

Qualitative and Quantitative Effects of Non-Thermal Atmospheric Pressure Plasma and ErCr: YSGG Laser Activation on Surface Remineralization and Fluoride Release of Three Different Fluoride Varnishes

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Abstract

Dental caries remains a prevalent oral health concern worldwide, prompting ongoing research into innovative approaches for caries prevention and management. This experimental investigation endeavors to conduct a comparative assessment of the bioactivity, fluoride release kinetics, shear bond strength, and compressive strength of glass ionomer cement (GIC) augmented with three distinct types of inorganic bioactive nanoparticles. Bioactive nanoparticles have garnered interest in dental materials for their potential to enhance material properties and promote remineralization. Three types of inorganic bioactive nanoparticles were incorporated into GIC formulations, and their effects on bioactivity, fluoride release kinetics, shear bond strength to enamel, and compressive strength were investigated using standardized laboratory tests. Results revealed significant variations in bioactivity, fluoride release profiles, shear bond strength, and compressive strength among the GIC formulations containing different bioactive nanoparticles. This study underscores the potential of NTAPP and ErCr: YSGG laser activation in enhancing the remineralization efficacy of fluoride varnishes, thereby contributing to the development of more effective caries prevention strategies. By elucidating the qualitative and quantitative effects of these activation techniques on surface remineralization and fluoride release, this research offers valuable insights into their application in clinical practice. Further investigations, including *in vitro* and *in vivo* studies, are warranted to validate these findings and explore the long-term efficacy and safety of activated fluoride varnishes in preventing dental caries. Understanding the effects of bioactive nanoparticles on GIC properties can inform the development of novel dental materials with improved clinical performance and therapeutic benefits.

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INTRODUCTION

“Innovation serves as the beacon guiding the trajectory of the future.” In this vein, the imperative for ingenuity and the integration of novel materials pervades every domain, with dentistry standing as a quintessential example. Dental materials are currently changing in behaviour from being passive

biomaterials that had no positive or negative effects to having constant bioactivity with positive and expected effects, such as cellular stimulation or antibacterial activity.

A bioactive material affects a living organism, including cells, tissues, and organs, as its name suggests. In 1996, Cao and Hench established the term “bioactivity,” which had previously been used in 1969. In general, it describes a particular characteristic of a substance that affects or will cause a reaction from a living tissue or cell, such as stimulating the creation of hydroxyapatite [1].

The glass ionomer cement (GIC) has been frequently utilised in dentistry since 1970. Because of its good mechanical qualities and transparency, it is regarded as being superior to other kinds of water-based cements. GIC is useful as a dental material because of its long-lasting fluoride release and strong biocompatibility. However, GIC has notable drawbacks, including a very high degree of brittleness and moisture sensitivity in the early stages of setting [2].

The inclusion of bioactive nanoparticles is one of the methods of improving bioactivity. Nanomaterials are used in dentistry to improve the qualities and functioning of dental materials as well as to create inventive, novel products that are beneficial to patients. According to their size, materials with nanoscales exhibit peculiar features. In comparison to larger size fillers, nanofillers offer superior tooth adhesion.

There has been a notable interest in developing new dental products using nanotechnology-based approaches and enhancing the functionality of existing dental products. By utilising nanoparticles composed of hydroxyapatite and fluoride, products based on nanotechnology have the potential to enhance the mineralization of hard dental tissues [3].

Forsterite in its pure form comprises magnesium, oxygen, and silicon, encapsulated in the chemical formula Mg_2SiO_4 . Research indicates that forsterite nanoparticles exhibit elevated surface energy and a tendency for heightened reactivity. Consequently, these nanoparticles have been shown to possess bioactive properties and demonstrate superior mechanical characteristics compared to microparticles of Mg_2SiO_4 [4].

Niobium pentoxide (Nb_2O_5) is a metal oxide typically found in the form of a monoclinic solid, insoluble in solvents, and believed to enhance mechanical properties. Beyond these improvements, Nb_2O_5 demonstrates bioactivity and enhances both the chemical and physical attributes of materials. Previous studies have shown that Nb_2O_5 triggers the formation of hydroxyapatite crystals upon contact with simulated human saliva or bodily fluids, further underscoring its potential in biomedical applications [5].

Considering the preceding conversation, the prudent utilization of bioactive materials could provide specific clinical benefits in restorative treatments. In light of the existing literature's limitations in directly comparing various bioactive materials, our study endeavors to investigate the synergistic effects of incorporating three distinct bioactive nanoparticle materials into conventional glass ionomer cement. Specifically, our goal is to evaluate the bioactivity, fluoride release, shear bond strength, and compressive strength of these composite materials. Through this research, we seek to evaluate and contrast the performance of different inorganic bioactive nanoparticles when integrated into conventional glass ionomer cement.

REVIEW LITERATURE

In recent times, there has been a notable progress in the field of dental materials. Modern techniques and materials have revolutionized dental practices and treatment strategies, encompassing everything from restorative and endodontic materials to bioactive compounds employed for bone regeneration. The utilization of “bioactive” materials for both restorative and reconstructive purposes has emerged as a prominent subject in contemporary dentistry. It must be emphasized that the definition of what is truly

termed “bioactive” varies depending on the application. The ability of a substance to generate hydroxyapatite crystals on its surface is referred to as being “bioactive” in the context of restorative dentistry [6, 7].

Various materials have been employed to address diverse dental issues; nevertheless, the efficacy of these treatments is restricted by the inherent properties of the biomaterials utilized. Due to NPs' distinctive qualities, such as their high surface-to volume ratio, antibacterial activity, physical, mechanical, and biological capabilities, and distinctive particle size, they are useful delivery systems for dental applications [8].

Bioactivity Evaluation

The pathophysiology of secondary caries and primary caries are similar. The bacterial microflora and their organic products form a biofilm that attaches to the tooth and secrete acids. The equilibrium between demineralization and remineralization of the tooth structure is affected as the pH value decreases as a result of acids, which results in the loss of calcium and phosphate ions. The minuscule gap between the tooth and the restoration defies mechanical cleaning, unlike accessible tooth surfaces that respond to routine oral hygiene methods by clearing away biofilm. Consequently, the microorganisms within this crevice persist undisturbed, leading to secondary caries over time. However, targeted interactions between dental materials and the oral environment can prevent secondary caries, a phenomenon recognized as bioactivity. Song Chen et al. studied the bioactivity of wollastonite nanoparticles incorporated into glass ionomer cement, while Sayyed FS et al. investigated the bioactivity of forsterite nanoparticles incorporated into glass ionomer cement. However, the addition of niobium pentoxide nanoparticles has been studied for the first time in this context [2, 4].

The addition of 3 wt% nanoparticles to glass ionomer cement was selected based on findings from two studies. Sayyed FS et al. concluded that incorporating 3 wt% forsterite nanoparticles into GIC enhanced bioactivity. Similarly, Isadora Martini Garcia et al. found that higher concentrations of Nb₂O₅ (5 wt% and above) negatively affected the material's physical properties [4, 6, 9].

Highest Ca levels were seen in Category 3 followed by Category 2, Category 4 and least Ca levels was seen in Category 1. Inter category pairwise comparison showed Category 1 had statistically notable lower ($p < 0.001$) Calcium levels were compared between Category 2 and Category 3, revealing notable differences. However, no significant difference was observed between Category 1 and Category 4. Similar results were found for phosphorus levels: the highest levels were observed in Category 3, followed by Category 2, Category 4, and the lowest activity in Category 1. Inter category pairwise comparison between four study categories showed that Category 1 had statistically notable lower ($p < 0.001$) P levels when compared to Category 2, 3, 4. The formation of a complex with high molecular weight is facilitated by the initiation of calcium and phosphorus nucleation during the apatite crystal formation, thereby enhancing remineralization. Consequently, the surface remineralization on bioactive materials can diminish or constrain the hydrolysis of collagen and resin constituents. Furthermore, the calcium and phosphate released from these materials can provide therapeutic advantages for the dental lesion.

Fluoride Release Evaluation

The prevention of caries is not directly attributed to fluoride. Fluoride's introduction into dentistry has been a notable step toward lowering the incidence of caries. Nearly 60 years ago, the use of fluoride in preventative dentistry was pioneered. The integration of fluoride into the lattice structure during the preeruptive stages of tooth development was considered to diminish the solubility of enamel. But it turned out that incorporating fluoride ions during the post-eruptive stages of tooth growth is made possible by the concomitant disintegration of tooth structure. Fluoride not only prevents caries but also slows its progression in a number of ways. Fluoride shields against demineralization and promotes remineralization by reconstructing the partially damaged hydroxyapatite crystal lattice. This fortifies the structure, rendering it less susceptible to acid erosion [10].

The fluoride ion release from the treated samples was assessed with Ion selective electrode (ISE) on day 01, 07 and 28. The samples were immersed in 5 ml deionized water for the categorical time and fluoride concentration in the water was measured in ppm. Previously, fluoride release of the Mg_2SiO_4 nanoparticles added to GIC has been studied but literature lacks studies of fluoride release of wollastonite and niobium pentoxide nanoparticles incorporated with GIC.

Upon pairwise comparison, significant statistical disparities were observed between each category, except for Category 2 and 4, where the distinction was not statistically significant ($p > 0.05$) on both day 1 and day 7. At 28th day, on pairwise comparison, there was high degree of statistical notable difference between each pair of categories.

Shear bond Strength and Mode of Failure Evaluation

Shear bond strength is a simple and frequently used test to evaluate how well a restorative material bonds to the tooth. It has been mentioned that the primary advantage of GIC lies in its chemical bonding to enamel and dentin. The capacity of restorative materials to adhere to tooth structure is a crucial aspect in contemporary restorative methods [11].

The nature of the dental base is among numerous factors that could influence the adhesive properties of a material. Dentin surfaces vary between individual teeth, and the bonding strength can fluctuate depending on the location within the tooth where bonding occurs, owing to changes in the size of dental tubules from the dentinoenamel junction (DEJ) to the pulp chamber. The standard deviation for each experimental category or the diverse results in the numerous research could both be influenced by this issue [12].

Compressive Strength Evaluation

The compressive strength of Glass Ionomer Cements (GICs) is a mechanical attribute of brittle substances associated with the forces exerted during chewing in vivo and the cross-linking of trivalent ions within the matrix. It was verified utilizing the universal testing machine. Multiple studies have indicated that the fillers in GICs could influence the mechanical properties of the cements, potentially yielding either positive or negative outcomes. Whether they can increase the cross-linking in the cured cement is dependent on their abilities. The mechanical strength frequently has adverse effects because of the additives used to boost bioactivity and antibacterial characteristics [13].

According to H. Prentice et al. [14], when nanoparticles are added, the strength of the cement is reduced, which shows that the nanoparticles notably interfere with the usual glass-ionomer reaction. The addition, however, might point to inappropriate wettability at the filler-matrix interface. Around the nanoparticles in this situation, cracks emerge, and the number of cracks is directly related with the amount of nanoparticles present. These fissures reduce mechanical strength by serving as stress concentration points.

In the present study, the results concluded that mean compressive strength was highest in Category 2 followed by Category 4, Category 1 and Category 3. There was high degree of statistical notable ($p < 0.001$) difference among four study categories. On pairwise comparison, the mean compressive strength of Category 1 was significantly lower than that of Categories 2 and 4 ($p < 0.001$). However, the mean compressive strength of Category 1 was slightly higher than that of Category 3, but this difference was not statistically significant ($p > 0.05$). Thus, the addition of the bioactive nanoparticles increased the compressive strength in the Category 2 and Category 4 while in the Category 3 it did not affect the compressive strength adversely.

MATERIALS AND METHODS

This in-vitro study was carried out in School of Dental Sciences, Krishna Institute of Medical Sciences, deemed to be University, Karad. The ethical clearance was obtained from the committee before the initiation of the study (protocol number 230/2020-2021).

The sample size of 160 was derived by a statistician using the following formula to obtain a statistically notable result.

$$n = \frac{(z_{\alpha} + z_{\beta})^2 (S_1 + S_2)^2}{(m_1 + m_2)}$$

$$z_{\alpha} = 1.96 \quad S_1 = 8.4 \quad m_1 = 57$$

$$z_{\beta} = 1.64 \quad S_2 = 27.7 \quad m_2 = 24.8$$

With level of significance = 5%, Power = 95%

Thus, n= 10 was the sample size per parameter to have notable results (40 samples per category) and total sample size of 160 was finalized.

The materials used in this study are listed below. The choice of materials includes GIC incorporated with three inorganic nanoparticles: Forsterite nanoparticles, wollastonite nanoparticles and Niobium pentoxide nanoparticles.

Inclusion Criteria

Morphologically intact non-carious, non-hypoplastic, non-restored premolars which are extracted for therapeutic orthodontic treatment.

Exclusion Criteria

Teeth with morphological variations, fractured crowns, fluorosed teeth and teeth with hypoplastic lesions.

The teeth samples selected for the study comprised 80 human premolars, which were extracted for therapeutic orthodontic purposes, as shown in Table 1.

Table 1. Sample distribution in different categories.

Materials	Bioactivity (material specimens)	Compressive strength (material specimens)	Shear bond strength. (restored teeth)	Fluoride release (restored teeth)
<i>Category 1:</i> GIC [GC Gold Label 2 glass ionomer cement (control category)]	10	10	10	10
<i>Category 2:</i> GIC + Forsterite nanoparticles	10	10	10	10
<i>Category 3:</i> GIC + Wollastonite nanoparticles	10	10	10	10
<i>Category 4:</i> GIC + Niobium pentoxide nanoparticles	10	10	10	10

Materials Used in the Study

- Extracted teeth.
- GC Gold Label 2 glass ionomer cement
- Forsterite nanoparticles
- Niobium pentoxide nanoparticles
- Wollastonite nanoparticles
- Simulated Body Fluid
- Straight fissure bur
- Diamond Disc
- Test-tubes with seals
- Deionized water

- Self-cure acrylic resin
- Nail varnish

OBSERVATION AND RESULTS

FEG-SEM Analysis

Calcium Levels Among the Four Study Categories

Table 2 displays the bioactivity data (calcium levels) for various study groups. Intra-group analysis was conducted using the One-Way ANOVA test, revealing a significant statistical variance ($p < 0.001$) among all categories. Highest Ca levels were seen in Category 3 followed by Category 2, Category 4 and least activity was seen in Category 1 depicted in Table 2.

Table 2. Overall Comparison of Bioactivity (Ca Levels) Using One-way Anova F Test.

Ca levels	Mean	SD	One-way Anova F test	P value, Significance
Category 1 (GIC)	60.84	0.39	F = 1671.0	$p < 0.001^{**}$
Category 2 (GIC + Forsterite)	75.82	1.23		
Category 3 (GIC + Wollastonite)	86.63	1.21		
Category 4 (GIC + Niobium)	61.94	0.67		

On pairwise comparison of bioactivity (mean calcium levels) among the four study categories, it was observed that Category 1 had statistically notable lower ($p < 0.001$) Ca levels when compared to Category 2 and Category 3. Category 1 had lower Ca levels when compared to Category 4, but the difference was not found to be of statistical significance ($p > 0.05$). Category 2 < Category 3 (Ca levels) but had highly statistical notable difference ($p < 0.001$). Category 2 > Category 4 (Ca levels) with highly statistical notable difference ($p < 0.001$). Category 3 > Category 4 (Ca levels) with highly statistical notable difference ($p < 0.001$) shown in Figure 1.

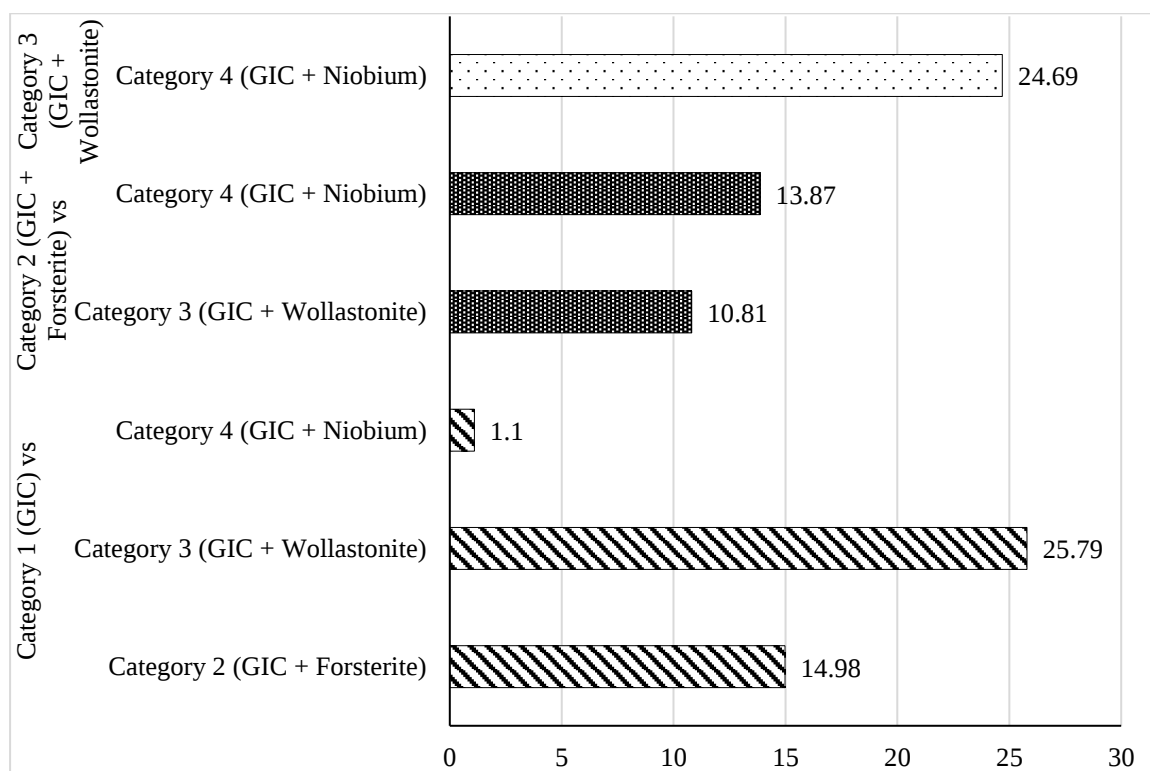


Figure 1. Pairwise Comparison of Mean Ca Levels Among Four Study Categories.

Phosphorus Levels Among Four Study Categories

The bioactivity statistics, specifically phosphorus levels, for the different study categories are presented in Table 3. Intra-category analysis using the One-Way ANOVA test revealed a highly significant difference ($p < 0.001$) across all categories. Highest P levels were seen in Category 3 followed by Category 2, Category 4 and least activity was seen in Category 1.

Table 3. Comparison of Bioactivity Results (P Levels) Using One-way Anova F Test.

P levels	Mean	SD	One way Anova F test	P value, Significance
Category 1(GIC)	25.95	0.56	F = 742.75	$p < 0.001^{**}$
Category 2 (GIC + Forsterite)	36.28	0.56		
Category 3 (GIC + Wollastonite)	45.04	1.13		
Category 4 (GIC + Niobium)	31.45	1.26		

On pairwise comparison in Figure 2 of bioactivity (P levels) between four study categories, it was observed that Category 1 had statistically notable lower ($p < 0.001$) P levels when compared to Category 2, 3, 4. Category 2 < Category 3 (P levels) with highly statistical notable difference ($p < 0.001$). Category 2 > Category 4 (P levels) with highly statistical notable difference ($p < 0.001$). Category 3 > Category 4 (P levels) with highly statistical notable difference ($p < 0.001$).

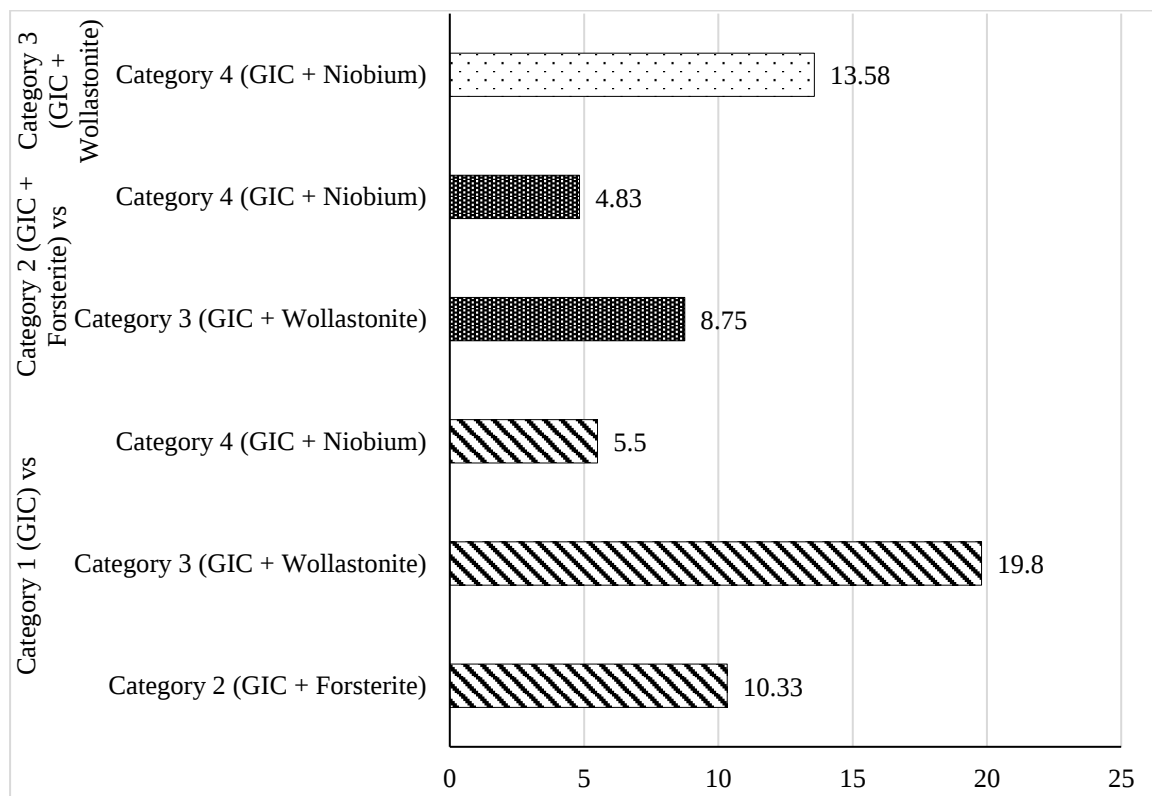


Figure 2. Graph Showing Pairwise Comparison of Mean P Levels.

Parameter 2: Fluoride Release (Ion Selective Electrode)

Day 1,7 and Day 28

Mean fluoride release was highest in Category 3 followed by Category 4, Category 2 and least activity was seen in Category 1. There was high statistical notable ($p < 0.001$) difference among four study categories at Day 1, Day 7. Mean fluoride release was highest in Category 3 followed by Category 2,

Category 4 and least activity was seen in Category 1. There was high degree of statistical notable ($p < 0.001$) difference among four study categories at Day 28 shown in Table 4.

Table 4. Comparison of Mean Fluoride Release Among Four Study Categories Using One Way Anova F Test.

	1 Day Mean (SD)	7 Day Mean (SD)	28 Day Mean (SD)
Category 1 (GIC)	2.68 (0.16)	1.84 (0.1)	1.04 (0.02)
Category 2 (GIC + Forsterite)	2.92 (0.05)	2.1 (0.1)	1.45 (0.13)
Category 3 (GIC + Wollastonite)	3.24 (0.07)	2.66 (0.15)	1.75 (0.08)
Category 4 (GIC + Niobium)	2.97 (0.01)	2.18 (0.05)	1.2 (0.05)
Anova F test value	F = 57.419	F = 91.56	F = 136.16
P value, Significance	$p < 0.001^{**}$	$p < 0.001^{**}$	$p < 0.001^{**}$

Day 1, Day 7 and Day 28

Mean fluoride release was highest in Category 3 followed by Category 4, Category 2 and least activity was seen in Category 1. There was high statistically notable ($p < 0.001$) difference among four study categories at Day 1, Day 7. Upon pairwise comparison, significant statistical differences were observed between each category pair. However, it was determined that the disparity between Category 2 and Category 4 did not reach statistical significance ($p > 0.05$). Mean fluoride release was highest in Category 3 followed by Category 2, Category 4 and least activity was seen in Category 1. There was high statistical notable ($p < 0.001$) difference among four study categories at Day 28 shown in Figure 3.

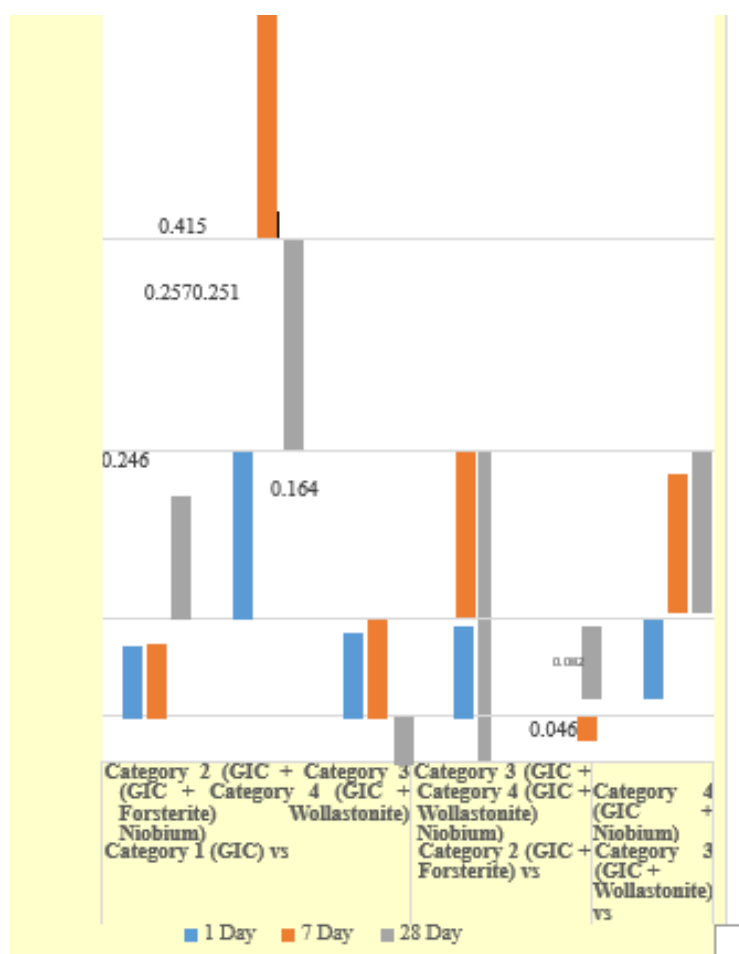


Figure. 3. Pairwise comparison of mean fluoride release among four study categories.

Shear bond strength (Universal testing machine)

Mean shear bond strength was highest in Category 4 followed by Category 2, Category 3 and least strength was seen in Category 1. There was high statistically notable ($p < 0.001$) difference among four study categories Shown in Table 5.

Table 5. Comparison of Average Shear Bond Strength Using One-way Anova F Test.

	Mean	SD	One-way Anova F test	P value, Significance
Category 1 (GIC)	1.8	0.12	F = 427.02	$p < 0.001^{**}$
Category 2 (GIC + Forsterite)	3.46	0.16		
Category 3 (GIC + Wollastonite)	2.43	0.21		
Category 4 (GIC + Niobium)	4.59	0.23		

On pairwise comparison in graph 4, mean compressive strength of Category 1 had notably notable ($p > 0.001$) lower shear bond strength as compared to Category 2, 3, 4. Category 2 > Category 3 (shear bond strength) with high notable statistical difference ($p < 0.001$). Category 2 < Category 4 (shear bond strength) with high notable statistical difference ($p < 0.001$). Category 3 < Category 4 (shear bond strength) with high notable statistical difference ($p < 0.001$) shown in Figure 4.

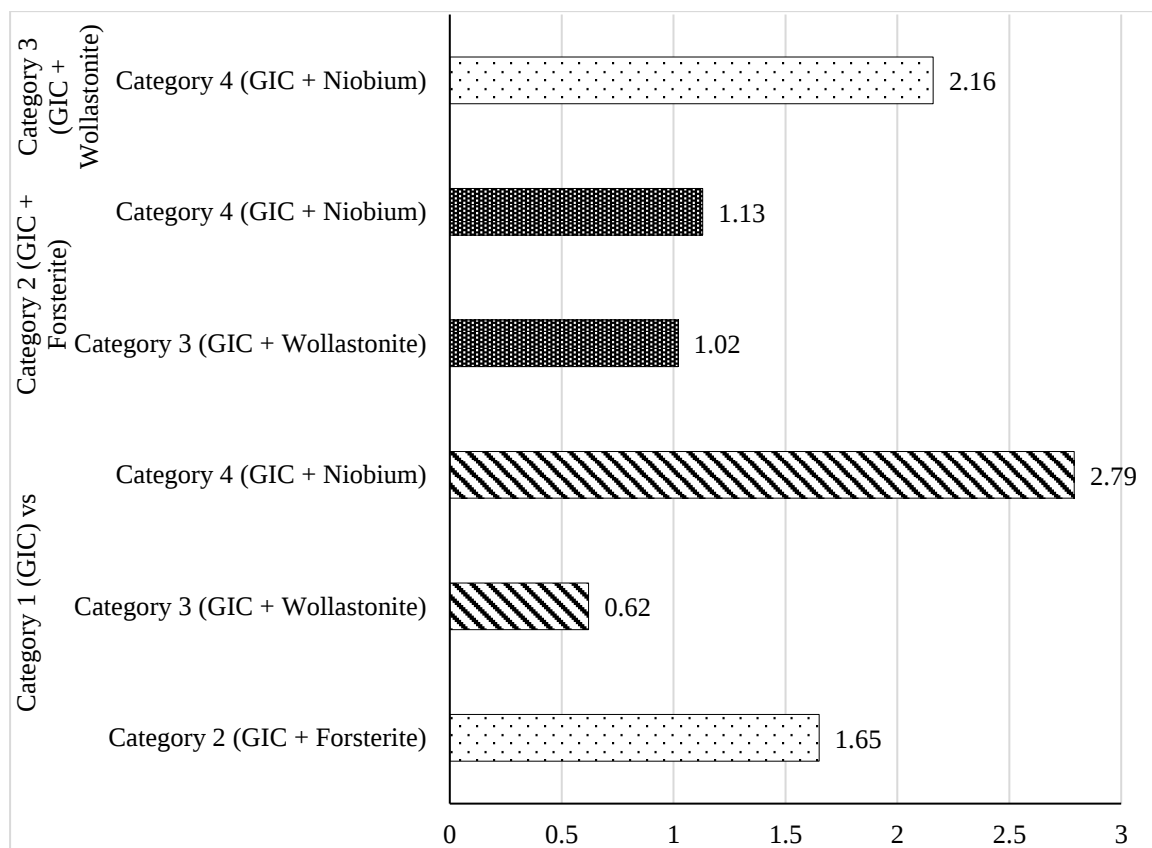


Figure 4. Graph Showing Pairwise Comparison of Average Shear Bond Strength.

Mode of Failure

On overall comparison in Table 6, there was notable statistical difference ($p < 0.05$) among four study categories. On comparison of bond failure between Category 1 and Category 2 & 4, there was found to be high degree of statistically difference ($p < 0.001$). On comparison of bond failure between Category 1 and Category 3, there was found to be notable statistical difference ($p < 0.05$). On comparison of bond

failure between Category 2 and Category 3, there was found to be no notable statistical difference ($p>0.05$). On comparison of bond failure between Category 2 and Category 4, there was found to be no notable statistical difference ($p>0.05$). On comparison of bond failure between Category 3 and Category 4, there was found to be no notable statistical difference ($p>0.05$).

Table 6. Comparison of Bond Failure Type Among Four Categories Using Chi Square Test.

	Adhesive Failure N (%)	Cohesive Failure N (%)	Mixed Failure N (%)
Category 1 (GIC)	1 (10%)	2 (20%)	7 (70%)
Category 2 (GIC + Forsterite)	9 (90%)	0 (0%)	1 (10%)
Category 3 (GIC + Wollastonite)	7 (70%)	0 (0%)	3 (30%)
Category 4 (GIC + Niobium)	9 (90%)	0 (0%)	1 (10%)
Overall Comparison	Chi square test value = 20.615, $p=0.002^*$		
Category 1 vs Category 2	Chi square test value = 56.5, $p<0.001^{**}$		
Category 1 vs Category 3	Chi square test value = 15.87, $p=0.032^*$		
Category 1 vs Category 4	Chi square test value = 56.5, $p<0.001^{**}$		
Category 2 vs Category 3	Chi square test value = 3.7, $p=0.108$		
Category 2 vs Category 4	Chi square test value = 0.0, $p=1.000$		
Category 3 vs Category 4	Chi square test value = 3.7, $p=0.108$		

Parameter 4: Compressive Strength (Universal Testing Machine)

Average strength was maximum in Category 2 followed by Category 4, Category 1 and least activity was seen in Category 3. There was high degree of statistical notable ($p<0.001$) difference among four study categories shown in Figure 5.

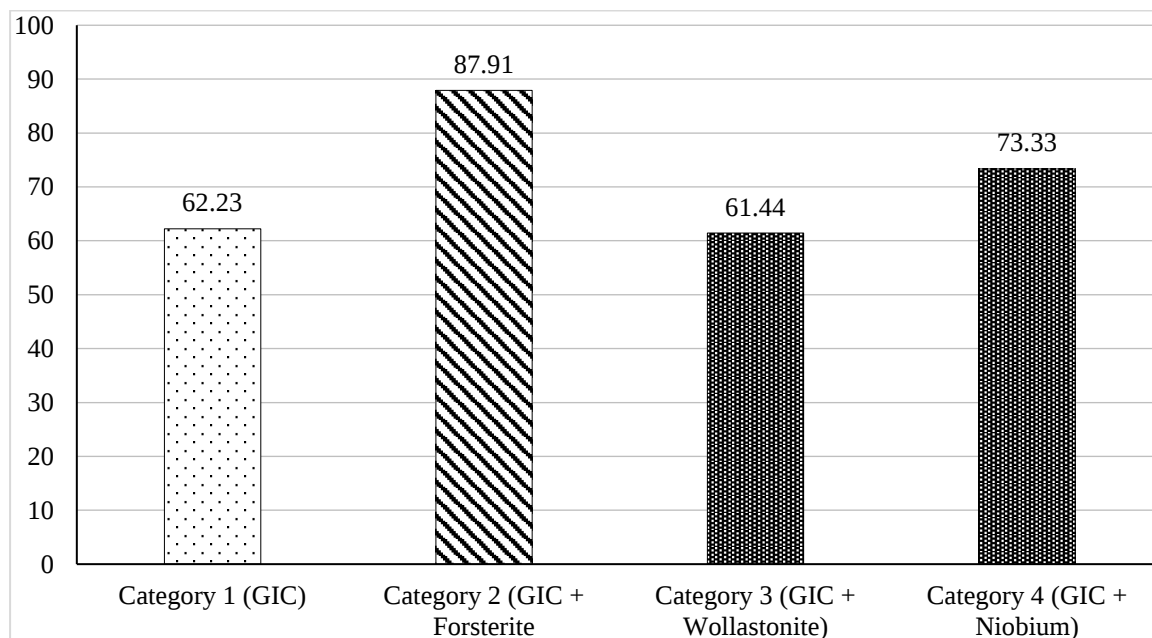


Figure 5. Graph Showing Mean Compressive Strength Among Four Study Categories.

On pairwise comparison shown in Table 7, average strength of Category 1 < Category 2, 4 with statistical notable ($p>0.001$) difference. Average strength of Category 1 > Category 3 but the difference was not found to be of statistical notable ($p>0.05$). Average strength of Category 2 > Category 3 with high notable statistical disparity ($p<0.001$). Average strength of Category 2 > Category 4 with high notable statistical disparity ($p<0.001$). Average strength of Category 3 < Category 4 with high notable statistical disparity ($p<0.001$).

Table 7. Inter Category Comparison of Mean Compressive Strength Between Four Study Categories Using Tukey's post Hoc Test.

Category	Comparison Category	Mean Difference	P value, Significance
Category 1 (GIC) vs	Category 2 (GIC + orsterite)	25.68	p<0.001**
	Category 3 (GIC + Wollastonite)	0.78	P=0.661 (NS)
	Category 4 (GIC + Niobium)	11.1	p < 0.001**
Category 2 (GIC + Forsterite) vs	Category 3 (GIC + Wollastonite)	26.46	p<0.001**
	Category 4 (GIC + Niobium)	14.58	p<0.001**
Category 3 (GIC + Wollastonite)	Category 4 (GIC + Niobium)	11.88	p<0.001**

CONCLUSION

Wollastonite nanoparticles incorporated to GIC showed the maximum increase in the apatite crystal formation followed by forsterite nanoparticles, niobium pentoxide nanoparticles and the control category i.e GC gold label 2 GIC on FEGSEM-EDX analysis. The results for the EDX analysis were similar and showed increase in the Ca and P content. Mean fluoride release was highest in wollastonite nanoparticles followed by niobium pentoxide nanoparticles, forsterite nanoparticles incorporated to GIC and least in the control category for day 1 and day 7. For day 28, mean fluoride release was highest in wollastonite nanoparticles followed by forsterite nanoparticles, niobium pentoxide nanoparticles, incorporated to GIC and least in the control category. The highest mean shear bond strength was observed in niobium pentoxide nanoparticles, followed by forsterite nanoparticles, and then by wollastonite nanoparticles integrated into GIC. Conversely, the control category exhibited the lowest level of activity. The findings from the stereomicroscope analysis revealed that the majority of fractures exhibited adhesive characteristics, suggesting that the measured values accurately represent the strength of the bonded interface. Mean compressive strength was highest in forsterite nanoparticles followed by niobium pentoxide nanoparticles, control category and wollastonite nanoparticles.

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