

## Comparison of micro-hardness of Conventional Glass ionomer cement, Glass ionomer cement reinforced with Calcium oxide and Hydroxyapatite nanoparticles

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### ABSTRACT:

#### Background

Glass Ionomer Cement (GICs) are commonly used in dentistry because of their chemical adhesion to dental tissues, biocompatibility, fluoride release ability, minimal microleakage at the tooth-restoration interface, and anti-cariogenic potential, but they have relatively poor mechanical properties. Hydroxyapatite[HA] is the primary mineral component of enamel and comprises more than 60% of Dentin weight. Hydroxyapatite, a naturally occurring form of calcium phosphate, is the main mineral component of teeth and bones. Natural hydroxyapatite and bone are biocompatible because they have relatively similar chemical and physical characteristics. The porous structure of hydroxyapatite is similar to that of natural bone due to its biodegradability, biocompatibility, and bioactivity. HA is commonly used in interdisciplinary fields of science commonly.

**Aim of the study:** The aim of the study is to compare the microhardness of conventional GIC and GIC-reinforced calcium oxide and hydroxyapatite nanoparticles.

#### Materials and methods:

15 disc-shaped samples of GIC restorative material with 2 mm of thickness were prepared using a customised mould. The samples were subdivided into three groups with 5 samples in each group. The Vickers microhardness test was carried out using a Shimadzu HMV-G microhardness tester with a load of 300 g with a well time of 15 seconds.

#### Results and discussion:

As a result, by comparing the microhardness of conditional GIC and GIC reinforced with calcium oxide and hydroxyapatite nanoparticles using SPSS ANOVA Test. The p-value is found to differ significantly from 0.00353, i.e.,  $p < 0.05$ .

#### Conclusion:

Conclusively, the value of the GIC reinforced with HA nanoparticles is higher when compared to CaO. So, GIC reinforced with HA nanoparticles has higher micro-hardness.

**Keywords:** Glass Ionomer Cement, Hydroxyapatite, Calcium oxide, ANOVA, Chemical adhesion

### INTRODUCTION

Glass Ionomer Cement (GIC) was introduced in the 1960s. It is an acid-base reaction between a polycarboxylic acid and an essential fluoro-alumino-silicate glass powder in the presence of water. (1,2)

Many changes and improvements to the original formulation have been made later. Conventionally, setting GICs (conventional GICs) are considered to be hybrid materials that contain inorganic and organic materials. It is made of aqueous solutions of acrylic acid-containing homopolymer and copolymers of tartaric acid and calcium fluoro-alumino-silicate glass powder. (3,4) GIC has numerous applications in dentistry due to its distinct properties. (5,6) It has anti-cariogenic properties, esthetics, fluoride-releasing property, low stimulation to the pulp, chemical adhesion to the tooth structure, microleakage at the tooth restoration interface, low cellular toxicity, and similar modulus of elasticity to the tooth structure and coefficient of thermal expansion. (7)-(8),(9) It seems to be the most popular amalgam substitute for primary tooth restoration. GICs are widely used in medicine, primarily in reconstructive surgery and otologic, as well as orthopaedics. Because this cement produces no heat during the setting process, it will not cause thermal damage to tissues and therefore will not affect the heat-labile drugs incorporated in the cement's matrix phase. (10)-(11) Their primary medical applications include the stabilisation of implanted devices, the resection or restoration of bone deformities, and the stabilisation of bony fragments. (12)-(13),(14) However, one of the most significant shortcomings of GIC is its susceptibility to moisture contamination, slow rate of setting, and dehydration during the early stages of setting, and it also has poor physical properties, such as low flexural strength and low fracture resistance, which prevents their use in high-stress bearing areas. (15) In order to enhance mechanical properties, RMGICs were developed in the late 1980s. (16)-(17) These materials are more wear-resistant, moisture-resistant, more fracture toughness, and they last longer. Resin-modified GIC(RMGIC) has been introduced to overcome the shortcomings of GIC, which has a higher initial strength than the original formulation. (18) Metal addition to the filler component has been suggested to strengthen conventional GICs. (19)-(20) After that, the powder is mixed with fluoro-alumino-silicate glass and a silver alloy (21), or the glass is sintered with silver. (22) The latter is widely recognised as cermet cement (**ceramics and metal**). Metal-reinforced GICs were proposed for core build-up and restoration. (23) Glass short fibres with a specific ratio (length/diameter) can be used as a reinforcing agent in glass-ionomer cement. (24) Glass fibres have several applications for the strengthening of plastic materials in general, and fibre-glass-reinforced plastics (FRP) are a common example. (25) The addition of glass short fibres to glass-ionomer cement is more likely to strengthen it if the composition of the short fibres is identical to that of the powder in the cement, which is fluoro-alumino-silicate glass. (26) Because of its similar composition, a glass fibre composed of CaO-P2O5-SiO2-Al2O3 (CPSA) is being used as an attempt to strengthen glass-ionomer cement. (4) The CPSA glass fibre was therefore developed as a biomaterial for medical and dental applications. (27)-(28),(1)

Hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ) is the primary mineral component of enamel and comprises more than 60% of Dentin weight. (29) The main mineral component of bones and teeth is hydroxyapatite, a naturally occurring form of calcium phosphate. Natural hydroxyapatite and bone are biocompatible because they have relatively similar physical and chemical characteristics. Its porous structure is similar to that of natural bone. GICs were found to bind with HA via the polyacid's carboxylate groups. (30) HA becomes one of the most bioactive and biocompatible materials, and its nanosized particles have a crystal structure, crystallinity, and morphology similar to apatite crystals found in tooth enamel. (31) In addition to improving mechanical properties, it creates a favourable environment for enamel remineralisation. (32) Nano-HA has a larger surface area and higher solubility than micro-HA due to its smaller particle size, which is similar to the size of minerals in teeth. (33)-(34) Microparticles of HA can be used to easily mix with GIC powder, including resin, while porous spherical HA particles are used to most effectively increase mechanical properties and fluoride ion release. (35)

Furthermore, various filler particles such as stainless steel powder, aluminosilicate fibres, silver, and hydroxyapatite have been previously incorporated into GIC in order to improve their mechanical properties. So, the aim of the study is to compare the microhardness of conventional GIC and GIC reinforced with calcium oxide and hydroxyapatite nanoparticles.

## MATERIALS AND METHODS:

15 disc-shaped samples of GIC restorative material with 2 mm of thickness were prepared using a customised mould. The samples were subdivided into three groups with 5 samples in each group.

### Calcium oxide (CaO) preparation :

The Seashells were washed and dried in the sunlight. Further, it undergoes the process of sintering at 800 degrees Celsius using a muffle furnace for 6 hours. Then the seashell is crushed using the mortar and pestle. As a result, fine particles of CaO are obtained.

### Nano-hydroxyapatite Powder (HAP) preparation:

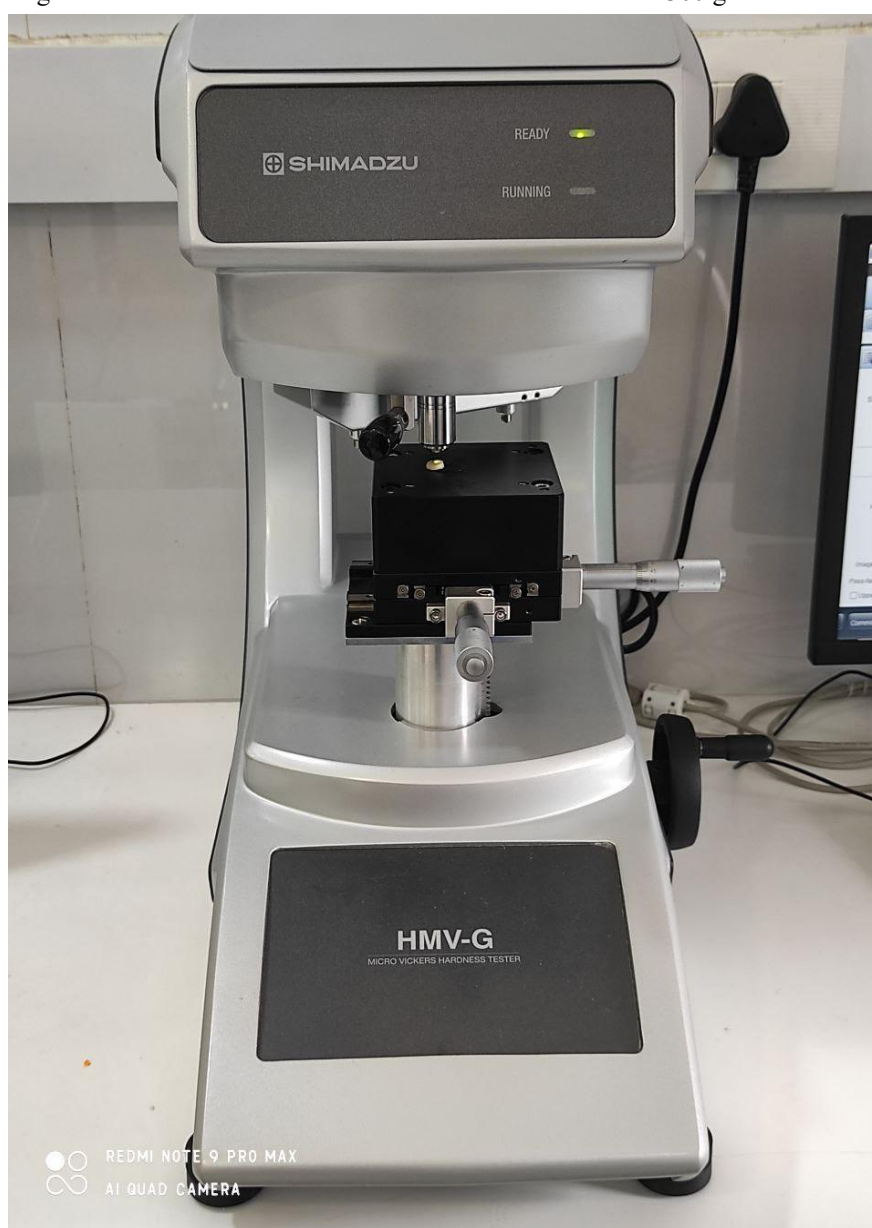
In the present work, calcium oxide (CaO) (S D Fine Chem Limited, Mumbai, India), orthophosphoric acid ( $\text{H}_3\text{PO}_4$ ) (Fisher Scientific, Mumbai, INDIA), and ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) (Fisher Scientific, Mumbai, India) were used as starting materials. Firstly, an analytical weighing scale was used to accurately weigh CaO powder. 1.42 mol (79.55 g) CaO powder was added to 500 ml of deionised water in a 1000 ml beaker and vigorously stirred at 1000 r/min at 20 C for 24 h to react and form a suspension of  $\text{Ca}(\text{OH})_2$  in an excess of

deionised water. The beaker was covered in order to avoid possible contamination via contact with atmospheric conditions. A thermostat-controlled water bath maintained the temperature of the reaction (20 °C). An analytical weighing scale was used to accurately weigh the required quantity of orthophosphoric acid. 97.32 g of 85%  $\text{H}_3\text{PO}_4$  was added to the  $\text{Ca}(\text{OH})_2$  solution at a rate of 1.5 ml/min. During the course of the acid addition, the pH of the solution was monitored via a handheld pH meter with an accuracy of 0.2. The reactants were stirred for a further 24 h to aid the maturation stage, under continuous stirring conditions at 1000 r/min, held at the respective reaction temperature of 20 °C. 0.28 mol (9.94 g)  $\text{NH}_4\text{OH}$  was added to the HA slurry after a 24 h ripening period to stabilise the pH of the supersaturation solution to above 9. To maintain a Ca/P ratio of 1.67, the solution was kept above 9[1,5]. A pH of 10 has been considered to produce the correct stoichiometric condition by which only pure single-phase HA is formed.

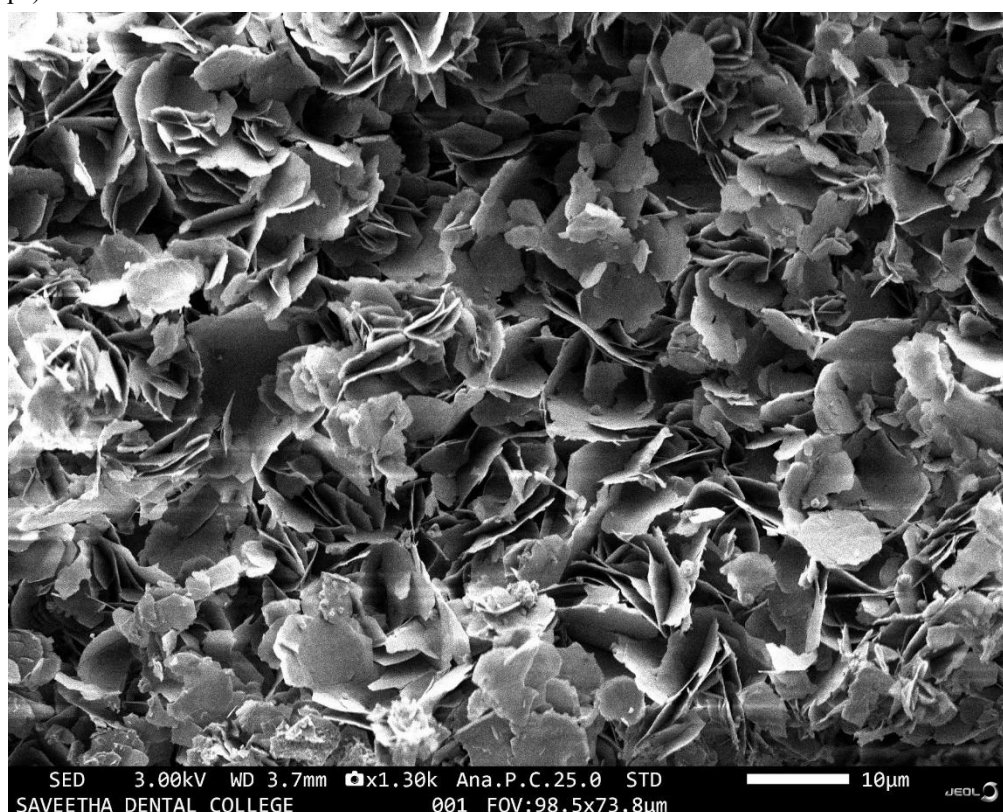
#### Micro hardness test :

The Vickers microhardness test was carried out using a Shimadzu HMV-G microhardness tester with a load of 300 g, with a well time of 15 seconds (Figure 1).

**Figure 1:** Microhardness testing of conventional GIC and GIC reinforced with calcium oxide and hydroxyapatite nanoparticles using a Shimadzu HMV-G microhardness tester with a load of 300 g.



**Figure 2:** Image of GIC reinforced with hydroxyapatite nanoparticles sample under a SEM (scanning electron microscope).



## RESULT

As a result, by comparing the microhardness of conventional GIC and GIC reinforced with calcium oxide and hydroxyapatite nanoparticles using SPSS ANOVA Test. The p-value is found to differ significantly from 0.00353, i.e.,  $p < 0.05$  (Table 1). The p-value is **0.003537**. The result is significant at  $p < 0.05$ .

|                    | Conventional GIC | GIC + HAP | GIC + CaO |
|--------------------|------------------|-----------|-----------|
| Sample size (n)    | 5                | 5         | 5         |
| Mean               | 15.7             | 27.22     | 16.96     |
| Standard deviation | 1.9429           | 7.0212    | 3.2921    |

**Table 1:** Tabular representation of the mean and standard deviation of conventional GIC is  $15.7 \pm 1.94$ , GIC reinforced with HAP is  $27.22 \pm 7.02$  and GIC reinforced with CaO is  $16.96 \pm 3.29$ . The mean value and standard deviation of the GIC reinforced with HAP nanoparticles are higher when compared to GIC reinforced with CaO. So, we can conclude that GIC reinforced with HAP nanoparticles has higher micro-hardness.

## DISCUSSION:

There are no previous studies comparing the microhardness of conventional GIC, GIC reinforced with CaO and HAP nanoparticles.

However, recent research that examined the impact of HAP nanoparticle inclusion on the flexural strength of RMGIC at 0, 1, 2, 5, 7, and 10 wt% found that the groups with up to 5 wt% HAP showed an increase in flexural strength. Although they were much weaker than the control group, the flexural strengths of the groups with more than 5 wt% HAP were still significantly lower.<sup>(36)</sup> This decline in flexural strength of the combination was attributed to the production of HAP nanoparticle agglomerates in the matrix and increased porosity, which may act as a weak spot. Furthermore, it has been shown that nanoparticles at high weight percentages can act as

nonreactive fillers and obstruct acid-base reactions.(37) Additionally, too much water may seep between the HAP nanoparticle agglomerates in the matrix, reducing strength in the end(38). The findings of the previous study demonstrated that the diametral tensile strength (DTS) of conventional GIC was unaffected by the insertion of 15 wt% of micro-HAP into the conventional GIC.(9),(30),(39) However, after micro HAP inclusion, the DTS of RMIG was considerably reduced. According to some earlier studies, adding HAP to GIC could enhance its mechanical characteristics and bond strength to the tooth structure. Moreover, after HAP was included in GIC, the fluoride ion release improved.(40) In the study conducted by Sadashiv et al, the nano-HA-added GIC group has greater bonding strength (13.88 MPa) than the micro-HA-added GIC group (11.05 MPa).(39,41) According to Jong-Jin Lee et al., more bone-like apatite particles were deposited in the nano-HA-added GIC group than in the micro-HA-added GIC group. In addition, their deposition narrowed the dentinal tubules.(42) There are two possible explanations for the increased deposition of bone-like apatite particles in the nano-HA-added GIC group. First, the high solubility of nano-HA may promote active ionic exchange with SBF, increasing the production of apatite particles that resemble bone. Second, nano-HA has a stronger affinity for tooth structure because its composition is identical to that of teeth.(42)

Furthermore, the addition of nano-hydroxyapatite and fluorapatite to the structure of conventional GIC resulted in higher compressive strength, DTS, and flexural strength when compared to conventional GIC. In the present study, smaller sample sizes were used, and further clinical and in vivo studies are required. It has to be done in class I, II, and V cavities, and it should examine the potential impacts of various HAP and CaO concentrations and particle sizes on the mechanical and binding strength characteristics of GICs, and it can also

be conducted using a variety of other filler particles to improve the micro-hardness and mechanical strength of GIC.

## CONCLUSION:

Conclusively, the mean value and standard deviation of the GIC reinforced with HAP nanoparticles are higher when compared to CaO. So, we can conclude that GIC reinforced with HAP nanoparticles has higher micro-hardness.

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## CONFLICT OF INTEREST

The authors hereby declare that there is no conflict of interest in this study.

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