

# IMPROVING THE MECHANICAL PROPERTIES OF GLASS IONOMER CEMENT

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## ABSTRACT

**Background:** Glass ionomer cement (GIC) exhibits limitations such as low mechanical strength, poor fracture resistance, and limited durability, restricting its clinical applications. This study aimed to evaluate the impact of nano-hydroxyapatite (nHA) incorporation on the mechanical properties of GIC, focusing on tensile strength, fracture resistance, fatigue limit, and microhardness.

**Materials & Methods:** Study type is experimental, the research is done in faculty of dentistry, University of Al Maarif, Al Anbar, Iraq. All samples were collected and tested in a duration of two years (2023 to 2025). A total of 50 GIC formulations were prepared, divided into control (25 samples without nHA) and modified (25 samples with nHA) groups. Mechanical tests were performed to measure tensile strength, fracture resistance, fatigue limit, and microhardness. Data were analyzed statistically to compare the two groups.

**Results:** The modified GIC group exhibited significantly higher tensile strength (25.41 MPa vs. 20.21 MPa), fracture resistance (101.48 N vs. 84.15 N), fatigue limit (12,659 cycles vs. 8,095 cycles), and microhardness (62.80 VHN vs. 48.27 VHN) compared to the control group ( $p < 0.05$  for all). Regression analysis highlighted tensile strength and fracture resistance as strong predictors of microhardness.

**Conclusion:** The incorporation of nHA significantly improved the mechanical properties of GIC, making it a promising material for dental restorations requiring enhanced strength and durability.

**KEY WORD:** Dental cements; Glass ionomer cement; Microhardness; Nano-hydroxyapatite; Tensile strength.

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## INTRODUCTION

Glass ionomer cement (GIC) is a cornerstone material in dentistry, widely utilized for its chemical adhesion to tooth structure and fluoride-releasing properties, which are essential for caries prevention and tooth remineralization. It is considered as one of the most used dental cements due to its good chemical bond to tooth structures and the restoration.<sup>1</sup> Its ease in use, bacteriostatic effect, chemical setting and bonding, good tensile strength make it always the cement of choice to be used as a permanent luting cement and temporary restoration. Despite many dental cement types, GIC can be used to lute both metal and ceramic restoration alongside cementing all types of dental

posts.<sup>2</sup> On the other hand, conventional GIC formulations face significant challenges, including poor mechanical strength and moisture sensitivity, limiting their use in high-stress areas of the dentition.<sup>3</sup> Advances in nanotechnology, such as the incorporation of nanoparticles, have shown promise in enhancing these mechanical properties and expanding clinical applications.<sup>1</sup> Moreover, resin modifications have improved durability and aesthetic outcomes, making GICs a viable alternative to traditional materials in minimally invasive dentistry.<sup>4</sup> Research continues to innovate GIC formulations to meet the demands of modern clinical dentistry, ensuring optimal outcomes for both patients and practitioners.<sup>5</sup>

Many researchers were conducting the improvement of GIC, such as incorporating nano-hydroxyapatite (nHA) into glass ionomer cement (GIC) to enhance biocompatibility and osteoconductivity, making it more suitable for dental restorations that require integration with bone and tooth tissues.<sup>1</sup> Researchers tried to improve the mechanical properties of the GIC by adding many materials to the cement in order to increase its lifetime and make it more able to resist masticatory force and provide good retention to the restorations. Improvement GIC is challenging due

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to its multiple uses in dentistry. It must have good chemical, mechanical and physical properties to overcome all the challenges in dental field.<sup>6</sup>

The modified glass ionomer with the incorporation of nHA enhances antibacterial properties, reducing the risk of secondary caries by mitigating bacterial growth around restorations.<sup>7</sup> The osteoconductive nature of nHA facilitates improved bonding strength to dental tissues, promoting better restorative outcomes.<sup>8</sup> Furthermore, its incorporation mitigates microleakage and cytotoxicity, ensuring long-term durability and safety in clinical applications.<sup>3</sup>

In term of mechanical properties, the addition of nHA could improve the compressive strength and fracture toughness, addressing limitations of conventional GIC in high-stress areas.<sup>4,6</sup>

The objectives of this study were focused on improving the mechanical and biological properties of glass ionomer cement through the incorporation of nano-hydroxyapatite. The study aimed to investigate whether the addition of nano-hydroxyapatite would lead to enhanced tensile strength, thereby improving the material's ability to withstand pulling forces. It also sought to determine the impact of this modification on fracture resistance, assessing whether the enhanced formulation could better resist breaking under applied forces. Another critical objective was to evaluate the fatigue limit, analyzing the material's ability to endure repetitive stress cycles without failure, which is a vital characteristic for dental and restorative applications. Furthermore, the study aimed to examine the microhardness of the modified glass ionomer cement to determine the surface durability and resistance to deformation. Through these objectives, the research sought to provide a comprehensive understanding of how nano-hydroxyapatite could be utilized to strengthen glass ionomer cement and enhance its performance in clinical applications.

## MATERIALS AND METHODS

Study type is experimental one, the research is done in faculty of dentistry, University of AlMaarif, Alanbar, Iraq. All samples were collected and tested in a duration of two year (2023 to 2025). 50 formulations were created, splitted equally into two groups. The first group in similar proxy terms consisted of 25 control formulations without nano-hydroxyapatite, while the second group consisted of 25 formulae with the addition of nano-hydroxyapatite. Tensile strength, fracture resistance, fatigue limit and microhardness of samples fabricated from each formulation were tested and evaluated. Statistical analysis was done using t-tests.

Two groups of formulations were prepared: Nano hydroxyapatite as a modifying agent, the second was in the form of a control group having no modifications. Measurement of glass ionomer cement powder was made with an analytical balance and na-

no-hydroxyapatite powder was separately weighed for the modified formulations calculated. The glass ionomer cement powder was thoroughly mixed with this additive, before mixing it all together thoroughly using a sterile glass spatula in a clean dry mixing dish. Measurements of the liquid component of the glass ionomer cement were made using a calibrated micropipette. Mixing of powder and liquid was performed in a sterilized manner.

The concentration of additive in all modified formulations were carefully measured based on nano hydroxyapatite particles using an analytical balance so that the concentration of additive was consistent. The additive nHA was blended with the glass ionomer cement powder before the liquid component is added for integration of nHA to the formulations. Finally, the blending process included placing the measured nHA powder in a sterilized glass mixing dish and the pre measured glass ionomer cement cement powder. A glass spatula was used to thoroughly mix the two components. A fixed duration was applied to this step so as to achieve a homogenous dry mixture. The powder mixture was then dry blended once finished, and then the liquid component of the glass ionomer cement was added. We measured the volume of the liquid delivered to each sample with a calibrated micropipette to ensure delivery with precise volume. Modified powder was then combined with the liquid and stirred during stirring of the mixture with a sterilized glass spatula. The additive was systematically added into glass ionomer cement formulations in order to effectively and consistently add nano-hydroxyapatite into the cement.

The samples were made to conform to the requirements for mechanical testing by means of molds of standardized dimensions. The molds were coated lightly with a thin layer of petroleum jelly. Carefully with a clean spatula, the glass ionomer cement paste was then placed into the molds to fill the cavities with the material so that no air could be entrapped. The molds were then cured under recommended curing conditions of glass ionomer cement. The samples were removed from the molds, and placed in distilled water at 37°C. The samples were prepared and shaped in standardized dumbbell form meeting the respective dimensions of the international testing standards. Each specimen was mounted on a universal testing machine grip. The sample was subjected to a controlled tensile force at a constant crosshead speed until breakage. The force needed to break each specimen was recorded and the tensile strength was calculated.

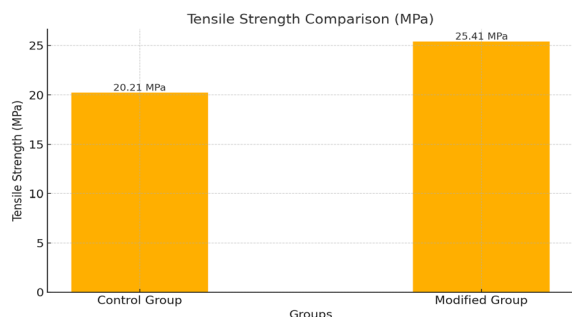
A controlled force was applied at the center of the beam and the specimen was positioned on a three point bending apparatus. The universal testing machine loaded samples at the midpoint at a constant rate until fracture, applying gradually increasing load in the process. Fracture resistance was recorded as the maximum force required to break the specimen.

The samples were mounted on a fatigue testing machine, which applied cyclic loading at a controlled frequency and amplitude to each sample. Repeated cycles of loading and unloading were used to subject the samples until fracture or a predetermined maximum number of cycles was achieved. Highly polished samples were fixed on the testing stage of a Vickers microhardness tester. A calibrated microscope was used to measure the size of the indentation, and then the Vickers Hardness Number (VHN) was calculated from applied load and impression dimensions. The surface variability was accounted for multiple measurements were taken at different locations on each specimen to ensure reliability of the data.

All data was collected and processed using SPSS and tested using t-tests for normally distributed data and Mann-Whitney U test for non-parametric data. The multiple linear regression analysis was conducted using the Ordinary Least Squares (OLS) method.

### RESULTS

A total of 50 GIC formulations were prepared, divided into control (25 samples without nHA) and modified (25 samples with nHA) groups. Mean tensile strength was significantly higher for the modified group, corresponding to improved resistance to pulling forces (Fig.1).



**Figure 1: Comparison of Tensile Strength (MPa) Between Control and Modified Glass Ionomer Cement Groups.**

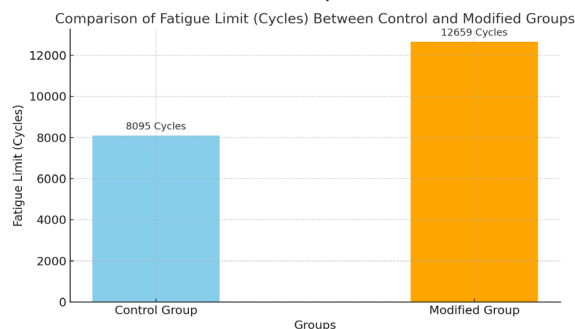
The mean value of fracture resistance for the modified group was considerably greater than control group in terms of fracture resistance. This result suggests that nano hydroxyapatite particles improved the material’s ability to withstand crack propagation under the applied forces or by normalizing the stress distribution of the cement matrix (Table 1).

Fatigue limit testing showed a huge increase in the

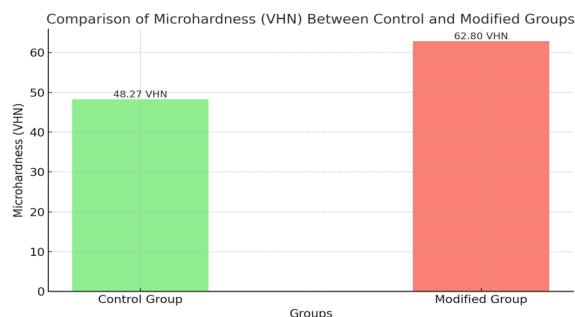
**Table 1: Statistical Comparisons between Control and Modified Glass Ionomer Cement Formulations**

Parameter	Control Group (Without Nano- Hydroxyapatite) (n=25)	Modified Group (With Nano-Hydroxyapatite) (n=25)	p value
Tensile Strength (MPa)	20.21 ± 1.69	25.41 ± 2.12	0.0000
Fracture Resistance (N)	84.15 ± 4.90	101.48 ± 6.14	0.0000
Fatigue Limit (Cycles)	8095.48 ± 870.39	12659.03 ± 922.17	0.0000
Microhardness (VHN)	48.27 ± 2.37	62.80 ± 4.16	0.0000

number of cycles that the modified formulations would endure before failure (Fig. 2). This improved performance then indicates the nano-hydroxyapatite inclusion not only strengthens the cement but also helps cement’s endurance under repeated stress over time.



**Figure 2: Comparison of Fatigue Limit (Cycles) Between Control and Modified Glass Ionomer Cement Groups**



**Figure 3: Comparison of Microhardness (VHN) Between Control and Modified Glass Ionomer Cement Groups.**

Microhardness values for the modified group were generally higher, implying that the inclusion of nano-hydroxyapatite provided a denser and more durable surface which resulted in greater material resistance to wear and deformation (Fig. 3).

The multiple linear regression analysis was conducted using the Ordinary Least Squares (OLS) method. It was found that tensile strength was an important predictor of microhardness. A positive coefficient suggests that greater tensile strength was related to increased values of microhardness. Fracture resistance is shown to be statistically significant due to its p value. Statistically, the coefficient of this

model of the fatigue limit was not significant. Thus, our result suggests that although the ability of the material to withstand repetitive stress may enhance microhardness, the stronger relationships with the tensile strength and fracture resistance effectively limited its contribution. In general, tensile strength and fracture resistance were found to be by far the most important predictors of microhardness. Tensile strength was a significant predictor of high microhardness, as shown by our logistic regression analysis (Table 3). Also statistically significant as a predictor were fracture resistance with a positive coefficient. Although the microhardness was positively associated with the fatigue limit in this model, the latter did not reach statistical significance.

Three steps of hierarchical regression were performed to evaluate how much tensile strength, fracture resistance and fatigue limit add to the prediction of microhardness (Table 4). It was then adding a new predictor each step and finding how R-squared and adjusted R-squared changed. The models above were used to create F-statistics and corresponding p-values to find the significance of each model.

In the third step, fatigue limit was included as an additional predictor. After fatigue limit addition, R-squared was increased even more, but this increase was much smaller than before. Overall, the hierarchical regression analysis showed that tensile strength and fracture resistance are the most dominant factors

contributing to the surface hardness of the material independently as well as collectively.

**DISCUSSION**

Accordingly, the rationale of this study was the limitations of conventional glass ionomer cement such as mechanical and biological properties, need to be resolved. To overcome these challenges, nano-hydroxyapatite, a biocompatible material was incorporated with GIC to improve its mechanical properties. The study was undertaken in order to systematically evaluate the influence of nano-hydroxyapatite on the glass ionomer cements mechanical properties. The results showed that additions of nHA significantly improved the fracture resistance of glass ionomer cement (GIC). This enhancement is probably due to stop crack propagation and improve stress distribution in the cement matrix by the nano-hydroxyapatite. The results point to the material’s suitability for clinical application where forces must be endured during application.

The finding is consistent with the comparison to recent work which demonstrated that when hydroxyapatite nanoparticles were added, compressive strength increased markedly.<sup>9</sup> Daokar et al. also observed that nano hydroxyapatite has increased bonding strength to dentin, which is a near identity to improved mechanical performance.<sup>8</sup> Based on Murugan et al., broad improvement in these properties of

**Table 2: Multiple Linear Regression Analysis of Microhardness (VHN) Using Tensile Strength, Fracture Resistance, and Fatigue Limit as Predictors**

Variable	Coefficient	Standard Error	t-value	p-value	Confidence Interval Lower	Confidence Interval Upper
Const	10.08254141	5.892448993	1.711095238	0.0938	-1.77834321	21.94342604
Tensile Strength (MPa)	0.902948755	0.274775158	3.286136785	0.001949	0.349855049	1.45604246
Fracture Resistance (N)	0.144895556	0.074373577	1.948212811	0.057506	-0.00481069	0.294601803
Fatigue Limit (Cycles)	0.001044018	0.00044815	2.329617053	0.024271	0.000141939	0.001946097

**Table 3: Logistic Regression Analysis of Predictors for High Microhardness in Glass Ionomer Cement**

Variable	Coefficient	Standard Error	z-value	p-value	Confidence Interval Lower	Confidence Interval Upper
const	-28.8655364	9.499331232	-3.038691429	0.002376	-47.48388349	-10.24718931
Tensile Strength (MPa)	0.562523231	0.338454664	1.662034212	0.096506	-0.100835721	1.225882184
Fracture Resistance (N)	0.109974202	0.084113854	1.307444574	0.191062	-0.054885923	0.274834327
Fatigue Limit (Cycles)	0.000523816	0.000464102	1.128665311	0.259039	-0.000385807	0.001433439

**Table 4: Hierarchical Regression Analysis of Predictors for Microhardness in Glass Ionomer Cement**

Model	R-squared	Adjusted R-squared	F-statistic	p-value
Step 1: Tensile Strength	0.587221526	0.578621975	68.28513368	8.83E-11
Step 2: + Fracture Resistance	0.669022823	0.654938688	47.50187464	5.19E-12
Step 3: + Fatigue Limit	0.703950923	0.684643374	36.45988108	3.23E-12

GIC, fracture toughness and antibacterial were found on inclusion of hydroxyapatite nanoparticles.<sup>1</sup> Pérez-Castro et al. also mentioned improved solubility and flexural strength that was achieved with GIC treated with nano hydroxyapatite nanoparticles and use in clinical outcomes.<sup>10</sup> Additionally, Ilancheran et al. demonstrated that green mediated hydroxyapatite addition to GIC increased compressive strength as was observed in current study with the increased fracture resistance.<sup>11</sup> This is in agreement with the prediction that nano hydroxyapatite reinforcement improves GIC's susceptibility to mechanical stresses. Ivanišević et al. found that modified GIC mechanical properties had a compressive modulus closer to the higher fatigue resistance in this work.<sup>12</sup> Furthermore, using the data from the present study, Jusoh et al. found that the compressive strength and density of GIC with time improved with increasing hydroxyapatite ratio.<sup>13</sup> Furthermore, Daokar et al. found increased bonding strength of nano hydroxyapatite reinforced GIC which also correlates with the fatigue resistance test data compiled in the current study.<sup>9</sup> The multifaceted evidence from all these studies is consistent with these studies and they point to the benefits above and beyond fatigue resistance improvement by nano hydroxyapatite incorporation in GIC.

Incorporation of nano hydroxyapatite in glass ionomer cement was shown to greatly increase its tensile strength in the current study. This improvement is attributable to the reinforcing effects of nano-hydroxyapatite that increase internal structural cohesion of the material making it more resistant to tensile failure. The current results were in agreement with a research made by Daokar et al., who concluded that nano-hydroxyapatite incorporated into conventional glass ionomer cement had a profound favorable effect on bonding strength to dentin, not because of superior structural interlocking, but by utilizing adhesive properties.<sup>8</sup>

Similar to the present study, Mederos et al. found that the addition of hydroxyapatite significantly increases the tensile strength of the conventional glass ionomer cement as it did in this study.<sup>14</sup> Piyush et al. then tested the influence of many different additives on glass ionomer, and concluded that nano sized hydroxyapatite enhanced compressive strength and micro hardness of glass ionomer cement, and are therefore good for clinical applications of GIC.<sup>15</sup> These findings are consistent with other work reported by Ravi et al., nanocomposite addition to hydroxyapatite and other nanoparticles enhances surface durability and wear resistance in glass ionomer cement.<sup>16</sup> Caesarianto and Nurlily also observed that the addition of fluorhydroxyapatite nanocrystals had a higher microhardness corresponding to a more integrity structure.<sup>17</sup> Furthermore, in the Sharafeddin et al. study, when hydroxyapatite and chitosan were added to the surface, increasing microhardness was found without affecting the surface roughness of glass ionomer cement as was observed

in the current study.<sup>18-20</sup> Piyush et al. evaluated glass ionomer cements containing novel additives and formulated with nano hydroxyapatite when compared to unmodified variants, the microhardness of which superior to unmodified ones suggests their suitability for critical clinical applications.<sup>15</sup>

Finally, the results of this study are in general agreement with the recent literature in that nano hydroxyapatite greatly influences the microhardness of the glass ionomer cements. This modification also enables longevity and performance in relevant clinical applications and improves mechanical properties. Variability was attributed to the differences in testing conditions, nanoparticle type and concentration between studies, representing a direction to further optimization.

## CONCLUSION

This study shows incorporation of nano hydroxyapatite into the glass ionomer cement improves its tensile strength, fracture resistance, fatigue limit and microhardness. The long term clinical performance should be examined and nHA concentration optimized in future work.

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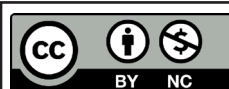
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#### AUTHORS' CONTRIBUTION

The following authors have made substantial contributions to the manuscript as under:

Conception or Design:	MA
Acquisition, Analysis or Interpretation of Data:	MA
Manuscript Writing & Approval:	MA

All the authors agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.



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