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Micro-hydroxyapatite reinforcement and surface microhardness of conventional glass ionomer cement: An *in vitro* study

Abhishek Sharma[1], Supriya Makam[2], Swati Sagar[3], Kunal Nischal[4], Siddharth Anil Shinde[5,*], Palak Abhishek Pandya[6] & Pratik Surana[7]

[1]Department of Prosthodontics and Crown & Bridge, Eklavya Dental College and Hospital, Kotputli, Rajasthan, India; [2]Department of Conservative Dentistry and Endodontics, Rajarajeswari Dental College and Hospital, Bangalore, Karnataka, India; [3]Department of Dentistry, Darbhanga Medical College and Hospital, Laheriasarai, Darbhanga, India; [4]Department of Prosthodontics and Crown & Bridge, SGT Dental College and Hospital, Gurgaon, Haryana, India; [5]Department of Orthodontics, Bharati Vidyapeeth Deemed to be University Dental College and Hospital, Pune, Maharashtra, India; [6]Department of Prosthodontics, Karnavati School of Dentistry, Karnavati University, Gandhinagar, Gujarat, India; [7]Department of Pedodontics and Preventive Dentistry, Maitri College of Dentistry and Research Centre, Durg, Chhattisgarh, India; *Corresponding author

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https://maitricollege.in

Author contact:
Abhishek Sharma - E-mail: drabhisheksharmaperfectsmile@gmail.com; Phone: + 91 9001443774
Supriya Makam - Email: drsupriyamakam@gmail.com; Phone: +91 7899419936
Swati Sagar - Email: doctor.swatisagar@gmail.com; Phone: + 91 7903320959
Kunal Nischal - E-mail: drkunal1001@gmail.com; Phone: + 91 8826778533
Siddharth Anil Shinde - E-mail: orthodontistshinde@gmail.com; Phone: + 91 9766000500
Palak Abhishek Pandya - E-mail: drpalakpandya28@gmail.com; Phone: + 91 9490681377
Pratik Surana - E-mail: suranadrpratik@gmail.com; Phone: +91 8871310111

Abstract:
Glass ionomer cement is often used in restorative dentistry because of its fluoride release, adhesion and biocompatibility, despite its limited mechanical strength. Therefore, it is of interest to evaluate the outcome of incorporating 5 wt% microhydroxyapatite into conventional GIC on surface microhardness using 30 standardized disc specimens divided equally into two groups. Microhardness was evaluated using a Vickers hardness tester. Thus, incorporation of 5 wt% microhydroxyapatite significantly enhanced the surface hardness of conventional GIC, suggesting improved mechanical performance and clinical durability.

Keywords: Glass ionomer cement (GIC), hydroxyapatite, microhardness

Background:
The preservation of natural tooth structure through minimally invasive restorative dentistry has become a fundamental goal of modern dental care **[1]**. An ideal restorative material should not only restore the form and function of lost tooth structure but also withstand the demanding mechanical and chemical challenges of the oral environment **[2]**. Although resin-based composite materials provide excellent esthetics and acceptable clinical performance, their technique sensitivity, polymerization shrinkage and susceptibility to marginal degradation continue to present challenges, particularly in stress-bearing posterior restorations **[3]**. Consequently, glass ionomer cements (GICs) remain an indispensable restorative option owing to their unique biological and therapeutic advantages **[4]**. Glass ionomer cements have been extensively used in pediatric and restorative dentistry because of their chemical adhesion to enamel and dentin, sustained fluoride release, biocompatibility and coefficient of thermal expansion closely matching that of tooth structure **[5]**. These characteristics contribute to reduced microleakage and enhanced prevention of secondary caries, making GICs especially valuable in atraumatic restorative treatment (ART), minimally invasive dentistry and restorations in high-caries-risk patients **[6, 7]**. However, their relatively low mechanical strength, brittleness, poor wear resistance and inadequate surface hardness limit their use in stress-bearing areas and limit their long-term clinical durability **[8]**. To overcome these shortcomings, considerable research has focused on reinforcing conventional GICs with bioactive fillers and nanostructured materials **[9]**. Among these, hydroxyapatite (HA) has attracted significant attention because it is the principal inorganic constituent of enamel, dentin and bone **[10]**. Its excellent biocompatibility, bioactivity and chemical similarity to dental hard tissues facilitate improved interaction with the glass ionomer matrix and may enhance the physical and mechanical performance of the cement. Incorporation of hydroxyapatite has been reported to improve properties and bond strength while preserving the inherent fluoride-releasing capability of GIC **[11]**. Microhydroxyapatite (μHA) improves the glass ionomer matrix by enhancing particle dispersion and reducing agglomeration. Its incorporation may increase surface microhardness, thereby improving wear resistance and the long-term clinical performance of restorations **[12]**. Therefore, it is of interest to assess the microhardness of conventional GIC and GIC reinforced with 5% microhydroxyapatite.

Materials and Methods:
Study design:
This experimental research was undertaken to evaluate the effect of 5% microhydroxyapatite (μHA) incorporation on the microhardness of conventional glass ionomer cement (CGIC). A total of 30 disc-shaped specimens were fabricated and equally allocated into two experimental groups (n = 15 per group).

Study materials:
The restorative material used was conventional glass ionomer cement, prepared according to the manufacturer's recommendations. Microhydroxyapatite (μHA) powder (Sigma-Aldrich, USA) was used as the reinforcing filler. Standardized

cylindrical stainless-steel molds measuring 7 mm in diameter and 3 mm in height were used for specimen fabrication.

Grouping of specimens:
The 30 specimens were randomly assigned into two equal groups. Group I (Control) consisted of conventional GIC, while Group II (Experimental) comprised conventional GIC reinforced with 5 wt% microhydroxyapatite (μHA).

[1] Group I: For the control group, CGIC was manipulated strictly according to the manufacturer's instructions. One scoop of powder was mixed with one drop of liquid on a cooled glass slab using a plastic spatula until a homogeneous consistency was achieved. The mixed cement was immediately packed into the cylindrical mold placed over a glass slab covered with a transparent celluloid strip. Another celluloid strip and glass slab were positioned over the mold and gentle pressure was applied to extrude excess material and get smooth, flat specimen surfaces. The assembly was maintained under pressure until the material had completely set. Following setting, the specimens were carefully removed from the molds and a thin layer of petroleum jelly was applied to all surfaces to protect the cement from early moisture contamination and dehydration.

[2] Group II: For the experimental group, the powder component consisted of 95 wt% CGIC and 5 wt% microhydroxyapatite powder. The powders were accurately weighed using a digital analytical balance and manually blended with a plastic spatula on a clean glass slab until a uniform mixture was obtained. The modified powder was then mixed with the manufacturer's recommended quantity of liquid following the same powder-to-liquid ratio and manipulation procedure used for the control group. The mixed material was packed into the molds, covered with celluloid strips and glass plates and allowed to set under uniform pressure. After complete setting, the specimens were removed from the molds and coated with a thin layer of petroleum jelly. All samples were stored in distilled water at 37°C for 24 hours before microhardness evaluation.

Microhardness testing:
Surface microhardness was determined using a Vickers microhardness tester. Prior to testing, the specimens were gently dried with absorbent paper to remove surface moisture. Three indentations were made on the top surface of each sample using a Vickers diamond indenter under a standardized load and dwell time as recommended by the instrument manufacturer. The indentation sites were positioned so that the distance between adjacent indentations and the specimen margins was at least 1 mm to eliminate the influence of overlapping stress fields. The diagonal lengths of each indentation were measured using the built-in optical microscope and the corresponding Vickers Hardness Number (VHN) was automatically calculated by the testing software. The mean value of the three measurements for each specimen was considered the final Vickers hardness value for statistical analysis.

Statistical analysis:
Statistical analysis was performed using IBM SPSS Statistics version 25.0 (IBM Corp., NY, USA). Descriptive statistics, including mean and standard deviation, were calculated for each group. Intergroup comparison of the mean Vickers hardness values was performed using an independent samples t-test.

Results:
The mean Vickers microhardness of Group I (Conventional GIC) was 58.42 ± 2.91 VHN, whereas Group II (Conventional GIC reinforced with 5% microhydroxyapatite) demonstrated a significantly higher mean microhardness of 69.15 ± 3.04 VHN. Surface microhardness was significantly higher in the 5% microhydroxyapatite-incorporated IC compared with the conventional GIC ($t = 9.84$, $p < 0.001$) **(Table 1)**.

Table 1: Comparison of Vickers Microhardness (VHN) Between the Study Groups

Group	N	Mean VHN	Standard Deviation (SD)	t-value	p-value
Group I – Conventional GIC	15	58.42	2.91	t = 9.84	<0.001*
Group II – Conventional GIC + 5% Microhydroxyapatite	15	69.15	3.04		

Discussion:
The current research assessed the influence of integrating 5 wt% microhydroxyapatite (μHA) into conventional glass ionomer cement (CGIC) on its surface microhardness. The findings showed a statistically significant increase in the Vickers hardness of the modified GIC compared with the conventional material, supporting the study hypothesis that μHA reinforcement enhances the mechanical performance of glass ionomer cement. Surface microhardness is a vital indicator of the capacity of a restorative material to resist surface indentation, abrasion and occlusal wear **[13]**. Materials with higher hardness generally exhibit improved resistance to masticatory forces and are less susceptible to surface degradation, thereby contributing to greater clinical longevity **[14]**. Since glass ionomer cement is frequently used in stress-bearing areas of primary and permanent dentition, improving its hardness is expected to increase its durability without compromising its biological advantages. The improvement performance in the present study may be associated to the bioactive nature of microhydroxyapatite and its chemical similarity to the mineral phase of tooth structure. The incorporation of μHA may promote better interaction between the glass particles and the polyacrylate matrix during the acid-base reaction, resulting in a denser and more homogeneous cement structure. Furthermore, the calcium and phosphate ions present in hydroxyapatite may contribute to enhanced matrix maturation and reduce structural

defects, leading to increased resistance to surface deformation **[4, 15]**. The outcomes of the current study are aligned with earlier investigations demonstrating the beneficial effect of hydroxyapatite incorporation on the mechanical properties of glass ionomer cement. Sharafeddin *et al.* reported that addition of 5 wt% and 15 wt% microhydroxyapatite reinforced the glass ionomer matrix, improved bonding between the glass core and matrix and significantly enhanced the microhardness of resin-modified GIC and Zirconomer **[16]**. Similarly, Maity *et al.* observed improved surface hardness following hydroxyapatite incorporation, suggesting that the bioactive filler strengthens the cement matrix. The increased microhardness observed in the present study may therefore be attributed to the reinforcing effect of microhydroxyapatite particles, which promote a denser cement structure while preserving the inherent biological advantages of conventional glass ionomer cement **[10]**. In current investigation, microhydroxyapatite was selected as the reinforcing material because its composition closely resembles that of natural enamel and dentin, providing excellent biocompatibility and favorable interaction with the cement matrix **[17]**. The concentration of hydroxyapatite plays a crucial role in determining the mechanical properties of glass ionomer cement. Gu *et al.* reported that the incorporation of hydroxyapatite/zirconia (HAP/ZrO_2) particles significantly improved the mechanical properties of GIC; however, excessive hydroxyapatite content may reduce the density of the set cement and adversely affect its microhardness **[18]**. Based on this rationale, the present study incorporated 5 wt% microhydroxyapatite, which provided effective reinforcement without compromising the setting characteristics or structural integrity of the cement. The significant increase in microhardness observed in the present study suggests that this concentration offers an optimal balance between matrix reinforcement and material stability. Strict standardization was maintained throughout specimen preparation to minimize variables that could influence hardness measurements. All specimens were fabricated under identical laboratory conditions using the manufacturer's recommended, standardized molds, uniform mixing procedures and identical setting conditions. Application of a protective petroleum jelly coating minimized early moisture contamination and dehydration, while standardized Vickers hardness testing ensured reliable comparison between the experimental groups **[19-21].** Despite the encouraging findings, this research has some limitations. As *ex vivo* investigation, the specimens were evaluated under controlled laboratory conditions that cannot completely reproduce the multifaceted oral environment, thermal fluctuations, cyclic loading, pH changes and long-term water exposure. In addition, only surface microhardness was evaluated, whereas other clinically relevant properties such as compressive strength, flexural strength and bond strength were not assessed. Within the limitations of the present study, incorporation of 5 wt% microhydroxyapatite significantly enhanced the surface microhardness of conventional glass ionomer cement. This modification has the potential to improve the clinical durability of GIC while preserving its favorable biological properties. Further long-term laboratory investigations and well-designed clinical studies are recommended to evaluate the performance of microhydroxyapatite-reinforced glass ionomer cement under functional oral conditions.

Conclusion:

Incorporation of 5 wt% microhydroxyapatite significantly improved the microhardness of conventional glass ionomer cement. This modification may enhance the mechanical performance and clinical durability of the restorative material. Microhydroxyapatite-reinforced glass ionomer cement may therefore serve as a promising alternative for restorations requiring improved mechanical strength.

Advancement to knowledge:

The present study provides evidence that incorporation of 5 wt% microhydroxyapatite can significantly improve the surface microhardness of conventional glass ionomer cement. This finding supports the potential use of microhydroxyapatite as reinforcing filler for enhancing the mechanical performance of conventional GIC.

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