The effect of incorporating hydroxyapatite into Type II glass ionomer cement on flexural strength and the examination of fractured surfaces using scanning electron microscopy

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ABSTRACT

Background: *Glass ionomer cement (GIC) is widely recognized as the prevailing direct esthetic restorative material. In order for a restoration material to be considered effective, possess favorable physical and mechanical properties. Incorporating with hydroxyapatite (HA) can provide these properties. This research is to investigate the impact of integrating HA into GIC on the flexural strength and scanning electron microscopy (SEM) of the fractured surfaces.*
Methods: *The study consisted of 28 samples of GIC, which are fabricated in the shape of rectangular prisms (25x2x2mm). The samples were divided into four groups n=7. Group 1 is a control group (GIC), Group 2 GIC+2%HA, group 3 GIC+2%HA, and GIC+4%HA. The flexural strength of the sample was evaluated by a Universal testing machine, followed by an examination of the fracture surface using SEM.*
Result: *The Brown-Forsythe was used as data analysis to examine the flexural strength values across all groups, resulting in a statistically significant p-value of less than 0.05. The addition of HA does not result in a significant increase in the flexural strength value of the glass ionomer cement (GIC). The integration of microstructure based on scanning electron microscopy (SEM) pictures demonstrates improved visual quality following the incorporation of HA.*
Conclusion: *The addition of HA to GIC did not result in a significant change in flexural strength compared to the control group. However, GIC group with 2% HA exhibited the highest average flexural strength value among all the groups*

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INTRODUCTION

Caries is a pathological condition affecting the calcified structures of the tooth, which is distinguished by the degradation of tissue on the tooth surface, namely in the pit, fissure, and interproximal regions.[1] In severe cases, the damage may progress to involve the pulp region. Based on the findings of the 2018 RISKESDAS survey, it was observed that a significant proportion, specifically 45.3%, of the Indonesian population exhibited dental caries in their teeth.[2,3] The presented data depicts a notable lack of awareness among the Indonesian community regarding the need of maintaining oral health, as evidenced by the fact that 45.3% of individuals in Indonesia have untreated dental caries. The issue of dental caries can be addressed with the conservative approach of tooth extraction. Tooth shedding is a dental procedure aimed at restoring teeth by eliminating carious tissue and applying restorative materials to damaged teeth. Direct esthetic restorative is a commonly employed restoration material within the realm of dentistry. The direct esthetic restorative material that is most frequently utilized is glass ionomer cement (GIC).[4] Wilson and Kent in 1971 first introduced GIC as a dental restoration material.[5] The GIC (Glass Ionomer Cement) is a composite material consisting of calcium fluoroaluminosilicate glass (silicate cement) and a polycarboxylate acid solution. Its purpose is to achieve translucency and release fluorine from the silicate cement, while also possessing the capacity to chemically bond to tooth structure through silica gel.[6] The classification of GIC is type I as lutting agent, type II.[1] as aesthetic filling materials, type II.[2] as bis reinforced filling materials, and type III as lining, base & fissure sealing materials. Glass ionomer cement (GIC) in the field of dentistry offers several notable advantages. Firstly, it is characterized by its ease of manipulation, allowing for convenient application. Additionally, GIC exhibits a coefficient of thermal expansion that closely matches that of tooth structure, therefore minimizing the risk of structural damage. Moreover, GIC demonstrates favorable biocompatibility, ensuring compatibility with the oral environment. Furthermore, GIC has the ability to release fluorine, contributing to its potential for dental caries prevention. Lastly, GIC possesses commendable physical properties, further enhancing its suitability for dental applications.[7] Nevertheless, it should be noted that GIC exhibits the drawback of being readily soluble in water.[8] The impact of a material's water absorption and solubility on the mechanical properties, such as malleability, compressive strength, tensile strength, hardness, and surface roughness, of restorative materials has been observed.[9] Based on the results of research by Hakim et al (2013), as many as 64% experienced GIC collision failure, which is easily broken (brittle).[10] This finding demonstrates the limitations of the GIC. In the context of restoration, it is imperative for the materials utilized to possess favorable physical and mechanical qualities. Various attempts were undertaken to address the deficit of GIC by incorporating fillers into its components. Hydroxyapatite (HA) can serve as a suitable filler in this context. HA has the potential to serve as a suitable filler for many repair materials, including composite resins, glass ionomer cement, among others. HA is a bioceramic that has the chemical formula $Ca_{10}(PO4)_6(OH)_2$ and has similarities with the main minerals that make up bones and teeth.[11] HA is proven to have good biocompatibility properties and can be tolerated very well by human oral cavity tissues.[12] HA is used as a filler for adding GIC mechanical strength. Based on Nilandasari's research (2018) showed that type II GIC powder experienced an increase in compressive strength after adding 8%

hydroxyapatite from purebred chicken egg shells.[13] While Khagani et al (2016) researchers showed that there were differences in compressive strength values in GIC added with nano and HA microparticles from bovine femur bones as much as 1%, 3%, 5%, and 7%. The maximum mechanical strength results occur with the addition of 1% hydroxyapatite and decrease in the addition of 3%, 5%, and 7% nano hydroxyapatite.[14] Based on the aforementioned context, the researchers want to investigate the impact of including pure hydroxyapatite at varying concentrations (2%, 3%, and 4%) into type II glass ionomer cement. The focus of this investigation is to assess the effects of these additions on the flexural strength and particle interaction of the composite material. The evaluation of these properties will be conducted using scanning electron microscopy (SEM).

Research Methods

Sample preparation

The sample was made from type II GIC material, namely the Fuji IX brand which consisted of 28 sample of GIC and fabricated in the shape of rectangular prisms with dimensions of 25x2x2mm. Group 1 represents the control group, wherein no HA is added to the GIC. Group 2 comprises the addition of 2% HA to the GIC. Group 3 involves the addition of 3% HA to the GIC. Lastly, group 4 encompasses the addition of 4% HA to the GIC. The GIC utilized in this study is the Fuji IX brand GC-GIC, specifically designed for restorations in posterior teeth. The GIC powder and liquid are combined on a paper pad in a specific ratio of 0.33 grams of powder to 0.13 grams of liquid. This mixture is mixed using an agate spatula in a folding motion on the paper pad for a duration of roughly 1 minute and 30 seconds. In the experimental group, the dough is combined with HA at varying concentrations, specifically 2%, 3%, and 4%.. Subsequently, the upper surface of the specimen is coated with celluloid plastic and subjected to a load of 1 kg. The specimen is then allowed to set for approximately 11 minutes before being carefully removed from the mold. The sample is comminuted using a stone bur and subsequently measured to meet the appropriate dimensions. Following this, the sample is immersed in distilled water at a temperature of 37°C for a duration of 24 hours.

Flexural Strength Test

The flexural strength of the test specimen was evaluated using a Shimadzu AGS-X Universal testing machine, manufactured in Japan. The length of the test rod was accurately measured and recorded along its center line using a pencil. The test rod that has been marked is positioned at the central location of the tool, following which the transverse indenter is affixed onto the Autograph tool. The test specimen is positioned on two transverse pedestals at a specific distance, and the cross head velocity is adjusted to 0.5 mm/min. The load button is activated in order to reset the number to the zero position. The machine's power button is activated, and then, the down button is pressed on the center line of the test bar using a tool ballast. This action is performed in order to apply the maximum load or force in the transverse direction, potentially resulting in the fracture or breakage of the sample.

Scanning Electron Microscopy (SEM) Analysis

The specimens subjected to flexural strength testing were coated with a thin layer of Osmium using a plasma Os coater (HPC-20, Vacuum Device Co., Ltd., Ibaraki, Japan). Subsequently, the coated specimens were examined using a scanning electron microscope (SEM) (S-4800, Hitachi High-Technologies Co., Tokyo, Japan).

Data Analysis

It is imperative to ascertain the normality of the data by doing normality tests such as the Shapiro-Wilk test, as well as assessing homogeneity using Levene's test. Following the application of the Shapiro-Wilk test, it was determined that the data exhibited normal distribution ($p<0.05$). Subsequently, the homogeneity test revealed that the data displayed heterogeneity ($p<0.05$). The data, which exhibited both normal and inhomogeneous distribution, was subjected to data analysis employing the Brown-Forsythe test in order to assess the variations in flexural strength values across all groups. Following the administration of the Brown-Forsythe test, the subsequent step involved conducting the Games-Howell test in order to ascertain the presence of statistically significant differences among the various treatment groups.

Results

The addition of fillers to the components of type II GICs has the potential to enhance the FS strength value. One potential filler material that might be utilized is hydroxyapatite.[15] According to a study conducted by Nilandasari (2018), it was observed that the addition of 8% hydroxyapatite derived from the eggshell of purebred chickens (Gallus gallus) resulted in an enhancement of compressive strength in Type II GIC powder.[13] Hydroxyapatite, a bioceramic with the chemical formula $Ca_{10}(PO4)_6(OH)_2$,exhibits resemblances to the primary minerals constituting bones and teeth.[11] Hydroxyapatite has been empirically demonstrated to possess favorable biocompatibility characteristics, rendering it highly compatible with human oral tissues.[12] The subsequent data presents the outcomes of the FS test conducted using predetermined maximum force (P) values, which will then undergo processing by inputting them into the FS calculation method.

$$S = \frac{3PL}{2bd^2} \text{ N/mm}^2$$

Information:
S : Flexural strength (MPa)
L : Support length/distance (mm)
b : Sample width (mm)
d : Sample thickness (mm)
P : maximal force (N)

Table 1. Mean and standard deviation of flexural strength of each group

Group	Mean	Std.Deviation
Control group	6.55	±0.2156
GIC+2%HA	6.65	±0.1694
GIC+3%HA	6.15	±0.3721
GIC+4%HA	4.48	±0.3459

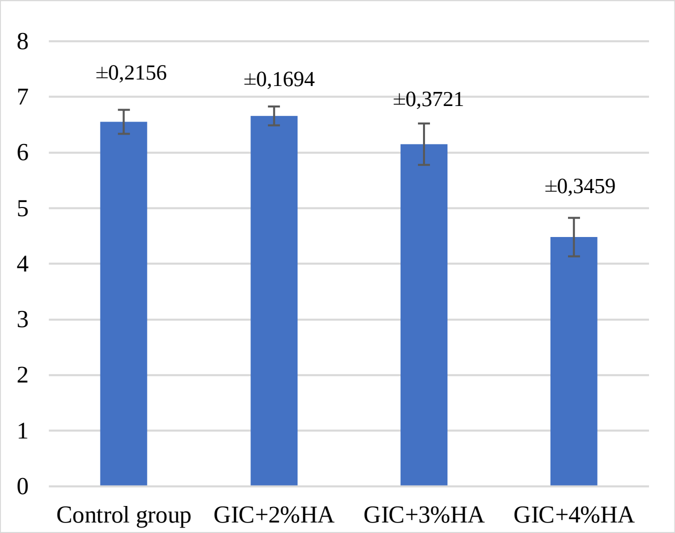

Figure 1. Mean and standard deviation of flexural strength of each group

The data in this investigation had a normal distribution and lacked homogeneity, necessitating the utilization of the Brown-Forsythe test for assessing differences. Subsequently, a Post Hoc analysis was conducted, followed by the Games-Howell Test.

Table 2. Difference Test: Robust Tests of Equality of Means

Flexural strength	Statistic	df1	df2	Sig.
Brown-Forsythe	12.257	3		0.000*

*Significance difference (p<0.05)

Table 3. Post Hoc Test – Multiple comparison Dependent Variable: Flexural strength (Games-Howell)

Group	Control group	GIC+2%HA	GIC+3%HA	GIC+4%HA
Control group		0.980	0.787	0.001
GIC+2%HA			0.613	0.000
GIC+3%HA				0,020
GIC+4%HA				

The purpose of employing the Post Hoc Games-Howell test is to investigate potential disparities between two distinct groups. The utilization of scanning electron microscopy (SEM) allowed for the characterization of HA particles, leading to the observation that the morphology of the resultant HA-in GIC consists of a combination of spherical silica particles and rod-shaped HA particles. The image presented below provides a summary of the scanning electron microscopy (SEM) conducted in each group, with magnifications of 250x and 15,000x.

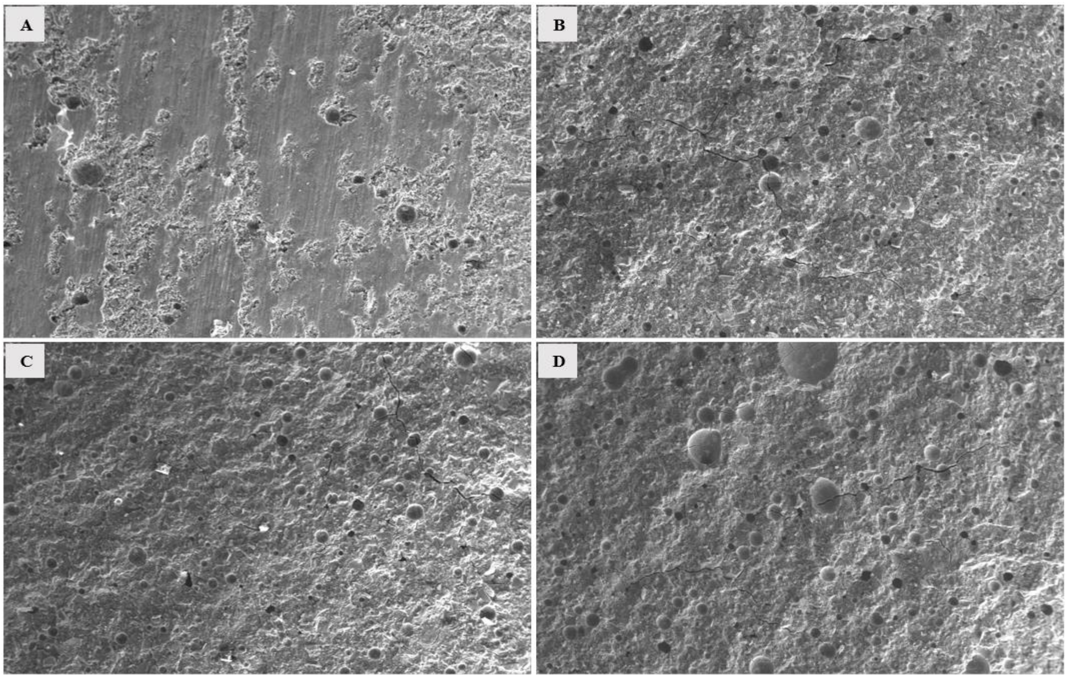

Figure 2. SEM results of sample fracture area after flexural strength test in each group with magnification of 250x (A. Control group, B. GIC group + 2% HA, C. GIC group + 3% HA, and HA + GIC group 4%)

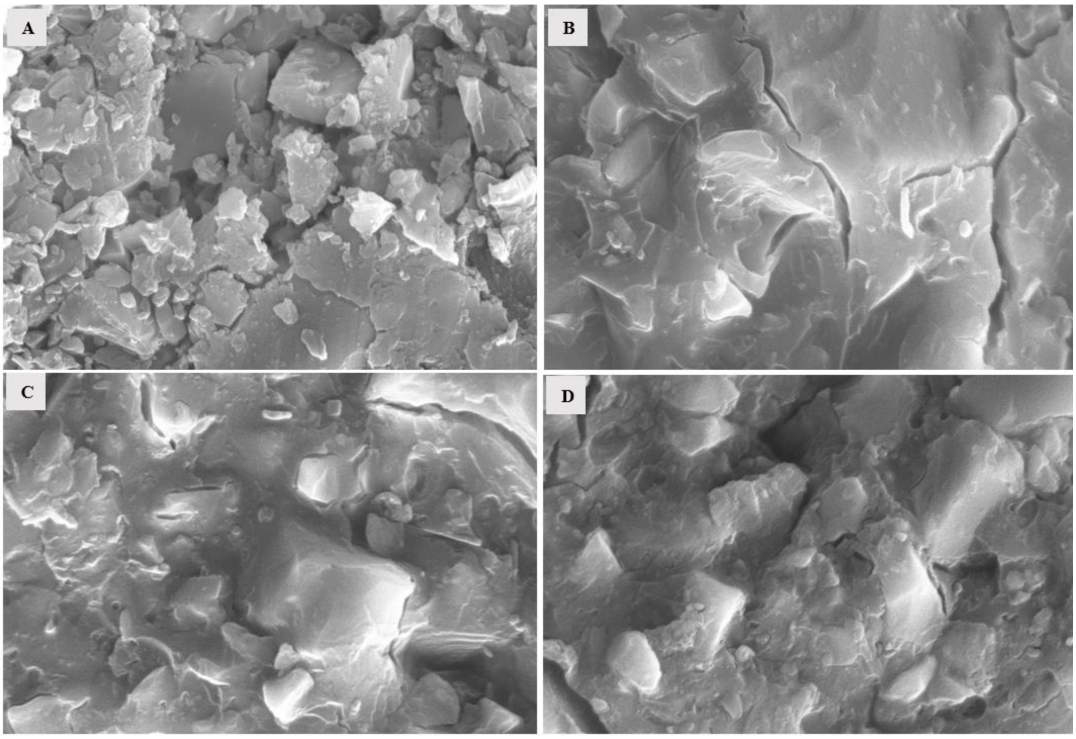

Figure 3. SEM results of sample fracture area after flexural strength test in each group with magnification\ of 15,000x (A. Control group, B. GIC + 2% HA group, C. GIC + 3% HA group, and GIC + 4% HA group).From the results of the Brown-Forsythe test above, it is known that the value of p = 0.000 < 0.05 means that there are significant differences in all groups

Discussion

In order for a repair material to be considered effective, it is imperative that it possesses favorable mechanical qualities. The possession of mechanical qualities is a significant characteristic exhibited by a restoration material.[16] The mechanical qualities of a material play a significant role in determining both its fragility and weakness. The control group had mean and standard deviation values of 6.55±0.21 MPa, while P1 had mean and standard deviation values of 6.65±1.69 MPa. The findings of the study demonstrated a notable enhancement in the flexural strength of type II GIC powder upon the incorporation of 2% HA. The enhancement in mechanical strength can be attributed to chemical transformations that take place during the early solidification of GIC materials, which incorporate HA. HAis a particulate substance that undergoes dissolution in the presence of acidic conditions. The solubility of HA exhibits a significant rise when it comes into direct contact with polycarboxylic acid. Under these circumstances, the release of calcium ions occurs from the HA surface, leading to an elevation in the acid-base reaction intensity. Consequently, salt bridges are introduced into the structure of GIC, resulting in the formation of cross-links and ultimately yielding a more robust cement. The use of HA into GICs has been found to enhance their density by effectively occupying the interstitial spaces between glass particles within the GIC matrix.

The response mechanism observed exhibits similarities to the adhesion mechanism between enamel and dentin, resulting in an enhancement of mechanical strength. The incorporation of HA particles into GIC powder has been found to enhance the fracture toughness of cement materials, enabling them to establish and sustain long-term adhesion with dentin[18]. The findings also indicated a reduction in flexural strength within the cohort that incorporated 3% HA and 4% HA. This assertion aligns with the findings of Khagani et al. (2016), who conducted a study demonstrating variations in compressive strength values of GICs when supplemented with nano and micro HA particles derived from bovine femur bones at concentrations of 1%, 3%, 5%, and 7%. The highest levels of mechanical strength are observed when incorporating 1% hydroxyapatite, while the addition of 3%, 5%, and 7% nano HA leads to a drop in mechanical strength.[14] The incorporation of HA at concentrations over 1% has been observed to adversely affect the mechanical characteristics. This is attributed to an imbalance in the ratio of HA powder to GIC powder, resulting in insufficient formation of polycarboxylate acid. Consequently, the hydrolysis of ionomeric HA is hindered, leading to the formation of an inadequate matrix and a subsequent drop in resistance.

Additionally, it suggests that there could be additional attributes of HA, such as particle size and particle surface area, that have an impact on the compressive strength of GIC[20]. The study's treatment group yielded statistically significant differences in its results. The control group, when compared to the treatment group receiving 2% HA in conjunction with GIC, yielded a p value greater than 0.05 (p = 0.98) in the Post Hoc Games-Howell method. This indicates that there is no statistically significant difference between the two groups. The GIC used as a control exhibits an FS value that does not demonstrate a statistically significant difference when compared to a GIC supplemented with an additional 2% HA. This finding suggests that the addition of HA does not result in an increase in the FS value of the GIC. The findings of the Post Hoc difference test indicate that there is a statistically significant difference between the control group and the GIC+4%HA group, as well as between the GIC+2%HA and GIC+4%HA groups, and between the GIC+3%HA and GIC+4%HA groups ($p<0.05$). These results suggest that there is a distinction between all groups when a combination of GIC with 4% HA is present. This is supported by the fact that the GIC+4%HA group exhibits the lowest FS value among all groups and shows a significant difference as more HA is added. Subsequently, the formation of polycarboxylate acid is shown to be inadequate in its ability to facilitate the hydrolysis of ionomeric HA, resulting in an insufficient matrix and a consequent reduction in resistance. This finding suggests a notable impact resulting from the incorporation of HA. The enhancement of the mechanical characteristics of GIC can be attributed to various aspects associated with HA, including the morphology of HA powder, the presence of HA pores, and the size of HA grains.[21] It is well acknowledged that nano HA, characterized by particles smaller than 100nm, exhibits remarkable reactivity and possesses a fine microstructure akin to the primary mineral constituents found in bones and teeth. This aligns with the findings of a study that utilized HA SIGMA Aldrich nanoparticles. The synthesis technique plays a significant role in influencing the morphology, crystallography, and phase purity of HA particles. Consequently, when HA material is combined with GIC fillers, it can impact the mechanical characteristics of the repair material.[22] The FS strength value is subject to variation based on several factors inside the test, such as the amount of variables involved, including manipulation methods, particle size, GIC types, and specimen sizes. Hence, challenges arise when attempting to establish a standardized measure for the mechanical strength of GICs.[23]

The therapeutic applications of GIC materials are significantly influenced by their flexural strength. GICs exhibit a notable capacity for high flexural strength, hence offering commendable stability and resistance to functional loads when employed as a dental restorative material. The structural integrity of the GIC enables it to endure the mechanical stress and masticatory forces exerted during the act of food mastication. Furthermore, it should be noted that a high FS can play a significant role in reducing the likelihood of cracking or rupture occurring in GIC restorations. This, in turn, can contribute to the longevity and overall effectiveness of these restorations.[18] In a broad sense, dental materials utilized for impact resistance must possess a sufficient factor of safety to endure the forces exerted during mastication, including pressure and chewing forces. The bending strength of dental restorative materials is often advised to fall within the range of 50-100 MPa.

Nevertheless, the aforementioned value may exhibit variability contingent upon the specific nature and geographical context of the intended dental intervention.[8] No particular suggested percentage of HA addition exists for the material used in tooth fillings. Nevertheless, previous research has incorporated varying percentages of HA into GICs, with concentrations ranging from 1% to 28%.[14,17] The proportion of hydroxyapatite (HA) addition may exhibit variability contingent upon the intended use and the specific attributes of the GIC material employed. Alternatively, it is advisable to seek guidance from the manufacturer or a dental expert in order to ascertain the optimal proportion of HA to be incorporated in accordance with the particular requirements of your application.

The SEM results reveal that the HA-silica powder is effectively incorporated within the GIC matrix. This integration allows the HA-silica powder to occupy the voids between GIC particles, leading to enhanced mechanical properties of the GIC material. The mechanical properties of GIC20 are influenced by various factors, including particle shape, size, particle surface area, and the presence of HA. The scanning electron microscopy (SEM) image of the control group, where no HA was added, reveals that there is a lack of binding between the glass particles and glass matrix. This deficiency is commonly observed in GIC and is attributed to a disparity in the mechanical properties of the glass ionomer matrix and the glass particles. Specifically, the modulus of elasticity plays a significant role in generating substantial stress accumulation between these two components. In the experimental group where HA was introduced, there was a reduction in the bonding of glass particles and the glass matrix. As a result, the fracture surface exhibited a smoother appearance and the integration between the glass particles and matrix was enhanced. The incorporation of H) leads to an improvement in microstructure integration. The SEM images reveal alterations in the morphology and dimensions of HA. This is likely attributed to the total encapsulation of HA by cementitious material. Consequently, it can be inferred that a robust interfacial adhesion exists between GIC and HA. However, additional investigation is warranted to comprehensively understand this phenomenon[20].

Conclusion

The findings pertaining to the flexural strength The inclusion of HA in the study did not yield a statistically significant difference when compared to the control group (which did not incorporate HA). However, it is worth noting that the average results in the group that utilized 2% HA in conjunction with GIC had the greatest FS value in comparison to the other groups. The integration of microstructure based on SEM images exhibits improved characteristics following the incorporation of HA. This is evident through the enhanced smoothness of the surface and the filling of void spaces with glass particles, glass matrix, and HA. Consequently, it can be inferred that a robust bond exists between GIC and HA, necessitating further investigation. Additional investigation is required to ascertain the optimal proportion of HA incorporation that can effectively enhance flexural strength in alignment with clinical applications within the domain of dentistry.

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