ORIGINAL ARTICLE

Evaluation and Characterization of some Properties of Glass Ionomer Cement Reinforced by Novel Boron Nitride Nanoplatelets

Hasanein A. AlNamel [1*], Rafid M. AlBadr [2], Farah Abdul Razzaq [3]

[1] *Department of Prosthetic Dentistry, College of Dentistry, University of Basrah, Basrah, Iraq*

[2] *Department of Basic Sciences, College of Dentistry, University of Basrah, Basrah, Iraq*

[3] *Department of Conservative and Aesthetic Dentistry, College of Dentistry, University of Basrah, Basrah, Iraq*

*Corresponding Author: Hasanein A. AlNamel
Email: Hasanen.muhsen@uobasrah.edu.iq

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Abstract

Purpose: This study characterized and incorporated a novel Boron Nitride Nanoplatelet (BNNP) into conventional cement known as Glass Ionomer Cement (GIC) with changed ratios (range from 1%, 3%, 5%, and 7%wt) Subsequently.

Materials and Methods: The study examined the impact of adding BNNP on the mechanical characteristics of GIC, including its Flexural Strength (FS), Diametral Tensile Strength (DTS), water sorption/solubility, and setting times. The BNNP was characterized using Physio-Chemical Characterization, and Brunauer-Emmett-Teller (BET) testing, and density was also measured. In addition to showing considerably greater DTS (16.34± 1.26MPa) and FS (24.037 ± 0.816 MPa), the results showed that 3% wt. BNNP-modified GIC specimens decreased in water sorption/solubility 19.358±2.40 and 2.979±0.65 µg/mm^3, respectively, compared with traditional GIC.

Results: In this work, a novel BNNP containing GIC was created, resulting in a 15% reduction in water sorption. When compared to commercial GIC, the demonstrated GIC can quadruple the DTS and FS.

Conclusion: For water-based cement types, the glass-ionomer formulations including BNNP exhibit equivalent and acceptable working qualities.

Keywords: Boron Nitride Nanoplatelets; Glass Ionomer Cement; Flexural Strength; Diametral Tensile Strength; Physical Properties; Water Sorption.

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1. Introduction

Research is always being done to find and create novel dental cement varieties with better mechanical and physical characteristics. Glass Ionomer kinds of Cement (GICs) are polymer-based composites that have specific benefits like as low thermal expansion coefficient, translucency, and superior biocompatibility in addition to chemical bonding to the tooth structure [1–3].

The versatile inorganic substance boron nitride nanoplatelets was initially investigated in dentistry. Due to several special qualities, including chemical inertness, environmental friendliness, excellent lubricity, high oxidation resistance, large thermal conductivity, good electrical insulation, and non-toxicity, it has drawn a lot of attention in a variety of industrial applications (such as ceramic composites, lubricants, insulators, surface coatings, etc.) [4, 5].

Recently, there has also been a lot of interest in Boron Nitride Nanoplatelets (BNNP) because of their enhanced mechanical, electrical, optical, and thermal properties [6, 7]. They have lately been utilized in dentistry to enhance the qualities of dental adhesives, denture bases, dental ceramics [8], and dental implants [9]. Two-dimensional hexagonal boron nitride, or h-BN, is a nanomaterial with superior chemical, mechanical, and physical properties that resembles graphene [12].

The white color and biocompatibility of BNNP made it a viable candidate for investigation as a reinforcing material in dental applications [8]. The mechanical characteristics of self-cured acrylic polymethyl methacrylate reinforced with stabilized zirconia nanoparticles and hexagonal boron nitride were documented in one study, these studies showed a significant increase in the improvement of flexural strength and the modulus of elasticity [8, 13]. An ideal replacement for current standard base materials is a denture base material enhanced with BNNP that has strong mechanical qualities and good antibacterial qualities [11].

Because glass-ionomer cements can have their physical qualities altered by adjusting their chemical formulation or powder/liquid ratio, they can be used in a variety of clinical applications. In the posterior dental area, GICs are mostly utilized as temporary filling materials. Many benefits come with GIC, including little microleakage, tooth adherence, biocompatibility, and a coefficient of thermal expansion comparable (~11.0 ppm K−1) to teeth (Dentin 8.3 ppm K−1 and enamel 11.4 ppm K−1) [14]. Furthermore, GIC is frequently used for luting, lining, and repair and has superior aesthetics to amalgam, gold, and porcelain. Low flexural strength was demonstrated by conventional GICs [15, 16].

Over the past few decades, cement formulation modifications have been used to improve the qualities of GIC, but the improvements have not been sufficient [5] added resin to GIC and discovered that the resin-modified GIC was less sensitive to moisture than the regular GIC. Since then, numerous attempts have been made to enhance the characteristics of GIC by adding additional fillers or altering it with nanomaterials like hydroxyapatite, ZrO_2, and TiO_2 [17, 18].

The purpose of this study was to investigate changes in glass ionomer cement in the mechanical and physical properties, such as Flexural Strength and Flexural Modulus as well as Diametric Tensile Strength measurement (DTS) and sorption kinetics, which are considered important properties in maintaining these materials within the oral environment by adding varying amounts of Boron Nitride Nanoplatelets (BNNP). The material created for dentistry has many potential applications when BNNP is included.

2. Materials and Methods

2.1. The Materials

ESK Ceramics GmbH & Co. (Germany) provided the Boron nitride Nanoplatelets (BNNP) with an average diameter of > 100 nm and width of less than 3 nm. GIC of the powder/liquid type, Cavex Glass Ionomer, Lot 2033134, from Cavex (Germany), was used. The ratio of liquid to powder was 2:1. The powder (glass made of aluminum silicate). To examine the mechanical and physical characteristics, BNNP was added to GIC at weight/weight ratios of 1%, 3%, 5%, and 7%. The GIC was manually mixed and injected into the metallic mold [18].

2.2. Physio-Chemical Characterization of Nanoplatelets

Using a 40 kV voltage, an X-ray source (Cu K) with a Philips PW 1700 series diffractometer (Leiden, Netherlands), captured patterns for XRD analysis. In

Bragg-Brentano geometry, the measurements were carried out in a continuous scan between the diffraction angles 2θ (10° and 90°) using a count time of 0.2°/min per step and a step size of 2θ = 0.02 ºθ in continuous mode.

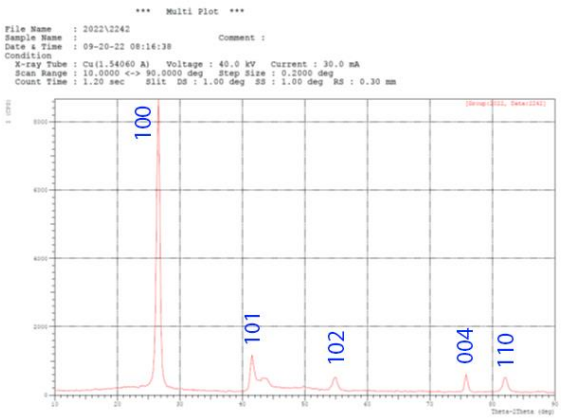

Figure 1. XRD pattern of BNNP

XRD was used to look at the BNNP's morphological characteristics. The XRD study of BNNP is displayed in Figure 1, which reveals a well-organized crystalline structure. With an interlayer spacing of 0.334 nm, the crystallographic plane (002) of the BNNP structure is responsible for the detection of the diffraction peak at 26.59 2θ [17]. The (100), (101), (102), (004), and (110) planes had much weaker peaks at 41.62, 43.9543, 54.82, and 75.79 2θ, respectively, according to (JCPDS No. 34-0421), with interplanar spacings of 0.216 nm, 0.208 nm, 0.167 nm, and 0.125 nm for planes. In accordance with equation Scherrer [18], the platelets are likely made up of numerous smaller crystallites that are invisible in the SEM pictures, as indicated by the lateral crystallite diameters, which range from 9.19 nm to 35.33 nm and form (101 and 110 planes).

Utilizing Scanning Electron Microscopy (SEM), the worn steel ball surface was examined. Using Energy-Dispersive X-ray (EDX) spectroscopy, the chemical composition was investigated. Images captured by scanning electron microscopy (SEM) were captured with a (VEGA TESCAN - Czech).

The samples' distribution characteristics, morphology, and sizes were determined using SEM. See Figure 2. The products have a high density of one-dimensional structures. The particles were sharp-edged polygonal. Most particles have diameters in the range of 70–100 nm.

The identical energy dispersive X-ray elemental mapping (EDX) to inspect the composition of BNNP nanoplatelet (Figure 3a) demonstrates the elemental signatures of nitrogen (N) and boron (B) weight denoted 61.95% and 38.05% from the resulting BNNP nanoplatelets. Moreover, the EDX elemental mapping (Figure 3b) demonstrates tribofilm formation, indicating that the material used is BN nanoplatelets.

Using a Michelet Magna-IR 550, Fourier-transform infrared (FT-IR) analysis was carried out in the 4000-400 cm^{-1} region. The FTIR spectra of BNNP are displayed in Figure 4; the five absorption bands are situated at 802, 1382, 2363, and 3432cm^{-1}. B–N stretching vibrations are represented by the bands at 802 cm-1 and B–N–B bending vibrations by the strong

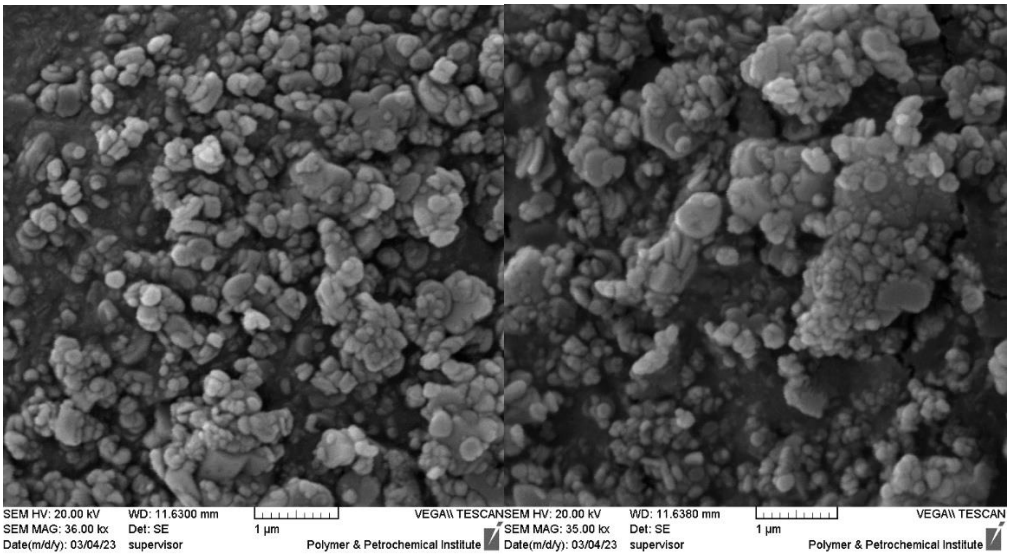

Figure 2. The morphology and diameters of BNNP measured by SEM

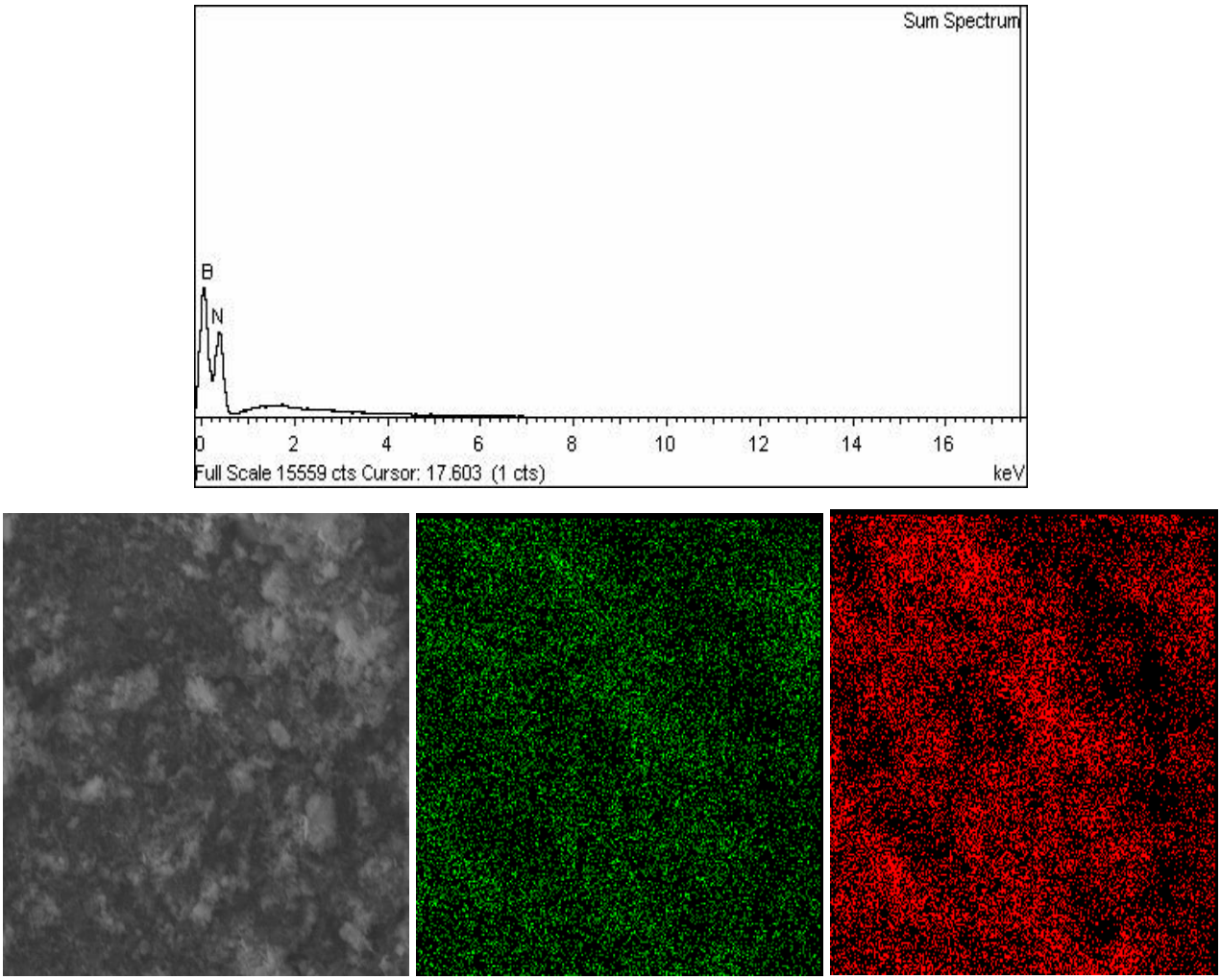

Figure 3. EDX spectra with corresponding elemental mapping results of h-BN powder

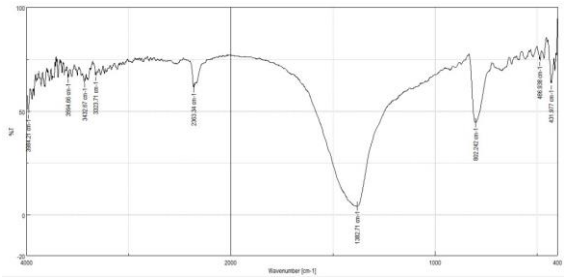

Figure 4. FTIR of BNNP

and wide bands at 1382 cm^{-1}, respectively [19]. At ~2363 cm^{-1}, the weakest band represents ambient CO_2. It is possible to attribute the band at 3432 cm^{-1} to OH stretching vibrations.

Using pyecnomtric techniques computations, the density of the BNNP was determined to be 2.131± 0.006 g/cm^3, while the theoretical density was 2.29 g/cm^3. The powders' specific surface area and porosity are measured using the Brunauer-Emmett-Teller (BET) surface analysis [20]. Nitrogen is the adsorption/desorption gas in a CHEMBET 3000 QUANTACHROME, which is used in the BET technique. BET technique was calculated using N2 adsorption/desorption data at 77 K, yielding 29.279 m^2/g. With a density of 2.29 g/cm^3 and an aspect ratio of 30.152, we can determine the aspect ratio using the specific surface area determined by BET and the particle size of BNNP [21]. This corresponds to a particle size of about 90 nm.

2.3. Production of Hybrid Nano Glass Ionomer Cement

The powder was aluminum silicate glass, and the liquid-to-powder ratio was 2:1. To enhance the mechanical and physical qualities of GIC, BNNP added various weight ratios: 1%, 3%, 5%, and 7%.

2.4. Water Absorption and Water Solubility

The water sorption procedure was carried out in compliance with ISO 4049: 2008, the specification standard for composite dental resins. For every GIC/BNNP ratio, four specimens were made in a cylindrical mold (Ø 15mm, 1mm). The values for water solubility (WS) and sorption (WA) were derived using the following Equations 1, 2:

$$WA\ (\frac{\mu g}{mm^3}) = \frac{M_1 - M_2}{V} \quad (1)$$

$$WS\ (\frac{\mu g}{mm^3}) = \frac{M_0 - M_2}{V} \quad (2)$$

By precisely measuring densities, the Archimedes principle was used to determine the volumetric changes of the specimens. Additionally, the available data on the densities of the dry (ρ_d) and the densities after immersion (ρ_i) are used to determine the volume increase, VI (%) (Equation 3):

$$Vl\ (\%) = \frac{(\frac{M_1}{\rho_i} - \frac{M_0}{\rho_d})\rho_d}{M_0} \times 100 \quad (3)$$

2.5. Setting Time

The ISO 9917 and BS 29917 standards were followed in measuring the setup times [1, 2]. After combining the ingredients, the model was examined by setting it in an ISO-recommended 37°C humid environment. Next, a 250 g flat-ended indenter needle with a diameter of roughly 1.0 mm was lowered vertically onto the cement surface and left there for five seconds. The time that passed between the conclusion of mixing and the point at which, when observed at a 2x magnification, the needle failed to make a complete indentation was recorded as the net setting time.

2.6. Mechanical Properties

A mold measuring 25 x 2 x 2 mm was utilized for the Flexural Strength and Flexural Modulus in accordance with ISO 9917-1:2007. Every ratio had four prepared specimens, and the overlap method was used to test each specimen. The specimens were subjected to a three-point bending test at a crosshead speed of 0.5 mm/min utilizing a universal testing machine (Zwick/Roell BT1FR2.5TN Germany). For a whole day, every specimen was submerged in distilled water. The following formulas were used to compute the flexural strength and flexural modulus (Equation 4):

$$FS = \frac{3pL}{2bd^2} \quad (4)$$

where L is the distance between the supports, b and d are the width and thickness of the specimens (mm), and p is the failure load (N) (Equation 5).

$$E = \frac{3PL^3}{4bd^3D} \quad (5)$$

Where F is the force at deflection, L is the distance (20 mm) between two points, b is the width, and D is the cutting limit [22].

Using the ANSI/ADA specification No. 27 technique, a diametric tensile strength measurement (DTS) test was conducted. Additionally, four specimens (Ø 6mm, T 3mm) were made and placed into a mold. After the mold was removed, the specimens were preserved in distilled water at 37º C for a whole day. a crosshead speed of 0.5 mm/min for a universal testing machine (Zwick/Roell BT1FR2.5TN, Germany). It was determined that the DTS (MPa) was [23] (Equation 6):

$$DTS = \frac{2p}{\pi DL} \quad (6)$$

where D is the specimens' diameter (mm), L is the thickness of the specimen (mm), and p is the stress at fracture (N).

3. Results

This study assessed the mechanical and water-sorption characteristics of glass ionomer cement modified with BNNP as a dental restorative material. The findings suggested that BNNP might improve these qualities when added to GIC.

3.1. Water Absorption and Water Solubility

The averages and standard deviations for water sorption and solubility (µg/mm^3) are presented in Table 1. Following sorption in every test, the GICs

were identified as GIC+3%BNnps and GIC+7%BNnps, respectively, and ranged from 19.35 to 24.11µg/mm^3. Since the measurements were taken every day, the specimen mass was deemed stable when five consecutive measurements were observed, with a mass difference of ±0.001g. All GIC-modified samples reached stability after 15 to 20 days, with the GIC (control) recording the longest stability times and the GIC+7%BNnps recording the shortest. GIC+1%BNnps and GIC+3%BNnps showed a little decrease in sorption in comparison to the other two, whereas GIC+7%BNnps showed the highest absorption rate. Compared to the control group, the water solubility of 7% was much higher. Between 0% and 7%, the change was negligible, nonetheless(nonsignificant).

3.2. Mechanical Testing

Figure 5 shows the means and standard deviations of flexural strength for tested GIC. As seen, the values of FS increased with the modification of a small amount of BNNP, with a significant difference between the GIC+1%BNpns and GIC+3%BNpns group and the GIC (control). However, the GIC+5%BNpns and GIC+7%BNpns showed an apparent decrease in values compared with the GIC (control).

Figure 6 shows the means and ±SD of diametral tensile strength (DTS) values with their statistical test results. An increase in the DTS was seen with an increasing amount of BNNP in the GIC. The addition of BNNP modified of GIC+1%BNnps and GIC+3%BNnps significant increase in the DTS values 11.80± 1.69 and 16.34±1.26Mpa respectively, while the GIC (control) recorded 9.03±1.02MPa. Further increasing the amount of BNNP led to a decrease in the DTS. This effect was more noticeable with the addition of GIC+7%BNnps record 6.84±1.68 MPa.

Diametral tensile testing was used to determine tensile strength indirectly because dental composite materials are fragile.

4. Discussions

GIC's mechanical and physical qualities are of modest quality, thus it has undergone a number of alterations in the past to improve its physical characteristics and increase its strength and fracture resistance. A variety of materials and products, including various nanomaterials [23, 24], were added to GIC in order to boost its mechanical and physical properties. The current study's findings support the hypothesis that BN nanoparticles

Table 1. Sorption, solubility (µg/mm3), Volume increase (%) and setting times (sec) of modified GIC with various concentrations (mean ± standard deviation)

Concentration	Sorption (µg/mm^3)	Solubility (µg/mm^3)	Volume increase (%)	Setting Times (Sec)
GIC (0%)	22.567±0.47	2.485±0.67	2.065±0.032	153±2
GIC+1%BNnps	20.122±2.25	2.942±0.88	2.082±0.077	187±10
GIC+3%BNnps	19.358±2.40	2.979±0.65	2.073±0.10	225±6
GIC+5%BNnps	21.008±3.04	3.004±1.24	2.357±0.218	251±2
GIC+7%BNnps	24.117±1.20	3.268±1.33	2.398±0.321	262±14

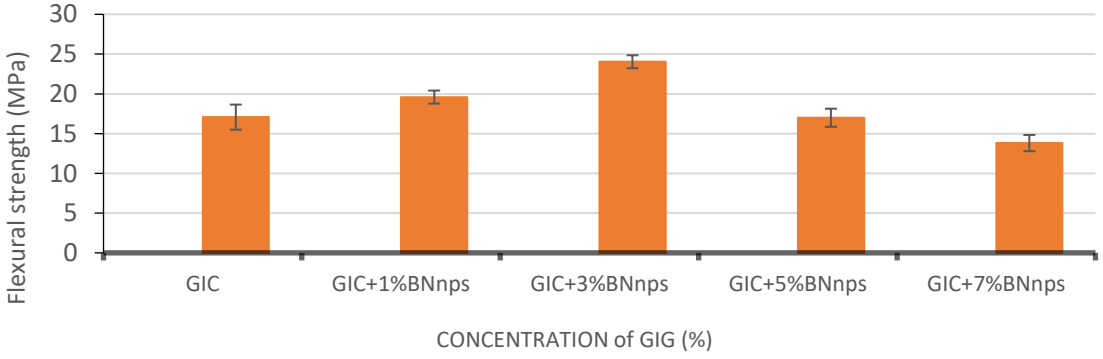

Figure 5. Flexural strength of modified GIC with various concentrations (mean ± standard deviation)

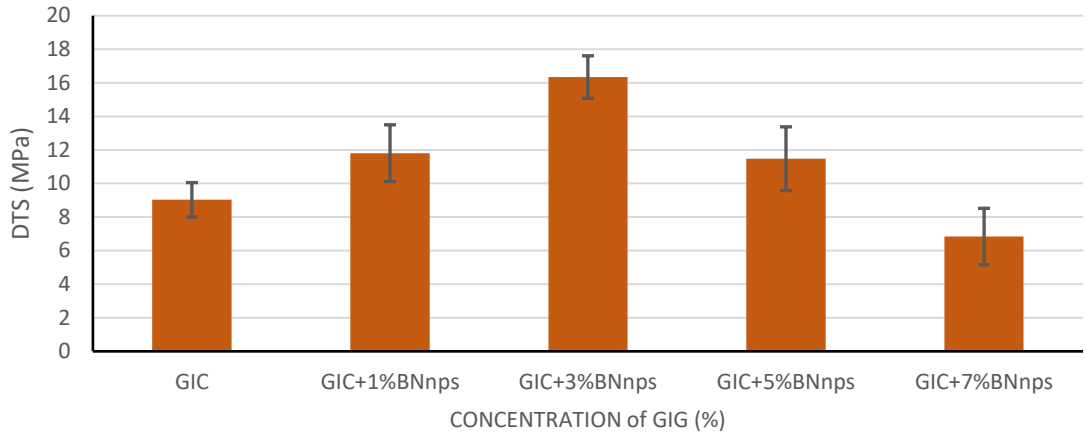

Figure 6. DTS of modified GIC with various concentrations (mean ± standard deviation)

added to glass-ionomer cement will result in GIC with altered characteristics.

Due to its hydrophilic nature and susceptibility to moisture, GIC may develop water sorption or hydrolytic degradation, which would reduce its mechanical performance and limit its service life [25]. The network of restorative materials may collect chemicals and water and release components to its surroundings since the mouth cavity is a moist environment [25, 26].

Table 1 summarizes the solubility volume, and water sorption increase for water sorption, with increasing BNNP (1% to 5%) with GIC, the water sorption decreased, but there was no noticeable difference in comparison to the control, while at 7% of BNNP, water sorption increased. Thus, the addition of low-concentration BNNP provides a compatible effect, due to the regular and non-clumpy behaviour of the overlapped nanoparticles. while at 7% of BNNP addition, water sorption increased, this increase may be caused by higher concentrations of nanoparticles clumping together, resulting in a greater powder of GIC-BNNP interaction than filler-matrix interaction. This reduces intrametric homogeneity and has a negative impact on the water sorption and solubility of the polymerized GIC. An increase in solubility was observed with a rise in the presence of nanoparticles. The increase is quite small, with a maximum of 10% for large concentrations of GIC+7%BNnps. Its structural stability and biocompatibility may be impacted by the solubility of GIC control and modification [27]. Conversely, the data show that the addition of NBBP particles causes a slight increase in volume. This can be explained by the fact that water molecules diffuse between the material's macromolecules, slightly separating them, while higher nanoparticle concentrations increase GIC's water sorption and solubility. Furthermore, the agglomeration of nanoparticles at higher concentrations may be responsible for the 7% increase in water sorption of BNNP. This agglomeration produces more powder of GIC-BNNP interaction than filler-matrix interaction, which lowers intra matrix homogeneity and negatively impacts the water sorption and solubility of the polymerized GIC water solubility. An increase in solubility was observed with a rise in the presence of nanoparticles. The increase is quite small, with a maximum of 10% for large concentrations of GIC+7%BNnps. Its structural stability and biocompatibility may be impacted by the solubility of GIC control and modification [30]. The findings also showed that, depending on the size, shape, and nature of the added nanoparticles, water sorption and solubility were strongly correlated with the concentration of the nanoparticles. Water sorption and solubility were determined in the current study using the International Organization for Standardization's (ISO 9917-1:2007) recommended methodology.

The setting times denoted slightly increased in the modified GIC ranged between 187 to 262 Sec (~ 3.1 to 4.3 min.) as shown in Table 1. The setting time increase was gradually proportional to the percentage of incorporated BNNP to GIC. The GIC mixture is

usually set within 2–8 minutes [3], with a view to control the setting times, an appropriate accelerator such as tartaric acid could be added to the liquid part of the Glass ionomer cement.

For assessing the mechanical properties of brittle dental cement the flexural strength test exhibited some superiority over the compressive strength test [28] because fracture in the matrix of GICs occurs due to shear and tensile loads on the atomic scale. Figure 5 shows that the mean flexural strength of BNNP was significantly higher than the Control (9.59±0.82MPa) and recorded the highest value at 3% wt. (14.372±0.81 MPa) after this percentage decreased to 3.8± 1.02MPa at 7%. The BNNP has a tubular shape, length > 100 nm, and diameter of 3 nm, giving them greater bonding strength than those with spherical particles. Therefore, that effect can be related to incomplete mixing, and distribution is better than high percentages. This finding may be due to the nonuniform and nonhomogeneous distribution of nanoparticles in GIC powder and the inefficient action of polyacrylic acid on these nanoparticles. Owing to this limitation, the cement is weak at the agglomerates site, and cracks initiate at these sites [29, 30]. However, the effect of the incorporation of BNNP on other properties requires further investigations.

Finally, a study involving the incorporation of BN nanoplatelets into GICs resulting in GIC material that these particular nanoparticles may improve mechanical properties and water sorption. Excellent mechanical properties were obtained by the addition of 3 wt.% of BNNP.

The DTS results made it clear that the modified GIC outperformed the traditional GIC at weights of 1% and 3% of BNNP. If the BNNP particle content was increased by more than 3 weight percent, there was no discernible change in the DTS value. Once more, as seen in Figure 6, the diametral tensile strength diminishes when the BNNP content increases to greater than 3% wt.

5. Conclusion

BN nanoplatelets, a newly developed nanomaterial, were investigated in this study as a reinforcement material for GIC. Within the study's limitations, the results showed that adding 3% weight BNNP towards the GIC exhibits improved its mechanical properties and reduced its water sorption without impairing the surface micro-hardness. Also, a higher than 3% weight adversely affects GIC properties. Finally, the unintended results showed that adding BNNP increased the whiteness of the GIC significantly, which qualifies it as a beautifying material for the GIC and improves its general properties such as mechanical and physical properties.

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