GIOMER. Therefore, porous HAp improves both the strength and bioactive functions of GIC without losing the advantages of conventional GIC.

Key words: glass ionomer cement, hydroxyapatite, ion release, bioactive functions

Introduction
Glass ionomer cement (GIC) was invented by Wilson and Kent in 1971 [1]. GIC is widely used as a dental material, due to its ease of use, low coefficient of thermal expansion, good biocompatibility with bone pulp tissue, and long-term bonding to tooth surfaces and metals [2,3]. In addition, its unique fluoride (F) ion release characteristics have anticaries, antimicrobial and remineralization effects [4,5]. However, its current uses are limited because of its inadequate strength. Several researchers have attempted to overcome these poor mechanical properties by adding reinforcements, but sufficient improvements in mechanical and chemical properties have not yet been achieved [6-8]. As hydroxyapatite (HAp) has great biocompatibility and a composition similar to apatite in the human tooth, Nicholson *et al*. [9] first attempted to in-corporate HAp into GIC prepared from oxide glass and F glass and concluded that the properties of GIC were not affected by the presence of HAp. However, other researchers reported that addition of HAp into commercially prepared GICs improved the mechanical properties [10,11]. In our previous studies, it was demonstrated that the addition of HAp particles was able to enhance both the mechanical and chemical properties of a conventional GIC used for dental restoration, Fuji IX GP™ (IX-GP; GC Corp., Tokyo, Japan) [12-14]. Moreover, we confirmed that the most suitable HAp for incorporation into GIC was porous HAp with a high specific surface area [14]. However, further investigations are necessary to clarify the mechanisms responsible for the improvement in mechanical and chemical properties by adding HAp to GIC.

The purposes of this study were to investigate the effects of HAp using the latest commercialized GIC, Fuji IX GP Extra™ (IX-Ex; GC Corp.), which is an improved formulation of IX-GP, and to investigate the mechanisms for the improvement of mechanical and functional properties with addition of HAp. Moreover, we compared the elemental release properties of our novel GIC containing porous HAp, referred to herein as apatite ionomer cement (AIC), and functional S-PRG filler-containing composite resin, which was developed and introduced recently [15], commonly known as GIOMER.

Materials & Methods

1. Investigation for HAp's adaptation to latest GIC

1) Preparation of AIC specimens

Conventional posterior glass ionomer restorative cement, IX-Ex (lot nos. 1205081 and 1312051), was used as the control and base material in AIC groups. IX-Ex is the latest GIC, having improved translucency and F release properties, as compared with IX-GP. IX-Ex-powder is mainly composed of fluoro-aluminosilicate glass containing silicon (Si), aluminum (Al), sodium (Na) and strontium (Sr). IX-Ex-liquid is composed of polyacrylic acid, polybasic carboxylic acid and distilled water. Each AIC powder was prepared by replacing 6, 12, 18 and 24 wt% IX-Ex-powder with spherical HAp (HApS; Taihei Chemical Industrial Co., Ltd., Osaka, Japan), followed by mixing with IX-Ex-liquid at a powder/liquid ratio (P/L) of 3.4, in accordance with instruction manual of IX-Ex (6%-, 12%-, 18%- and 24%-AIC, respectively). In addition, IX-GP (lot no. 1012241) was also prepared as a negative control.

2) Mechanical properties

(1) Flexural strength tests

Beam-shaped samples of IX-GP, IX-Ex and AICs measuring 20 mm × 3 mm × 3 mm for flexural strength test were prepared in a stainless-steel split mold (n = 6/group). Cement mixtures were condensed into the mold and clamped under a 500 g load for 10 minutes at room temperature, and were then unclamped and kept for 50 minutes at 100% humidity at 37°C, followed by storage in artificial saliva (Saliveht™ Aerosol, Teijin Ltd., Osaka, Japan) for 23 hours. Samples for flexural strength test were then subjected to a three-point bending test using a universal testing machine (AGS-X, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 0.5 mm/min.

(2) Compressive strength test

Cylindrical samples of IX-GP, IX-Ex and AICs measuring 4 mm in diameter × 6 mm in height for compressive tests were prepared in a stainless-steel split mold, following the procedures outlined in ISO9917-1 (n = 6/group). After 24 hours of storage in artificial saliva, samples were tested using a universal testing machine (AGS-X, Shimadzu Corp.) at a crosshead speed of 1 mm/min.

3) Fluoride ion release test

For F ion release measurement, IX-GP (n = 6), IX-Ex and AIC (n = 4/group) samples measuring 10 mm in diameter × 2 mm in thickness were prepared using a polyethylene split mold. Mixed IX-GP, IX-Ex and AICs were placed into a mold on a glass plate, covered with celluloid strips and pressed with another glass plate under 500g load for 10 minutes. Specimens were kept at 37°C and 100% relative humidity for a further 50 minutes, and were then gently removed from molds. Samples were individually suspended in 8 mL of deionized water in sealed containers and stored at 37°C. For measurements, each disk was removed from water, washed with 2 mL of deionized water over the immersed water, dried on filter paper, and immediately immersed in 8 mL of fresh deionized water for further measurement. F ion concentrations were measured every day for 30 days using an F ion-selective electrode (Orion 9609 BNWP, Thermo Fisher Scientific Research, Boston, MA, USA) connected to an ion analyzer (Orion 2115010 Dual Star pH/ion-meter, Thermo Fisher Scientific Research). Next, 1.0 mL of total ionic strength adjustment buffer III solution (TISAB III; Orion 940911, Thermo Fisher Scientific Research) was added to provide a constant background of ionic strength. All measured concentrations were expressed as mass/surface area ($\mu g/cm^2$).

2. Evaluation of functional properties of AIC based IX-Ex

1) Scanning electron microscopy (SEM) observation

SEM (SU-70, Hitachi High-Technologies Co., Tokyo, Japan) was used to observe the microstructure of HApS particles. The fractured surfaces of IX-Ex and 18%-AIC samples after flexural strength test were observed by SEM (S-4800, Hitachi High-Technologies Co.) in combination with energy dispersive X-ray spectrometry (EDS; IncaPentaFETx3, Oxford Instruments PLC., Oxford, UK). Before SEM observations, specimens were sputter coated with osmium using a plasma multi coater (PMC-5000, MEIWAFOSIS Co., Ltd, Tokyo, Japan). Moreover, cross-sectional SEM specimens were prepared using a cross-section polisher (SM-09020CP, JEOL, Tokyo, Japan) and backscatter electron imaging was performed using a field-emission SEM system (JSM-6701F, JEOL).

2) Multi-mineral release test

In multi-mineral release test, S-PRG filler-containing resin Beautifil II^{TM} (GIOMER; SHOFU INC., Kyoto, Japan: lot no. 121318), which is a known functional material, was used as a positive control. IX-Ex and 18%-AIC samples measuring 10 mm in diameter $\times$ 2 mm in thickness was prepared, as described in Experiment 1 (n = 4/group). GIOMER was placed into the mold, covered with celluloid strips and pressed between two glass plates to the thickness of the mold, and was cured using a dental light-cure device (Newlight VL-II, GC Co.) for 20 seconds on each side. Samples were individually suspended by a thread in 18 mL of deionized water for 24 hours at 37℃. For measurements, each disk was removed from the water, and washed with 2 mL of deionized water over the immersed water. Concentrations of Al, Si, phosphate (P), calcium (Ca) and Sr released from the samples were assessed using inductive coupled plasma atomic emission spectroscopy (ICP-AES; ICPS-8100, Shimadzu Co.). F ion concentration was measured using an ion analyzer (D-53, Horiba Ltd., Kyoto, Japan) and F electrode (6561-10C, Horiba Ltd.).

3. Statistical analysis

Differences in test results were analyzed by one-way ANOVA and Tukey's test, and p-values of less than 0.05 being regarded as significant.

Results

1. Experiment 1

1) Mechanical properties of AIC based on IX-Ex

The results of flexural and compressive strength tests for IX-GP, IX-Ex and AICs are shown in Table 1. The flexural strength of IX-Ex was significantly lower than that of IX-GP ($p<0.01$), while the flexural strength of 12%-AIC was significantly higher than that of IX-Ex ($p<0.01$). Furthermore, there were no significant differences in flexural strength between IX-GP and 12%- and 18%-AICs ($p>0.05$), and although 6%- and 18%-AICs tended to have increased flexural strength when compared with IX-Ex, the differences were not significant. Conversely, 24%-AIC tended to have decreased flexural strength. The compressive strength of IX-Ex and AICs was also significantly lower when compared with that of IX-GP. There were no significant differences in compressive strength between IX-Ex and AICs.

Table 1 Flexural and compressive strength of IX-GP, IX-Ex and AICs
ANOVA/Tukey's test; *: $p<0.05$, **: $p<0.01$, ***: $p<0.001$

Group	Flexural strength (MPa)	Compressive strength (MPa)
IX-GP	27.3 ± 4.2	155.2 ± 11.1
IX-Ex	18.9 ± 1.6	119.7 ± 8.6
6%-AIC	19.8 ± 3.4	122.1 ± 16.5
12%-AIC	27.6 ± 5.9	127.2 ± 7.4
18%-AIC	20.7 ± 2.7	129.0 ± 9.0
24%-AIC	17.9 ± 4.0	115.6 ± 12.0

2) Fluoride ion release properties of AIC-based IX-Ex

The accumulated F ion release for IX-GP, IX-Ex and AICs is shown in Fig 1. F release for IX-Ex was significantly higher than that for IX-GP after 30 days, and the accumulated F release concentration of IX-Ex on 30th day was about eight-fold higher when compared to that of IX-GP. Moreover, 6%-, 12%- and 18%-AICs tended to have higher levels of F release when compared with IX-Ex for 30 days after mixing. In particular, the accumulated F release dose of the 18%-AIC specimen up to 26th day after mixing was significantly higher when compared that of IX-Ex ($p<0.05$). Moreover, 18%-AIC had 1.2-fold higher F release dose on the 30th day (1049.6 ± 50.2 μg/cm^2) than that of IX-Ex (849.6 ± 180.3 μg/cm^2). In contrast, F release for the 24%-AIC specimen (752.8 ± 92.1 μg/cm^2) was lower, but was not significantly different from that for IX-Ex.

2. Experiment 2
1) SEM observation

Figure 2 shows a representative image of a HApS particle used in AIC. HApS particles were spherical and about 20 μm in diameter (Fig. 2-A and B), and were formed by aggregation of innumerable nano-sized HAp particles (Fig. 2-C and D). Figure 3 shows compositional SEM images of IX-Ex and 18%-AIC. Innumerable nano-particles from the HApS were found to be dispersed within the matrix of 18%-AIC (Fig. 3-B), as compared with IX-Ex (Fig. 3-A).

Two representative images of HApS particles in 18%-AIC are shown in Fig. 4-A and B. In Fig. 4-A, HApS were integrated within the matrix of AIC. The matrix components of AIC infiltrated into HApS particles, which is indicated by the asterisk in Fig. 4-B-2. Based on the results of EDS point analysis, it was confirmed that the structure was matrix like.
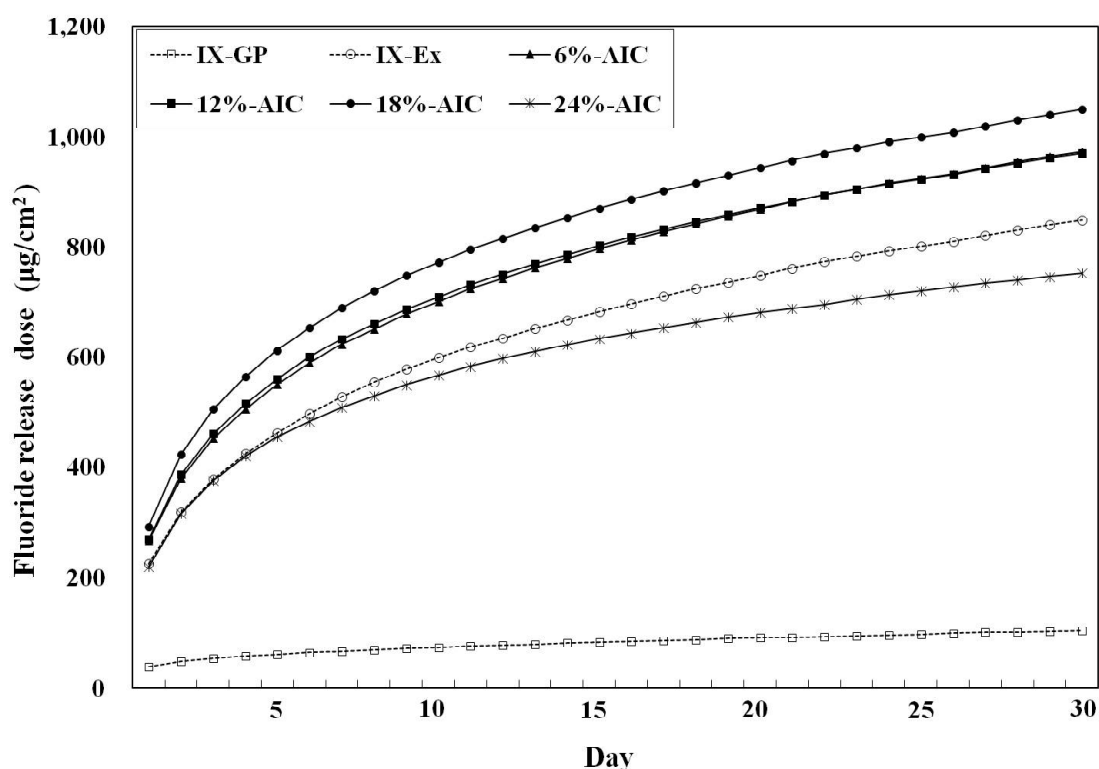

Fig. 1 Accumulated fluoride release from IX-GP, IX-Ex and AICs
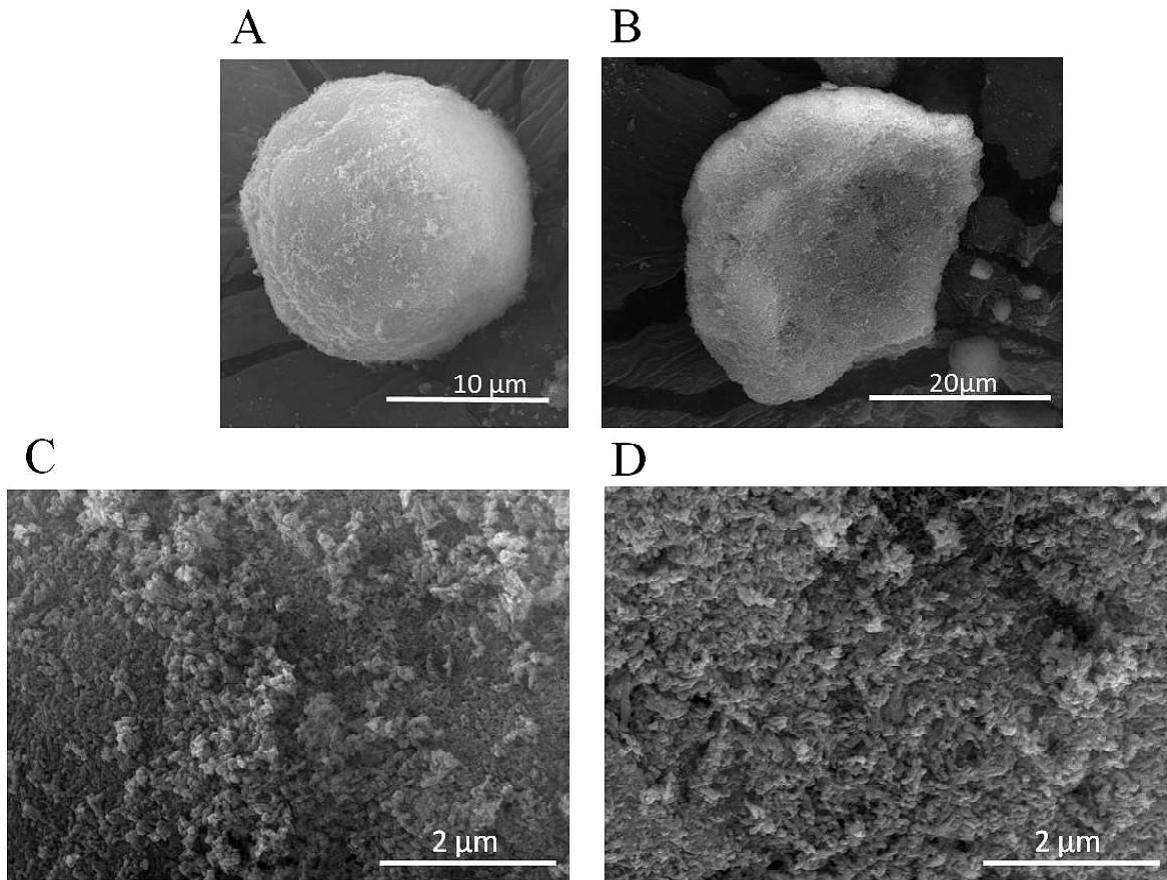
A

B

C

D

Fig. 2 SEM images of HApS
A: HApS powder, B: Broken HApS particle, C: Surface of HApS (high magnification of A),
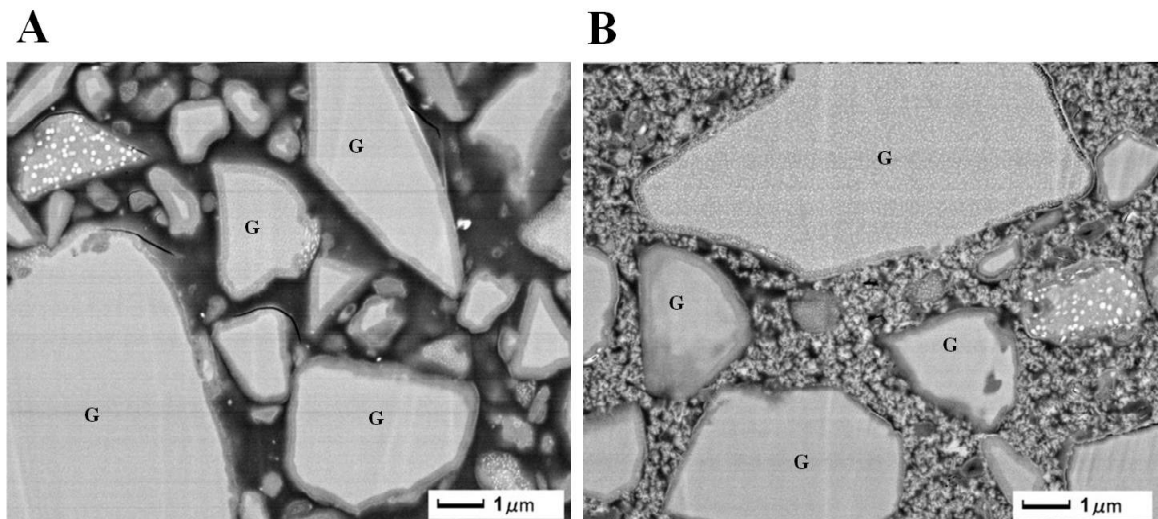D: Interior of HApS (high magnification of B)

A

B

Fig. 3 Compositional SEM image of IX-Ex (A) and 18%-AIC (B) in backscattered electron mode. Abbreviations:
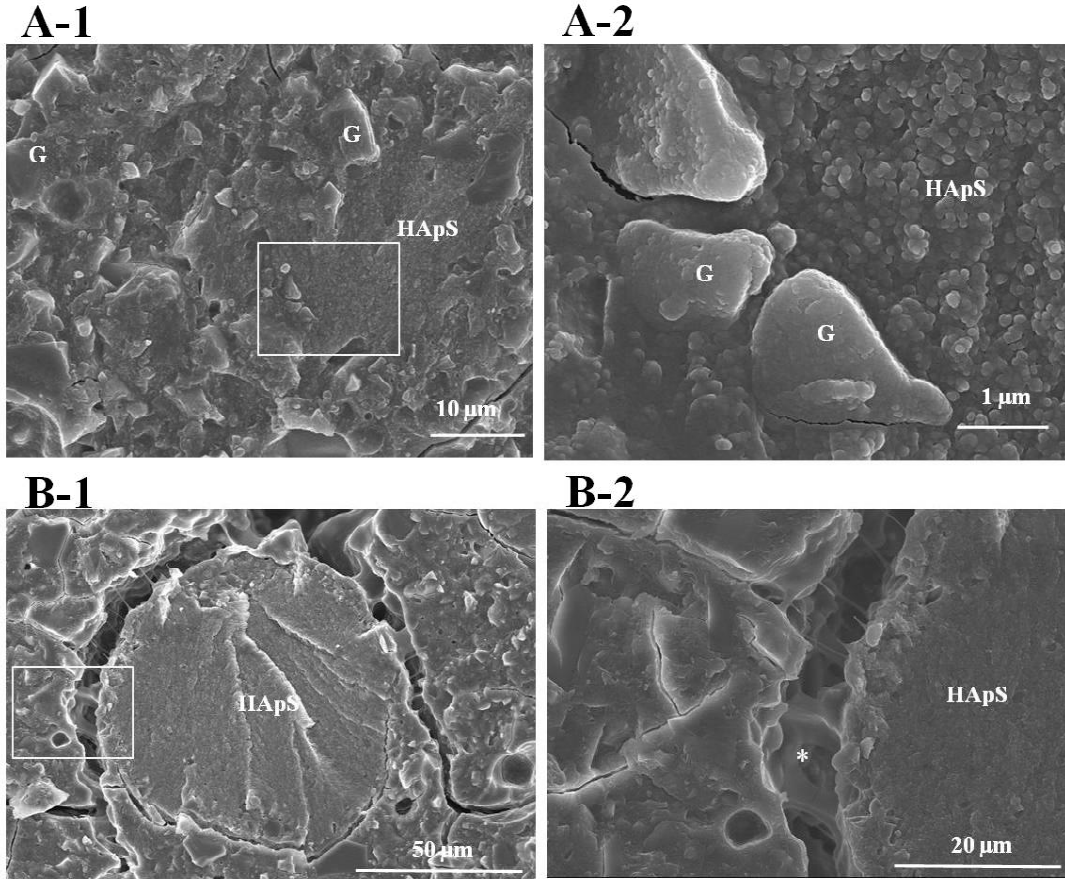G: IX-Ex-glass-core

Fig. 4   Representative images of HApS particles in 18%-AIC
A-1: HApS were integrated in matrix of AIC, A-2: High magnification of A-1,
B-2: AIC matrix component infiltrated into HApS particles,
B-2: High magnification of B-1
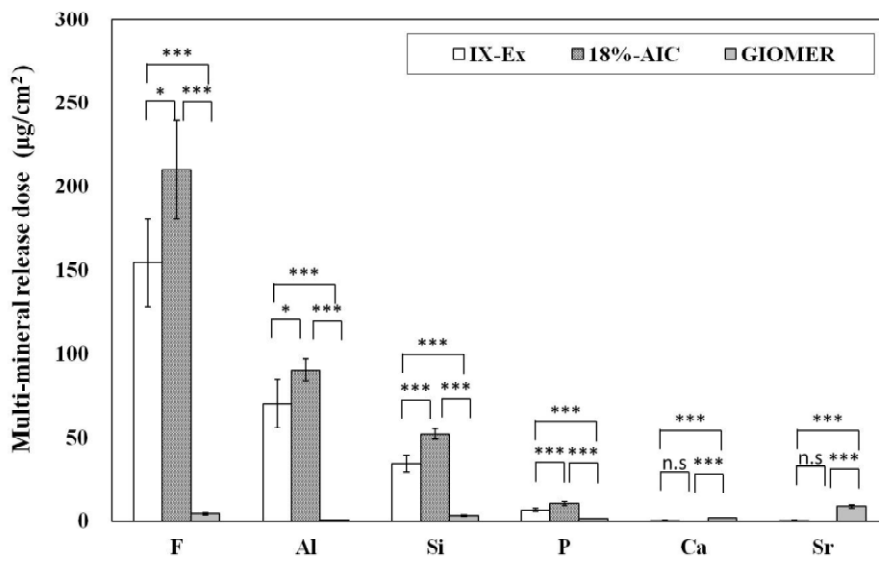Abbreviations: G: IX-Ex-glass-core, *:EDS analysis point.

Fig. 5   Mineral release properties of IX-Ex, 18%-AIC and GIOMER
ANOVA/Tukey's-test; *: $p<0.05$, **: $p<0.01$, ***: $p<0.001$, n.s.: no significant difference

2) Multi-mineral release test

Figure 5 shows the results of ICP analysis (Al, Si, P, Ca and Sr) and F ion release tests for IX-Ex, 18%-AIC and GIOMER. The release of F, Al, Si and P from 18%-AIC was significantly higher when compared with IX-Ex and GIOMER. The release of Ca and Sr from AIC was significantly lower when compared with GIOMER. There were no significant differences in Ca and Sr release dose between IX-Ex and 18%-AIC.

Discussion

IX-GP is one of the strongest commercially available restorative GICs. Therefore, it has been widely used for atraumatic restorative treatment (ART) [16-19]. In our previous study, IX-GP was used as a base material for AIC, and it was confirmed that HAp enhanced both the mechanical and chemical properties of the conventional restorative GIC [12-14]. In recent years, IX-Ex has been commercialized as a GIC with unsurpassed wear resistance, compressive strength and durability, and has been promoted as containing a next generation glass filler, Smart-Glass™, with higher translucency, extremely high (about six times higher) F release and faster setting, as compared to IX-GP. Several researchers have demonstrated its excellent F release properties [20,21] and translucency [22]. In the present study, the mechanical properties and F release properties of IX-Ex were first compared with those of IX-GP. According our results, the flexural and compressive strengths of IX-Ex were significantly lower than those of IX-GP. However, porous HApS was able to enhance the flexural strength of IX-Ex to a comparable level as IX-GP (Table 1). F release from IX-Ex was already eight-fold greater when compared IX-GP. And furthermore, F release from AIC was markedly higher than IX-GP (Fig. 1).

In our preliminary study, it was confirmed that the most suitable ratio for HAp addition in AIC depended on the base GIC; for example, the best ratio of HAp for AIC based on IX-GP was 8% [12-14]. According to the results of "Experiment 1", we decided the optimal HApS ratio for IX-Ex-AIC would be 18%, and performed further examinations in "Experiment 2", as the

rate of HApS may produce further improvement in F release properties without reducing the mechanical properties of IX-Ex (Table 1 and Fig. 1).

Numerous studies have focused on F release from GIC [20,21,23,24], because F release is an important property of GIC and may assist in the prevention of secondary caries, acceleration of remineralization of dentin, and antibacterial properties [5,25-27]. In the present study, it was found that AIC offers improved F ion release properties, despite being produced by simply adding HAp to IX-Ex (Figs. 1 and 5). With regard to F components, they were present only in the IX-Ex-powder, i.e., fluoro-aluminosilicate glass, and AIC releases greater amounts of F ions than IX-Ex, despite the lower F content in AIC (Fig. 1 and 5). It has been reported that the F released from GIC is mainly from the matrix [28,29]. SEM observations of the AIC fracture surface in the present study indicate that the matrix had infiltrated HApS particles (Fig. 4). It is also possible that the HApS, glass cores, and matrix may react with one another, as the compositional SEM image of AIC (Fig. 3) indicated that the hydroxyapatite became dispersed and dissolved. In addition, HAp is a well-known biocompatible functional material for use in ion exchange and adsorbent materials, and as a catalyst. Therefore, HAp appears to improve the movement of F ions from the IX-Ex glass core to the matrix layer. However, some researchers have concluded that F release depends on GIC surface degradation or dissolution of the glass core [30,31]. We have not investigated the relationship between F release and solubility of AIC, and this requires further study.

Moreover, we examined the release properties of several minerals from AIC in addition to F, and this was compared with IX-Ex and S-PRG filler containing composite resin. S-PRG filler, which forms a stable glass-ionomer phase on the surface of filler by pre-reacting acid-reactive glass containing F with polyacrylic acid in the presence of water, was recently introduced [15]. It has been reported that S-PRG filler has good ion release and pH buffer properties [32], and that S-PRG filler exhibits antibacterial activity via the release of metal ions from

the composite resin [33]. In this study, it was shown that the release of Si, P and Al ion from AIC was significantly higher when compared with that from IX-Ex and GIOMER (p<0.001) (Fig.5). This suggests that soluble ions were discharged to the matrix gel phase, due to the source of ion release. In particular, Besinis *et al.* reported that the infiltration of demineralized dentin with Si and HAp nanoparticles may provide a suitable scaffold for the remineralization of dentin [34]. The release of Al is known to be associated with enhanced F release due to the alumino-fluoro complex in GIC glass [35]. In addition, glass in the Fuji IX family is mainly composed of fluoro-alumino-silicate containing Si, Al, Na, F and Sr, but not Ca. Shiozawa *et al.* demonstrated that Ca enhances the formation of GIC matrix, thereby increasing the surface hardness [21]. Ca is therefore necessary for reinforcement of GIC mechanical strength, and HAp could be a useful source of Ca in AIC made with IX-Ex, one of the Fuji IX family. In addition, GIC is able to adhere to the tooth surface. It has been reported that "the intermediate layer" between GIC and dentin contains Ca and P from HAp [36], and we believe that a layer similar to "the intermediate layer" forms around HApS in the AIC matrix. In this study, it was observed that innumerable HAp nano-particles were scattered in the AIC matrix on SEM images (Fig. 3-B), and that the flexural strength of AIC was significantly higher than that of IX-Ex (Table 1). This suggests that HAp enhances the GIC matrix, leading to GIC reinforcement.

According to the results of this study, HAp is able to enhance the mechanical and chemical properties of IX-Ex, latest conventional restorative GIC. In a previous study, it was found that a granular HAp-containing GIC had improved fracture toughness and long-term bonding to dentin [13]. In addition, we reported that AIC continues to mature over time [14]. That is to say, AIC has potential as a reliable restorative material without losing the advantages of GIC. Furthermore, the release of F and Si from AIC suggests the possibility of antibacterial activity and inhibition of secondary caries.

Conclusion

A novel GIC containing porous HAp, known as AIC, based on the latest conventional restorative GIC, IX-Ex was evaluated for mechanical properties and F release properties. Porous HAp was able to improve both strength and F release, without losing the advantages of conventional GIC. Moreover, the release of several minerals from AIC was higher when compared with S-PRG filler containing composite resin, commercialized as a functional and bioactive restorative material. Porous HAp may play an important part in improving both mechanical strength and F release of GIC, as well as inhibiting secondary caries and antibacterial properties.

Acknowledgments

References
1) Wilson AD, Kent BE. The glass-ionomer cement, a new translucent dental filling material. J Appl Chem Biotechnol 1971; 21: 313.
2) Burke FM, Lynch E. Glass polyalkenoate bond strength to dentine after chemomechanical caries removal. J Dent 1994; 22: 283-291.
3) Noffsinger DP, Jedrychowski JR, Caputo AA. Effects of polycarboxylate and glass ionomer cements on stainless steel crown retention. Pediatr Dent 1983; 5:68-71.
4) Tam LE, Chan GP, Yim D. In vitro caries inhibition effects by conventional and resin-modified glass-ionomer restorations. Oper Dent 1997; 22: 4-14.
5) Nakajo K, Imazato S, Takahashi Y, Kiba W, Ebisu S, Takahashi N. Fluoride released from glass-ionomer cement is responsible to inhibit the acid production of caries-related oral streptococci. Dent Mater 2009; 25: 703-708.
6) Irie M, Nakai H. Mechanical properties of silver-added glass ionomers and their bond

strength to human tooth. Dent Mater J 1988; 7: 87-93.

7) Mitra SB. Adhesion to dentin and physical properties of light-cured glass-ionomer liner/base. J Dent Res 1991; 70: 72-74.

8) Xu X, Burgess JO. Compressive strength, fluoride release and recharge of fluoride-releasing materials. Biomaterials 2003; 24: 2451-2461.

9) Nicholson JW, Hawkins SJ, Smith JE. The incorporation of hydroxyapatite into glass-polyalkenoate ("glass-ionomer") cements: a preliminary study. J Mater Sci Mater Med 1993; 4: 418-421.

10) Yap AUJ, Pek YS, Kumar RA, Cheang P, Khor KA. Experimental studies on a new bioactive material: HAIonomer cements. Biomaterials 2002; 23: 955-962.

11) Moshaverinia A, Ansari S, Moshaverinia M, Roohpour N, Darr JA, Rehman I. Effects of incorporation of hydroxyapatite and fluoroapatite nanobioceramics into conventional glass ionomer cements (GIC). Acta Biomaterialia 2008; 4: 432-440.

12) Arita K, Lucas ME, Nishino M. The effect of adding hydroxyapatite on the flexural strength of glass ionomer cement. Dent Mater J 2003; 22: 126-136.

13) Lucas ME, Arita K, Nishino M. Toughness, bonding and fluoride-release properties of hydroxyapatite-added glass ionomer cement. Biomaterials 2003; 24: 3787-3794.

14) Arita K, Yamamoto A, Shinonaga Y, Harada K, Abe Y, Nakagawa K, Sugiyama S. Hydroxyapatite particle characteristics influence the enhancement of the mechanical and chemical properties of conventional restorative glass ionomer cement. Dent Mater J 2011; 30: 672-683.

15) Ito S, Iijima M, Hashimoto M, Tsukamoto N, Mizoguchi I, Saito T. Effects of surface pre-reacted glass ionomer fillers on mineral induction by phosphoprotein. J Dent 2011; 39: 72-79.

16) Frencken JE, Pilot T, Songpaisan Y, Phantumvanit P. Atraumatic restorative treatment (ART): Rationale, technique, and development. J Public Health Dent 1996; 56: 135-140.

17) Frencken JE, Taifour D, van't Hof MA. Survival of ART and amalgam restorations in permanent teeth of children after 6.3 years. J Dent Res 2006; 85: 622-626.

18) Smales RJ, Gao W. In vitro caries inhibition at the enamel margins of glass ionomer restoratives developed for the ART approach. J Dent 2000; 28: 249-256.

19) Marczuk-Kolada G, Waszkiel D, Łuczaj-Cepowicz E, Kierklo A, Pawińska M, Mystkowska J. The effect of glass ionomer cement Fuji IX on the hard tissues of teeth treated by sparing methods (ART and CMCR). Adv Med Sci 2006; 51 (suppl.1): 138-141.

20) Mousavinasab SM, Meyers I. Fluoride release and uptake by glass ionomer cements, compomers and giomers. Res J Biol Sci 2009; 4: 609-616.

21) Shiozawa M, Takahashi H, Iwasaki N. Fluoride release and mechanical properties after 1-year water storage of recent restorative glass ionomer cements. Clin Oral Invest 2014; 18: 1053-1060.

22) Tanaka K, Kato K, Noguchi T, Nakaseko H, Akahane S. The change in Translucency of posterior restorative glass-ionomer cement. IADR 2007 abstract #2025.

23) Swartz ML, Phillips RW, Clark HE. Long-term F release from glass ionomer cements. J Dent Res 1984; 63:158-160.

24) Wiegand A, Buehalla W, Attin T. Review on fluoride-releasing restorative materials –fluoride release and uptake characteristics, antibacterial activity and influence on caries formation. Dent Mater 2007; 23: 343-362.

25) Alsaffar A, Tantbirojn D, Versluis A, Beiraghi S. Protective effect of pit and fissure sealants on demineralization of adjacent enamel. Pediatr Dent 2011; 33: 491-495.

26) Kantovitz KR, Pascon FM, Correr GM, Borges AFS, Uchôa MNS, Puppin-Rontani RM. Inhibition of mineral loss at the enamel/sealant interface of fissures sealed with fluoride- and non-fluoride containing dental materials in vitro. Acta Odontol Scand 2006; 64: 376-383.

27) Trairatvorakul C, Kladkaew S, Songsiripradabboon S. Active management of incipient caries and choice of materials. J Dent Res 2008; 87: 228-232.

28) Matsuya S, Matsuya Y, Yamamoto Y, Yamane M. Erosion process of a glassionomer cement in organic acids. Dent Mater J 1984; 3: 210-219.

29) Meryon SD, Smith AJ. A comparison of fluoride release from three glass ionomer cements and a polycarboxylate cement. Int Endod J 1984; 17: 16-24.

30) Gandolfi MG, Chersoni S, Acquaviva GL, Piana G, Prati C, Mongiorgi R. Fluoride release and absorption at different pH from glass-ionomer cements. Dent Mater 2006; 22, 441-449.

31) Chan WD, Yang L, Wan W, Rizkalla AS. Fluoride release from dental cements and composites: A mechanistic study. Dent Mater 2006; 22: 366-373.

32) Wang Y, Kaga M, Kajiwara D, Minamikawa H, Kakuda S, Hashimoto M, Yawaka Y. Ion release and buffering capacity of S-PRG filler-containing pit and fissure sealant in lacted acid. Nano Biomedicine 2011; 3: 275-281.

33) Saku S, Kotake H, Scougall-Vilchis RJ, Ohashi S, Hotta M, Horiuchi S, Hamada K, Asaoka K, Tanaka E, Yamamoto K. Antibacterial activity of composite resin with glass-ionomer filler particles. Dent Mater J 2010; 29: 193-198.

34) Besinis A, Noot RV, Martin N. Infiltration of demineralized dentin with silica and hydroxyapatite nanoparticles. Dent Mater 2012; 28: 1012-1023.

35) Czarnecka B, Nicholson JW. Ion release by resin-modified glass-ionomer cements into water and lactic acid solutions. J Dent 2006; 34: 539-543.

36) Sennou HE, Lebugle AA, Grégoire GL. X-ray photoelectron spectroscopy study of the dentin-glass ionomer cement interface. Dent Mater 1999; 15: 229-237.

Corresponding author*:*

Dr. Takako Nishimura, D.D.S.
Department of Pediatric Dentistry,
Osaka Dental University,
1-5-17 Otemae, Chuo-ku, Osaka 540-0008, Japan
Tel: +81-6-6910-1515
Fax: +81-6-6910-1038
E-mail: nisimura@cc.osaka-dent.ac.jp