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# Effect of adding nano titanium dioxide and chitosan on compressive strength of glass ionomer cement

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**Abstract**

**OBJECTIVE:**

To evaluate the compressive strength of glass ionomer cement incorporating nano-titanium dioxide as well as chitosan.

**MATERIALAND METHODS:**

The samples were prepared in six groups, including non-modifed GIGs (NMGIC, n = 12), chitosan incorporated GICs (CHGIC, n = 12),3% nanotitanium incorporated GICs (nano$Tio_2$GIC, n = 12), 5% nanotitanium incorporated GICs (nano$Tio_2$GIC, n = 12), chitosan/3% nanotitanium incorporated GICs (CH/nano$Tio_2$/GICs, n = 12),chitosan/5% nanotitanium incorporated GICs (CH/nano$Tio_2$/GICs, n = 12) .The compressive strength was evaluated using a universal testing machine. Data were analyzed using the ANOVA and Tukey tests. Statistical significance was set at the 0.05 probability level.

**RESULTS:**

With the dual-modification, a significant improvement in thecompressive strength was found.

**CONCLUSION:**

Under the limitations of the present investigation, the following conclusion can be drawn:5% wt $TiO_2$ NP modified GIC powder with the chitosan modified GIC liquid **(Group 5)** exhibited the highestcompressive strength.

## I. Introduction:

Conventionally, glass ionomer cements ( GICs) consist of two main components: a powder of fluoro-aluminosilicate glass and aqueous solution of a mixture of organic acids . Polyacrylic acid is the main constituent of the aqueous component. Less viscous polyacids such as maleic and itaconic acids may also be present in the solution to improve manipulation .[1, 2]

Tartaric acid is usually added to the liquid component to act as a chelating agent, which allows setting reaction time control and improve the handling properties. Conventional glass ionomer cements set via an acid-base reaction between the aqueous solution of polyacrylic acid and fluoro-aluminosilicate glass particles .[3, 4]

Glass ionomer cement (GIC) possesses certain properties of chemical adhesion to tooth structure ,white color ,low coefficient of thermal expansion similar to the tooth structure, biocompatibility, and fluoride releasingand their protective properties against tooth decay , which have led to beused for a wide range of applications in dentistry as luting , base , liners and restorative materials.[5, 6]

GICs also has a number of disadvantages such as brittleness , low wear resistance, inappropriate surface properties , low tensile and flexural strengths as well as high early moisture sensitivity. These drawbackswere limited its use for many clinical cases. So, many modifications have been applied to the conventional GICs to overcome the poor mechanical properties.[7-9]

Nanotechnology involves the use of systems, modifications or materials ranging of 1 – 100 nm. Several ways have been attempted to improve the physical and mechanical properties of GIC using nano sized materials made by nano technology.[10]

Titanium dioxide ($TiO_2$) is an inorganic additive which has some advantages such as its chemically stability, nontoxicity and biocompatibility. A recent previous studies has attempted to incorporate $TiO_2$ nanoparticles (NP) in the powder component of GICs resulting in significant improvement in the physical and mechanical properties.[11, 12]

The effectiveness of the antibacterial effect of conventional GICs against Streptococcusmutans, whichhave a major role in the formation of tooth decay, remains questionable and required.Alteration of GICs with different antibacterial agents is therefore important if it is not associated with adverse effects on physical or mechanical properties.[13, 14]

Chitosan (CH) is derived from chitin by deacetylation. It is a weak base, insoluble in water and organic solvents, but soluble in dilute aqueous acidic solution. It is a cationic, non-toxic, biodegradable, biocompatible and have various potential biological effects such as antibacterial and antifungal properties.[15, 16]

Chitosan displays a wide range of antibacterial activity against Gram-positive and Gram-negative bacteria. Liquid phase modification of GICs with chitosan was reported previously with significant improvement in antibacterial properties at optimum chitosan concentration of 10% (v/v).[17, 18]

Glass ionomer cement (GIC) dually modified with chitosan (CH) in the liquid phase and titanium dioxide nanoparticles (TiO2/NP) in the powder phase will be made to investigate both mechanical and antibacterial properties for dental restorative applications.

## II. Material and methods:

The materials used in this study along with pertinent information (specification, manufacturers and batch numbers) are listed in Table (1)

| **Material** | **Specification** | **Manufacturer** | **Batch no.** |
|---|---|---|---|
| **Medifil** | Glass ionomer filling cement containing:<br>Al-Ca-La fluorosilicate glass and polyalkenoic acid, tartaric acid | Domagkstrasse 31 24537 Neumuenster Germany | 1924439 |

| | | | |
|---|---|---|---|
| **Titanium dioxide(NP)** | Mixture of rutile and anatasenanopowder<100 nm particle size<br>MW:79.87 | Sigma-Aldrich, St. Louis, MO, USA | 634662 |
| **Chitosan** | - $C_6H_{11}NO_4X_2$<br>- Degree of Deacetylation: Min. 90% | Oxford Lab Fin CHEM LLP,India | 4464 |

**Preparation of Chitosan Solution:**
0.3N acetic acid was used as a solvent for chitosan. 1.8 ml ofglacial acetic acid was made up to 100 ml with distilled water in a 100 ml standard flask to get 0.3N acetic acid. 20 mg of Chitosan were weighedseparately and dissolved in 0.3 N acetic acid and made upto 100 ml with the same acetic acid in a 100 ml standardflask to get 0.2mg / ml Chitosan Solution. 0.1ml of 0.2mg/ml of Chitosan solution wasadded to 0.9ml of GIC liquid to get 10 v/v%Chitosan modified glass ionomer solution .(19)

**Preparation of Titanium Dioxide Nanoparticles Modified GIC:**
$TiO_2$ nanotubes were weighed using a laboratory scale (Analytical balances KERN ABJ 220-4NM, KERN & SOHN GmbH, Balingen – Germany) (with weight range from 220g to 0.1mg) and adjusted to zero, then was added to the GIC's component powder prior to hand mix manipulation. Each GIC was blended with 3% or 5% (w/w) $TiO_2$ nanoparticles of rutile and anatasenanopowder<100 nm particle size (Sigma-Aldrich, St. Louis, MO,USA). GIC powder and TiO2 NPs were mixed for one minute with vortex mixer(VM-300 Vortex Mixer, power: 220V / 50 Hz, Gemmy industrial corp., Taiwan) to obtain the most uniform distribution possible of the nanoparticles into GIC powder.(11)

**Compressive strength (CS) testing:**
.A specially designed split mold was fabricated to form cylindrical specimens with dimensions of (4 mm in diameter x 6 mm in height).These dimensions were determined according to the recommendation of international standards organization ISO NO.9917 (2007) .Each group was mixed with GIC liquid on a glass slab using plastic cement spatula. The mixed cement was condensed in the metallic mold which was placed on glass plate. Specimens were covered with celluloid strip and pressed with another glass plate. Specimens were removed from the mold after setting and stored in distilled water at 37 °C for 24 hours prior testing.
Specimens were compressed using a universal testing machine. Each sample diameter was measured three times and an average taken. A crosshead speed of 0.5 mm/min was used with a load cell of 10 kN and the samples compressed to failure.
The maximum force of each sample was recorded and used to calculate the CS (in MPa) using Equation:(20)
CS= 4 F/π r2
Where
F is the force applied at fracture,
π is the mathematical constant equal to 3.14 and
r is the average radius calculated from three measurements.

## III. Statistical Analysis:
One-way ANOVA tests were used to compare compressive strength of the different GIC groups. For all analyses, F - test was used for pair wise mean comparison among the tested groups. Calculations were handled by the software PASW Statistics 17 (SPSS Inc., Chicago, IL, USA), and all of the tests' accuracy was set at a significance level of 0.05.

## IV. Results:

**Compressive strength:**

The results of statistical analysis showed that; 5% wt $TiO_2$ NP modified GIC powder with the chitosan modified GIC liquid **(Group 5)** exhibited the highestcompressive strength. While control group; unmodified glass ionomer cement **(Group 1)** recorded the lowest compressive strength means value. Pair-wise comparisons among the groups revealed that; all groups were statistically significant difference as shown in table (2)

**Table (2): Compressive strength measurements results (Means ± SDs) for allinvestigated groups**

| Compressive strength | G1 | G2 | G3 | G4 | G5 | G6 |
|---|---|---|---|---|---|---|
| Range | 199.9 – 210.2 | 220.1 – 223.4 | 229.1 – 234.5 | 239.1 – 244.7 | 245.1 – 249.3 | 210.1 – 219.3 |
| Mean ± SD | 204.11 ± 3.74 | 221.46 ± 1.29 | 231.48 ± 1.63 | 241.71 ± 1.76 | 246.93 ± 1.58 | 214.62 ± 3.50 |
| F test | 84.360 | | | | | |
| P value | 0.001* | | | | | |

| P1 | P2 | P3 | P4 | P5 | P6 | P7 | P8 | P9 | P10 | P11 | P12 | P13 | P14 | P15 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * | 0.001 * |

Where (*) significant ($p<0.05$) , ns; non-significant ($p>0.05$)

## V. Discussion:

Glass ionomer cement was invented decades ago by Wilson and Kent in 1969. These materials form part of contemporary restorative dentistry largely due to their ability to chemically bond to tooth structure with insignificant heat formation or shrinkage, biocompatibility with pulp and periodontal tissues and fluoride releasing properties . They are used today in a variety of clinical situations such as restorative, lining, basing, luting and sealing materials.[(21)]

Various modifications and the developments of glass powder and polymer liquid have been introduced to improve its antimicrobial effect in turn mechanical and physical properties of GICs. The nature of setting reaction of conventional glass ionomer is an acid base reaction between the acidic polyectrolyte and the aluminosilicate glass.[(22)]

Two concentration of titanium dioxide were selected to be studied after mixing with powder of glass ionomer cement at 3% and 5% by weight for efficiency and their effect on the mechanical and physical properties.[(11)]

Chitosan, a widely used natural biocompatible linear biopolyaminosaccharide, has proven its potent antibacterial effect against oral biofilms, specifically S. mutans, thus paving the way for its use as a preventive and therapeutic agent to control dental caries and to effect on mechanical properties. [(18)]

Chitosan can be considered a strong base as it possesses primary free amino groups (NH3+) when dissolved with polyacrylic acid. The reaction had taken place between amino (-NH2) group of CH and the functional group (OH group and C=O group) of GIC. Since CH possess hydroxyl and acetamide groups, they bind to hydroxyl group of powder particles and carboxylic groups of poly acrylic acid by hydrogen bonding .[(23)]

Titanium dioxide ($TiO_2$) is an inorganic additive which has some advantages such as its chemically stability and biocompatibility , non toxicity, and improvement of mechanical properties in composites and hybrid materialswhere they be used on assessing its effect on GICs' antibacterial, physical and mechanical performance.[(24)]

The compressive strength (CS) is an important property in restorative materials, particularly in the process of mastication. To test compressive strength of a material, two axial sets of force are applied to a sample in an opposite direction, in order to approximate the molecular structure of the material.[25]

Modification of the liquid phase of GIC with 10% v/v CH solution improved compressive strength where chitosan chains carry many hydroxyl groups and acetamide groups which are able to bind to hydroxyl groups of the GIC particles and to polyacrylic acid (PAA) carboxyl groups by hydrogen bonding. The network formed by CH and PAA around the inorganic GIC particles might reduce the interfacial tension among the GIC components, improving the mechanical performance.[26]

The result of this study showed also agreed with the result of some authors[26]where modifying the liquid phase of conventional GIC with 10% v/v CH increased the compressive strength values when compared to non-modified GIC.

The addition of $TiO_2$ nanoparticles increased the compressive strength values with the increasing the addition ratio till 5 wt. %. The reason for increased these strength values with increased the ratios of added TiO2 due to the surface of $TiO_2$ has rich hydroxyl groups and it covalently bonded with GIC matrix.[27]

Also, the modifided glass ionomer powder mixed with $TiO_2$ filler had a wider range of particle-size distributions, and the $TiO_2$ nanoparticles fill the spaces between GIC macromolecules. This nanoparticles may have served as a reinforcement agent, which may improve mechanical properties .[11,28]

This is supported by the studies of some researchers [11,20] which concluded that the addition of 3 wt.% , 5 wt.% $TiO_2$ NP to the powder phase of GICs resulted in improvement of the strength with the increasing the addition ratio till 5 wt. % .

The dual modification of GIC powder with $TiO_2$ NP and the GIC liquid with chitosan as with **(Group 4&5)** resulted in significant enhancement in the compressive strength of the GICs compared to all groups. This could be explained, mechanically, by the combined effect of $TiO_2$ NP which act as additional inorganic fillers reinforcing the GIC matrix and the chitosan which form multiple hydrogen bonds holding the GIC glass particles and matrix together.[29]

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