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EFFECT OF ADDING NANOTITANIUM DIOXIDE AND CHITOSAN ON FLEXURAL STRENGTH, DIAMETRAL TENSILE STRENGTH, AND HARDNESS OF GLASS IONOMER CEMENT

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ABSTRACT

Objective: To determine the flexural strength, diametral tensile strength, and hardness of a glass ionomer cement containing nano-titanium dioxide and chitosan. **Material and Methods:** This study analyzed a total of 216 samples. The samples were classified into six major categories: Group 1 (non-modified GICs as a control group), Group 2 (3 wt% nanotitanium incorporated GIC powder), Group 3 (5 wt% nanotitanium incorporated GIC powder), Group 4 (3 wt% nanotitanium incorporated GIC powder and chitosan 10 v/v% incorporated GIC liquid), Group 5 (5 wt% nanotitanium incorporated GIC powder and chitosan 10 v/v% incorporated GIC liquid) and Group 6 (10 v/v% chitosan modified GIC liquid without any modification in the powder). According to the testing method, each main group was broken into three subgroups (n= 12). Flexural strength, diametral tensile strength, and hardness of modified glass ionomer cement were determined. The data were analyzed using one-way analysis of variance (ANOVA) and Tukey's analysis. At a level of probability of 0.05, statistical significance is achieved. **Results:** Group 5 exhibited the highest statistically significant flexural and diametral tensile strength means values, while the control group (Group 1) recorded the lowest strength mean values. However, the results revealed that (Group 3) showed the highest statistically significant surface hardness mean value compared to the dual modification of GIC, non-modified GIC, and the chitosan-modified GIC**. Conclusions:** The addition of TiO_2 nanoparticles or chitosan to GIC boosts its flexural and diametral tensile strengths, and the optimum improvement will be obtained with the dual modification. The incorporation of TiO_2 NPs will increase the surface hardness of GIC, while chitosan incorporation will decrease the surface hardness of the material.

KEYWORDS: Glass ionomer, nanotitanium dioxide, chitosan

INTRODUCTION

Formerly, glass ionomer cements (GICs) were composed of two major components: an aqueous solution containing a mixture of organic acids and fluoro aluminosilicate glass powder. The major component of the water-based component is polyacrylic acid. To facilitate handling, solutions containing lower viscosity polyacids, including itaconic acid and maleic acid, may be administered [1, 2].

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Tartaric acid is commonly used as a chelating agent in liquid components, which helps to reduce reaction time and increase processing performance. Traditional glass ionomer cements are cured by the acid-base reaction between the fluoroaluminosilicate glass particles and the polyacrylic acid aqueous solution [3, 4].

Glass ionomer cement chemically attaches to tooth surfaces, is white in color, has a thermal expansion's low coefficient equivalent to the tooth structure, is biocompatible, and possesses fluoride-releasing capabilities in addition to its anti-cavity characteristics. It is a very versatile material that is used in a range of dental applications, including restorative materials, luting, bases, and liners [5, 6].

Brittleness, limited abrasion resistance, insufficient surface characteristics, low tensile and flexural strengths, and strong early moisture sensitivity are all downsides of GICs. Because of these flaws, it cannot be used in a lot of clinical situations. As a result, various modifications to the traditional GIC have been made to compensate for its weak mechanical performance [7-9].

Nanotechnology utilizes systems, changes, or materials with a diameter of 1 to 100 nanometers. Using nanoparticles created by nanotechnology, Numerous strategies have improved GIC's physical and mechanical characteristics [10].

Titanium dioxide (TiO_2) is an inorganic additive with chemical stability, non-toxicity, and biocompatibility, among its benefits. The latest research aimed to enhance the GIC's mechanical and physical characteristics by adding TiO_2 nanoparticles (TiO_2/NP) to the powder component [11, 12].

The antibacterial impact of conventional GIC on Streptococcus mutans is still debatable, and more research is needed. Streptococcus mutans is a pathogen that causes dental caries. As a result, it is critical to modify the GIC with a different antimicrobial agent if it does not have a negative impact on the physical or mechanical qualities [13, 14].

Deacetylation is used to generate chitosan (CH) from chitin. It is a water-insoluble weak basic that dissolves in dilute acidic aqueous solutions but not in water or organic solvents. It is cationic, biodegradable, non-toxic, and biocompatible, and has several possible biological effects, among which are antibacterial and antifungal [15, 16].

Chitosan possesses antibacterial characteristics that work equally for gram-negative and gram-positive bacteria. At ideally 10% chitosan (v/v) concentration, liquid phase modification of GIC with chitosan has been shown to improve antibacterial activity considerably [17, 18].

To investigate the effects on mechanical properties for therapeutic applications, Glass ionomer cement (GIC) has been double altered in the liquid phase using chitosan (CH) and powder nanoparticles with titanium dioxide (TiO_2/NP).

MATERIAL AND METHODS

The materials for this investigation were; Glass ionomer filling material (Medifil, PROMEDICA Dental Material, Germany), TiO_2 nanoparticles with two concentrations 3wt% and 5 wt% (Sigma-Aldrich, Inc., USA), and chitosan powder with concentration 10 v/v% (Oxford Lab. FINE CHEM LLP, India).

Grouping of samples:

This study evaluated a total of 216 samples. The samples were classified into six major groups (n=36) based on their alterations:

Group 1: Glass ionomer cement without any additives in the powder and liquid (control group).

Group 2: Powdered glass ionomer cement with a content of 3% by weight TiO_2 NP , liquid GIC without any additives.

Group 3: Powdered glass ionomer cement with a 5% TiO2 NP weight percentage and no existing additives in GIC liquid.

Group 4: Glass ionomer cement powder with a TiO_2 NP content of 3% by weight and a chitosan solution in GIC liquid at a concentration of 10% (v/v).

Group 5: Glass ionomer cement powder with the addition of 5% wt TiO_2 NP and addition of chitosan solution in GIC liquid at 10%(v/v).

Group6: Glass ionomer cement powder without any additives and addition of chitosan solution in GIC liquid at 10%(v/v).

Each main group was then divided into three subgroups (n= 12) based on the type of test used: flexural strength, diametral tensile strength, and surface hardness.

Preparation of chitosan modified GIC liquid:

Chitosan was dissolved in acetic acid (0.3N) to create 0.3N acetic acid, the volume of 1.8ml glacial acetic acid in a normal 100ml flask was increased to 100ml using distilled water. 20 mg chitosan, taken separately and dissolved in 0.3N acetic acid, was introduced to a 100 ml standard flask containing the same acetic acid to generate a chitosan solution containing 0.2 mg/ml chitosan. To make a 10% chitosan modified glass ionomer cement liquid, 0.1 ml of 0.2 mg/ml chitosan sample was added to 0.9 ml of GIC liquid [19].

Preparation of titanium dioxide nanoparticles modified GIC powder:

TiO_2 nanotubes were weighed using a laboratory scale (Analytical balances KERN ABJ 220-4NM, KERN & SOHN GmbH, Balingen – Germany), adjusted to zero, and then added to the GIC powder according to the required concentrations 3% and 5% (w/w). Each GIC powder modified with TiO_2 NP (3% and 5% (w/w) was mixed for one minute with a vortex mixer (VM-300 Vortex Mixer, power: 220V / 50 Hz, Gemmy industrial corp., Taiwan) to achieve the most homogenous distribution of TiO2 nanoparticles feasible in GIC powder [11].

Flexural strength (FS) testing:

Each group's samples were made by mixing the respective GIC powder with the associated liquid per the manufacturer's guidelines. The mixed GIC material from each group was condensed in split molds (25mm long x 2mm thick x 2mm wide). GIC was poured into the mold and covered with a glass slide until the initial setting occurred. Following solidification, prior to the experiment, specimens (n=12 per group) were stored in distilled water at 37°C for 24 hours.

Three-point bending tests were conducted using universal test equipment (Instron 3365 universal testing machine, UK). Each sample was adjusted on a bending attachment comprised of two parallel supports 20mm apart and loaded using a thin rod placed centrally between the two supports at a cross-head speed of 0.5 mm/min (MPa). To determine the flexural strength (MPa), the continuity formula was utilized: [11]

$$O' = 3PL/2bd^2$$

Where O' stands for flexural strength (MPa), P stands for fracture load (N), L stands for the distance between two endorses (mm), b stands for specimen width (mm), and d stands for specimen thickness (mm) (mm).

Diametral tensile strength (DTS) testing:

Each group's GIC samples (n=12) were made as described before for the flexural strength test (FS), but with the addition of a split mold (6 mm diameter x 4 mm height). (DTS) presses the specimens diagonally to ascertain the material's tensile strength[20].

Samples were ground to failure across the diameter using an Instron test machine with a 0.5 mm/min cross-head speed. The maximum force applied at fracture for each sample was recorded and used to calculate the (DTS) in (MPa) according to this equation: [21].

$$DTS = 2\,Fmax/\pi dh$$

Where Fmax is the greatest force exerted at fracture (N), (d) is the sample's diameter (mm), (h) is the sample's height (mm), and π its standard is equal to 3.14.

Surface hardness testing:

The GIC samples (n = 12 per group) were prepared as previously described in the flexural strength test (FS), but by using a split mold (diameter 6mm x height 4mm).

The surface microhardness was determined using a Vickers diamond indenter and a 20X objective lens on a Vickers Microhardness Tester with a digital display (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd., China).

For ten seconds, a load of 200 g is applied to the sample's surface. Three indentations were made on the surface of each sample and equally placed on a circle, and the distance between the adjacent indentations was not less than 0.5 mm. The notch's diagonal length was determined using a built-in scale microscope.

The Vickers hardness number (VHN) was determined using the equation below:

$VHN = 1.8544 \times P/d^2$

Where P denotes the applied force in kilograms and d denotes the diagonal length (mm) [22].

In each sample, three indentations were made, and the average of all measurements was used for statistical purposes. Any indentation produced in the pores or defects of the cement surface was rejected, and the test was repeated.

Statistical Analysis:

The antibacterial activity, water sorption, and solubility of several GIC groups were compared using a one-way analysis of variance (ANOVA). The F test is utilized to compare paired means between test groups in all analyses. The computation is carried out using the software PASW Statistics 17 (SPSS Inc., Chicago, IL, USA), with all reach a certain threshold to an accuracy of 0.05.

RESULTS

Flexural strength:

The statistical analysis results showed that; **(Group 5)** showed the highest statistically significant flexural strength mean value (12.33±0.32 MPa). While; control group; **(Group 1)** recorded the lowest flexural strength mean value (6.81±0.40 MPa) followed by **(Group 6)** (8.07±0.25 MPa) followed by **(Group 2)** (9.13±0.37 MPa) followed by **(Group 3)** (10.19±0.21 MPa) followed by **(Group 4)** (11.13±0.29 MPa).

Comparing the groups pairwise demonstrated that all groups had a substantial difference, as seen in Figure (1)

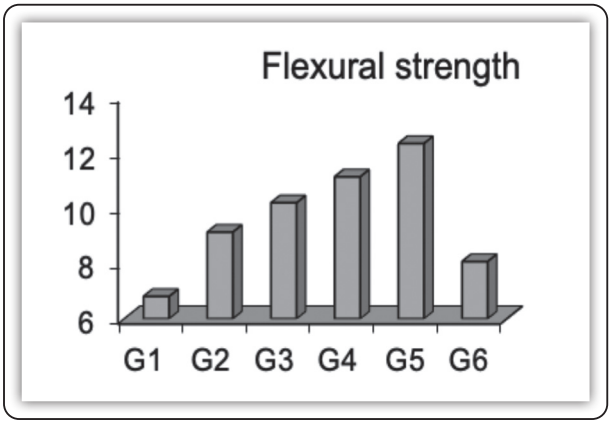

FIG (1) Column chart representing means of flexural strength for all groups

Diametral tensile strength:

The statistical analysis outcomes showed that; **(Group 5)** showed the highest statistically significant diametral tensile strength mean value (42.73±1.75 MPa). While; control group; **(Group 1)** recorded the lowest diametral tensile strength mean value (15.16±1.48 MPa) followed by **(Group 6)** (20.73±1.70 MPa) followed by **(Group 2)** (26.34±2.14 MPa) followed by **(Group 3)** (32.71±1.44 MPa) followed by **(Group 4)** (37.64±2.10 MPa).

Comparing the groups pairwise demonstrated that there was a statistically significant difference between all groups, as illustrated in Figure (2)

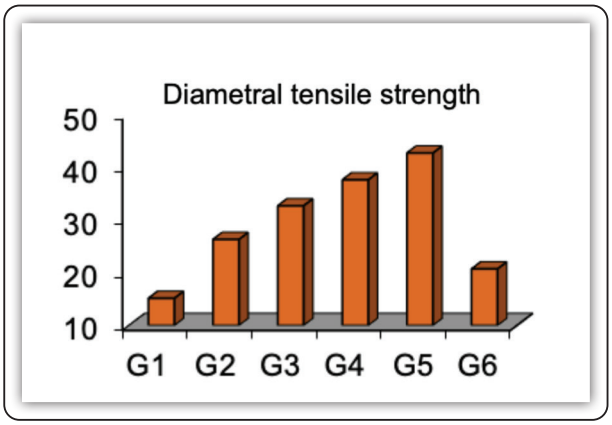

FIG (2) Column chart representing means of diametral tensile strength for all groups

Surface hardness:

The findings of statistical analysis showed that; **(Group 3)** showed the highest statistically significant surface hardness mean value (132.51±2.43 VHN). While; **(Group 6)** recorded the lowest surface hardness mean value (82.11±1.35 VHN) followed by **(Group 4)** (91.89±2.26 VHN) followed by **(Group 5)** (98.97±2.81VHN) followed by control group; **(Group 1)**(110.00±2.94 VHN) followed by **(Group 2)** (122.23±3.27 VHN).

Comparing the groups pairwise demonstrated that there was a statistically significant difference between all groups, as illustrated in Figure (3)

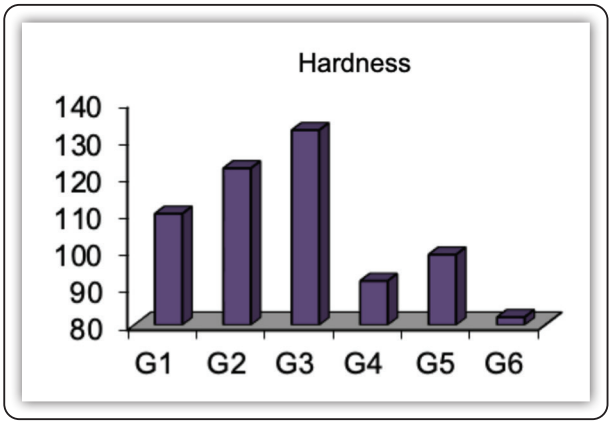

FIG (3) Column chart representing means of surface hardness for all groups

DISCUSSION

The focus of this thesis was to identify the effects of Chitosan and titanium dioxide nanoparticles on a variety of properties of glass ionomer cement (surface hardness, flexural strength, and diametral tensile strength).

Wilson and Kent invented glass ionomer cements (GICs) in 1969, decades ago. Due to their chemical attachment to the tooth structure without considerable shrinkage, biocompatibility, and fluoride-releasing characteristics, they are used in a variety of clinical conditions today, including restorative, base, luting, and sealing materials [23].

However, (GICs) have fundamental limitations in usage as restorative materials, including their poor mechanical qualities, including low fracture toughness, low tensile strength, and low wear resistance, as do all materials. GICs are therefore unsuitable for application in high-stress situations[1].

To improve the mechanical features of GICs, numerous modifications and advancements to the glass powder and polymer liquid have been made[24].

Chitosan and titanium dioxide nanoparticles were utilized as chemical additives to alter and enhance the varied characteristics of glass ionomer cement. A concentration of 10% v/v percent was used in this study. Chitosan-modified glass ionomer liquid was utilized since previous tests demonstrated that it possessed enhanced properties [24, 25].

Two concentrations of titanium dioxide were selected in this study after mixing with powder of glass ionomer cement. 3 wt% and 5 wt% were used depending upon optimum properties obtained by these concentrations in the previous study [11].

Chitosan is a biocompatible, natural linear biopolyaminosaccharide. It has been demonstrated to have a high antibacterial effect on oral biofilms, allowing it to be used to prevent tooth cavities and improve mechanical properties [18].

Titanium dioxide (TiO_2) is an inorganic additive with chemical stability, non-toxicity, biocompatibility, and increased mechanical qualities in composite and mixed materials, where it is utilized to examine its effect on GIC mechanical performance [26].

The addition of a 10% v/v CH solution to the liquid phase of GIC improved the strengths in regions where the chitosan chains contain multiple hydroxyls and acetamide groups potential for hydrogen bonding with the hydroxyl groups of GIC particles and the carboxyl groups of polyacrylic acid (PAA). The CH and PAA network that forms around inorganic GIC particles may contribute to the reduction of interfacial tension between the GIC ingredients, hence enhancing mechanical properties[27].

The results of this work corroborate those of several authors[27], who found that adding 10% v/v CH to the liquid phase of conventional GIC enhanced the flexural and diametral tensile strengths when contrasted to unmodified GIC.

The inclusion of TiO_2 nanoparticles boosted the flexural and diametral tensile strengths when the addition ratio was raised up to 5% by weight. The rationale for the increase in these strengths values as the ratio of TiO2 introduced increases is that TiO_2 includes a large number of hydroxyl groups on its surface and is covalently bonded to the GIC matrix[28].

Also, the particle size distributions of the modified glass ionomer powder with TiO_2 filler were broader, and the TiO_2 nanoparticles filled the gaps between GIC macromolecules. These nanoparticles could act as a reinforcing agent, enhancing mechanical qualities [11, 29].

This is supported by the studies of some researchers [11,30] that their conclusions were that adding 3 wt. %, 5 wt. % TiO_2 NP to the powder phase of GICs produced in an increase in strength when the additional ratio was increased to 5 wt. %.

Double modification of GIC powder with TiO_2 NP and GIC liquid with chitosan, as detailed in (Group 4&5), increased the flexural and diametral tensile strengths of the GICs significantly in comparison to the other groups. This mechanism may be explained by the simultaneous action of TiO_2 nanoparticles, which act as additional inorganic fillers reinforcing the GIC matrix, and chitosan, which forms numerous hydrogen bonds that hold the GIC glass particles and matrix together. [31]

Modification of the liquid phase of GIC with 10% v/v CH solution decreased hardness when chitosan was added. The polymeric structure was integrated into the matrix network at the expense of inorganic crystals. The surface hardness of polymers is lower than that of glass particles. As a result, the addition of chitosan has a detrimental effect on the hardness of the surface. Additionally, the hydrophilic nature of chitosan had a detrimental influence on surface hardness, leading to an increase in water content and a plasticizing impact on the structure [32].

The findings of this investigation corroborate those of several authors [32], who reported that modifying GIC liquid with chitosan to produce 10% (v/v) chitosan modified GIC lowered surface hardness in comparison to unmodified GICs.

The results of this investigation indicated that treating GIC powder with TiO_2 NP in the manner described in **(Group 2&3)** resulted in an increase in surface hardness when compared to unmodified GIC. The rationale for significantly increased surface hardness with the increased TiO_2 ratios is that TiO_2 has a harder surface than the parent material. Nanoparticles, with their smaller particle sizes, may act as fillers, filling the residual GIC glass particles' vacant small gaps, so strengthening the binding formed by the extra TiO_2 and GIC material particles. All these variables contribute to the surface's increased resistance to plastic deformation [33].

The findings of this study agree with those of researchers [28,33] where 5 wt. % TiO_2 modified GIC

recorded the highest surface hardness followed by 3 wt.% TiO_2 modified GIC while the non-modified GIC recorded the lowest surface hardness.

Dual alteration of GIC powder with TiO2 NP and chitosan as described in **(Group 4&5)** caused a reduction in surface hardness compared to the control group **(Group 1)**, where the addition of chitosan increased surface hardness. The presence of titanium dioxide nanoparticles may be the primary reason for the increased surface hardness of (Group 4 & 5) as contrasted to chitosan-modified GIC **(Group 6)** [31].

CONCLUSION

The synergetic impact of dual alteration of GIC powder with TiO2 nanoparticles (3&5 percent w/w) and GIC liquid with chitosan (CH) solution (10% v/v) greatly improves GIC's flexural and diametral tensile strengths. The addition of TiO2 NP to GIC powder increases its surface hardness, but the addition of chitosan decreases its surface hardness.

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