Original Study

COMPRESSIVE STRENGTH AND FLEXURAL STRENGTH OF TITANIUM NANO-ENRICHED GIC AT DIFFERENT PERCENTAGES AN IN VITRO STUDY

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ABSTRACT

Background: The use of nanoparticles in dentistry has become an important part of the studies. The main aim of this research was to measure the flexural and compressive strength of GIC (GC Universal Restorative Cement 2) compared to GIC (GC Universal Restorative Cement 2) containing titania nanoparticles at 3% and 5% (w/w).

Materials and Methods: This experimental in-vitro study was carried out in the Pakistan Institute of Engineering and Applied Sciences (PIEAS) and the duration of this study was two and a half months from 1st January 2019 to 15th March 2019. The powder of GIC (GC Universal Restorative Cement 2) was mixed with titania nanoparticles at 3 % and 5% (w/w). Group 1, Group 2, and Group 3 received 0%, 3%, and 5% titania, respectively. UTM (Universal Testing Machine) was used for compressive strength and flexural strength analysis. Cylinders of 9.5×1 mm were prepared for all three groups in a metal mold. A total of 18 samples for flexural strength and 18 samples for compressive strength were prepared. Surface analysis of all three experimental groups specimens, before and after flexural strength test were analyzed under SEM.

Results: The study showed significant improvement in flexural and compressive strength ($p<0.05$). SEM showed improved packing of particles in modified GIC at 3% and 5% (w/w) titania nanoparticles.

Conclusion: Considering the durability of titania containing GIC and the ability of this material to resist masticatory forces, the titania containing GIC is thought to be encouraging material for filling.

Key words: GIC, Titania nanoparticles, UTM .

Introduction

Dental caries is a significant health problem and the most chronic disease worldwide. Caries is an infectious disease that is distinguished by a multifactorial ethology and slow evolution that ultimately causes the destruction of hard dental tissues. Dental caries involves a continuous demineralization process, affecting the mineralization of dental tissues. It is the most common oral disease and the main reason for tooth loss in the population [1].

So, the most suitable treatment of dental caries with penetration into dentin is scrapping of demineralized and infected tooth tissue by the process of cavity preparation. After that, the removed dental hard tissues are restored with dental materials such as composite resin, ceramics, GIC, and amalgam [2].

The advantages of amalgam include its ease to handle and it is inexpensive. While mercury release leading to toxicity and poor esthetic are the major drawbacks of amalgam [3]. The resin composite shows good physical properties and is esthetic. But it is time-consuming, expensive, and sensitive [4, 5].

Hence, GIC can be used in various clinical processes because its physical properties can be changed by modifying the powder/liquid ratio or chemical materials. Moreover, GIC has anti-cariogenic properties due to the presence of fluorine and chemical adhesion to the tooth structure [6].

Actually, GIC is the most promising bioactive dental material that is formed from ion leachable fluoro-aluminosilicate glasses and polyacrylic acid solutions. These glass polyalkenoate cements have been successfully applied in dentistry for more than 30 years. A composite gel phase that is coated with unreacted glass particles is formed as a result of acid-base reaction [7].

GIC is considered as a vital tool in the fight against caries. GIC is a rich source of fluoride and other ions in the oral cavity. Fluoride is the mechanical barrier that basically shields the surface of the tooth against bacteria. Most importantly it can give a long-term seal under the most challenging clinical situations. In preventive dentistry, GIC can be used as a therapeutic coating. Also, for the management of the initial childhood rampant caries, the clinical application of the internal remineralization concept is very significant [8].

Nevertheless, GIC is avoided in the stress-bearing areas [9]. For posterior teeth filling, GIC is used as a temporary filling material. GIC is considered a bioactive dental material and Wilson and Kent first introduced them. To replace mercury-based amalgam in dentistry, a tooth-colored and biocompatible dental restorative material was needed. So, GIC was invented [8].

As a result of all of these materials having fillers, fluorides and copolymers such as polycarboxylic acid became available. The latest feature of these polyacrylate cements was their ion binding potential to the hydroxyapatite phase of dentin and enamel [5]. After sometime first practical GIC (ASPA) was introduced to the market in 1972 [10].

As the use of fluoride, in caries prevention, is very well known in dentistry. In glass ionomer cement, the acid part degrades the glass structure, bonds of the glass network hydrolyze and releases aluminum and calcium cations that are chelated by the carboxylate groups and serves to crosslink the polyacrylic chains. The degree of crosslinking mainly affects the properties of the resulting cement. Young's modulus of set cement is enhanced due to the high crosslinking [11].

Minor amounts of resins such as Bis-GMA and HEMA to liquid give beneficial physical properties. The addition of photopolymerizable resin to polyacid liquid component hardens the material on the application of visible light beam. After the resin composition has been cured, the glass ionomer setting reaction continues, protected from moisture and overdrying by the hard resin framework [12].

Since the development of GIC, numerous filler components have been added including Silver amalgam particles, spherical silica, zirconia, glass fibers, hydroxyapatite, and bioactive glass particles [13]. Nanoparticles reinforce dental resin composite and epoxy. Their addition to GIC improves its fracture toughness, compressive strength, flexural strength, and hardness [14]. Recently, it is reported that mechanical properties and bond strength to dentine are enhanced due to the incorporation of hydroxyapatite and fluorapatite nano bioceramics into conventional GIC [15].

Titanium dioxide (TiO_2) being an inorganic additive has many significant qualities as it is chemically stable, biocompatible, occurs abundantly in nature, cheap, less toxic, and antibacterial [16]. For use in dentistry, titanium can be found in various combinations. The composition of pure titanium is 99.5 titanium and 0.5% of interstitial elements such as carbon, oxygen, and nitrogen [17].

More importantly, the low density of titanium gives an opportunity to make a significantly light and resistant prosthesis. Besides all the advantages of titanium, its high melting point requires extremely special melting procedures, cooling cycles, and equipment to save it from contamination. Titanium can be thought out to be a resourceful, versatile, and adaptable material due to its combination of strength and lightness [17].

A study has been reported that the addition of titania at 3% and 5% (w/w) into GIC enhanced its mechanical properties [14]. Another study showed that the formulation containing TiO_2 nanoparticles was significantly stronger than those without TiO_2 nano particles ($p>0.01$). This study reported that ChemFil® Rock with no titania nanoparticles in it gave a compressive strength of 33.0 (9.9) MPa but after the addition of titania nano particles, the compressive strength was enhanced to 47.2 (5.3) MPa. Similarly, EQUIA™ Fil without titania nanoparticles gave a compressive strength of 32.3 (2.4) MPa and after the addition of titania nanoparticles in it showed a compressive strength of 42.1 (5.3) MPa [17].

Objective:

This study aimed to analyze the flexural and compressive strength of GIC containing titania nanoparticles at 3% and 5% (w/w) in comparison to GIC without titania nanoparticles addition.

Materials and Methodology:

Titania nanoparticles anatase phase were mixed with GIC (GC Universal Restorative Cement 2) at concentrations of 3% and 5% (w/w) in a mortar and pestle. Ethanol was used as a medium for the intimate mixing of nanoparticles with GIC (GC Universal Restorative Cement 2). Ethanol evaporated after some time leaving behind a mixture of GIC (GC Universal Restorative Cement 2) and titania nanoparticles. Three groups were prepared: Group 1 (no titania also referred to as control group), Group 2 (3% w/w titania), and Group 3 (5% w/w titania).

Flexural strength:

For flexural strength testing, samples of all three types of GIC were prepared by mixing GIC powder and liquid according to the manufacturer's recommended ratio on a glass slab using a mixing spatula. Firstly the mixture was poured into the cylindrical metal mold (9.5×1mm). Excess material was removed using a glass slide. After setting the GIC cylinder was removed from the metal mold. Each sample was measured using a digital vernier caliper. Before testing, 6 cylinders of group 1 (0% titania) were prepared and stored in a container labeled as 0% titania, 6 cylinders of group 2 (3% titania) were prepared and stored in a container labeled as 3% titania, and 6 cylinders were prepared of group 3 (5% titania) and stored in a container labeled as 5% titania. A total of 18 samples were prepared for flexural strength testing.

Compressive strength:

For compressive strength testing, samples were prepared by mixing GIC powder and liquid according to the manufacturer's recommended ratio on a glass slab using a mixing spatula. Firstly the mixture was poured into the cylindrical metal mold (9.5×1mm). Excess material was removed using a glass slide. After setting the GIC cylinder was removed from the metal mold. Each sample was measured using a digital vernier caliper. Before testing, 6 cylinders of group 1 (0% titania) were prepared and stored in a container labeled as 0% titania, 6 cylinders of group 2 (3% titania) were prepared and stored in a container labeled as 3% titania, and 6 cylinders were prepared of group 3 (5% titania) and stored in a container labeled as 5% titania. A total of 18 samples were prepared for compressive strength testing.

Flexural and compressive strength testing:

For flexural strength testing, the prepared samples were subjected to three points bending in the universal testing machine (SHENZHEN SANS TESTING MACHINE CO., LTD. CHINA). The sample was placed in a cylinder with an opening diameter of 10 mm and the load was applied with a gradual increase at the cross speed of 1 mm/min (Mpa). When the sample broke, the load application was stopped and all details were measured. This process was repeated for all 6 samples of each group and the results were obtained.

For compressive strength testing, the prepared samples were subjected to the compressive load using two flat metal disks in a universal testing machine (SHENZHEN SANS TESTING MACHINE CO., LTD. CHINA) at a cross speed of 1 mm/min(Mpa). The load was applied until the sample broke and all the details were measured. All 6 samples of each group were tested and the results were obtained.

One-way ANOVA analysis was used to compare the mean differences in flexural strength, compressive strength between the three groups. Post-hoc Turkey analysis was done to compare inter-group mean differences. An arbitrary significance value of 0.05 was thought to be significant. All data were entered and analyzed using SPSS v.23.0. The study showed significant improvement in the flexural and compressive strength ($p<0.05$).

Results:

Flexural strength Analysis:

Specimens with 5% Titania had the greatest flexural strength [22.34 (0.32); Table 1). The intergroup comparisons have been illustrated in Table 2 and Graph 1.

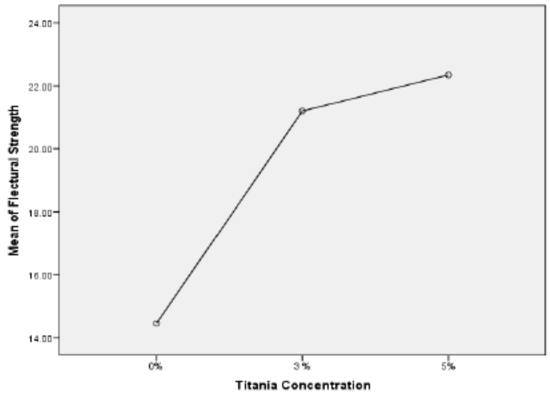
Titania Concentration	Mean (Standard Deviation)
0%	14.46 (1.25)
3%	21.20 (0.15)
5%	22.34 (0.32)
Total	19.33 (3.64)

Table 1: Mean and standard deviation for Flexural Strength (N = 18)

The flexural strength for 0%-Titania group was significantly less than both the 3% (p = 0.000) and 5% (p = 0.000) groups. The 3%-Titania samples were also found to be significantly less than the 5% samples (p = 0.04).

Titania Concentration	Comparison Group Concentration	Mean Difference (SE)	P-Value
0%	**3%**	**-6.74 (0.43)**	**0.000**
	5%	**-7.88 (0.43)**	**0.000**
3%	**5%**	**-1.14 (0.43)**	**0.04**

Table 2: Intergroup Mean Differences Analysis for Flexural Strength; SE = Standard Error



Graph 1: flexural strength plot for different titania concentrations.

Compressive Strength Analysis:

The samples with 5% Titania had the greatest compressive strength [7.40 (1.26); Table 3]. The intergroup comparisons have been illustrated in Table 4 and Graph 2.

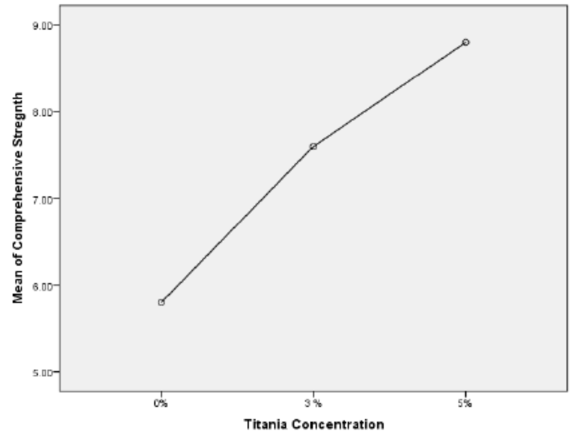
Titania Concentration	Mean (Standard Deviation)
0%	5.80 (0.05)
3%	7.60 (0.07)
5%	8.80 (0.03)
Total	7.40 (1.26)

Table 3: Mean and standard deviation for Compressive Strength (N = 18)

The compressive strength for 0% Titania group was found to be significantly lower than the groups containing 3% and 5% titania (p=0.000). The compressive strength of the group containing 3% titania was also found to be significantly lower when compared to 5% titania containing group (p=0.000) as shown in Table 4.

Titania Concentration	Comparison Group Concentration	Mean Difference (SE)	P-Value
0%	**3%**	**-1.80 (0.03)**	**0.000**
	5%	**-3.00 (0.03)**	**0.000**
3%	**5%**	**-1.20 (0.03)**	**0.000**

Table 4: Intergroup Mean Differences Analysis for Compressive Strength; SE = Standard Error



Graph 2: Compressive strength plot for different titania concentrations

Discussion:

In dentistry, compressive and flexural strength tests are used for laboratory simulation of the stresses that may be associated with the loads applied clinically to a restorative material. The accurate value of masticatory forces is not known but almost all mastication forces are compressive in nature. Hence, it is of extreme importance to conduct an investigation about the role of compressive forces in fracture failure during the mastication process. The lowest values required to withstand the mastication forces in primary and posterior teeth are 100 and 125 MPa, respectively [18].

According to a previous study, adding 3% (w/w) titania nanoparticles into glass ionomer cement improved its mechanical properties to some extent. But when the concentration of titania nanoparticles in GIC was increased to 5% (w/w) the resultant material showed excellent compressive strength properties. Compressive strength increased with an increase in the concentration of titania nanoparticle showing a direct relationship [19]. Prosser et al have mentioned that the most suitable method to measure the strength of GIC is a flexural strength test.

According to this study, with an increase in the concentration of titania nanoparticles into GI powder the flexural strength has been increased. Samples of 5% (w/w) titania nanoparticles showed significantly higher flexural strength 22.34 (0.32) than both of the other groups. 3% (w/w) titania nanoparticles containing group showed improved results 21.20 (0.15) when compared with 0% (w/w) titania nanoparticles-containing group.

The group containing 0% titania nanoparticles showed a significantly lower flexural strength value of 14.46 (1.25) MPa. 0% (w/w) titania nanoparticles containing GIC showed a mean difference of -6.74 (0.43) MPa when compared with 3% (w/w) titania nanoparticles containing GIC and -7.88 (0.43) when compared with 5% (w/w) titania nanoparticles containing GIC. The comparison of 3% (w/w) titania nanoparticles containing GIC with 5% (w/w) titania nanoparticles containing GIC showed a mean difference of -1.14 (0.43).

This study also showed that samples containing 0% (w/w) titania nanoparticles have significantly lower compressive strength 5.80 (0.05) when compared to samples containing 5% (w/w) titania nanoparticles (p=0.000). The group containing 3% (w/w) titania nanoparticles in GIC showed a lower compressive strength of 7.60 (0.07) in comparison to the group containing 5% (w/w) titania nanoparticles in GIC, which was 8.80 (0.03).

There was a difference observed in the compressive strength between groups containing 0% (w/w) titania nanoparticles and 3% (w/w) titania nanoparticles. 0% (w/w) titania nanoparticles containing GIC showed a mean difference of -1.80 (0.03) when compared to 3% (w/w) titania containing GIC and showed a mean difference of -3.00 (0.03) when compared to 5% (w/w) titania nanoparticles containing GIC.

Mechanical and bond strength properties are affected by the Powder/Liquid ratio of GIC. Factors such as integrity of the interface between the glass particles and the polymer matrix, particle size, number of voids, and size of voids play an important role in determining mechanical properties. GIC microstructure determines its mechanical properties. High flexural strength was closely related to integrated microstructure while the compressive strength was related to smaller glass particles [9].

GIC resistance to fracture is very low and the reason behind that is the presence of pores in the cement matrix. The entrapment of air during the mixing process of GIC gives rise to these pores. These voids in cement matrices look like air bubbles. The presence of these porosities explains a reason for the low compressive strength of GIC [20].

According to a study, GIC containing titania nanoparticles showed higher compressive strengths and improved packing of particles within the cement matrix. The incorporation of titania nanoparticles in GIC reduces porosity. The weakening of glass particles and matrix interface leads to internal defects and brittleness of cement ultimately resulting in poor mechanical properties of GIC [19].

When acid is mixed with GIC powder containing metallic nanoparticles, the metal ions are released. These released metal ions act as a cross-linking species resulting in a stable cement. As mentioned in the previous study that low strength values can be attributed to metal ions that cannot leach out to form cross-linking [19].

Enhancement in flexural strength of the GIC was notable at the concentration of 3% (w/w) and 5% (w/w) titania nanoparticles. This improvement in flexural strength can be awarded to the small size of titania nanoparticles combined with glass particles of GIC powder. The resultant product had a broad range of particle sizes showing markedly increased mechanical properties [21].

Scanning Electron Microscope Analysis:

SEM analysis of before and after flexural strength analysis of all three experimental groups (0%, 3%, and 5%) containing titania are shown in figure 1 (a,b), 2 (a,b), and 3 (a,b). Images showed intimate contact between particles of 3% and 5% titania containing groups when compared with 0% titania containing group. Images of 0% titania containing group specimens showed the highest crack lines indicating the lowest strength and microhardness.

Images of 3% titania containing group specimens show moderate crack lines indicating high strength compared to 0% titania containing group. While images of 5% titania containing group show minimum crack lines indicating the highest strength among all three experimental groups. Spaces between particles of Group 2 and Group 3 were occupied by Titania nanoparticles.

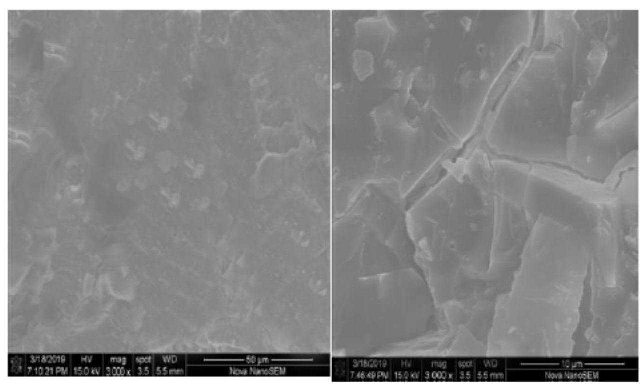


Figure 1a- GIC sample containing 0% Titania before flexural strength testing. Figure 1b- GIC sample containing 0% Titania before flexural strength testing

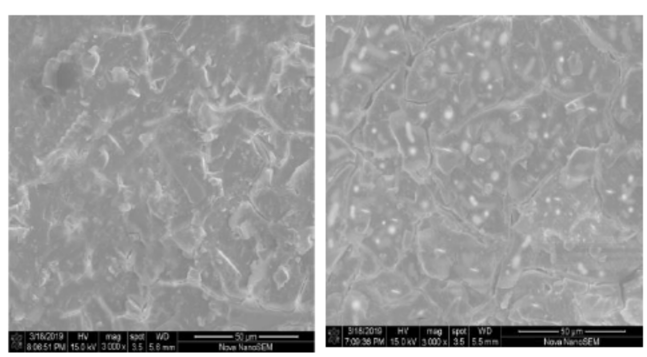


Figure 2a- GIC sample containing 3% Titania before *flexural strength testing. Figure 2b- GIC sample containing 3% Titania before flexural strength testing*

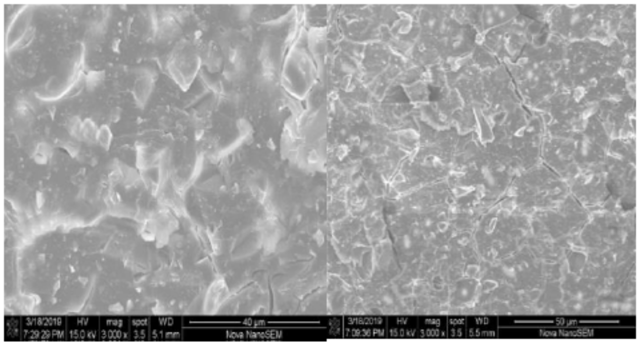


Figure 3a- GIC sample containing 5% Titania before flexural strength testing. Figure 3b- GIC sample containing 5% Titania before flexural strength testing.

Conclusion:

Considering the durability of titania containing GIC and the ability of this material to resist masticatory forces, the titania containing GIC is thought to be an encouraging material for filling.

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