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Enhancement of Compressive Strength of Glass Ionomer Cement Byincorporation of Calcium And Hydroxyapatite Nanoparticles- An Invitro Study

C. Sunil kumar[1], M. Kishore[2], S. Datta prasad[3], N. Vamsee krishna[4], S.Sunil kumar[5], K. S. Chandrababu[6]

[1]*(Department of conservative dentistry and endodontics/ CKS Theja institute of dental sciences and research, India)*

[2]*(Department of conservative dentistry and endodontics/ CKS Theja institute of dental sciences and research, India)*

Abstract:

Introduction:*Since its introduction, Glass Ionomer Cement [GIC] has been very popular with various advantages. However, the major disadvantage which limits its use in stress bearing areas is its poor compressive strength. Many studies in literature aim to improve the compressive strength by addition of various Nanoparticles. Hence calcium and hydroxyapatite Nanoparticles are used in the present study.*

Aim:*To evaluate and compare the compressive strength of Glass Ionomer Cement after Incorporation of Calcium Nanoparticles and Hydroxyapatite Nanoparticles.*

Materials and methods: *This Invitro study was conducted at CKS THEJA institute of dental sciences and research, Tirupati, India. Calcium and hydroxyapatite nano particles are used in the study. The calcium nano particles are synthesized and characterization was done using dynamic light scattering machine. A total of 24 samples were divided into 4 groups. Group 1 was used as control group without Nanoparticles, group 2 was calcium nanoparticles in 3wt% incorporated in GIC, group 3 was 5wt% calcium nanoparticles incorporated in GIC and group 4 was 5wt% of commercially available hydroxyapatite Nanoparticles incorporated in GIC and material manipulation was done according to manufacturer's instructions. Freshly mixed cement samples were placed in metalmolds, stored in distilled water for 24 hours and compressive strength test was done using universal testing machine. Statistical analysis was done using one way ANOVA Post hoc tukey test. $p<0.05$ was considered as statistically significant.*

Results: *There was a statistically highly significant difference ($P < 0.001$**) between the groups when the overall compressive strength means were compared. The highest fracture resistance value was demonstrated by Group 4 with mean value of 147.95 MPa, whereas the least fracture resistance values were observed in Group 1 with mean value of 128.55 MPa. Inter group comparison showed that group 3 is statistically significant and group 2 and 4 are statistically highly significant compared to group 1.*

Conclusion:*The compressive strength of conventional GIC can be enhanced by the addition of various Nanoparticles. The compressive strength of the GIC modified with calcium and hydroxyapatite Nanoparticles is significantly higher. So these Nanoparticles can be considered promising additives for glass ionomer restorative dental materials.*

Key Word*: Glass ionomer cement; hydroxyapatite nanoparticle; calcium nano particles; compressive strength.*

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I. Introduction

Since its introduction in 1972, glass-ionomer cement (GIC) has been very popular among dentists due to its exclusive properties such as chemical adhesion to mineralized tissues of the tooth, low coefficient of thermal expansion and moisture insensitivity which is similar to that of tooth structure [1-3]. Moreover, the superior biocompatibility, fluoride release, and rechargeability of GIC have attained its anticariogenic property [1-3]. Neutral aqueous solution exposure brings many changes in set Glass Ionomer Cement, that is, it absorbs water and releases various ions such as sodium, silica, calcium, and fluoride [4, 5]. A fast burst during the early period (1–7 days), and a long-term diffusive process [6] are the two processes that occur in relation to fluoride release. Despite all these advantages, there is a disadvantage of Glass Ionomer Cement that compromises its durability in stress-bearing areas [7] that is the low compressive strength.

Many attempts have been made to enhance the compressive strength of conventional GIC, such as the addition of resin, alumina, carbon particles, glass particles and various nano sized particles like hydroxyapatite

Nanoparticles, and fluoroapatite Nanoparticles [8]. Surprisingly these additives did not compromise the fluoride-release properties of GIC [8].

The composition and crystal structure of hydroxyapatite (HA) is similar to the apatite in human dental structure. Moreover, it has excellent biocompatible properties. The tooth enamel primarily consists of calcium phosphate minerals, HA also belongs to the same type. Therefore, modification of Glass Ionomer Cement with incorporation of hydroxyapatite improves biocompatibility as well as enhancing the most important mechanical property which is compressive strength [9]. The addition of the Hydroxyapatite particles to the GIC has the ability to increase the fracture toughness and that maintained long term bond to the dentin [9]. The utilization of Chicken Egg Shell Particles as a filler material to enhance the mechanical properties of GIC has some advantages, as it affects the mechanical properties, as well as its fluoride and calcium release [8]. As calcium is the major content of the chicken egg shell [10]. Apart from using chicken egg shell nano particles, calcium nano particles which are indigenously synthesized are used in the study.

The aim of this study was to incorporate Calcium Nanoparticles and Hydroxyapatite Nanoparticles in conventional glass ionomer cement and evaluate and compare the compressive strength of modified Glass Ionomer Cement with that of conventional cement.

II. Materials and methods

This Invitro study was conducted at CKS THEJA institute of dental sciences and research, Tirupati, India.
A total of 24 samples were divided into 4 groups of 6 samples each (n=6)
Group 1: GIC without Nanoparticles (n = 6).
Group 2: GIC with 3%wt. Calcium Nanoparticles added to the powder component (n = 6).
Group 3: GIC with 5%wt. Calcium Nanoparticles added to the powder component (n = 6).
Group 4: GIC with 5%wt. nano HA added to the powder component (n = 6).

PREPARATION OF NANOSCALE CALCIUM OXIDE (N-CaO) PARTICLES

Nanoscale calcium oxide particles (n-CaO) were prepared using sol–gel method. 1% of calcium nitrate (Tetrahydrate purified LR, Sd-fine chemicals Ltd, Mumbai, India) was mixed with 0.05% of sodium citrate tribasic dehydrate (extra pure AR, Sd-fine chemicals Ltd, Mumbai, India) and stirred at 60℃for 3 hours. Then the solution was filtered using filter paper (Whattmanno. 1) and dried at 100℃for 6 hours [11]. The collected powder was used for further characterization and experimental studies.

PARTICLE SIZE AND ZETA POTENTIAL MEASUREMENTS

The dynamic light scattering (DLS) technique was used to measure the hydrosol's hydrodynamic ratio with the help of dynamic light scattering machine and to measure average calcium nano particle size. The recording of the scattering angle of laser light which was obtained from synthesized hydrosols was done using Scanning electron microscope and was measured [11].

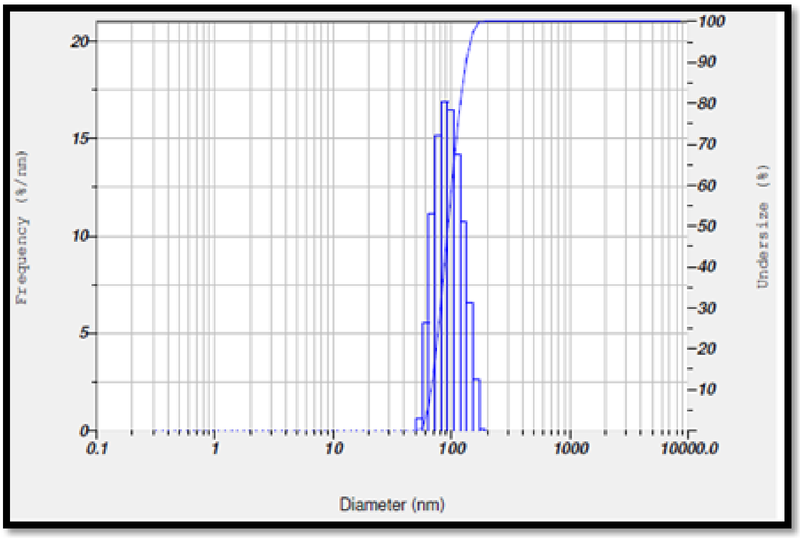
Glass Ionomer Cement was mixed according to the manufacturer's instructions, with 1:1 (Powder:Liquid) (Group 1) and Calcium nanoparticles prepared as described earlier were then added to the powder component with proportions of 3wt% (Group 2) and 5wt% (Group 3) and hydroxyapatite with 5wt% (Group 4) using electrical balance. The specimens were thus categorized into 4 groups.
Cylindrical samples were fabricated using metal molds with 4-mm diameter and 6-mm height and stirred in distilled water for 24 hours before compressive strength test. Universal testing machine (Dak system series 7200, AGS-10 kNG - SHIMADZU) was used to evaluate the compressive strength of set samples at a cross-head speed of 1 mm per min until failure occurred. The maximum load before failure by the surface area is the formula used by the machine operating software to automatically calculate the compressive strength [11].

Statistical analysis

The results obtained were tabulated and subjected to statistical analysis using One way ANOVA carried to determine if there was a significant difference in the overall group means and post hoc tukey test was used to compare mean of one group against each other. P value <0.05 was considered as statistically significant.

III. Results

The DLS results showed that the average calcium nano particle size is 84.1 nm [table/fig no. 1 and 2]. The recording of the scattering angle of laser light which was obtained from synthesized hydrosols was done using Scanning electron microscope and was measured [table/fig no. 3].



Table/fig 1– Histogram showing the particle size distribution and hydrodynamic diameter of the n-CaO particles

Calculation Results

Peak No.	S.P.Area Ratio	Mean	S. D.	Mode
1	1.00	97.3 nm	24.9 nm	87.9 nm
2	---	--- nm	--- nm	--- nm
3	---	--- nm	--- nm	--- nm
Total	1.00	97.3 nm	24.9 nm	87.9 nm

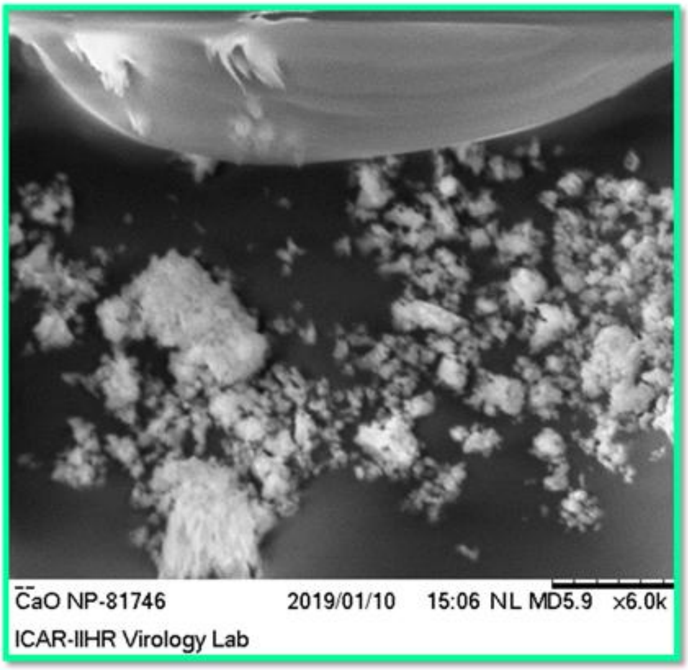
Cumulant Operations

Z-Average : 84.1 nm
PI : 0.425

Molecular weight measurement

Molecular weight : ---
Mark-Houwink-Sakurada parameters : ---

Table/fig 2– calculation results of the particle size distribution and hydrodynamic diameter of the n-CaO particles.



Table/ fig 3- calcium nano particle SEM image.

Table/fig 4 - shows the results of One way ANOVA to determine if there was a significant difference in the overall group means. There was a statistically highly significant difference (p< 0.001**) in compressive strength values between the groups. The highest fracture resistance value was demonstrated by Group 4 mean value of 147.95 MPa, whereas the least fracture resistance values were observed in Group 1 mean value of 128.55 MPa.

	GROUP 1 (control - GIC)	GROUP 2 (3% nano calcium + GIC)	GROUP 3 (5% nano calcium + GIC)	GROUP 4 (5% nano HA + GIC)
Mean	128.556	146.4477	140.5635	147.9577
SD	5.304319	6.033366	4.897196	8.226909
F value	11.93704			
P value	0.000106* Highly Significant			

Table/fig 4 – compressive strength test results] (in MPa)

One way ANOVA was carried out to determine if there was a significant difference in the overall group means

Treatments pair		Tukey HSD p-value
GIC (Group 1)	3% nano calcium + GIC (Group 2)	0.0010**
	5% nano calcium + GIC (Group 3)	0.0162*
	5% nano HA + GIC (Group 4)	0.0010**
3% nano calcium + GIC (Group 2)	5% nano calcium + GIC (Group 3)	0.3857(NS)
	5% nano HA + GIC (Group 4)	0.8999(NS)
5% nano calcium + GIC (Group 3)	5% nano HA + GIC (Group 4)	0.2038(NS)

Fig 5- Post hoc tukey, P<0.05* statistically significant, p<0.001** statistically highly significant, NS not significant

Table/Fig 5 shows the inter group comparison of compressive strength, significant difference was observed between group 1 with remaining groups (group 2, group 3, group 4) and no significant differences were observed between group 2 with remaining group (group 3, Group 4) and between group 3 and group 4.

IV. Discussion

Dental caries continues to be one of the most common oral diseases despite the advancements in the field of oral health. [14]. As a result, the major priority is given to the treatment of decayed teeth [12]. GIC is a favorable choice to restore the teeth with caries especially in non-stress bearing areas [12]. Since its introduction, GIC has undergone various improvements, which significantly improved the strength and handle GIC in stress bearing areas [15].

GIC can be modified with incorporation of various nanoparticles which leads to wider particle size distribution, which is the main cause for higher mechanical values [12]. The Nanoparticles are believed to occupy the empty spaces which are present between the particles of glass ionomer material and reinforce the composition of the material [13].

To evaluate the strength of the restorative material, the most routine test to be done is the compressive strength testing [16, 17, 18& 23]. Hence, we focused on this part of the mechanical property of the restorative cement to evaluate using universal testing machine.

In the present study, it was evident that incorporation of calcium and HA nanoparticles to conventional GIC significantly increased the mechanical property of the cement in terms of compressive strength.

GIC modified by HA group showed higher compressive strength compared to other groups and is an evidence for its structure and function [19&24].However, there was no statistically significant difference between the compressive strengths of GIC modified by the addition of Calcium or HA nanoparticles.

Williams Billington (1989) analyzed the compressive strength of glass ionomer cements after 30 minutes, 1 hour and 24 hours following the specifications proposed by ISO 7489:19868. It was observed that a 24-hour storage period has been the most widely chosen, since most of these materials reach their limit strength value within this period, which is recommended by the British Standard (BS 6039:1981) and by the International Standard specified by ISO (7489: 1986)58 due to which in this study 24 hour storage period was used according to British Standards [24].

The 24 hour compressive strength test aimed at obtaining preliminary data is considered as a corner stone for investigations of compressive strength [22]. Hence we have done compressive strength test after 24 hours of manipulation of the cement.

The DLS result of calcium Nanoparticles showed the mean size of 100 NM which are in close agreement with previous studies by M. Deepa et al (113 NM) [11]in which similar method is employed to synthesize nanoparticles. In addition, the Scanning Electron Microscope results showed that the synthesized Nanoparticles are spherical in shape.

Alireza et al reported that the addition of 5% nano-HA into commercial GIC enhanced the mechanical properties (compressive, diametral tensile and biaxial flexural strength) of the resulting cements and their bond strengths to dentin [10]. The addition of the hydroxyapatite particles to the glass-ionomer powder has the ability to increase the fracture toughness of the cement which maintained long-term bond to dentin [20& 21].

Both nano HA and calcium Nanoparticles are involved in the acid base reaction of GIC and reacts with organic or inorganic components of the GIC network via their phosphate and calcium ions thereby incorporating HA and calcium Nano with GIC powder composition, after H+ attack the ceramic particles, there would be more calcium ions available for cement formation, polysalt bridge formation and cross linking, all of which reinforce the GIC matrix [9]. In this study both modified GIC powders exhibited high value in compressive strength tests in comparison with commercially available GIC.

The possibility of formation of hydrogen bond may be greater because of the presence of additional hydroxyl, phosphate and fluoride ions in the matrix. Without doubt, stronger bonds between organic and inorganic network of the set cement lead to higher mechanical strength of final set cement [9].

V. Limitations

Since the mechanical properties of Glass Ionomer Cement increases with time, compressive strength test done at extended frames would have added extra value to this research.

VI. Conclusion

Within the limitations of the study, it can be concluded that the compressive strength of conventional GIC was significantly enhanced by the addition of various Nanoparticles. So these bioceramic particles can be considered as promising additives for restorative dental materials like Glass Ionomer Cement.

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