



THE ENHANCEMENT OF GLASS IONOMER CEMENT HARDNESS BY ADDING GOURAMI SCALES POWDER NANOPARTICLES

PENINGKATAN KEKERASAN SEMEN IONOMER KACA DENGAN MENAMBAHKAN NANOPARTIKEL BUBUK SISIK GURAMI

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ABSTRACT

Background: Material hardness in dentistry is used as a measuring tool to determine the ability of a restoration material to withstand the compressive force of masticatory. The hardness of Glass Ionomer Cement (GIC) is a mechanical property related to the abrasion and compressive strength of the GIC. If the material's hardness is low, it results in low material resistance and causes a fracture. Therefore, it is necessary to add materials that contain hydroxyapatite (HA), which can increase the hardness of GIC, as in gourami fish scales. **Purpose:** This research aims to determine the influence of adding Gourami Fish Scale Powder (GFSP) nanoparticles to GIC hardness. **Method:** The twenty five sample is in tablet form and divided into five sample groups, namely G0: GIC without the addition of GFSP, G1: GIC powder + 0.5% GFSP, G2: GIC powder + 1.5% GFSP, G3: GIC powder + 2.5% GFSP, G4: GIC powder + 3.5% GFSP. Hardness test was using Vickers Hardness Tester. Data were analyzed using a One Way ANOVA test. **Result:** The average values of the hardness results from lowest to highest were the G0 (55.5 ± 2.70 VHN), G1 (56.9 ± 3.36 VHN), G2 (63.7 ± 7.73 VHN), G3 (66.3 ± 1.44 VHN), and G4 (70.1 ± 4.72 VHN), while the One Way ANOVA test results were significant among all groups (p -value < 0.05). **Conclusion:** The addition of GFSP nanoparticles increased GIC hardness. The highest hardness value was obtained by adding 3.5% GFSP nanoparticles.

ABSTRAK

Latar belakang: Kekerasan material dalam kedokteran gigi digunakan sebagai alat ukur untuk menentukan kemampuan suatu material restorasi untuk menahan gaya tekan mastikasi. Kekerasan Semen Ionomer Kaca (SIK) adalah sifat mekanik yang terkait dengan abrasi dan kuat tekan SIK. Jika kekerasan material rendah akan menghasilkan ketahanan material yang rendah dan menyebabkan fraktur. Oleh karena itu, perlu ditambahkan bahan dengan kandungan hidroksiapatit (HA) yang dapat meningkatkan kekerasan SIK, seperti pada sisik ikan gurami. **Tujuan:** Penelitian ini bertujuan untuk mengetahui pengaruh penambahan nanopartikel Bubuk Sisik Ikan Gurami (BSIG) terhadap kekerasan SIK. **Metode:** Dua puluh lima sampel dalam bentuk tablet dan dibagi menjadi lima kelompok sampel, yaitu G0: SIK tanpa penambahan BSIG, G1: bubuk SIK + BSIG 0.5%, G2: bubuk SIK + BSIG 1.5%, G3: bubuk SIK + BSIG 2.5%, G4: bubuk SIK + BSIG 3.5%. Uji kekerasan menggunakan vickers hardness tester. Data dianalisis menggunakan uji ANOVA satu arah. **Hasil:** Nilai rata-rata hasil kekerasan dari terendah ke tertinggi adalah G0 (55.5 ± 2.70 VHN), G1 (56.9 ± 3.36 VHN), G2 (63.7 ± 7.73 VHN), G3 (66.3 ± 1.44 VHN), dan G4 (70.1 ± 4.72 VHN), sedangkan hasil uji ANOVA satu arah signifikan diantara semua kelompok (p -value < 0.05). **Kesimpulan:** Penambahan nanopartikel BSIG meningkatkan kekerasan SIK. Nilai kekerasan tertinggi diperoleh dengan penambahan nanopartikel BSIG 3.5%.

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INTRODUCTION

Conventional *Glass Ionomer Cement* (GIC) is often used in dentistry as a restorative material because it has advantages such as being biocompatible with pulp tissue, tooth-colored restorative material, binding well to the tooth structure, and releasing fluoride as an anti-cariogenic (Diansari *et al.*, 2016; Roeroe *et al.*, 2015). However, it has disadvantages, such as low fracture and wear resistance, so it is not applied for dental restoration with large loads, brittle, sensitivity to moisture, and porosity (Chen *et al.*, 2016). The hardness properties of GIC influence that characteristic (Rizzante *et al.*, 2015; Barandehfard *et al.*, 2016; Rodrigues *et al.*, 2015; Roeroe *et al.*, 2015; Barandehfard *et al.*, 2016).

Hardness is a mechanical property related to restorative materials' abrasion and compressive strength (Khan *et al.*, 2015). Material hardness in dentistry is used as a measuring instrument to determine the ability of restorative material to withstand masticatory pressure. The eligible surface hardness of the restorative material affects the restoration's clinical success by increasing its resistance to abrasion and preventing it from deforming under various forces. The restorative material must have high hardness values to preserve the integrity of the restoration against chewing forces in the oral cavity (Karakas *et al.*, 2021). Low hardness leads material to have less resistance, which leads to a fracture (Anusavice *et al.*, 2022). Therefore, adding *hydroxyapatite* (HA) to glass ionomer cement aims to improve the mechanical properties (Almuhaiza, 2016).

Hydroxyapatite can improve the compressive strength, diametrical tensile strength, and flexural strength of GIC (Gjorgievska *et al.*, 2020; Kheur *et al.*, 2020). The content of HA (w/w) can be obtained in the scales of gourami fish (*Osphronemus gourami*) by 16% to 59% (Sockalingam *et al.*, 2015; Dewanti *et al.*, 2020). Previous study found that the addition of *Gourami Fish Scale Powder* (GFSP) to GIC with concentrations of 2.5%, 5%, and 10% influenced the character of GIC. The addition of 2.5% GFSP caused a decrease in the level and diameter of pores, marginal gap, and increased compressive strength. Raising 5% and 10% concentrations increases the level and diameter of pores, marginal gap, and compressive strength (Dewanti *et al.*, 2022; Wulandari *et al.*, 2022). This result may occur because the GFSP used is not nano-sized.

Nano-size improves the material's mechanical properties (Al-Hamaoy *et al.*, 2018). The nanoparticles will be perfectly embedded in the GIC matrix and form good adhesion bonds (Alatawi *et al.*, 2019; Alobiedy *et al.*, 2019). *Nano-hydroxyapatite* (Nano-HA) was added to conventional GICs and improved mechanical properties such as biaxial flexural strength, compressive strength, and diametral tensile strength compared to the conventional GICs (Amin *et al.*, 2021). Previous studies

showed that adding 0.5%, 1.5%, 2.5%, and 3.5% GFSP nanoparticles increases GIC tensile strength (Cahyanisa *et al.*, 2023) and significantly enhances the antibacterial activity of GIC against *S. aureus* and *S. mutans* (Wulandari *et al.*, 2023). Thus, this research determines the influence of adding 0.5%, 1.5%, 2.5%, and 3.5% GFSP nanoparticles to the GIC hardness. It is hoped that the addition of GFSP nanoparticles can increase the hardness of GIC.

MATERIAL AND METHOD

This research is an experimental laboratory study with a post-test only control group design. The research was conducted in the Bioscience Laboratory Jember Dental and Oral Hospital (RSGM) University of Jember. The number of samples was in a 25 tablets form divided into five groups, each group containing five samples, as described (1) G0/control group: GIC, (2) G1: GIC + 0.5% nano GFSP, (3) G2: GIC + 1.5% nano GFSP, (4) G3: GIC + 2.5% nano GFSP, (5) G4: GIC + 3.5% nano GFSP.

Manufacturing of gourami fish scales powder nanoparticle

The clean gourami scales were dried by freeze drying, mashed with a blender (Cosmos, Jakarta, Indonesia), and sieved using a 200 mesh sieve (ABM Test Sieve Analysis, Jakarta, Indonesia) (Wulandari *et al.* (2022). *Nano-sized GFSP* (nGFSP) was obtained by grounding the powder fish scales using a ball mill (Piras *et al.*, 2019). Furthermore, the pH of nGFSP was measured, and particle size was analyzed using a Particle Size Analyzer (Horiba-SZ 100z, California, US) (Cahyanisa *et al.*, 2023).

Sample preparation

Nano-sized GFSP (nGFSP) and *Glass Ionomer Cement* (GIC) powder (GC Fuji 9 Gold Label High Strength Posterior Extra, Tokyo, Japan) were weighed using analytical scales (Adam type p 254, England, UK) and mixed using a vortex (Labinco L46, Breda, Netherland) to be homogeneous (Mawadara *et al.*, 2016). The ratio and composition of GIC powder and nGFSP can be seen in Table 1

Powders in each group were manipulated into GIC liquid in a ratio of 3.4 : 1 over a paper pad using an agate spatel for 25 seconds (according to the manufacturer's instructions). The homogeneous dough was put into an acrylic mold (Riya printing, Jember, Indonesia) with a diameter of 5 cm and a height of 2 cm, then condensed with a cement stopper (Schezher, Berlin, Germany). The top surface of the sample was covered with celluloid strips (Tor VM Transparent Striprill, Moscow, Russia), loaded with 0.5 kg for 20 seconds, then put in a closed container (Onyx Jakarta, Indonesia) for 24 hours. The sample was removed from the mold, immersed

with aquadest in a sealed container, and then put in an incubator (Biobase BJPX-H123II, Karnataka, India) at 37 °C for 24 hours. The sample was dried using an air spray (Spray Nozzles, Tokyo, Japan) for 20 seconds (Mawadara et al., 2016).

The measurement of hardness

Samples were tested for hardness using the Vickers method with a load of 50 gf for 15 seconds (Vickers Hardness Tester, Future-Tech FM-800, Tokyo, Japan). This method aims to determine the hardness of a material in the form of material resistance to a pyramid-shaped

diamond indenter that is quite small with a peak angle of 136° which is emphasized on the surface of the test material. The hardness test was carried out randomly on each sample at three different places, then the results were averaged (Yudhit et al., 2021).

Statistical analysis

The data obtained were tested for normality using *Shapiro-Wilk* and *Levene* homogeneity tests. A statistical test was performed using a *One Way ANOVA* (p -value > 0.05), followed by a *Least-Significant Difference* (LSD) test (p -value > 0.05) using SPSS version 25 (Windows, US).

Table 1. Ratio and composition of conventional *Glass Ionomer Cement* (GIC) powder and *Nano-Sized GFSP* (nGFSP)

Groups	Ratio of GIC powder to nGFSP	GIC powder (g)	nGFSP (g)
GIC	100 : 0	0.2347	0
GIC + nGFSP 0.5 %	99.5 : 0.5	0.2335	0.0012
GIC + nGFSP 1.5 %	98.5 : 1.5	0.2312	0.0035
GIC + nGFSP 2.5 %	97.5 : 2.5	0.2288	0.0059
GIC + nGFSP 3.5 %	96.5 : 3.5	0.2265	0.0082

RESULT

The normality test results showed a significant value (p -value > 0.05) in all groups, meaning the data were normally distributed. Based on *Levene's* test, the significance value shows (p -value > 0.05), so it can be concluded that the data are homogeneous. The result of the *One Way ANOVA* test showed that each

group's average hardness value differed significantly (p -value < 0.05). The *Least Significant Difference* (LSD) test showed that there was a significant difference (p -value < 0.05) between groups G0 and G2, G3, G4; G1 with G2, G3, G4; G2 with G4. Meanwhile, the G0 with G1, G2 with G3, and G3 with G4 did not show a significant difference (p -value > 0.05). The average hardness value from the highest to the lowest, namely G4, G3, G2, G1, and G0 as shown in Figure 1.

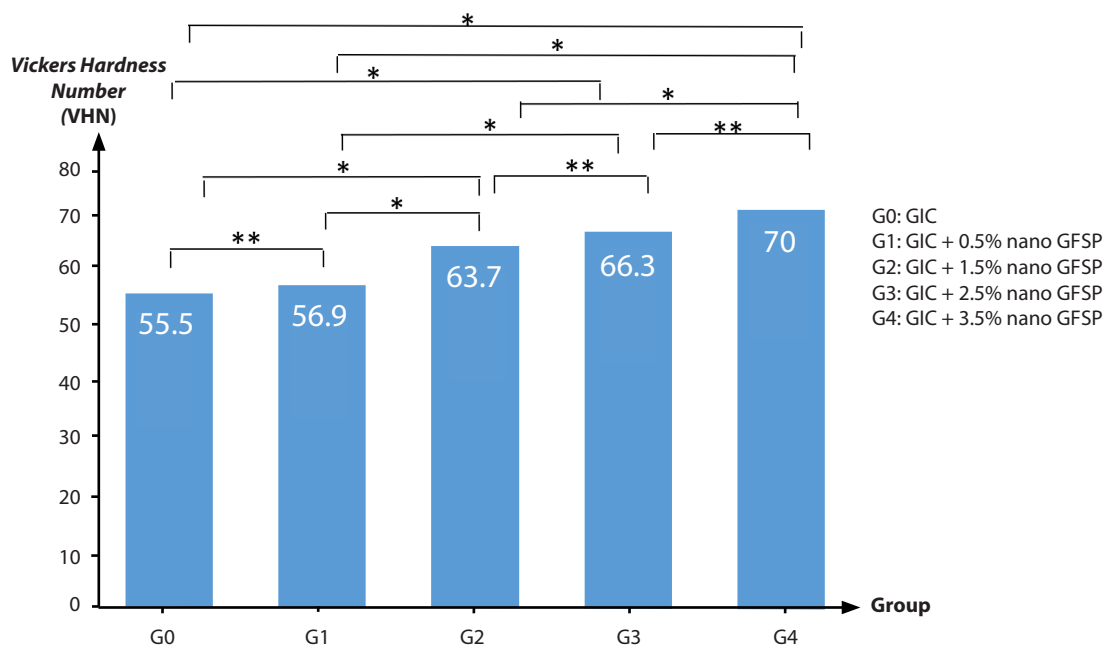


Figure 1. The average hardness value and test for group differences (* p -value < 0.05, ** p -value > 0.05)

DISCUSSION

The results showed that the sample group with the addition of *Gourami Fish Scale Powder* (GFSP) nanoparticles (G1, G2, G3, G4) had a higher hardness value than the control group (G0). This is because when GFSP is added to *Glass Ionomer Cement* (GIC) powder mixed with the GIC liquid, calcium ions from GFSP, which act as additional calcium in GIC powder, will be released from *hydroxyapatite* (HA). It will initiate an acid-base reaction and release aluminum, sodium, and fluoride ions from the surface of GIC powder to form more cross-linking structures (Mozartha, 2015). It can increase the degree of acid-base reaction in the GIC structure to create a denser and stronger cement. HA from GFSP fills the gap between the glass particles in the empty GIC, preventing pores formation. The absence of pores increases material hardness (Mozartha, 2015).

The results also showed that the higher the addition of GFSP concentration, the higher the GIC hardness value. This is thought to be due to an increase in the concentration of HA in the GFSP into GIC powder, resulting in more saturated GIC powder, which will have an impact on improving the degree of density of GIC powder (Mozartha, 2015). The higher the concentration of HA, the more calcium binds to the polyacrylic acid in the GIC fluid, increasing the degree of acid-base reaction in the GIC structure. It will cause an increase in cross-linking in the GIC, thereby increasing the density and strength of the GIC and preventing the formation of pores (Mozartha, 2015; Barandehfard *et al.*, 2016).

Hydroxyapatite (plays a role in the chemical changes that occur during the initial hardening of the GIC. HA will dissolve rapidly at a pH below 2.05 when mixed with polyacrylic acid with a pH of 1.23 (Khurshid *et al.*, 2015). An acidity degree (pH) test was carried out to ensure the degree of acidity of the GFSP. The results show that the GFSP is alkaline (7.82), added to the alkaline GIC powder to react with GIC liquid through an acid-base reaction. GIC liquid is an acidic proton donor, and GIC powder is an essential proton acceptor (Anusavice *et al.*, 2022).

The gourami fish scale powder in this research had a size of 51.77 nm (nanoparticle). Nano-size particle aims to better mix the HA in GFSP and GIC powder with a particle size of 10 μm (Rahman *et al.*, 2014). Nanoparticles are small particle sizes with a larger contact surface area, increasing the adhesion force between particles of a mixture of GFSP and GIC powder (Alobiedy *et al.*, 2019). The addition of HA nanoparticles to GIC showed better mechanical properties than conventional GIC (Rahman *et al.*, 2014; Moheet *et al.*, 2020). HA nanoparticles are perfectly embedded in the GIC matrix, increasing the formation of salt bridges and filling the space between the GIC particles, thereby improving mechanical and physical properties (Al-Hamaoy *et al.*, 2018; Alatawi *et al.*, 2019). The addition of HA nanoparticles in GFSP is proven to increase the hardness of GIC.

The sample in this research was immersed in water to obtain a completely hardened sample. Water plays an important role in the setting reaction. It acts as a reaction medium in releasing the remaining GIC monomer to complete the GIC polymerization. Water is a reaction medium that releases and transports calcium and aluminum ions in GIC powder to react with polyacids to form a polyacrylate matrix (Anusavice *et al.*, 2022).

CONCLUSION

The research concludes that adding GFSP nanoparticles increased the hardness of conventional GIC. The highest hardness value was obtained with the addition of 3.5% GFSP. Further research is needed regarding adding GFSP nanoparticles to improve other mechanical and physical properties of GIC.

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