

Evaluation of Microtensile Bond Strength of Zirconia-Reinforced Glass Ionomer After Addition of Nano-Hydroxyapatite: An In-Vitro Study

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ABSTRACT

Objectives: To evaluate and compare the microtensile bond strength of Zirconomer Improved before and after adding nano-hydroxyapatite particles.

Materials and Methods: An in-vitro study was conducted using 32 non-carious extracted human premolar teeth, divided into Group I and Group II. Each group included sixteen specimens for microtensile bond testing. Group I: Zirconomer Improved and Group II: Zirconomer Improved with 5% nano-hydroxyapatite incorporated. The microtensile bond strength was measured using a universal testing machine. The normally distributed data were analyzed using independent t-tests.

Results: Group II exhibited significantly higher microtensile bond strength values (8.95 ± 1.89 MPa) compared to Group I (5.85 ± 1.02 MPa; $p < 0.05$).

Conclusions: These findings support nano-hydroxyapatite as a promising reinforcement for improving the performance of Zirconomer Improved. These enhancements result from the small particle size and bioactive nature of n-HA, which boost adhesion and mechanical strength.

Keywords: Microtensile bond strength, Nano-hydroxyapatite particles, Zirconia-reinforced glass ionomer.

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INTRODUCTION

Glass ionomer cements (GICs) are popular because they adhere well to tooth structure, release fluoride, and are biocompatible, which helps improve restoration durability and reduces treatment complication time [1]. Their self-adhesion to tooth structure eliminates the need for bonding agents and enhances durability [2].

A newer version, Zirconia-reinforced GIC (Zirconomer Improved), combines the strength of amalgam with the fluoride release of GIC, leading to improved aesthetics and durability [3,4]. Adding HAp to GICs further enhances their mechanical properties, including tensile strength, fracture toughness, and bonding, due to their biocompatibility and similarity to natural apatite [5].

Bond strength testing is crucial for assessing adhesion and durability. Macro-tests (such as shear, tensile, or push-out tests) evaluate areas larger than 3 mm². Conversely, micro-tests, especially microtensile bond strength tests, offer a more precise evaluation of the material's adhesive performance [6-8].

Tensile strength is the maximum force a material can withstand when pulled perpendicularly [9]. The μ TBS test is commonly used to measure restorative bond strength [10]. Specimens can be either trimmed or left untrimmed,

with the trimmed ones being more technique-sensitive [11]. The μ TBS also helps identify regional differences and reduces cohesive failures. However, it requires specialized equipment, is technique-sensitive, and its small samples are prone to dehydration and damage, with limited accuracy below 5 MPa [12].

Bond strength testing provides an in vitro measure of the resistance of the restorative-tooth interface to tensile loading. μ TBS testing uses relatively small bonded specimens and can provide greater discrimination of interfacial performance than conventional macro-tests [13]. However, μ TBS is technique-sensitive, and the statistical unit must be considered carefully when multiple beams originate from the same tooth [14].

Therefore, this study aimed to evaluate and compare the microtensile bond strength of a zirconia-reinforced glass ionomer cement (Zirconomer Improved) before and after incorporating nano-hydroxyapatite (n-HA) particles.

MATERIALS AND METHODS:

Materials

The materials used in the study, their composition, manufacturer, and batch numbers, are listed in Table 1 and displayed in Figure 1.

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Table 1. Materials used in the study

Material Name	Composition	Manufacturer	Batch No.
Zirconomer Improved	Zirconia-reinforced glass ionomer Powder: Fluoroaluminosilicate glass; zirconium oxide, pigment, and others. Liquid: ▪ Polyacrylic acid solution and tartaric acid.	Shofu Dental, Tokyo, Japan.	02210480
Nanoparticles Hydroxyapatite	Particles: powder ▪ Calcium phosphate compound $\text{Ca}_{10}(\text{PO}_4)_2\text{OH}_2$	Nanogate lab Egypt	HAp20223



Figure 1. The materials used in the study, (A) Zirconomer-Improved Glass-ionomer, (B) Hydroxyapatite Nanoparticles added to Zirconomer-Improved Glass-ionomer.

Methods

Characterization of Nano-hydroxyapatite:

Hydroxyapatite nanoparticles were synthesized using a modified wet chemical method. A calcium chloride solution was gradually combined with ammonium hydrogen phosphate under constant stirring, and the pH was adjusted to 10 with ammonia. The mixture was kept at 40 °C for four hours, then stirred at room temperature for an additional 16 hours to produce a white hydroxyapatite precipitate [15,16]. The nanoparticles were characterized with a high-resolution transmission electron microscope (TEM; JOEL JEM-2100,

200 kV). TEM analysis showed that the hydroxyapatite nanoparticles have a needle shape with an average width of 10 ± 5 nm and an average length of $80 \text{ nm} \pm 10$ nm. Nano-hydroxyapatite-modified zirconia-reinforced glass ionomer was prepared by adding 5% w/w nHAP powder to Zirconomer Improved, then milling the mixture for 30 minutes at 200 rpm in a planetary ball mill to ensure uniformity.

Sample Grouping

Table 2 summarizes the sample grouping and the tests conducted on various material groups.

Table 2. Tests applied to different Material groups

Group	Microtensile bond strength
Zirconomer (Improved) alone	16
Zirconomer (Improved) with nanohydroxyapatite (nHA)	16
Total	32

Tooth Preparation

Thirty-two healthy human premolars, extracted for orthodontic purposes (Faculty of Dentistry, Suez Canal University), were used in this study. The teeth were stored in physiological saline to be used within one month of extraction. Each tooth was mounted in cold-cured acrylic resin blocks (1.5 mm apical to the cemento-enamel junction, CEJ), aligned parallel to the molds (Fig. 2A). The occlusal enamel of the crowns was removed at the dentino-enamel

junction (DEJ) using a low-speed automated diamond saw under running water on a hard-tissue microtome (IsoMet 4000, Buehler Ltd., Lake Bluff, IL, USA) (Fig. 2B) to expose flat mid-coronal dentinal surfaces. Exposed dentin was examined under a microscope to ensure the absence of residual enamel, pulp exposure, or cracks (Fig. 2C). To prevent dehydration, the teeth were kept covered with damp tissue until used shortly after dentin exposure.

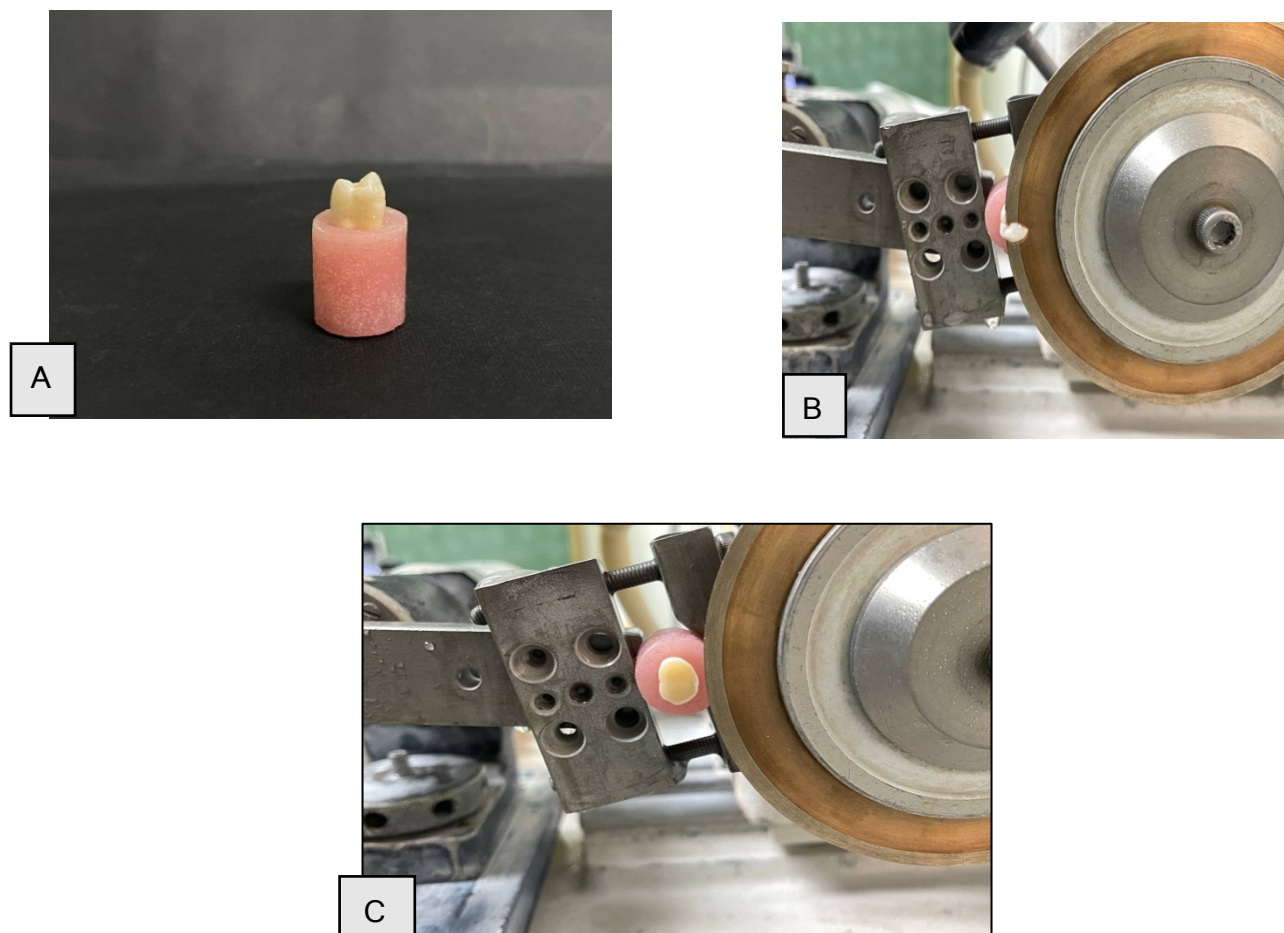


Figure 2. (A) The standard mold used to mount the teeth, (B) Enamel removal in a Microtome (IsoMet 4000, Buehler Ltd., Lake Bluff, IL, USA), (C) Tooth with occlusal enamel removed.

Materials were mixed at a 2:1 powder-to-liquid ratio according to the manufacturer's instructions and placed into 4x4 mm Teflon molds, which were later split open (Fig. 3).

Group I: Zirconomer Improved (control), and Group II: Zirconomer Improved modified with nanohydroxyapatite.

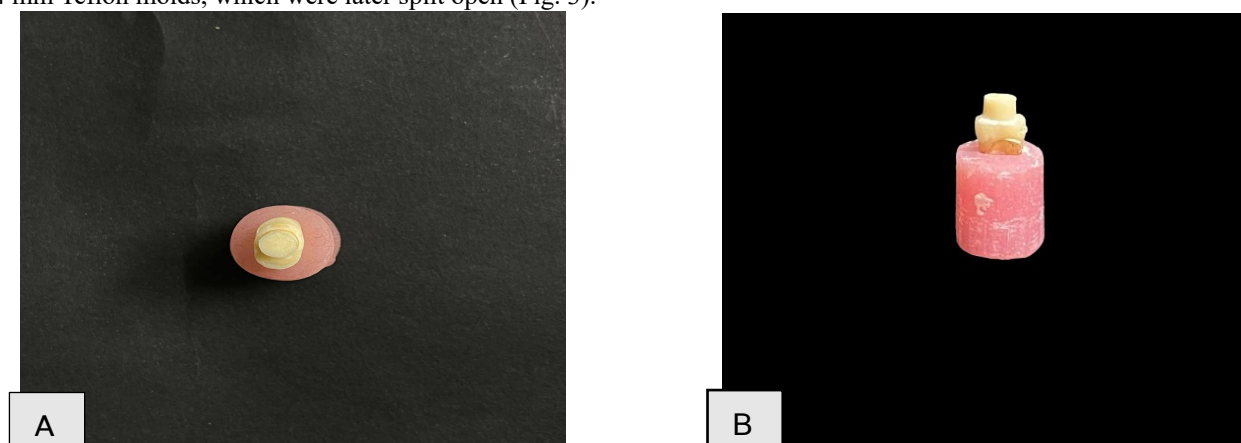


Figure 3. The sample with built-up restoration, (A) Occlusal view, (B). Lateral view.

The specimens underwent artificial aging via thermocycling (1000 cycles between 5 °C and 55 °C), with 10-second dwell times in each bath, to mimic oral temperature fluctuations (Fig. 4).



Figure (4): Thermocycling machine used in the study (SD Mechatronic Thermocycler Germany)

Micro-tensile bond strength test (μ TBS)

The bonded specimens were prepared using the non-trimming technique. Each tooth was sectioned into beams

(0.9 ± 0.1 mm thickness and 7 ± 1 mm length), and cut at the root base from the acrylic supports (Six beams were obtained from each tooth) (Figs. 5A, B, C).

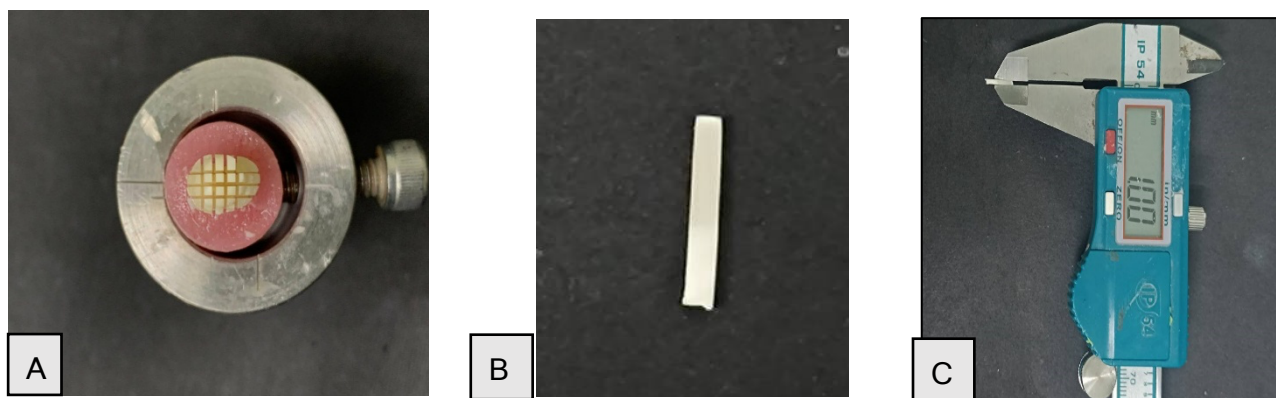


Figure 5. Micro-sectioning of the bonded teeth (A) Tooth longitudinally sectioned in both mesio-distal and buccal-lingual directions, (B) The obtained beam (8mm length, 1mm thick), (C) Digital caliper confirming the beam dimensions.

Each beam was fixed to the Geraldellis jig parallel to the tensile force axis with a cyanoacrylate adhesive (Fig. 6A) and stressed in tension using an Instron universal testing

machine (model 3345, England) with BlueHill Instron software at a crosshead speed of 0.5 mm/min until failure (Fig. 6B).

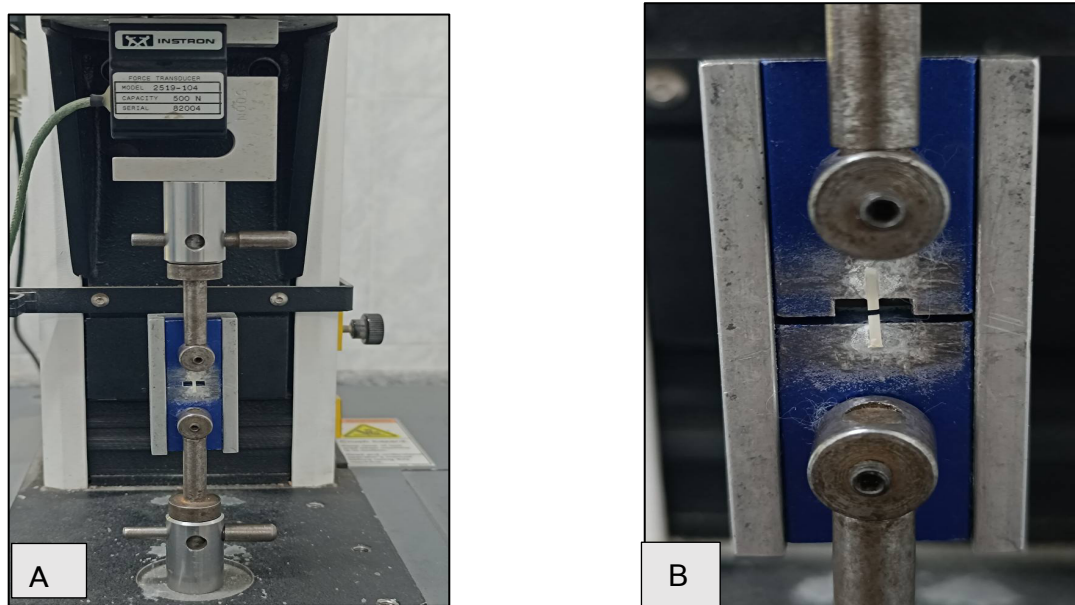


Figure 6. (A) Universal testing machine with the specimen attached, (B) Failure of the specimen.

Statistical analysis and data interpretation

Data were presented as mean and standard deviation (SD) values. Normality was assessed using the Shapiro–Wilk test. Data that were normally distributed were analyzed with independent t-tests. The significance level was set at $p < 0.05$. Statistical analysis was conducted using R software, version 4.4.2, for Windows.

RESULTS:

Microtensile bond strength

The cement with incorporated nano HA particles demonstrated significantly higher bond strength values (8.95 ± 1.89 MPa) than the cement without nanoparticles (5.85 ± 1.02 MPa) ($p < 0.001$), as shown in Table (3) and Figure (7).

Table (3): Intergroup comparison and summary statistics of micro-tensile bond strength (MPa).

Micro-tensile bond strength (MPa) (Mean $\pm$ SD)		Mean difference (95% CI)	Test statistic	p-value
Cement without nano HA	Cement with nano HA			
5.85 ± 1.02	8.95 ± 1.89	-3.10 (-4.64; -1.56)	4.28	<0.001*

CI Confidence interval, * significant ($p < 0.05$).

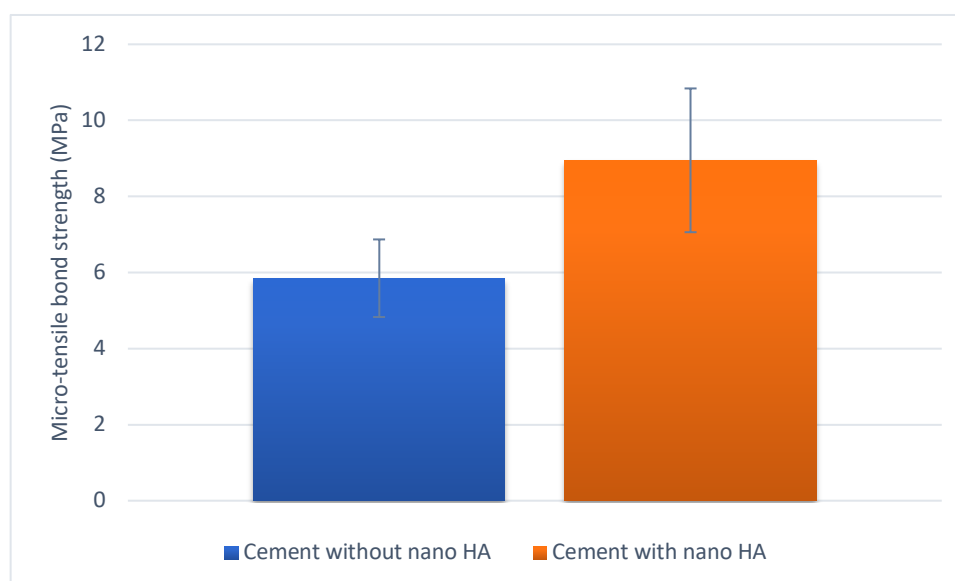


Figure (7): Bar chart showing means and standard deviation values for micro-tensile bond strength (MPa) specimens of tested materials.

Discussion:

Glass ionomer cements (GICs) chemically interact with tooth tissues and offer fluoride release and biocompatibility. However, GICs have low abrasion resistance, are moisture sensitive, and have poor aesthetics, which restricts their application in areas exposed to high masticatory forces. To address these issues, modified formulations have been developed [17]. Zirconia-reinforced GICs, including Zirconomer Improved, was introduced to provide the strength of amalgam along with the fluoride benefits of GICs [3]. This material contains micronized glass and evenly dispersed zirconia particles for enhanced mechanical performance [18].

Recently, Modification of GICs with hydroxyapatite has also been investigated as a means of changing their physicochemical and mechanical behavior [19].

The clinical effectiveness of restorative materials relies on the durability of their adhesion to dental tissues [20]. A weakened bond leads to compromised interfacial sealing, ultimately causing sensitivity, staining, and caries [21].

This study assessed bond strength using μ TBS, which offers better differentiation of adhesive performance than traditional macrotensile and macroshear tests. It also correlates more closely with clinical outcomes than microshear tests. Smaller specimens show increased strength due to fewer critical defects, allowing more efficient use of dental hard tissues by producing multiple micro-specimens from a single tooth [22,23].

The specimens underwent thermal cycling (5°C to 55°C for 1000 cycles with 10-second intervals) to simulate typical intraoral temperature fluctuations. This approach helps predict in-vivo performance by mimicking long-term marginal deterioration [24].

The mean microtensile bond strength of Zirconomer Improved with nano HA (8.95 ± 1.89 MPa) exceeded that of Zirconomer Improved without nano HA particles (5.85 ± 1.02 MPa). Zirconomer Improved chemically bonds to dentin through polyalkenoic acids that interact with hydroxyapatite. The higher bond strength with nano HA results from its calcium ion content, which enhances ionic interactions with the tooth. Additionally, the smaller particle size of nano HA increases surface area, solubility, and penetration into dentin, further strengthening the bond.

These findings support the statement of Gupta et al. (2018), who noted that nano-HAp increases the strength of glass ionomer cements and enhances bonding between the glass core and the matrix. The formation of apatite from nano-HAp, combined with ion release from the glass ionomer, improves the cement's mechanical properties [25].

Similarly, Lee et al. (2010) showed that nano-HA-modified GICs have better bonding strength because of their increased surface area and solubility, which helps them integrate more effectively into enamel and dentin [26].

The bond between the restorative material and the tooth structure provides resistance against dislodging forces in the oral environment, preserving marginal integrity and minimizing the risk of microleakage [7].

One limitation of the study was that it was conducted in vitro with short-term thermocycling. Therefore, the results

may not fully reflect the challenging oral environment or the long-term clinical performance of the tested materials. Future research with different n-HA concentrations and longer clinical follow-ups is needed to confirm these findings.

CONCLUSIONS:

Adding nano-hydroxyapatite (n-HA) to Zirconomer Improved significantly increased its microtensile bond strength, indicating enhanced interfacial integrity.

DECLARATIONS

Abbreviations

μ TBS	Microtensile bond strength
CEJ	Cemento-enamel junction
DEJ	Dentino-enamel junction
GICs	Glass ionomer cements
HAp	Hydroxyapatite
MOD	Mesio-occluso-distal
MPa	Megapascal
nHA	Nano-hydroxyapatite
SEM	Scanning electron microscope

Ethics approval and consent to participate:

This in vitro study was approved by the Research Ethics Committee (REC) of the Faculty of Dentistry, Suez Canal University, with approval number (624/2023). The study was conducted in accordance with the ethical standards of the Declaration of Helsinki. This study used extracted or exfoliated teeth with no patient identifiers, and the need for informed consent was waived by the Ethics Committee of the Faculty of Dentistry, Suez Canal University.

Consent for publication

Not applicable

Data availability

All data generated or analyzed during this study are included in this manuscript

Competing Interests:

The authors have no conflicts of interest relevant to this article.

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Authors' contributions

Conception and study design were done by AS, MD, MF and RB. AS carried out the practical work, discussed the practical steps, performed data analysis and wrote the first draft of manuscript. MD, MF revised the manuscript. RB discussed the practical steps, edited and revised the manuscript. All authors read and approved the final manuscript

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