

Incorporation of Cellulose Microfibers, Cellulose Nanocrystals and Nano Hydroxyapatite in Glass Ionomer Cement to Evaluate and Compare Compressive Strength - An Invitro Study

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Citation of this Article: M. Kishore, C. Sunil Kumar, S. Datta Prasad, S. Sunil Kumar, N. Vamseekrishna, K. S. Chandrababu, “Incorporation of Cellulose Microfibers, Cellulose Nanocrystals and Nano Hydroxyapatite in Glass Ionomer Cement to Evaluate and Compare Compressive Strength - An Invitro Study”, IJDSIR- December - 2020, Vol. – 3, Issue - 6, P. No. 344 –350.

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Type of Publication: Original Research Article

Conflicts of Interest: Nil

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 cellulose microfibers, cellulose nanocrystals and hydroxyapatite Nanoparticles are used in the present study.

Aim: To evaluate and compare the compressive strength of Glass Ionomer Cement after Incorporation of cellulose microfibers, cellulose nanocrystals and Hydroxyapatite Nanoparticles.

Materials And Methods: This Invitro study was conducted at CKS THEJA institute of dental sciences and research, Tirupati, India. cellulose microfibers, cellulose nanocrystals and hydroxyapatite nano particles are used in the study. A total of 40 samples were divided into 4 groups. Group 1 was used as control group without

Nanoparticles, group 2 was conventional Glass Ionomer Cement with 9.8 wt% of cellulose microfibers group 3 conventional Glass Ionomer Cement with 0.4 wt% of cellulose nanoparticles and group 4 was Conventional Glass Ionomer cement with 5 wt% of nano Hydroxyapatite and material manipulation was done according to manufacturer’s instructions. Freshly mixed cement samples were placed in metal molds, 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. ($p < 0.001$) between the groups when the overall compressive strength means were compared. The highest fracture resistance value was demonstrated by Group 3 with mean value of 217.79 MPa, whereas the least fracture resistance values were observed in Group 1 with mean value of 110.12 MPa. Inter group comparison showed that group 2 and 4 are statistically significant and group 3 is 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 cellulose microfibrils, cellulose nanocrystals and hydroxyapatite Nanoparticles is significantly higher. So these Nanoparticles can be considered promising additives for glass ionomer restorative dental materials.

Keywords: Glass ionomer cement; cellulose microfibril; cellulose nanocrystals; hydroxyapatite nanoparticle; compressive strength.

Introduction

Since its introduction in 1972, glass-ionomer cement (GIC) has been popular among clinicians due to its exclusive properties such as chemical adhesion to mineralized tissues and low coefficient of thermal expansion, which is close to that of tooth structure. Moreover, Glass Ionomer Cement has superior biocompatibility, fluoride release, and rechargeability, which impart its anticariogenic properties. Despite all these advantages, Glass Ionomer Cement has low mechanical strength that compromises its durability in stress-bearing areas. Many attempts have been made to enhance the mechanical properties of conventional Glass Ionomer Cement, such as the addition of resin and the incorporation of alumina, carbon, glass and various nanoparticles etc.¹

. In order to improve the properties and to overcome these shortcomings, active research is in progress, such as the addition of cellulose fibers, hydroxyapatite and fluoroapatite and nanotechnologies.²

Cellulosic fibers have been studied for several years, with the goal of incorporating them into materials, mainly polymers, as a reinforcing agent. The authors found that the addition of the ideal proportion of fibers to Glass Ionomer Cement did not interfere with its working and

setting times, solubility, disintegration in water, and diametral tensile strength, which remained similar to those of the conventional Glass Ionomer Cement meanwhile significantly, improved the mechanical properties.³

Hydroxyapatite nanoparticles have been used widely in medicine and dentistry. Its similar composition with teeth and bone make it a biocompatible substance for the physiological process. It is a natural calcium phosphate ceramic, predominant in 97% enamel. The hardest tissue of our body is tooth enamel which has Hydroxyapatite nano crystals as the building blocks. Incorporation of nano-sized particles may improve the mechanical properties of polymeric dental materials. The incorporation of nanoparticles into glass powder of Glass Ionomers led to wider particle size distribution, which resulted in higher mechanical values. Consequently they can occupy the empty spaces between the Glass Ionomer particles and act as reinforcing material in the composition of the Glass Ionomer cements.⁴

The compressive strength of a material is defined as the amount of stress required to distort the material in an arbitrary amount.⁵

The compressive strength of a material is any important factor to be considered in relation to masticatory forces. This property is the resistance exhibited by a restorative material against intraoral compressive and tensile forces which are produced both in function and parafunction. It is the amount of stress required to distort the material in an arbitrary amount. Compressive strength testing is commonly used as a measure by which clinicians and researchers predict the performance of a restorative material in oral environment.⁶ Hence the purpose of the present study was to evaluate compressive strength of glass ionomer cement with different types of nanoparticles incorporated into it.

Aim of the Study

The main aim of the study was to evaluate the compressive strength of conventional Glass Ionomer Cement and Glass Ionomer Cement modified with cellulose microfibrils, cellulose nanocrystals and nanohydroxyapatite.

Methodology

Cellulose Microfibre Preparation: The cellulose microfibrils (CM) were obtained by processing 6 g of eucalyptus cellulose fibers hydrolyzed with hydrochloric acid. The suspension was filtered and washed with distilled water until the pH was equal to the distilled water. The product was mixed with distilled water and sonicated for five cycles of 2 min each; at a controlled temperature and filtration was done using filter paper. The material obtained was frozen in liquid nitrogen and freeze-dried.

Cellulose Nano Crystal Preparation: To obtain the cellulose nanocrystals (CN) the eucalyptus fibers were hydrolyzed with sulphuric acid at 50°C for about 40 min. The dispersion was diluted twice and washed three times with deionized water by centrifugation, dialyzed against deionized water until it attained pH~6, immediately ultrasonicated for 5 min and filtered.

Hydroxyapatite Nano Crystal Preparation: Nano-hydroxyapatite was produced by an ethanol-based sol-gel method according to the methods similar to Kuriakose et al.¹⁹ and Feng et al.²⁰ by using diammonium phosphate [

(NH₄)₂HPO₄] and calcium nitrate dihydrate[Ca (NO₃)₂·4H₂O].

Compressive Strength Testing

Sample Preparation: Forty samples were divided into four groups.

Group I: conventional Glass Ionomer Cement

Group II: conventional Glass Ionomer Cement with 9.8 wt% of cellulose microfibrils

Group III: conventional Glass Ionomer Cement with 0.4 wt% of cellulose nanoparticles

Group IV: Conventional Glass Ionomer cement with 5 wt% of nano Hydroxyapatite

The nanoparticles and Glass Ionomer Cement were weighed in an electrical balance. The nanoparticles were mixed with the Glass Ionomer Cement powder by using plastic spatula to obtain uniform distribution and the powder was mixed with liquid using nonabsorbent paper pad and plastic spatula. The freshly mixed cement was placed in a metal mold (4X6 mm) covered with glass slab at the bottom and Mylar strip at the top. Slight compression was made to make uniform mass and smooth surfaces and left for 1hr to allow setting. The specimens were removed from the molds and placed in distilled water at 37°C for 24 hours. The compressive strength was then tested using Universal Testing Machine at a crosshead speed of 1 mm/min and the readings were recorded in Mega Pascal. The data collected was subjected to statistical analysis.

Observations and Results

Comparison of compressive strengths between the study groups

Groups	N	Minimum	Maximum	Mean ± SD	F value	p value
Conventional Glass ionomer cement	10	103.72	114.67	110.12		
Glass ionomer cement with cellulose micro fibers	10	131.83	140.5	135.64		

Glass ionomer cement with cellulose nano crystals	10	208.19	226.63	217.79	1193.33 1	<0.05*
Glass ionomer cement with hydroxyapatite nano crystals	10	138.72	145.63	141.83		

Anova P<0.05* (significant) P>0.05 (not significant)

INFERENCE: The above table shows Anova test done for the comparison of compressive strength between the study groups. There is statistically significant difference between all the groups (p<0.05).

Discussion

The quest to search an ideal restorative material has been a challenge in restorative dentistry. Glass Ionomers are a class of biomaterials in widespread use in modern dentistry.⁷ Over the past 50 years, many changes have occurred in restorative materials in development and on availability. Restorative dentistry, in its infancy, was dominated by the simple principle of “Extension for Prevention” laid down by GV Black and which was partially dictated by the restorative material available at that time. The ideal requisites for restorative material are that it should have good color stability, have a coefficient of thermal expansion similar to that of natural tooth structure, with excellent marginal seal, and the ability to adhere chemically to enamel and dentin.⁵ Glass ionomer cement (GIC) seems to meet most of these requirements along with certain drawbacks due to its low physical properties and poor long-term performance.⁸

The present study involves two nanoparticles, namely cellulose nano particles and hydroxyapatite nanoparticles. They were selected because they exhibit relatively low toxicity when present in other dental restorative formulations.⁹ For many years there have been attempts to incorporate fibers into the composition of these materials as agents to reinforce their physical structure.¹⁰⁻¹¹

The nanocrystals present in cellulose, in addition to carbon nano-tubes, are considered excellent options for

reinforcement, favoring improvements of the material’s mechanical properties. Cellulose nanocrystals (CNs) have excellent dispersion in the matrix besides maintaining the integrity and purity of the material. In addition to these characteristics, Cellulose Nanocrystals have a much lower cost when compared to carbon nanotubes.¹² Cellulose Nanocrystals-modified Glass Ionomer Cement resulted in greatly improved resistance to abrasion, and increased compression, diametral tensile strength and modulus of elasticity, using a lower concentration of cellulose in the material composition, besides being biocompatible.¹³

Hydroxyapatite, the main mineral component of the tooth structure and bone, is a bioceramic containing calcium and phosphorus. The Hydroxyapatite particles were added to Glass Ionomer powder due to their biocompatibility and similar composition to apatite in human dental and skeletal systems. Several studies reported improvements in mechanical properties of these materials such as diameter, tensile strength, fracture toughness, bonding and compressive strength compared to conventional glass-ionomers.¹⁴

Thus, the present study was conducted to compare and evaluate the compressive strength of conventional Glass Ionomer Cement incorporated with Nanohydroxyapatite, cellulose microfibers and cellulose 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, which considers specimens with 4mm in diameter and 6 mm in height. 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) due to which in this study 4mm in diameter and 6mm in height of samples and 24 hour storage period were used according to British Standards.¹⁵

Various storage media were used in order to simulate oral condition. The ability of restorative dental materials to withstand the functional force and exposure to various media in the mouth is an important requirement for their clinical performance for a considerable period of time. In this study distilled water is used as storage media.¹⁶

The compressive strength (CS) is an important property in restorative materials, particularly in the process of mastication. This test is more suitable to compare brittle materials, which show relatively low result when subjected to tension.

In the present study, results showed that the compressive strength was highest for cellulose nano crystals incorporated glass ionomer followed by Nano Hydroxyapatite incorporated Glass Ionomer cement then cellulose microfiber incorporated Glass Ionomer cement and least for Conventional Glass Ionomer cement.

The reason for the increased bonding strength of Glass Ionomer Cement with the addition of Hydroxyapatite is that calcium ions from Hydroxyapatite may participate in chemical ionic bonding between the tooth and the material. Due to their small size, the incorporation of nanoparticles into glass powder of Glass Ionomers cements, led to wider particle size distribution (the average particle size of Glass Ionomer particles were around 10–20µm) which resulted in higher mechanical values which is in accordance with the present study.¹⁷

The addition of cellulose fibers to glass ionomer cement modified the material structure so that it can be used in areas of great masticatory efforts, such as in restoration of

posterior teeth, especially in the (atraumatic restorative treatment) ART technique. The addition of a small amount of a nano particulate renewable material in the form of cellulose nanocrystals significantly increased the compressive and diametral tensile strengths of restorative Glass Ionomer Cements. Modification of Glass Ionomer Cements with Cellulose Nanocrystals seems to produce very promising restorative materials to be indicated as alternatives to dental amalgams. It is necessary for further studies to validate these findings and to verify the required conditions to lead us to an outstanding material for physical-chemical and biological performances.¹⁸

Conclusions

The following conclusions were drawn from the study:

1. The compressive strength was highest for Cellulose nano crystal incorporated glass ionomer cement followed by hydroxyapatite nano crystals incorporated glass ionomer cement and cellulose microfiber incorporated glass ionomer cement, and least for the Conventional Glass Ionomer cement.
2. Nanoparticles improves the compressive strength of the Conventional Glass Ionomer cements.

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