# The radiopacity of Glass Ionomer cement after addition of nHA powder: experimental study

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## ABSTRACT

**Introduction:** One of the restorative materials often used in dental restoration treatment is GIC. One type of GIC is conventional GIC. Conventional GIC has the disadvantage of having low compressive strength compared to resin-modified GIC and high viscosity. One of the efforts to improve the mechanical properties of GIC is by adding nano hydroxyapatite (nHA) which can be obtained from natural materials such as eggshells. The addition of nHA to GIC can increase the compressive strength of GIC and the addition of nHA is thought to result in a change in the radiopacity properties of GIC. The research aims to analyze the radiopacity of GIC after the addition of nano-hydroxyapatite powder. **Methods:** The number of samples was 24 discs with a diameter of 7 mm and a height of 3 mm which were divided into 4 group. The research group was K0 (GIC) as the control group, K1 (GIC+ 2% nHA), K2 (GIC+ 3% nHA), and K3 (GIC + 4% nHA). Radiopacity measurements were carried out using the J Image application. **Results:** The results of the ANOVA test was $p<0.05$, which means that the radiopacity values in each group had a significant difference. The average gray value of GIC without the addition of nHA (K0) was 206.5, GIC + 2% nHA (K1) was 233.0, GIC + 3% nHA (K2) was 237.8 and GIC + 4% nHA (K3) was 241.0 measured with Image J. **Conclusions:** The addition of nHA powder can increase the radiopacity of GIC and 4% nHA powder added to GIC has the highest radiopacity value compared to 2 and 3% nHA powder added to GIC.

## Key words

radiopacity, glass ionomer, cement, nHA

# Radiopasitas semen glass ionomer setelah penambahan bubuk nHA: studi eksperimental

## ABSTRAK

**Pendahuluan**: Salah satu bahan restoratif yang sering digunakan dalam perawatan restorasi gigi adalah GIC. Salah satu jenis GIC adalah GIC konvensional. GIC konvensional memiliki kekurangan yaitu kekuatan tekannya rendah dibandingkan dengan GIC yang dimodifikasi resin dan viskositas tinggi. Salah satu upaya untuk meningkatkan sifat mekanik GIC adalah dengan menambahkan nanohidroksiapatit (nHA) yang dapat diperoleh dari bahan alami seperti cangkang telur. Penambahan nHA ke GIC dapat meningkatkan kekuatan tekan GIC dan penambahan nHA diduga mempengaruhi sifat radiopasitas GIC. Penelitian ini bertujuan untuk menganalisis radiopasitas GIC setelah penambahan bubuk nanohidroksiapatit. **Metode**: Jumlah sampel adalah 24 cakram dengan diameter 7 mm dan tinggi 3 mm yang dibagi menjadi 4 kelompok. Kelompok penelitian ini adalah K0 (GIC) sebagai kelompok kontrol, K1 (GIC+2% nHA), K2 (GIC+3% nHA), dan K3 (GIC+4% nHA). **Hasil:** Hasil uji ANOVA diperoleh nilai $p<0.05$ yang berarti bahwa nilai radiopasitas pada setiap kelompok memiliki perbedaan yang signifikan. Rata-rata nilai gray value GIC tanpa penambahan nHA (K0) adalah 206,5, GIC + 2% nHA (K1) adalah 233,0, GIC + 3% nHA (K2) adalah 237,8 dan GIC + 4% nHA (K3) adalah 241,0. **Simpulan:** Penambahan bubuk nHA dapat meningkatkan radiopasitas GIC dan 4% bubuk nHA yang ditambahkan ke GIC memiliki nilai radiopasitas tertinggi dibandingkan dengan 2 dan 3% bubuk nHA yang ditambahkan ke GIC.

## Kata kunci

radiopasitas, semen, glass ionomer, nanohidroksiapatit

## INTRODUCTION

GIC is often used in the treatment of dental restorations as one of the restorative materials.[1] GIC is often used in dentistry because it has advantages such as being able to release fluorine, as an anti-bacterial, biocompatible with pulp tissue and binds well to the tooth structure.[1,2] Conventional GIC is one type of GIC. Conventional GIC has the deficiency of low compressive strength compared to resin-modified GIC and high viscosity.[3,4]

One attempt to improve the mechanical properties of GIC is to add hydroxyapatite to GIC therefore it transpires the GIC modification.[5] The addition of hydroxyapatite to GIC is known to increase the compressive strength of GIC.[6,7] Hydroxyapatite can be derived from natural materials such as eggshells, while eggshells are often found as waste. The eggshell contains $CaCO_3$ (calcium carbonate) that it used as a precursor in the synthesis of hydroxyapatite.[8,9] The addition of hydroxyapatite from eggshells affects the mechanical properties of conventional GIC, that is increasing the compressive strength of conventional GIC. Hydroxyapatite nanoparticles or nHA is known to affect the mechanical properties of GIC. Instead, the addition of nano nHA into GIC can increase the compressive strength and fracture toughness.[10-12]

GIC has radiopacity properties, the radiopacity is known as X-ray attenuation ability. Specifically, the inability of X-rays to penetrate a material due to reduced intensity of X-rays passing through the material or material raises a white image on the radiograph. The white image on the radiograph occurs because the beam of light from the X-rays passing through the object is absorbed.[13-16] The radiopacity of restorative material is very important as a requirement for dental materials because the radiopacity of the material allows dentists to assess restoration integrity, diagnose secondary caries, and differentiate between healthy tooth structures, restorative materials, and caries.[17]

The addition of nHA is thought to increase the radiopacity of GIC due to hydroxyapatite can increase the density.[7] An increase in the density of a material can cause an increase in X-ray attenuation, namely a decrease in the intensity of X-rays passing through the material. Increased X-ray attenuation can cause the radiopacity of a material to increase.[13,16,18,19] The purpose of this study was to analyze the radiopacity of GIC after the addition of nHA powder from eggshells.

## METHODS

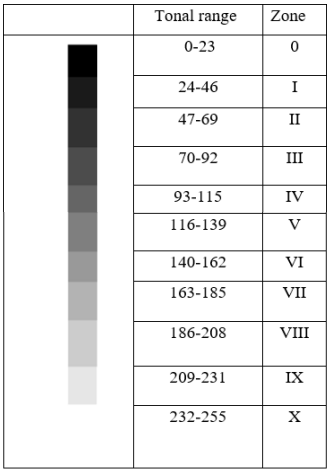
Posttest Only Control Group Design was a type of experimental research applied in this study. The research was conducted at the Technology and Bioscience Laboratories, Conservation Clinic, and the Radiology Installation of the Faculty of Dentistry, University of Jember. The sample form in this study was disc. The number of samples was 24 discs in diameter of 7 mm and height of 3 mm which were divided into 4 groups, each group containing 6 samples.[20-24] The samples were divided into four groups, as follows: The K0 (control) group consisted of GIC without added nHA. The K1 group contained GIC powder and 2% nHA (by weight), where the weights of the GIC powder and nHA were 0.2301 and 0.0046 g, respectively. The K2 group contained GIC powder and 3% nHA (by weight), where the weights of the GIC powder and nHA were 0.2277 and 0.0070 g, respectively. Group K3 contained GIC powder and 4% nHA (by weight), where the weights of the GIC powder and BnHA were 0.2254 and 0.0093 g, respectively

The research group was K0 (SIK) as the control group, K1 (SIK + 2% nHA), K2 (SIK + 3% nHA), and K3 (SIK + 4% nHA). GIC and nHA powders were measured with an analytical scale and mixed with a vortex. GIC powders and liquids were manipulated on a paper pad. The manipulation was carried out by dividing the GIC powder into two parts equally. A portion of the GIC powder was directed into the liquid and then stirred in a folding motion using an agate spatula. Stirring was carried out for 10 seconds. Next, the rest of the powder was added and the whole material stirred within 15-20 seconds resulting in a putty-like consistency and putty was obtained.[25-27] The homogeneous dough put into a metal mold and condensed with a cement stopper. The surface of the sample was smeared with cocoa butter 30 seconds after printing and coated with a celluloid strip, then a weight of 500 grams was given on top for 20 seconds. The sample was left for 24 hours in a metal mold and then put in a closed container. After 24 hours, the celluloid strip was removed. Next, the sample released from the metal mold using a cement stopper.[8,11,26,28-30] The sample that has been removed from the mold immersed in purchased artificial saliva until the entire surface of the sample was submerged in the medicine pot. Furthermore, the sample was put into an incubator at 37°C for 24 hours. The sample was then dried on all surfaces using a tissue and then stored in a plastic clip.[30-32]

Production of radiographs uses digital intraoral x-ray (KaVo Dental, Bieberich, Germany). The imaging plate/film plate was placed on a table with a distance between the source - imaging plate/film plate of 20 cm and at an angle of 90°. After adjusting the position of the sample, then adjust the voltage and current on the X-Ray unit, namely 70 kV and 7mA current. Exposed each group of samples for 4 seconds. Next, scanning and reading on digital intraoral imaging. Then, the SIK radiograph file was saved in png format onto a CD-RW and then the file was transferred to a laptop.[33-37]

Radiopacity measurements were carried out using the J Image application. Image J is a software developed by researchers at the Research Services Branch of the National Institute of Mental Health in Bethesda, Maryland, USA. Image J as a digital image analysis can be used in the field of health.[38] The image to be measured with the Image J application was opened by selecting the file menu and selecting the open menu. Next, select the image file that will be measured. Then select the analyze menu and select set measurements. Put a tick in the area, mean gray

value boxes, and other required values, then select Ok. Perform calibration and measurement by selecting the elliptical menu, then make a drag at the location being measured. The measured area must be the same for consistency in measurement. The area that has been created can be shifted to the measured position by pressing the arrow keys on the keyboard. The size of the measuring area was adjusted to the object to be measured so that it does not exceed the limit. Next, take a Mean gray value measurement by selecting the analyze menu and then selecting the measurement menu. The results would appear according to the selected parameters: area and mean gray value. Gray value was displayed in the Mean column (mean gray value). The gray value was adjusted to the Ansel Adam zone system and the greater the gray value indicates that the material was more radiopaque and has a greater material density[39-40]



| | Tonal range | Zone |
|---|---|---|
| | 0-23 | 0 |
| | 24-46 | I |
| | 47-69 | II |
| | 70-92 | III |
| | 93-115 | IV |
| | 116-139 | V |
| | 140-162 | VI |
| | 163-185 | VII |
| | 186-208 | VIII |
| | 209-231 | IX |
| | 232-255 | X |

**Figure 1.** Ansel Adam's zone system

Data from radiopacity measurements were analyzed using the normality test using the Shapiro-Wilk test and the homogeneity test using the Levene-statistic test. Then, the parametric One-Way ANOVA test was carried out and continued with the Least Significant Difference test to see significant differences between treatment groups.

## RESULTS

The samples used in this study were 24 samples of GIC in the form of discs which were divided into 4 groups. Measurement of GIC and GIC radiopacity with the addition of nHA powder was carried out using digital radiography, namely Image J (Figure 1).

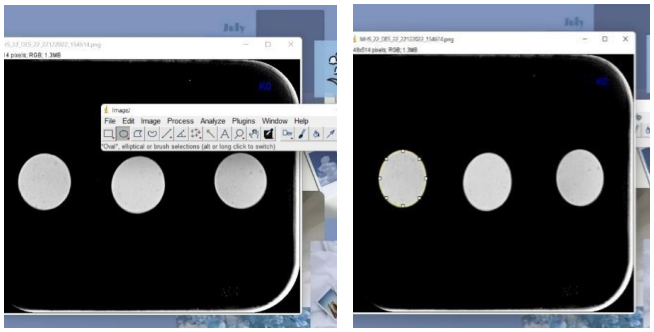


**Figure2.** Radiopacity measurement with Image J

The results of further radiopacity measurements from each sample were presented in the table. The results of the average radiopacity measurement in each study group can be seen in Table 1.

**Table 1.** The Average value of GIC and GIC radiopacity with the addition of nHA

| Sample group | n | Average |
|---|---|---|
| K0 | 6 | 206.5 |
| K1 | 6 | 233.0 |
| K2 | 6 | 237.8 |
| K3 | 6 | 241.0 |

The results of the normality test using the Shapiro-Wilk test and the results of the homogeneity test using the Levene Test (Table 2) obtained a value ($p>0.05$). Hence, the data was distributed and homogeneous. The results of the normality and homogeneity tests were continued by a one-way analysis of variance (ANOVA) parametric statistical test (Table 3) to determine whether there is a difference in radiopacity between each treatment group.

**Table 2.** Shapiro Wilk normality test results and Levene homogeneity

| Test | Sample group | Grade P |
|---|---|---|
| Shapiro Wilk | K0 | 0.303 |
| | K1 | 0.440 |
| | K2 | 0.190 |
| | K3 | 0.246 |
| Levene | | 0.061 |

**Table 3.** Test results one-way analysis of variance (ANOVA)

| Group | n | Deviation standard | p value |
|---|---|---|---|
| Control | 6 | 2.66 | 0.001 |
| K1 | 6 | 4.27 | |
| K2 | 6 | 2.14 | |
| K3 | 6 | 1.52 | |

According on the results of the ANOVA test, the value ($p<0.05$) was obtained, which means that the radiopacity values in each group had a significant difference, meaning that there were differences in radiopacity between treatment groups, with the result that it was continued with the LSD (Least Significant Differences) test to determine the extent of the radiopacity differences in each treatment group. The LSD test results are presented in Table 4.

**Table 4.** Test results of LSD radiopacity between treatment groups

| Sample group | Control | K1 | K2 | K3 |
|---|---|---|---|---|
| Control | - | 0.001 | 0.001 | 0.001 |
| K1 | | - | 0.009 | 0.001 |
| K2 | | | - | 0.061 |
| K3 | | | | - |

According to the results of the LSD test, there was a significant difference between the K0 and K1 treatment groups; K0 with K2; K0 with K3; K1 with K2; and K1 with K3 with p value ($p<0.05$). This shows that the addition of 2, 3 and 4% BnHA into GIC has a significant effect on GIC radiopacity so that the GIC radiopacity increases.

## DISCUSSION

Based on this study, the average gray value of GIC without the addition of nHA was 206.5, GIC samples with the addition of 2% nHA was 233.0, GIC with the addition of 3% nHA was 237.8 and GIC with the addition of 4% nHA was 241.0. The research results of 24 GIC samples in the form of discs, it was shown that the GIC sample group added with nHA powder from eggshells (K1, K2, and K3) had higher radiopacity than the GIC sample group without the addition of nHA from eggshells (K0).

Radiopacity in this study was measured with Image J. The resulting value in radiopacity measurements using Image J is called the gray value.[40,41] Gray value that can be obtained is between 0-255. Gray value 0 is for radiolucent, while 255 is for total X-ray absorption, causing a white or radiopaque image.[40] Based on research conducted by Kuter and Uzel.[20] GIC radiopacity of 148.3 was measured using Image J. According to the research of Manja and Harahap.[42] GIC radiopacity as measured using Image J was 186.8. Based on this study, the average gray value of GIC without the addition of nHA was 206.5, for GIC samples. The GIC radiopacity obtained was different from previous studies and the different brands of materials used allowed for differences in the GIC radiopacity obtained.

nHA is a hydroxyapatite nanoparticle with a particle size of 1-100 nanometers which can be added to GIC as an additive to improve the mechanical properties of GIC.[12,43] Nanoparticles have a small particle size. The small particle size causes a larger contact surface area. The larger contact surface causes an increase in the adhesion force between the particles of the nHA powder mixture from eggshells and GIC powder. Thus nHA can increase the formation of salt bridges thus improving the mechanical properties of GIC.[10,44] nHA has been suggested as an additive to GIC because it has a crystal structure resembling dental apatite and is proven to increase remineralization and reduce restoration microleakage.[45]

In this study, the GIC samples mingled with nHA were put into a closed container in purpose it would not interacted among the air, whereas in threatened the sample surface to crack due to evaporation from the air.[46] In addition, the dry condition of GIC due to water loss due to liquid evaporation can be prevented by applying a layer of cocoa butter on the surface of the GIC after the GIC is placed in the mold.[28,29,46] The sample that has been removed from the mold is soaked with artificial saliva to match the condition of the liquid in the oral cavity.[47] The factitious saliva in this study was saliva with a normal pH (6.7) because immersion of GIC in factitious saliva with a normal pH showed a lower surface roughness value compared to artificial saliva with an acidic pH.[31,48]

Based on research conducted by Showkat et al.[49], namely regarding the addition of nHA to GIC, it showed an increase in the compressive strength of GIC from 132.09 MPa to 148.80 MPa. Research conducted by Alatawi et al.[10] showed an increase in the compressive strength of GIC from 136.49 MPa to 147.12 MPa after the addition of nHA to GIC. Previous studies have shown that the addition of nHA can affect the mechanical properties of GIC, which can increase the compressive strength of GIC, and in this study, it is known that nHA can affect the radiopacity of GIC, namely increasing the radiopacity of GIC.

The results showed that the addition of nHA particles to GIC caused an increase in GIC radiopacity. GIC powder containing nHA is mixed with GIC liquid, calcium ions in nHA will be involved in acid-base reactions with GIC liquid and react with organic or inorganic components of GIC.[50] The increase in nHA from eggshells in GIC causes the calcium ions contained to increase so that it is possible to form more cross-linked structures. Furthermore, the nHA in GIC will fill the gaps between particles in GIC which causes an increase in GIC density.[11,50,51] The high density can affect X-rays, namely the type of radiation from the spectrum, generated by the oscillations or acceleration of electric charges and composed of energy waves that have different distances and frequencies.[19,52] Increasing the density of the material causes the X-rays to experience increased attenuation and less to reach and react with the film.[18,19]

Attenuation is the reduction or weakening of the X-ray radiation intensity after passing through the material. Attenuation can be affected by the density of a material because the denser the material, the less X-rays that can penetrate the imaging plate/film plate so that the intensity or number of X-rays that reach the imaging plate/film plate is smaller and will produce a brighter radiographic image or increased radiopacity. Solid materials appear radiopaque (white) on a radiograph due to absorption of the X-ray beam as it passes through the material.[13,16]

## CONCLUSION

The addition of nHA powder increase the radiopacity of GIC and the distinction in the percentage of addition of nHA powder affects the difference in GIC radiopacity. 4% nHA powder added to GIC has the highest radiopacity value compared to 2 and 3% nHA powder added to GIC.

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