Investigation on an Alternative Restorative Material: Composites of Nanoxhydroxyapatite from a Natural Resource With Glass Ionomer Cement

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**Abstract:** Hydroxyapatite's characteristics are improved in its nanoscale form, such as its enhanced surface area and reactivity, which can greatly improve how well it interacts with biological tissues[(*Article Detail*, 2014)](https://paperpile.com/c/wCBtds/QW5F) .Because of its special characteristics, such as its chemical adherence to oral tissues, its biocompatibility, and its capacity to release fluoride, glass ionomer cement (GIC) has long been a mainstay in restorative dentistry[(Wan Jusoh et al., 2021)](https://paperpile.com/c/wCBtds/EoCf) . GICs are particularly helpful for patients with high caries risk and in pediatric dentistry because of their capacity to produce fluoride, which aids in halting more tooth decay[(Moheet et al., 2019)](https://paperpile.com/c/wCBtds/eOQq) .Notwithstanding these benefits, GICs are sometimes criticized for their mechanical characteristics, especially when it comes to their inferior wear resistance and compressive strength when compared to resin-based composites [(*Website*, n.d.)](https://paperpile.com/c/wCBtds/Pia4).This restriction may have an impact on repair performance and longevity, especially in load-bearing places [(Ajay, Rakshagan, et al., 2022)](https://paperpile.com/c/wCBtds/1YGmK).In order to overcome these mechanical drawbacks and maintain the bioactive advantages of both materials, nHA has been integrated into GIC (Almatrafi et al., 2024). Studies have indicated that adding nHA to GIC can greatly improve its mechanical characteristics, such as wear resistance, flexural strength, and compressive strength[(*Mechanical and Two‐body Wear Characterization of Micro‐nano Ceramic Particulate Reinforced Dental Restorative Composite Materials*, n.d.)](https://paperpile.com/c/wCBtds/FmNg) .These enhancements are ascribed to the nHA particles' reinforcing action inside the GIC matrix, which aids in more uniformly distributing stress during masticatory forces [(“Development of Remineralizing, Antibacterial Dental Materials,” 2009)](https://paperpile.com/c/wCBtds/TaG8). Furthermore, nHA's bioactive properties encourage the remineralization of demineralized tooth tissues, which is advantageous when it comes to early carious lesions [(Cramer et al., 2010)](https://paperpile.com/c/wCBtds/j8Th). In order to preserve the integrity of the tooth structure and stop more decay, this remineralization process is essential[(Ajay, Suma, et al., 2022)](https://paperpile.com/c/wCBtds/u1NmS) [(Katyal et al., 2021)](https://paperpile.com/c/wCBtds/Y7hLQ).

**keywords:** Development of Remineralizing, Antibacterial Dental Materials, Synthesis ,XRD Analysis

# Introduction

The search for effective and innovative dental restorative materials has intensified in recent years, driven by the need for materials that not only restore function but also contribute positively to oral health [(Harsha & Subramanian, 2022)](https://paperpile.com/c/wCBtds/ztSAj)[(Deepika et al., 2022)](https://paperpile.com/c/wCBtds/kOttB)[(Solanki et al., 2022)](https://paperpile.com/c/wCBtds/A1PcK). Among the various materials explored, nanoxhydroxyapatite (nHA) derived from natural resources has emerged as a particularly promising candidate[(Chidambaram et al., 2022)](https://paperpile.com/c/wCBtds/Lpteu).[(Ajay, Sasikala, et al., 2022)](https://paperpile.com/c/wCBtds/JoZ0j). Hydroxyapatite, a naturally occurring mineral form of calcium phosphate, is the primary inorganic component of bone and teeth, making it an ideal material for dental applications due to its excellent biocompatibility, bioactivity, and similarity to the mineral structure of dental tissues[(“Modification of Glass Ionomer Cement by Incorporating Hydroxyapatite-Silica Nano-Powder Composite: Sol–gel Synthesis and Characterization,” 2014)](https://paperpile.com/c/wCBtds/LXub) .Hydroxyapatite's characteristics are improved in its nanoscale form, such as its enhanced surface area and reactivity, which can greatly improve how well it interacts with biological tissues[(Article Detail, 2014)](https://paperpile.com/c/wCBtds/QW5F) .Because of its special characteristics, such as its chemical adherence to oral tissues, its biocompatibility, and its capacity to release fluoride, glass ionomer cement (GIC) has long been a mainstay in restorative dentistry[(Wan Jusoh et al., 2021)](https://paperpile.com/c/wCBtds/EoCf) . GICs are particularly helpful for patients with high caries risk and in pediatric dentistry because of their capacity to produce fluoride, which aids in halting more tooth decay[(Moheet et al., 2019)](https://paperpile.com/c/wCBtds/eOQq) .Notwithstanding these benefits, GICs are sometimes criticized for their mechanical characteristics, especially when it comes to their inferior wear resistance and compressive strength when compared to resin-based composites [(Website, n.d.)](https://paperpile.com/c/wCBtds/Pia4).This restriction may have an impact on repair performance and longevity, especially in load-bearing places [(Ajay, Rakshagan, et al., 2022)](https://paperpile.com/c/wCBtds/1YGmK).In order to overcome these mechanical drawbacks and maintain the bioactive advantages of both materials, nHA has been integrated into GIC (Almatrafi et al., 2024). Studies have indicated that adding nHA to GIC can greatly improve its mechanical characteristics, such as wear resistance, flexural strength, and compressive strength[(Mechanical and Two‐body Wear Characterization of Micro‐nano Ceramic Particulate Reinforced Dental Restorative Composite Materials, n.d.)](https://paperpile.com/c/wCBtds/FmNg) .These enhancements are ascribed to the nHA particles' reinforcing action inside the GIC matrix, which aids in more uniformly distributing stress during masticatory forces [(“Development of Remineralizing, Antibacterial Dental Materials,” 2009)](https://paperpile.com/c/wCBtds/TaG8). Furthermore, nHA's bioactive properties encourage the remineralization of demineralized tooth tissues, which is advantageous when it comes to early carious lesions [(Cramer et al., 2010)](https://paperpile.com/c/wCBtds/j8Th). In order to preserve the integrity of the tooth structure and stop more decay, this remineralization process is essential[(Ajay, Suma, et al., 2022)](https://paperpile.com/c/wCBtds/u1NmS) [(Katyal et al., 2021)](https://paperpile.com/c/wCBtds/Y7hLQ).Furthermore, the usage of nHA derived from natural resources is consistent with the growing trend of environmentally friendly and sustainable dental procedures(Saadh et al., 2024). Eggshells and fish bones are examples of natural sources of nHA that not only offer a renewable resource but also lessen the environmental impact of manufactured materials[(“Effect of Aluminium Oxide, Titanium Oxide, Hydroxyapatite Filled Dental Restorative Composite Materials on Physico-Mechanical Properties,” 2022)](https://paperpile.com/c/wCBtds/WTwg) . The capacity to create nHA from these natural sources can help create dental materials that are both highly performing and more environmentally friendly[(Jabin et al., 2021)](https://paperpile.com/c/wCBtds/VuGfX)[(Balaji Ganesh S & Sugumar, 2021)](https://paperpile.com/c/wCBtds/tugpm) [(Govindaraj & Dinesh, 2021)](https://paperpile.com/c/wCBtds/nTWuW) .Numerous investigations have documented the synthesis, characterisation, and integration of nHA into GIC, exhibiting enhancements in diverse attributes such microhardness and shear bond strength . For example, studies have demonstrated that GIC treated with nHA demonstrates improved adherence to tooth structure, which is essential for dental restoration lifespan[(Rahman et al., 2014)](https://paperpile.com/c/wCBtds/y2JP) . Furthermore, the use of nHA can further improve the fluoride release characteristic of GIC, adding another level of caries prevention [(Keshi et al., 2022)](https://paperpile.com/c/wCBtds/jtR0).Improved clinical outcomes may result from the potential of nHA-GIC composites to function as efficient restorative materials, especially for patients with high caries risk and in pediatric dentistry[(“Alternative Direct Restorative Materials to Dental Amalgam,” 2024)](https://paperpile.com/c/wCBtds/qUJx) . These composites are a good substitute for more traditional materials like amalgam and resin-based composites because of their capacity to both restore function and support the health of dental tissues [(Tiwari & Jain, 2023)](https://paperpile.com/c/wCBtds/ANHyU)[(Graf et al., 2023)](https://paperpile.com/c/wCBtds/mKJzf).To sum up, the study of nHA composites with GIC fills a demand for materials that improve the mechanical and biological characteristics of dental restorations, which is a major step forward in restorative dentistry. To completely comprehend the consequences of these composites in clinical practice, including long-term performance and patient outcomes, more study in this field is necessary. The incorporation of novel materials like nHA into GIC may open the door to more efficient and long-lasting dental procedures as the field of restorative dentistry develops.

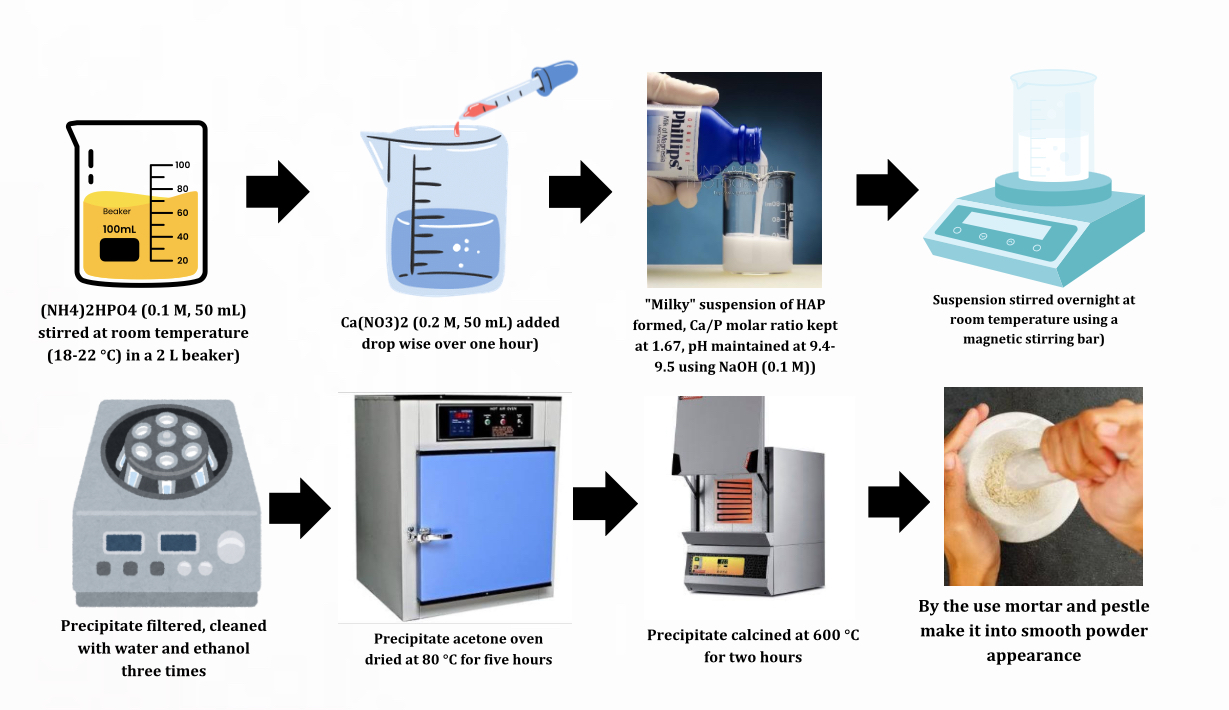
# MATERIALS AND METHODS

# MATERIALS

Ammonium dihydrogen phosphate (N H 4) 2 H P O 4 (NH 4 ) 2 HPO 4, formulated as a 0.1 M solution with a volume of 50 mL, is one of the materials used in the manufacture of hydroxyapatite (HAP) powder. This phosphate source is required for the reaction. In a 0.2 M solution with a volume of 50 mL, calcium nitrate Ca(NO3) 2 Ca(NO 3 ) 2 is utilized to provide the calcium ions necessary for HAP production. In order to facilitate the precipitation of hydroxyapatite, the pH of the solution is adjusted and maintained between 9.4 and 9.5 using sodium hydroxide (NaOH) at a concentration of 0.1 M.To ensure that contaminants and unreacted compounds are removed from the precipitate, water and ethanol are used in the washing process. Lastly, to help remove any remaining moisture from the precipitate and produce a purer hydroxyapatite product, acetone is employed throughout the drying process.

# SYNTHESIS

The picture shows how hydroxyapatite (HAP) powder is made. The first step is to prepare a 50 mL solution of 0.1 M ammonium dihydrogen phosphate (N H 4) 2 H P O 4 (NH 4 ) 2 HPO 4 °C at room temperature in a 2 L beaker. The phosphate solution is then mixed with a 50 mL solution of 0.2 M calcium nitrate (Ca(NO3)2 Ca(NO 3 ~) 2 ~) dropwise over the course of an hour, preserving a 1.67 calcium-to-phosphorus (Ca/P) molar ratio. 0.1 M NaOH is used to bring the pH down to 9.4–9.5, creating a "milky" HAP suspension. To guarantee homogeneity, this suspension is agitated with a magnetic stirring bar overnight at room temperature.After filtering and washing the precipitate three times with ethanol and water to get rid of any remaining contaminants, it is dried with acetone for five hours at 80 °C in the oven. Lastly, to increase crystallinity, the dried precipitate is calcined at 600 °C for two hours. It is then pulverized with a mill and pestle to a fine powder.

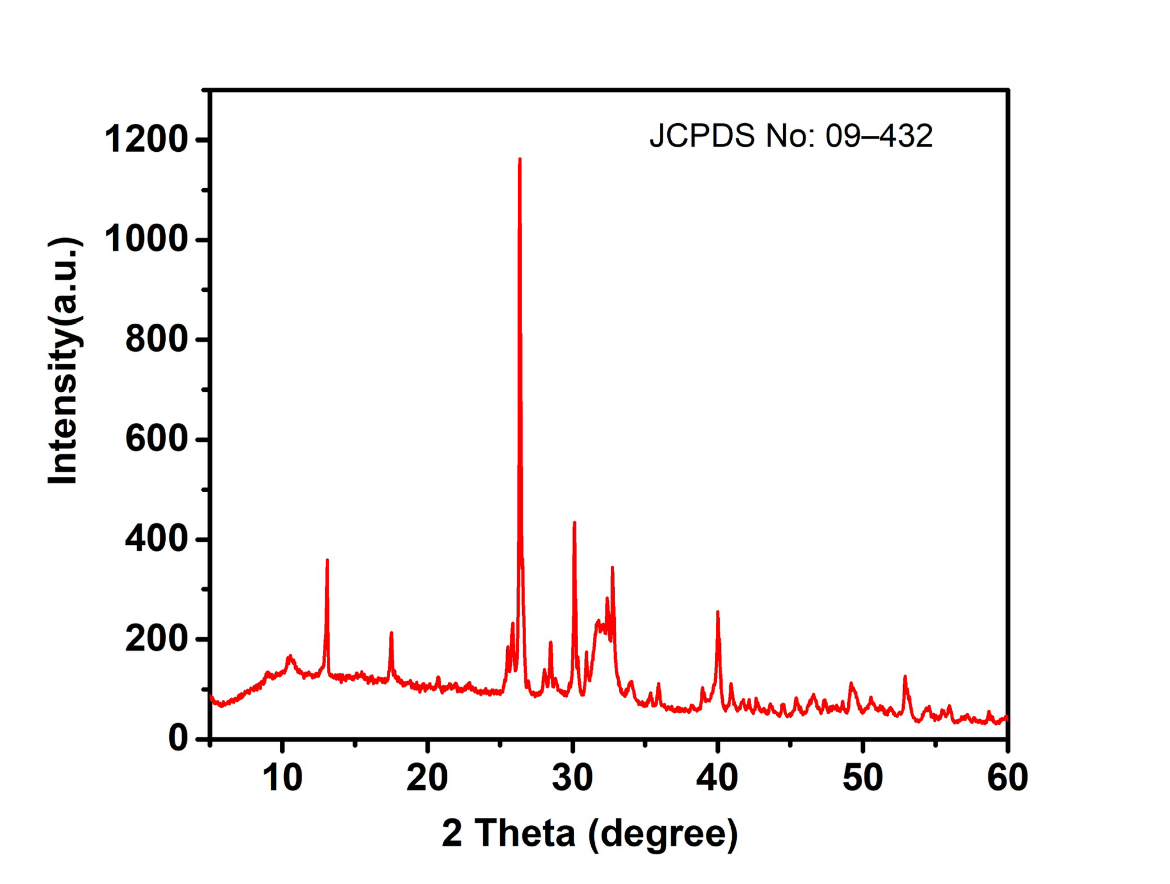


**Fig:** Schematic representation of synthesis of composites of nano hydroxyapatite from a natural resource with glass ionomer cement

# Result and discussion

## XRD Analysis

The XRD pattern of the HAP powder demonstrates that the predominately detected crystalline phase is assigned to hydroxyapatite. High intensity hydroxyapatite peaks at crystal planes (002), (211), (112), and (300) at 2θ = 26.0°, 31.9°, 32.1°, and 32.9°, respectively, match perfectly to the Joint Committee on Powder Diffraction Standards (JCPDS) standard PDF card No. 09-432.



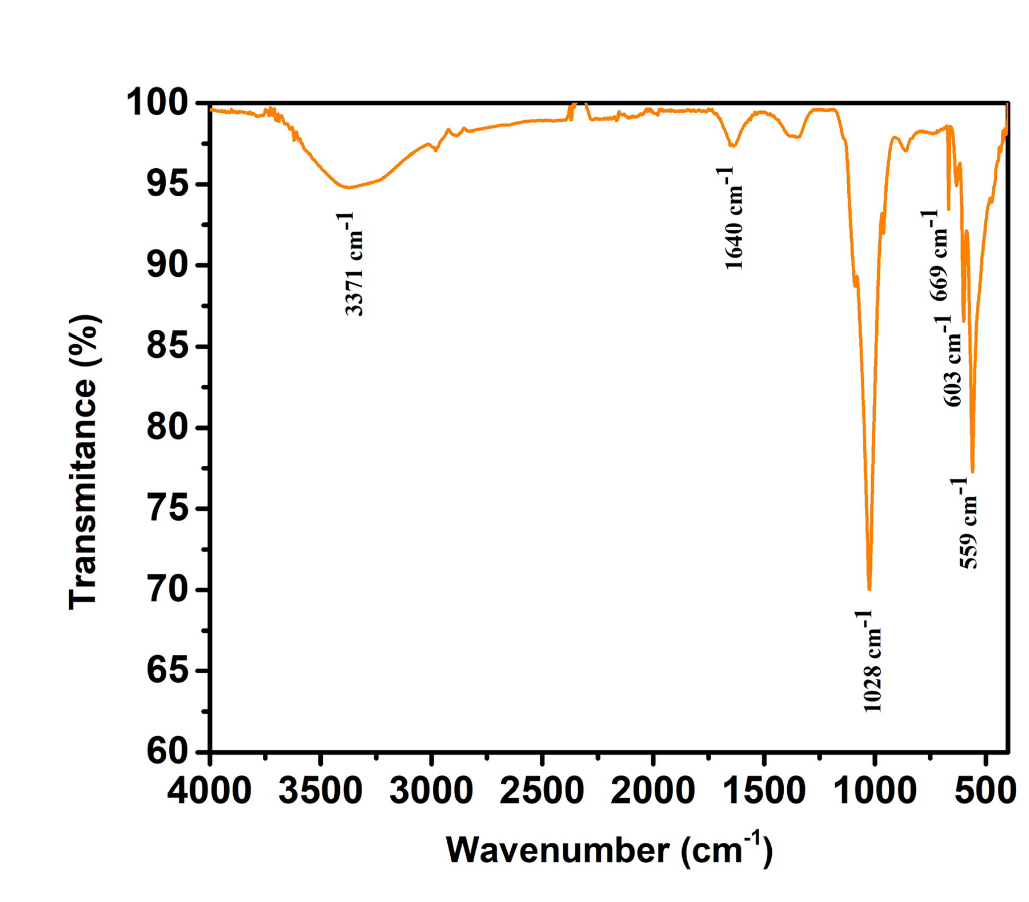
**Figure.1.**  XRD spectrum of nanohydroxyapatite.

## FTIR Analysis

The Fourier transform infrared (FT-IR) spectroscopy spectrum of HAP indicates characteristic absorption bands of natural hydroxyapatite at 463, 569, and 603 cm-1, which are attributed to the

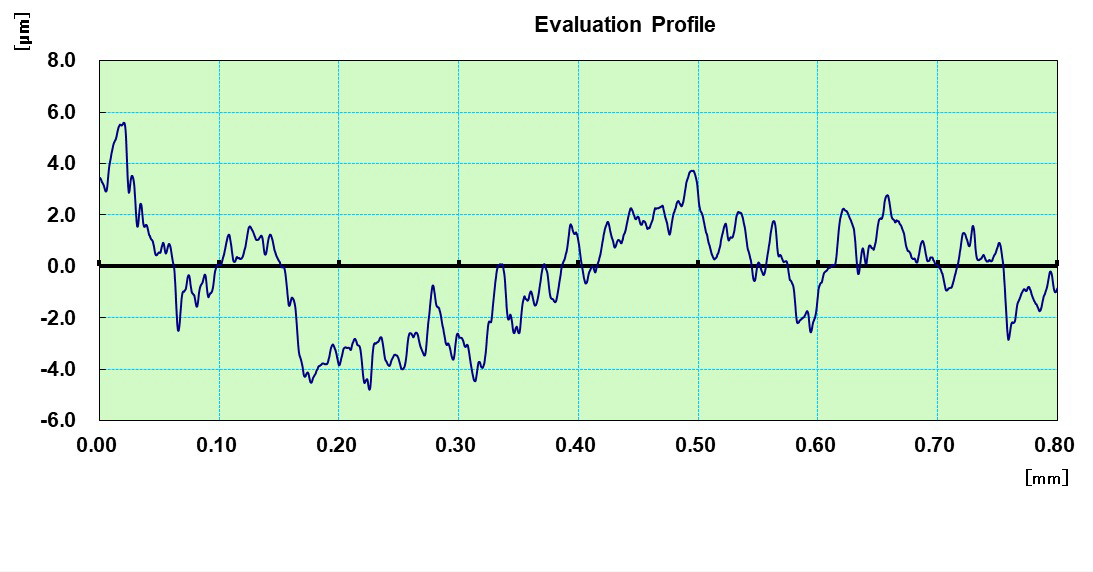
P-O bending of phosphate groups. In the composite HAP, three absorption bands of the PO43-group are clearly distinguished at 1092, 1038, and 960 cm-1, corresponding to the 0-P-0 phosphate ions of the hydroxyl site. A characteristic band at 3572 cm-1 and a weak broad band at 634 cm-1 can be attributed to the stretching and vibrational modes of the structural OH groups of the hydroxyapatite lattice. The absorbance bands at 1420 and 1460 cm-1 correspond to asymmetrical stretching vibrations of CO32- ions, indicating predominantly B-type hydroxyapatite.

Additionally, a vibration band located at 1640 cm-1 corresponds to the skeletal vibration of Gr due to aromatic sp2 hybridized C=C vibration stretching, confirming its presence in the synthesized .

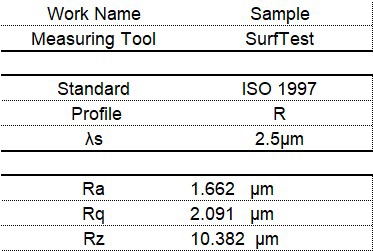


**Figure 2**: FTIR spectrum of dental composite containing nanohydroxyapatite

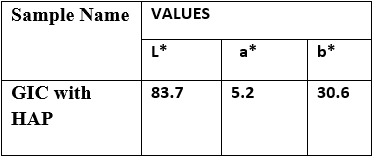
When comparing GIC with and without nanohydroxyapatite, the lab-prepared GIC with nanohydroxyapatite had a smoother surface (roughness less than 1) compared to plain GIC (roughness of 1.622), making it better for fillings. The colour of the GIC with nanohydroxyapatite is similar to normal dentin, making it suitable for front teeth restorations. The compressive strength of normal dentin is 90, while GIC with nano hydroxyapatite has a compressive strength of 79.8, showing it is still a good material for restorations.



**Figure 3:** Roughness test for GIC without nanohydroxyapatite



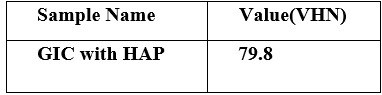
(a)



(b)

**Figure 4:** (a) (b) MICRO HARDNESS RESULTS

## Shimadzu HMV-G31DT Vickers Micro Hardness Tester



**Figure 5:** Force: HV0.3(2.942N)

## Holding time:15 sec

Color and Hardness Analysis of GIC with HAP

The table presents the colorimetric properties and Vickers hardness number (VHN) of Glass Ionomer Cement (GIC) with Hydroxyapatite (HAP).

Color Measurements (CIE Lab\* System)

The color of the sample was evaluated using the CIE Lab\* color space, which represents colors in three-dimensional coordinates:

• L\* (Lightness): 83.7 (indicates brightness, with 0 being black and 100 being white).

• a\* (Red-Green Axis): 5.2 (positive values indicate a shift toward red, while negative values indicate green).

• b\* (Yellow-Blue Axis): 30.6 (positive values indicate a shift toward yellow, while negative values indicate blue).

Vickers Hardness Number (VHN)

The hardness of the GIC with HAP sample was measured using the Vickers microhardness test, which determines resistance to localized deformation. The obtained VHN value was 79.8, indicating the material’s mechanical strength and wear resistance.

This data suggests that incorporating HAP into GIC influences both optical properties (color) and mechanical properties (hardness), which are critical for dental and biomedical applications

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