

Effects of N-Vinylcaprolactam Containing Polyacids and Zirconia on Mechanical Properties of Commercial Glass Ionomer Cements

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Abstract

Introduction: This study aimed to investigate the impact of N-vinylcaprolactam (NVC) and Nano-sized yttria-stabilized zirconia (YSZ), separately and simultaneously, on the mechanical properties of the commercial glass ionomer cements (GICs). **Methods:** The NVC is able to ameliorate the mechanical and surface properties of glass ionomers; however, its effect hasn't been investigated in conjunction with zirconia yet. In order to perform the current research, the liquid of glass ionomer was synthesized by adding the NVC to its copolymer and then was characterized by proton nuclear magnetic resonance and Fourier transforms infrared. In addition, Nano-sized YSZ was added to the powder of glass ionomer and then was characterized by scanning electron microscopy. Afterward, the specimens for both flexural strength (FS) and microhardness were prepared by mixing the powder and liquid of the modified glass ionomer. Eventually, the aforementioned properties were evaluated after 24 h and 1 week of immersion in distilled water in an incubator. Furthermore, the one-way analysis of variance was used to study the statistical significance of FS. **Results:** The obtained results demonstrated that microhardness and FS properties of the glass ionomer were clearly improved as zirconia and NVC were added to the powder and liquid of the glass ionomer, respectively ($P<0.05$). Moreover, the best result was achieved for the group in which the modification of powder and liquid of glass ionomer occurred concurrently. **Conclusion:** Based on the findings of the present study, it was deduced that the modified GIC is a promising dental material with improved mechanical properties.

Keywords: Microhardness, Glass ionomer, Flexural strength, N-vinylcaprolactam (NVC), Zirconia

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Introduction

The use of glass ionomer cements (GICs) as restorative compounds has rapidly increased during the past few decades. The GICs which are known as poly-alkenoate cements, were invented by Wilson and Kent at the Laboratory of the Government Chemist in 1970 (1) leading to the establishment of a strong usage base in the field of dentistry (2). The generic name of glass ionomers refers to a certain reaction between silicate glass powder and aqueous solution of polyacrylic acid. In fact, the setting reaction of these cements is fundamentally an acid-base reaction (3). There are three stages in the setting mechanism of GICs. Initially, insoluble polysalts are formed by the reaction of polyacid liquid with the released calcium from the glass. Then, aluminum carboxylate structures are created as the complex of anionic alumino-ions gradually quit their metal atoms as relatively discrete entities. Finally, diffusionally controlled crosslinking of the polymer matrix elevates the strength, hardness, and insolubility of the cements (4). The GICs are well-known for properties, such as good chemical bonding to the tooth structure and base metals, stability in an aqueous environment, low coefficient of thermal expansion similar to the tooth (5, 6) anticariogenic properties due to the release of fluoride (7) and minimized micro leakage at the tooth-enamel interface due to the low shrinkage (8, 9). However, their present major problems, such as lack of strength, hardness, brittleness, and poor wear characteristics (10-12). To be more specific, during the GICs setting, not all the carboxylic acid groups will convert to carboxylate groups, and very little polysalt bridge (Ca^{2+} dicarboxylate and Al^{3+} tricarboxylate complexes) formation will take place, leading to weak mechanical properties. In order to overcome these problems, scientists performed numerous investigations and evaluated diverse additives effects. One of the most efficacious additives has been N-vinylcaprolactam (NVC) monomers and amino acid derivatives. The NVC is an initial monomer for a water-

soluble polymer with a low critical dissolving temperature (36–37°C). In addition, the NVC has a higher ability to form complexes than other kinds of same compounds, such as N-vinylpyrrolidone (NVP) and it precipitates in a separate phase from aqueous solution in a range of physiological temperatures (13, 14). These monomers in glass ionomers can play the role of spacers between the carboxylic acid groups. This helps the polymeric backbone to be more flexible (15) and allows greater access to acid/base reaction (greater freedom for COOH groups to react with Al³⁺ and Ca²⁺ ions). As a result, the NVC modified polyacid compound improves the strength of the GICs (16, 17). Culbertson et al. (18) reported that acrylic acid–Itaconic acid–NVC polymers with the different molar ratios have the ability to improve the mechanical properties of the GICs. In addition to the NVC monomers, Nano-sized yttria-stabilized zirconia (YSZ) is one of the most useful ceramic systems which has been investigated extensively due to its interesting critical applications. For instance, the polymorph has been used as an advanced structural ceramic in tooth crowns and jet engines since it has high toughness (19–21). Moreover, it has been widely used for strengthening the bio glasses in biomedical applications (22). As well as, the ZrO₂ powders can be used in the fabrication of a high strength core for dental implants (23). All of these are reasonable evidence for considering the zirconia in the amelioration process of the glass ionomers. Since in dental materials; two of the most important physical properties are microhardness and flexural strength (FS) (24), it was decided to investigate these additives' effect on the mentioned properties in the current research. The used tests which were related to the application of flexural, compressive, and tensile strength, were all practical.

Materials and Methods

2.1. Materials

Acrylic acid (AA), Itaconic acid (IA), NVC, ammonium persulfate ((NH₄)₂S₂O₈), methanol (CH₃OH), anhydrous ethyl acetate (CH₃COOC₂H₅), calcium hydride (CaH₂), and calcium oxide (CaO) are all the reagents used in the present study. They were of analytical grade and applied as received from Sigma–Aldrich Chemical Corporation. The ZrO₂ powder was purchased from Tosoh Corporation, Japan. The glass ionomer powder and polyacid that were used in the experiments were in commercial-grade (Fuji IX, GC International, and Tokyo, Japan).

2.2. Polymer synthesis

The experimental procedure employed in the current study is a slight modification of a method first reported by Crisp et al. and Yamazaki et al. (25, 26). Details of reaction steps are as follows: In the first step, 1 wt.% (NH₄)₂S₂O₈ as the initiator of the polymerization reaction was dissolved in 75 mL of distilled water in a 250 mL three-necked flask. In the next step, 0.4 mol AA, 0.05 mol NVC, and 0.05 mole IA were measured and dissolved in 37.5 mL of distilled water in a beaker. A third solution was prepared, consisting of 1wt% (NH₄)₂S₂O₈ dissolved in 22.5 ml of distilled water in a beaker. The first solution was stirred with a magnetic stirrer and heated continuously until 98°C under constant flowing nitrogen gas. The second and third solutions were added to the flask after the temperature reached about 98°C, respectively, in a dropwise manner using a glass-dropping funnel at ~5 ml min⁻¹. Finally, after the completion of these additions, polymerization was allowed to proceed for an additional overnight by maintaining stirring under a blanket of nitrogen at the 95–98°C range. The polymer was freeze-dried for 24 h at 266 mbar (Operan Co., Yangcheon-meyon, Gimpo, Korea). In order to remove residual monomers, the dried polymer was completely dissolved in anhydrous methanol for five times and then precipitated from anhydrous ethyl acetate. The chemical structure of the synthesized terpolymer is indicated in Figure 1.

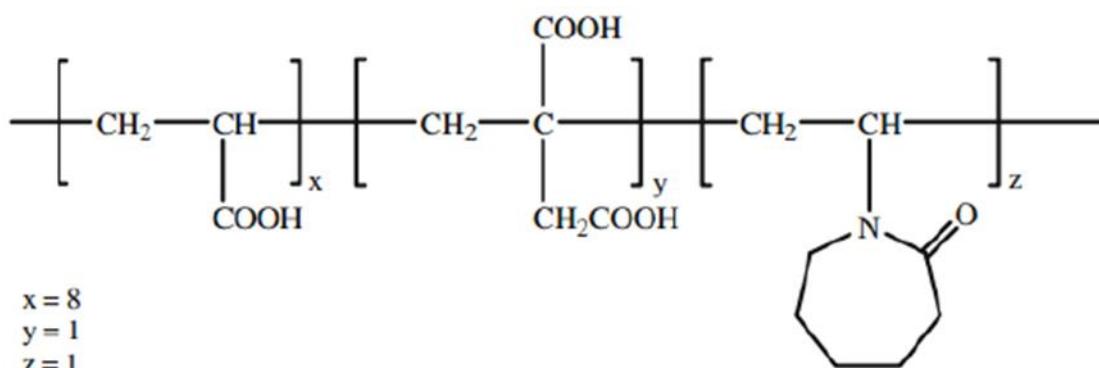


Figure 1. Chemical structure of the prepared terpolymer of AA–IA–NVC

2.3. Characterization

Fourier transform infrared (FTIR) analyses of the synthesized polymer were carried out using an FTIR-8400S spectrophotometer where the polymer sample was cast on K Br crystal to obtain spectra. Moreover, proton nuclear magnetic resonance (¹H NMR) analyses of the polymer were conducted using a Bruker 500 MHz ¹H NMR using D₂O as the solvent (Bruker Avance DRX, Bremen, Germany). The particle morphologies of the ZrO₂ and ZrO₂-GIC powders were observed by an AIS2100 (Seron Technology) scanning electron microscopy (SEM).

2.4. Formulation and evaluation of glass ionomer samples

2.4.1. Specimen preparation

In order to perform the experiments, four groups were considered. The first group was a control group (Fuji IX) and the second was the glass ionomer that was modified by 10%wt YSZ. In the third group, the liquid of glass ionomer was amended with NVC. In the last group, powder and liquid were simultaneously modified with zirconia and NVC, respectively. Firstly, the powder for the second and last group was prepared by adding the YSZ to the glass ionomer's powder. In order to achieve a uniform mixture of zirconia and aluminosilicate glass powders, the mortar was used. Then, the synthesized terpolymer was dissolved in the deionized water with 1:1 molar weight at room temperature. For the microhardness test, two specimens were prepared for each interval time of immersion, 24 h and 1 week (totally 16 specimens). For FS, five samples were provided for each interval time of soaking (totally 40 samples). The mold for microhardness samples was cylindrical PTFE (polytetrafluoroethylene) in size of 6×3 mm and for FS, it was a rectangular cube PTFE in size of 2.5×2.5×2 mm. The samples were fabricated in the following manner: first, in order to make a soft and uniform surface, a celluloid tape was placed under the mold. Then, glass powder and liquid were mixed on a special pad with a ratio of 3.6/1 as reported by Fuji company, after that the mixture was transferred to the mold. In the next step, the celluloid tape was again placed on the top-full mold. A uniform pressure was applied to the pad for two reasons:

filling the mold completely and removing the bubbles. Finally, the prepared samples were taken place in an incubator for 15 min. At the end of the process, the samples were taken out of the incubator, immersed in a DI solution, and again placed in an incubator for 24 h and 1 week.

2.4.2. Mechanical properties measurement

Mechanical testing of samples was performed on a screw-driven machine (Model 4202, Instron Corp., Canton, MA, USA) with a crosshead speed of 0.5mm/min. The FS was calculated from the relationship $FS=3PL/2BD^2$ where P is the load (N) at the fracture point, L =Length, B =width, and D =height. The microhardness (HV) of fabricated samples and the control group was determined using a microhardness tester (Model MVK-E, M 400, LECO, St. Joseph, MI, USA). A diamond indenter with 100 g load and a dwell time of 10 s were employed.

2.5. Statistical analysis

One-way analysis of variance followed by Tukey's test was used to determine if there is a significant difference between the strengths of the experimental cements and the control group. A level of $\alpha=0.05$ was used to indicate statistical significance.

Results

The SEM images of YSZ, GIC powder, YSZ-GIC powder, and YSZ-NVC-GIC samples after 1h setting reaction are shown in Figure 2 (A and D), respectively. In the aforementioned figure, (A) and (B) clearly indicate that the ZrO₂ particles are uniform in shape and have a particle size of about 70 nm; while the glass powders are angular in shape and the particle size of the powders fall in the range of 1-2 μ m. In Figure 2, (C) dedicates 10%wt YSZ-GIC powder morphology. It is clear that the YSZ and GIC powders are mixed well and spread out uniformly. In Figure 2, (D) shows the SEM micrographs of 10%wt YSZ-NVC GIC sample after 1h setting reaction in distilled water. The morphology of the sample indicates that there is a good connection between glass powders and a matrix of the hydrogel.

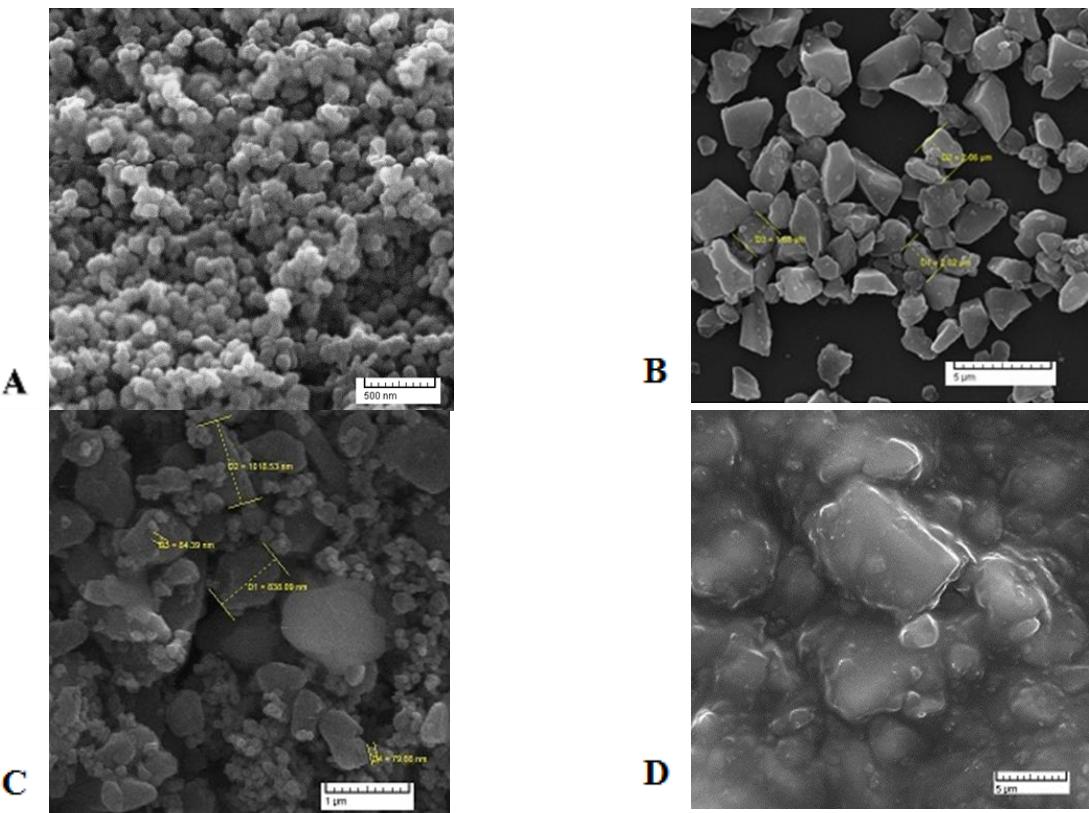


Figure 2. Scanning electron microscopy micrographs of (A) ZrO_2 powders, (B) glass ionomer cement powders, (C) 10% wt YSZ-GIC powders, and (D) 10% wt YSZ-NVC-GIC after 1h setting reaction in distilled water

The ^1H NMR spectrum of the terpolymer is shown in Figure 3. The displayed signals at $\delta=1.67$ (CH_2 from acrylic acid and NVC, respectively), $\delta=4.3$ (C-H from NVC backbone), $\delta=3.3$ (CH_2 on NVC ring), $\delta=2.3$ (CH from acrylic acid), and $\delta=3.1$ (CH_2 of Itaconic acid). These peaks are in good agreement with previously reported data and confirm the presence of functional groups in the synthesized terpolymer (26, 27). Therefore, the amount of impurities is very low and the samples are

prepared with the highest quality. As well as, the Fourier transforms infrared spectrum of the terpolymer (Figure 4) exhibits no peak at $1,620 \text{ cm}^{-1}$ due to the completion of polymerization and the disappearance of $\text{C}=\text{C}$ bonds, and indicates the absence of any unreacted monomer. Furthermore, the strong peaks at 1719, 1645 and 1540 cm^{-1} are associated with carbonyl and amide bonds that are in NVC structure.

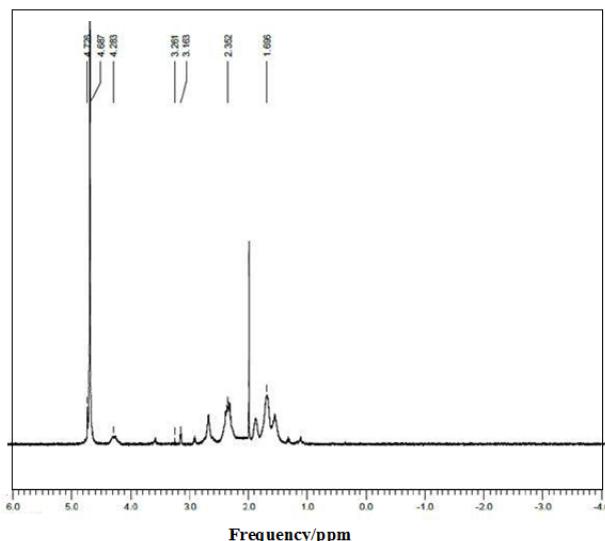


Figure 3. Proton nuclear magnetic resonance spectrum of the prepared terpolymer of AA-IA-NVC

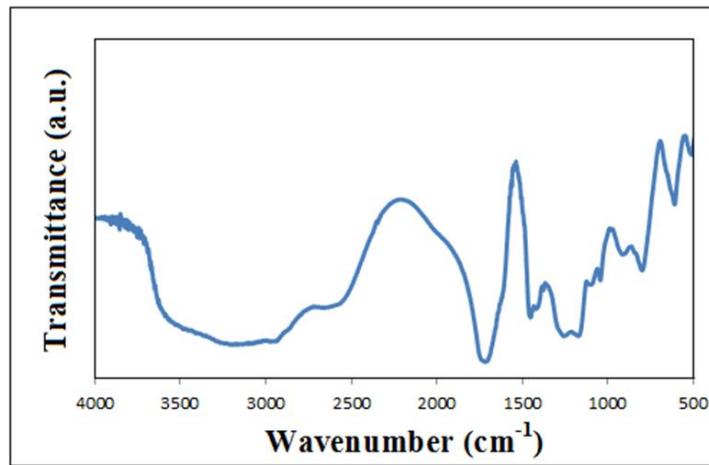


Figure 4. Fourier transforms infrared spectrum of the prepared terpolymer of AA-IA-NVC

The FS test results of the control group, 10%wt.YSZ-GIC, NVC-GIC, and 10%wt.YSZ- NVC-GIC after 24h and 1week immersion in distilled water are listed in Table I. Results were compared to the FS values of Fuji IX commercially available GIC as the control group. The FS values for 24 h (13.07 ± 0.51 MPa, 13.72 ± 0.97 MPa, and 14.44 ± 1.41 MPa) of the 10%wt.YSZ-GIC, NVC-GIC, 10%wt.YSZ-NVC-GIC groups, respectively, were higher than those of the control group (Fuji IX) ($FS=11.78\pm1.62$ MPa). However, according to statistical analysis, this increase wasn't meaningful ($P>0.05$). Furthermore, it must be mentioned that NVC had more effect on the FS property than the YSZ. The same results were achieved for 1week immersion (16.74 ± 1.17 MPa, 16.95 ± 0.91 MPa, and 19.19 ± 0.69 MPa) of the 10%wt.YSZ-GIC, NVC-GIC, 10%wt.YSZ-NVC-GIC groups, respectively, and $FS=13.94\pm0.74$ MPa for the control group. However, the difference is that according to the statistical analysis, the increase was meaningful for this soaking time ($P<0.05$). Therefore, it can be concluded that there was a direct relationship between FS values and immersion time. In other words, by elongating the soaking time, the FS increased. It stands to reason that, when maturation time grows, the polymer

molecule's cross-links with GIC's powder increases as well. In the end, the best result was for the 4th group in which the modification of powder and liquid occurred concurrently. The values of microhardness for the experimental and control samples are listed in Table II. The average microhardness values for 24 h of soaking time (61.371kg/mm^2 , 57.69 kg/mm^2 , and 63.30 kg/mm^2) of the 10%wt.YSZ-GIC, NVC-GIC, 10%wt.YSZ-NVC-GIC groups, respectively, were higher than the Fuji IX control group [Vickers hardness number (VHN)= 52.39 kg/mm^2]. Correspondingly, for 1week storage period at 37°C distilled water, there were significant differences in VHN for prepared samples as compared to the Fuji IX GIC control specimens. However, the amount of increase by adding YSZ and NVC for FS was higher than the increase for microhardness test (37% increase of FS for 10%wt.YSZ-NVC-GIC after 1 week of immersion in distilled water and 13% increment for VHN for the same group after the same amount of soaking in DI solution). Furthermore, the impact of YSZ on microhardness in both immersion interval time was higher than NVC. While the effect of NVC for improving the FS was more than YSZ.

Table I. Flexural strength test results of the GIC samples after 24 h and 1 week of storage in distilled water at 37°C (each entry is the mean value with standard deviations)

GIC group	Sample size	Mean flexural	Mean flexural
		strength (MPa)-24 h	strength (MPa)-1 week
Control group (Fuji IX)	(n=10)	11.78±1.62	13.94±0.74
10% wt. YSZ-GIC	(n=10)	13.07±0.51	16.74±1.17
NVC-GIC	(n=10)	13.72±0.97	16.95±0.91
10% wt. YSZ- NVC-GIC	(n=10)	14.44±1.41	19.19±0.69

GIC: glass ionomer cements

Table II. Microhardness test results of the GIC samples after 24 h and 1 week of storage in distilled water at 37°C (each entry is the mean value)

GIC group	Sample size	Microhardness (VHN)-24 h	Microhardness (VHN)-1 week
Control group (Fuji IX)	(n=4)	52.39	59.55
10%wt. YSZ-GIC	(n=4)	61.37	63.46
NVC-GIC	(n=4)	57.69	60.54
10%wt. YSZ- NVC-GIC	(n=4)	63.30	67.83

GIC: glass ionomer cements

Discussion

The soaking time has a pivotal role in the achievement of appropriate mechanical properties of glass ionomers. In fact, an increase in the immersion time leads to the improvement of setting reaction. To be more specific, after 24 h of soaking time, samples did not reach the final setting. However, when they were allowed to float in distilled water for 1 week, the number of cross-links between molecules of polymer and powder of GIC increased. Furthermore, it is possible that setting reaction lasts for a few days or even a few weeks because it is involved in the formation of the aluminum polysalt bridge. Aluminum has 3+ which can cause numerous cross-links in the polyacid phase resulting in a high degree of strength (4). The improvement of FS and microhardness by adding zirconia is due to the fact that zirconia is intrinsically a rigid material with high strength and stability. Therefore, adding these particles would have a positive effect on both FS and microhardness.

Additional to the innate nature of zirconia, it must be considered that nanoparticles can provide suitable particle distribution and increase the ratio of surface area to the volume of samples. This ameliorates the reactions between the polymer field and powder particles in interfaces. Nanoparticles in a composite prevent the formation of agglomerative structures. Excessive amount of agglomerative structure exerts negative effects on the sample properties. Furthermore, mixing of glass ionomers (with larger scales) and nanoparticles increase the density and compression of particles mixture. In fact, the widespread distribution of nanoparticles in larger particles results in a high density of glass and increases mechanical properties. In addition, one other reason may be related to the ionization of ZrO₂. In other words, nanoparticles of zirconia after mixing with glass ionomer liquid can be altered to ZrO²⁺ or other varieties of oxy-zirconium. These cations can make cross-links with COO⁻ like other cations of aluminofluorosilicate powder; therefore, increasing the strength. About the reason of

NVC's positive effect on the aforementioned properties, we can state that the carboxylic groups in the copolymer of GIC are often inhibited from reacting with cations due to the very close configuration in the polymer backbone (27). It is noteworthy to restate that the reaction between released Ca^{2+} and Al^{3+} ions and COO^- cause the formation of Al^{3+} and Ca^{2+} polycarboxylates. Therefore, in order to avoid this problem, NVC can be used. The NVC has a flexible lateral chain in its main backbone; consequently, it acts as a spacer and allows the greater mobility of the carboxylic acids in the main backbone (28). This mobility allows more carboxylic acid groups to participate in reactions with the glass powder and desired configuration to be achieved in the final polymer structure. Accordingly, the weak strength and toughness of glass ionomers, which could be the result of this steric hindrance, can be improved by adding NVC. Furthermore, it is assumed that the amide groups of NVC can cause an increase in hydrogen bonds, which not only increases the mechanical properties but also causes improvement in hydrophilicity.

Conclusion

In the present research, glass ionomer cement was modified with novel-NVC and YSZ and then, it was

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characterized and compared with the control group (Fuji IX commercial GIC). The mechanical properties of the resultant cement were evaluated and it showed that YSZ-NVC-GIC was a promising restorative dental material. The present study indicated that the addition of YSZ to the powder and NVC into the terpolymer had the ability to significantly enhance the mechanical strength of the glass ionomers (Microhardness and FS). The working properties of YSZ-NVC-GIC were acceptable and met the requirements of water-based cements. The microhardness and FS were increased by adding YSZ due to the zirconia's intrinsic rigidity, appropriate distribution of Nano-particles among the glass's powder, and the reaction between ZrO^{2+} and COO^- . Correspondingly, the NVC reduced the steric hindrance in the main backbone of the polymer and amended the mechanical properties. Furthermore, because of the continuous formation of poly salt bridges, the FS and microhardness increased by elongating the soaking time in the distilled water.

We have no conflicts of interest to disclose.

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