

THE EFFECTS OF NANO-SIZED HYDROXYAPATITE ON DEMINERALIZATION RESISTANCE AND BONDING STRENGTH IN LIGHT-CURED GLASS IONOMER DENTAL CEMENT

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Abstract

The aim of this study was to evaluate the effect of incorporated nano HA on the demineralization resistance and bonding strength of LC GIC in comparison with micro HA. Fuji II LC GIC was used as the control group and a base material for experimental groups. Two experimental groups were prepared. One was prepared by adding 15% micro HA to LC GIC by weight ratio (Exp. 1), and the other was prepared by adding 15% nano HA instead (Exp. 2). According to the results, the following conclusions could be obtained.

1. Observing under the CLSM, the control group showed thicker enamel demineralization layer than in the experimental groups, and the Exp. 2 group showed the thinnest demineralization layer.
2. In SEM analysis, there was greater enamel demineralization in the control group. The Exp. 2 group was more resistant to demineralization compared to the Exp. 1 group.
3. The bonding strength was found to be in the increasing order of control, Exp. 1, and Exp. 2 group ($p < 0.05$).
4. Observing the fractured surfaces under SEM after the bonding strength test was performed, there were bone-like apatite particles formed in HA-added experimental groups, and a greater number of bone-like apatite particles were formed in the Exp. 2 group compared to the Exp. 1 group.

Key words: Light-cured glassionomer cement, Nano-sized hydroxyapatite, Demineralization resistance, Bonding strength

I. Introduction

Recently there are many studies being conducted to see the effect of the addition of hydroxyapatite (HA) to various dental materials in an attempt to improve the physical properties. The use of HA in restorative dentistry offers several promising advantages, including its biocompatibility, intrinsic radiopaque response, enhanced polishability, and improved wear performance, because synthetic HA has a hardness similar to that of natural teeth. In addition, HA contains a significant amount of calcium and phosphate, which can promote remineralization¹⁾. The incorporation of HA into dental

restorative materials, such as resin or GIC, has been found to enhance flexural strength and Young's modulus²⁾, compressive strength, and bonding strength³⁾. It has also been reported that HA incorporation into chewing gums, toothpastes, mouthrinse improves enamel remineralization⁴⁻⁶⁾. The remineralization effect of HA may be because HA can act as a mineral reservoir, which dissociates to form calcium, phosphate ions and hydroxyl ions and fill the micro-sized fine pores of the demineralized enamel surfaces. However, because the micropores between enamel prisms are about 0.1 μm wide, it was hard to expect the conventional 0.2~0.3 μm HA to directly fill up the micropores on demineralized

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enamel surfaces.

Through nanotechnology, it is now possible to convert micrometer particles into nano-sized particles and produce new physical properties from the exact same material and chemical compositions of conventional restorations^{5,7}. The HA particles on enamel surface have an average size of 100 μm , which is fairly large compared to those in bone tissue or dentin⁸. The particles are also well aligned, paralleled to each other. But because the enamel does not include collagen-like organic materials as does the bone tissue and dentin⁹, however, it is necessary to consider the conventional bonding strength to enamel when developing artificial HA. Compared to the previously widely used micro-sized synthetic HA, the greatly minimized nano-sized particles of the nano HA have greater surface areas¹⁰ and solubility¹¹ that they are expected to be outstanding in filling micropores of demineralized enamel surfaces, and also in providing inorganic ions such as calcium and phosphate¹², thus increasing the bonding strength to the tooth. The aim of this study was to evaluate the effect of incorporated nano HA on the demineralization resistance, and bonding strength of LC GIC in comparison with micro HA.

The results of this study include the data of the control group and 15% micro HA LC GIC group tested according to ISO standards, which have been published in Key engineering material in 2009¹³, and compare it with data of 15% nano-HA LC GIC group.

II. Materials and Methods

1. Materials

We prepared commercially available LC GIC and HA of two different particle sizes (micro-sized HA, nano-sized HA). Fuji II LC (GC Co., Japan) was used as the control group and a base material for experimental groups and EliparTM FreeLight (3M/ESPE, USA) was used as a light curing source.

Calcium phosphate tribasic (Sigma-Aldrich Inc., USA) was the micro-sized HA powder of choice. Its molecular formula is $\text{Ca}_5(\text{OH})(\text{PO}_4)_3$ and its particle diameter about 5~10 μm .

Medical grade extra pure nano powder hydroxyapatite (OssGen Inc., Korea) was selected to represent HA with nano-particles, and the molecular equation of the materials is $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, and the average particle size 100~150 nm.

2. Methods

A. Preparing of HA-LC GIC

In that 15% micro HA-Fuji II LC GIC has presented the best physical characters in previous studies, 15% micro HA-Fuji II LC GIC and 15% nano HA-Fuji II LC GIC were used.

15% mixture ratio was measured by weight ratio, and in order to acquire even mixtures, Ball mill (Samwoo Scientific Co., Korea) was used under 400 rpm for 24 hours.

The recommended powder-liquid ratio was 2 for Fuji II LC GIC.

Fuji II LC GIC was used in the control group and the experimental groups were 15% micro HA-Fuji II LC GIC and 15% nano HA-Fuji II LC GIC (Table 1).

B. ISO test

The depth of cure, sensitivity to ambient light, and flexural strength, which are the necessary requirements for dental water-based light activated cements, were determined under regulations of ISO 9917-2:1998. They were tested using Fuji II LC GIC, 15% micro HA-Fuji II LC GIC and 15% nano HA-Fuji II LC GIC, maintaining the constant liquid powder ratio.

(1) Depth of cure

LC GIC was condensed into a mold (6 mm long and 4 mm in diameter) and was then pressed between two matrix strips and two glass plates. The glass plate covering top of the mold was removed and the exit window of light curing unit was gently placed on the matrix strip covering the mold. The LC GIC was then cured for 20 seconds as recommended by the manufacturer. After curing, the specimen was immediately removed from the mold, and the uncured portion was gently removed with a knife. The height of cured portion was measured in micrometers and was divided by two.

(2) Sensitivity to ambient light

Thirty milligrams of LC GIC was placed as a spheroid mass on a glass slide and was exposed to a Xenon lamp for 30 seconds to provide an illuminance of 8,000 lux. The glass slide was then removed from the illuminated area and a second microscope slide was immediately pressed against the material with a slight shear action to produce a thin layer. LC GIC was visually inspected to see whether it was homogenous with no discernible

voids of defects. The whole procedure was repeated twice using new samples of LC GIC for each test.

(3) Flexural strength

Specimens with a dimension of 25 × 2 × 2 mm were prepared using stainless-steel split molds. Materials were packed into the mold placed on a polyester film. A second polyester film was placed on the material and was covered with a glass plate. Pressure was applied to cause excess material to extrude. The light curing unit was then placed at the center of the specimen and irradiated for 20 seconds. After curing, the assembly was placed in the water bath of 37°C for 24 hours. Then, a load was applied onto the specimen with crosshead speed of 0.75 mm/min using a universal testing machine (Instron, UK) until the specimen fractured.

C. Acid demineralization test and surface observation by CLSM and SEM

Box-shaped cavities (6mm × 2mm × 1.5mm) were prepared along the cemento-enamel junction of human molars. The cavities were then filled with LC GIC, which was irradiated for 20 seconds with the light curing unit. The cavities were slightly overfilled to compensate the curing shrinkage of cement, and they were polished using Sof-Lex™ disk after curing. Two coats of an acid-resistant varnish were then applied to the tooth surface, leaving a 1 mm window without varnish around the cavity margins. The teeth were then placed in 25 ml of acid buffer solution containing 2.2 mM CaCl₂, 2.2 mM NaH₂PO₄, and 50 mM acetic acid for 4 days at 37°C (Table 2). The acid solution was replaced every 24 hours.

After 4 days, the teeth were embedded into cylindrical epoxy-resin to be made into slide specimens. By using

an EXAKT diamond band saw (EXAKT Co., Germany), the teeth were sawed parallel to the long axis, passing through the center of the restoration material. The sectioned surface of specimens was observed under a confocal laser scanning microscope (CLSM) (LSM 510, Carl Zeiss Meditec AG, Germany) and a scanning electron microscope (SEM) (S 2000, Hitachi, Japan) to examine the demineralization patterns of enamel adjacent to the cement material.

For microscopic observation under CLSM examination, the samples were dyed in 0.1 mM rhodamine B solution for 1 hour, washed with distilled water and dried. The CLSM causes the demineralized lesions on the enamel to fluoresce under the He-Ne laser light, which has a 543 nm excitation wavelength and a 560 nm long-pass barrier filter.

D. Bonding strength and surface observation by SEM

Teeth were embedded into cylindrical epoxy-resin molds (25 mm × 25 mm × 25 mm), with the intended site for bonding facing the bottom of the mold. The embedded specimens were ground flat perpendicular to the long axis of each tooth until the exposed enamel or dentin surface was large enough to bond a cylinder of restorative material (Fig. 1, a). Next, each cylindrical mold (6 mm in diameter, 6 mm in height) was placed on the etched tooth surface and the material was poured into the mold. The cylindrical specimens were attached to the tooth surface by light curing for 20 seconds (Fig. 1, b). Afterwards, each sample was kept under pH 7.4 simulated body fluid (SBF), at 37°C, and the fluid was changed every week. After four weeks, the cylindrical specimens were loaded by a metal rod on a universal testing machine (Instron, UK) in a direction perpendicular to the long axis at 1 mm/min until fracture occurred.

Table 1. Sample identification of LC GIC

Sample I. D.	LC GIC	wt% of HA
LC GIC (Control 1)	Fuji II	0
Micro HA-LC GIC (Exp. 1)	Fuji II	15
Nano HA-LC GIC (Exp. 2)	Fuji II	15

Table 2. Content 's ingredients of acid buffer solution (pH 5.0)

Materials	Molecular weight (g/mol)	Vol. (ml)	Weight (g)	Concent (M)
CaCl ₂	110.98	1500	0.3662472	2.2 mM
NaH ₂ PO ₄ · 2H ₂ O	156.01	1500	0.51	2.2 mM
CH ₃ COOH	60.05	1500	4.503942	50 mM

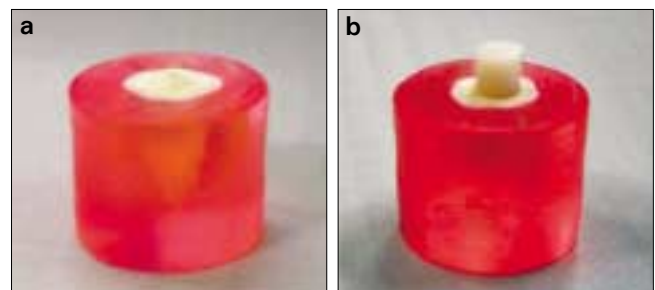


Fig. 1. Sample for the investigation of bonding strength.

The shear bonding strength was calculated as a ratio of fracture load over bonding area, and was expressed in mN/mm².

After measuring the shear bonding strength, the fractured surfaces of the specimens were observed under various magnifications using SEM (Hitachi S-800, Japan).

E. Statistical analysis

Statistic analysis was done on SAS 8.2 version. In order to examine the differences in depth of cure, flexural strength and bonding strength between the control, experimental groups, the Kruskal-Wallis test was used (p value = 0.05). A post-hoc test was then carried out through the Dunn procedure, in order to verify the groups that showed differences. Each test result was presented as the median and the range.

III. Results

1. ISO test

A. Depth of cure

Compared to the control group, both experimental groups showed smaller depth of cure (Table 3). Through the Kruskal-Wallis test, significant differences were found between the three groups (p < 0.05). Through the post-hoc test using the Dunn procedure, significant differences were found between the control group and the Exp. 2 group (critical value = 2.393). All groups satisfied the ISO requirement for the minimum curing depth

Table 3. Depth of cure (mm)

	Control	Exp. 1	Exp. 2
Median	4.720*	4.350	3.010*
Range	4.530 4.870	4.210 4.410	2.930 3.120

* : Statistically significant at critical value = 2.393 by Dunn procedure (minimum curing depth = 1 mm)

of 1 mm.

B. Sensitivity to ambient light

In all groups, homogeneity was maintained after the completion of the test, showing no clefts or voids. Sensitivity to ambient light, therefore, was not observed in any group.

C. Flexural strength

The Exp. 1 group showed 84.086 MPa of flexural strength that is greater than 70.082 MPa of the control group, whereas the Exp. 2 group showed a smaller flexural strength of 58.680 MPa (Table 4).

Through the Kruskal-Wallis test, significant differences were found between the three groups (p < 0.05). Through the post-hoc test using the Dunn procedure, significant differences were found between the Exp. 1 group and the Exp. 2 group (critical value = 2.393).

All groups satisfied the ISO requirement for the minimum flexural strength of 20 MPa.

2. Surface observation by CLSM after acid demineralization

CLSM allows the demineralized surface layers of enamel to be visualized down to approximately 100 μm. The control group had a relatively thick fluorescent layer compared to the two experimental groups. Comparing the two experimental groups, the Exp. 2 group had the thinnest fluorescent layer (Fig. 2).

Table 4. Flexural strength (MPa)

	Control	Exp. 1	Exp. 2
Median	70.082	84.086*	58.680*
Range	59.826 73.648	76.989 112.769	50.089 62.876

* : Statistically significant at critical value = 2.395 by Dunn procedure (minimum flexural strength = 20 MPa)

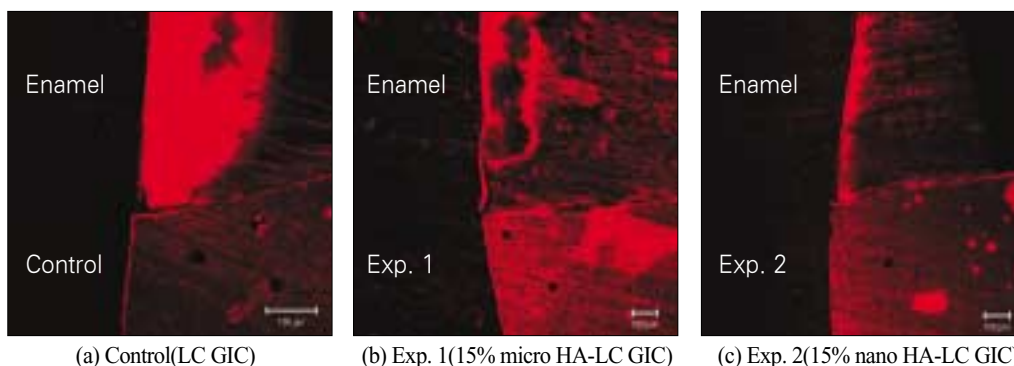


Fig. 2. Interface between tooth and cement observed under CLSM after acid demineralization (× 10).

3. Surface observation by SEM after acid demineralization

The SEM images of micro surfaces demonstrated small morphological differences between the control group and the experimental groups (Fig. 3). The control group showed more irregular granular and porous surfaces than the experimental groups after acid demineralization. Comparing the two experimental groups, there was

less demineralization occurred in the Exp. 2 group, and thus less irregular and porous surfaces were created in the Exp. 2 group.

The control group (a) showed greater irregularity and porosity on the enamel surface compared to the Exp. 1 group (b) or the Exp. 2 group (c). The Exp. 2 group (c) showed the least irregularity and porosity after acid demineralization.

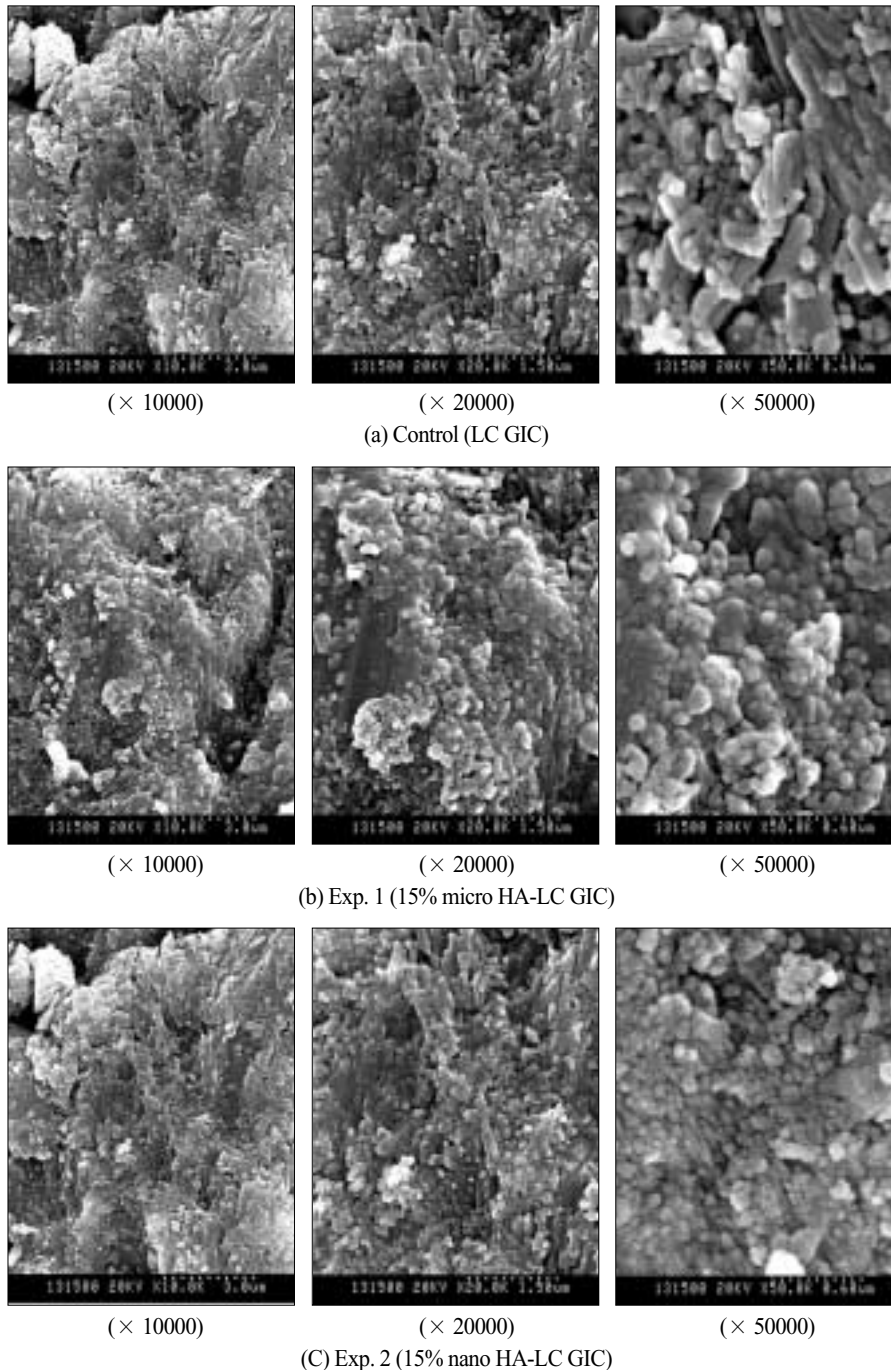


Fig. 3. SEM images of the sagittally sectioned enamel surface adjacent to the cement after acid demineralization.

Table 5. Bonding strength (mN/mm²)

	Control	Exp. 1		Exp. 2	
Median	419.20*	1075.60		1911.50*	
Range	329.60 445.30	989.70 1217.20	1816.90	2002.50	

* : Statistically significant at critical value = 2.395 by Dunn procedure

4. Bonding strength

The bonding strength of cement to dentin increased to 419.20 mN/mm² in the control group, 1075.60 mN/mm² in the Exp. 1 group, 1911.50 mN/mm² in the Exp. 2 group (Table 5). Through the Kruskal-Wallis test, significant differences were found between the three groups ($p < 0.05$). Through the post-hoc test using the Dunn procedure, significant differences were found between the control group and the Exp. 2 group.

5. Observation of fracture surfaces by SEM after measuring bonding strength

When the fractured surfaces were observed under SEM at low magnification after the bonding strength test, the cohesive failure ratio increased in following order: the control group, the Exp. 1 group, and the Exp. 2 group (Fig. 4).

At higher magnifications the dentinal surfaces were fairly flat in the control group, but there were bone-like apatite particles formed in the experimental groups. A greater number of bone-like apatite particles were formed in the Exp. 2 compared to the Exp. 1 group (Fig. 5).

Increased appearance of cohesive failure was observed in the experimental groups (b, c) compared to the control group (a). The Exp. 2 group (c) had greater cohesive failure compared to the Exp. 1 group (b).

IV. Discussion

Hydroxyapatites (HA) contain a significant amount of calcium and phosphate, which can promote remineralization of enamel subsurface lesions in animals and humans. Up to now, many researchers have confirmed that the biocompatibility of HA contribute to the enhancement of mechanical properties of various dental materials.

There are many studies conducted to apply nanotechnology to dental materials in the recent dental field. Because nano HA have greater surface areas and solubility compared to the micro HA, it is expected to show superior properties in filling micropores on demineralized enamel surfaces and in providing such inorganic ions as calcium and phosphate.

In this study, we attempted to compare the physical properties, the demineralization resistance and the bonding strength of not only the micro HA added to light-cured glass ionomer cement (LC GIC), but also the nano HA, produced by the recently spotlighted nanotechnology.

Fuji II LC GIC (GC Co., Japan), used in this study, is a dental water-based light-activated cement. Fuji II LC GIC was used in the control group and the experimental groups used were 15% micro HA-Fuji II LC GIC and 15% nano HA-Fuji II LC GIC. To evaluate their suitability as dental cements, their curing depth, sensitivity to ambient light and flexural strength were assessed according to ISO 9917-2:1998 regulations. The control, Exp. 1, and Exp. 2 group showed the depth of cure in the decreasing order ($p < 0.05$). This may be due to the light-scattering effect of HA particles¹⁰.

Sensitivity to ambient light is a necessary parameter

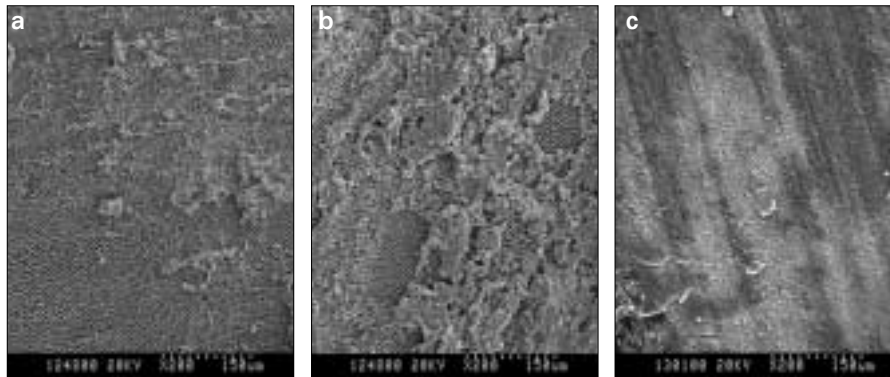


Fig. 4. SEM images of fracture surface after measuring bonding strength (x 200).

for light-activated materials, as ambient light may cause premature curing and result in crack and void formations during their use. There were no detectable changes in the homogeneity of any group after being exposed to the test light for 30 seconds, indicating that sensitivity to ambient light was not observed in any group.

Flexural strength gives an indication of the durability of restorative materials and it is considered to be the most appropriate and reliable estimate of the strength of LC GIC¹⁴⁾. Flexural strength was measured to be greater in the Exp. 1 group than in the control group, and was smaller in the Exp. 2 group than in the control group (p

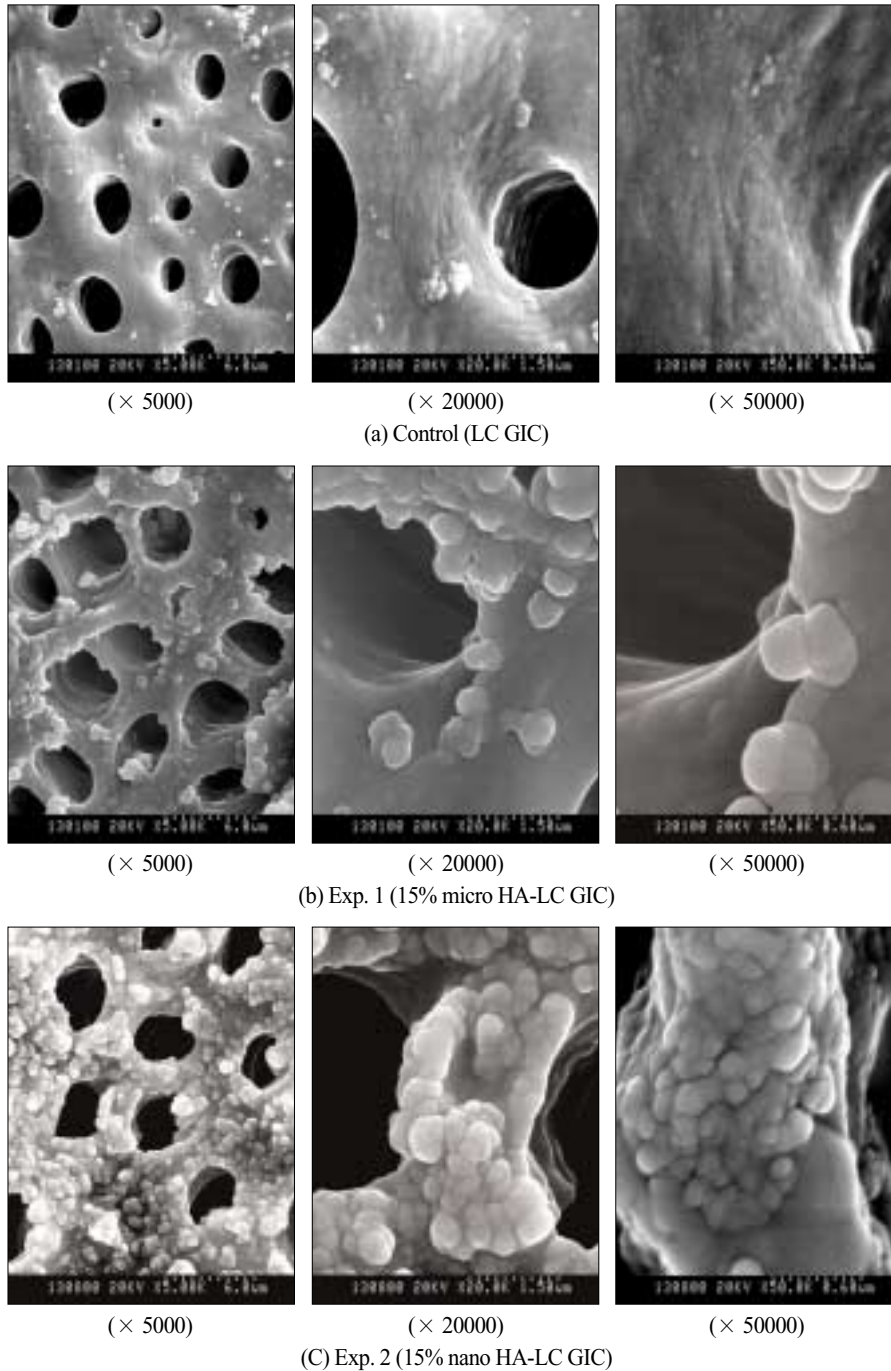


Fig. 5. SEM images of fracture surface after measuring bonding strength.
 (a) Control group, no bone-like apatite particles were observed on the tooth surface.
 (b) Exp. 1 group, the expected particles of bone-like apatite were formed on the tooth surface.
 (c) Exp. 2 group, the expected particles of bone-like apatite were formed on the tooth surface more than Exp. 1 group (b).

(< 0.05). Number of reasons for the Exp. 1 group to present a greater flexural strength than the control group could be speculated. The increase in the flexural strength after the addition of HA is thought to be dependent primarily on a chemical reaction between the HA, glass powder and polyacid. Although the exact underlying chemical mechanism is yet unknown and requires further investigations, it is known to involve calcium ions from the HA. In LC GIC, when the polyacid reacts with the glass powder, it selectively binds to calcium ions first and then reacts with other ions. Therefore, when HA is added, the number of calcium ions in the initial reaction increase and thus the initial ionic bonds increase. Since the bonding between polyacrylic acid and glass ions is the main reaction in LC GIC, the increase of flexural strength could be explained this way.

The size difference between the HA and GIC particles may also contribute to the increase of flexural strength, for the smaller particled HA may flow into the ionomer matrix of the bigger particled HA and induce a "packing effect"¹⁵⁻¹⁷.

As the reason for the Exp. 2 group to show weaker flexural strength than the control group, we may consider the increased solubility of nano HA¹¹. In case of nano particles, the "packing effect" induced by flowing into the ionomer matrix would obviously be greater than the micro particles, thus would increase the light scattering effect of the HA and decrease its flexural strength. This seems logical, observing the flexural strength increase as the density of HA increase and begin to decrease back at a certain point, while the density continue to increase¹⁸. The increase in HA content delays the polymerization of LC GIC, which in turn decreases the role of light polymerization while increasing that of autopolymerization. A previous study has reported a decrease in material strength cured by autopolymerization than cured by light polymerization¹⁸. Likewise, if the light-scattering effect of HA hinder light polymerization and shift the dual-setting reaction towards autopolymerization, a decrease in the material strength may eventually result.

In order to observe the demineralization resistant effect of each material, we kept the teeth in a pH 5.0 acid buffer solution for 4 days and cut them into slide specimens using a EXAKT diamond band saw. Under CLSM, the control, Exp. 1, and Exp. 2 group showed demineralization of the enamel in the decreasing order. From this, the assumption that demineralization resistance may increase with the addition of nano HA was con-

firmed. The demineralization observed under CLSM was found to be subsurface demineralization rather than enamel surface demineralization. This may be from the "coupled diffusion" resulting from the influx of acid and outflux of the dissolved materials^{1,19,20}.

Observing under SEM, the enamel surface of the control group was more uneven and showed greater loss of inorganic material between the enamel prisms than the HA-added experimental groups. Because the nano particles filled up the micropores formed by the loss of inorganic materials, the enamel surface of the Exp. 2 group was more smooth and even than that of the Exp. 1 group.

The reasons for increased demineralization resistance of nano HA are considered to be the effect of direct filling of micropores on the demineralized enamel surface by the smaller size particles, and the effect of dissolving and providing the main inorganic components of HA, calcium and phosphate, with the high solubility^{12,21}. Since the micropores between the enamel prisms are about 0.1 μm , it was hard to expect it to be directly filled up by the 5~10 μm micro HA, but the nano HA with an average size of 100~150 nm showed an outstanding performance in doing so. The nano particles embedded into the micropores are also expected to impede the movement of calcium soaring from the enamel surface by the means of balancing the inorganic ions⁷.

The control, Exp. 1, and Exp. 2 group presented the bonding strength in the increasing order ($p < 0.05$). LC GIC is known for its ability to chemically bond to the tooth substrate, but the exact mechanism of chemical bonding of LC GIC to dental structures is not completely known. The probable mechanism of adhesion is based upon both diffusion and adsorption phenomena. The polyalkenoic acid of the glass ionomer will penetrate the tooth structure, releasing phosphate ions, each of which will take with it a calcium ion from the tooth surface to maintain electrical neutrality. These ions will combine with surface layer of cement and form an intermediate layer of a new material, which is firmly attached to the tooth surface²². This has been described as a "diffusion based adhesion"²³. There is also a degree of adhesion available to the collagen of dentin through either hydrogen bonding or metallic ion bridging between the carboxyl groups on the polyacid and the collagen molecules²⁴. The reason for increased bonding strength by adding HA, one of the main components of the tooth, to LC GIC that chemically bonds to tooth surface, was con-

sidered to be from the participation of calcium ions from the HA in the chemical bonding of the tooth and the material¹⁵⁾. The bonding strength was greater in the Exp. 2 than in the Exp. 1 group, in that smaller particles make the surface areas greater, and the adhesion rate of the particles to the surface greater that they invade into the enamel surface and between the micropores and eventually provide greater bonding strength. Additional insight into this field was contributed by recent X-ray photoelectron spectroscopy studies. Sennou²⁵⁾ have shown the formation of an intermediate dentin-glass ionomer layer on the surface of the materials that enables the exchange of mineral and organic elements and consequently the adhesion of the material to dentin.

In the SEM observation of the fractured surfaces, cohesive failure was more evident in HA-added experimental groups, and more in the Exp. 2 group than in the Exp. 1 group, as a result of increased bonding strength.

The reason why the samples were kept in the SBF fluid was to induce the formation of bone-like apatite between the interfaces of the tooth and the material, and as a result observed under SEM after 4 days, the control group revealed to have a greater porosity compared to the HA-added experimental groups. There were observable bone-like apatite particles formed on the surfaces, in the HA-added experimental groups. This is reported to be contributed by the ion exchange between the SBF and the HA soaring from the material^{2,26)}. We could observe a greater number of bone-like apatite particles in the Exp. 2 group compared to the Exp. 1 group, and their dentinal tubules were hence narrowed by the formation of these particles. There are two possible reasons that can explain why a greater number of bone-like apatite particles were formed as the size of HA particles decreased. The first reason is that because the solubility of the nano HA is greater, the particles can more actively participate in the ion exchange with the SBF fluid^{11,27,28)}, and the second reason is because the nano HA has a strong adhesion strength and because it is similar to tooth components. As it was mentioned as the reason for increased bonding strength, the strong adhesion strength and the similarity to tooth components facilitate its infiltration into the micropores of the tooth surface and the subsided nano HA particles facilitate additional particles to subside.

V. Conclusion

This study was conducted to evaluate the effect of incorporated nano HA on the demineralization resistance and bonding strength of light-cured glass ionomer cement in comparison with micro HA. According to the results, the following conclusions could be obtained.

1. Observing under the CLSM, the control group showed thicker enamel demineralization layer than in the experimental groups, and the Exp. 2 group showed the thinnest demineralization layer.
2. In SEM analysis, there was greater enamel demineralization in the control group. The Exp. 2 group was more resistant to demineralization compared to the Exp. 1 group.
3. The bonding strength was found to be in the increasing order of control, Exp. 1, and Exp. 2 group ($p < 0.05$).
4. Observing the fractured surfaces under SEM after the bonding strength test was performed, there were bone-like apatite particles formed in HA-added experimental groups, and a greater number of bone-like apatite particles were formed in the Exp. 2 group compared to the Exp. 1 group.

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국문초록

광중합형 글라스아이오노머 시멘트의 탈회 저항성과 결합 강도에 대한 나노미터 입자의 하이드록시아파타이트의 효과

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본 연구의 목적은 광중합형 글라스아이오노머 시멘트에 마이크로 입자의 하이드록시아파타이트와 나노미터 입자의 하이드록시아파타이트를 첨가하였을 때 물리적 성질과 탈회 저항, 결합 강도의 차이를 비교하기 위함이다. 실험에 사용된 광중합형 글라스아이오노머 시멘트는 Fuji II LC 였고 순수한 Fuji II LC GIC는 대조군으로, 15% micro HA- Fuji II LC GIC는 실험군 1, 15% nano HA- Fuji II LC GIC는 실험군 2로 설정한 후 실험을 진행하였고 다음과 같은 결론을 얻었다.

1. CLSM으로 탈회 표면 깊이를 관찰한 결과 대조군 보다 실험군에서 범랑질의 탈회가 덜 발생하였고, 실험군 1 보다 실험군 2에서 범랑질의 탈회가 적게 관찰되었다.
2. SEM을 이용한 탈회면 관찰시 대조군에서 범랑질의 탈회가 더 많이 일어났고, 실험군은 하이드록시아파타이트의 영향으로 탈회가 덜 일어나 표면입자가 보다 규칙적이었다. 두 실험군을 비교했을 때 실험군 2가 실험군 1 보다 탈회에 저항하였다.
3. 결합 강도는 대조군, 실험군 1, 실험군 2 순으로 증가했으며 세 군간에 통계학적으로 유의할 만한 차이가 있었다 ($p < 0.05$).
4. SEM 상에서 결합 강도 측정 후 파절된 면을 관찰한 결과 하이드록시아파타이트를 포함하는 실험군에서 골 유사 아파타이트 추정 입자가 관찰되었으며 실험군 1 보다 실험군 2에서 더 많은 입자가 형성되었다.

주요어 : 광중합형 글라스아이오노머 시멘트, 나노미터 하이드록시아파타이트, 탈회 저항성, 결합 강도