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Reinforcement of Glass Ionomer Cement by Yttrium Oxide Nanoparticle Substitution

Zuha Ayad Jaber

University of Kufa, Iraq

Abstract

Objective: To determine the surface microhardness after 24 hours of maturation effect of partial weight-based substitution of conventional GIC (glass ionomer cement) powder with Y2O3 nanoparticles. **Materials and Methods:** Four groups with 12 specimens per group were prepared. One control without any nanoparticle addition and GIC powder partially substituted with 1wt% Y2O3, 3 wt% Y2O3, and 5 wt% Y2O3 nanoparticles. The control and substitute groups had the same powder-liquid ratios overall. Specimens were prepared as per the manufacturer's instriction and transported away for storage for a duration of 24 hours. Surface microhardness was carried out on all groups with the use of a Vickers microhardness tester (200 g load, 15 sec). Intergroup comparisons were done with significance set at $p < 0.05$. **Results:** An increase in microhardness dependent on concentration was observed with increasing substitutions with nanoparticles ($p = 0.036$). **Conclusion:** Regular powder of GIC partially replaced with nanoparticles of yttrium oxide produced a significant improvement of surface microhardness at 24 h. Composition with lower proportions did not produce any significance changes.

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Email: Zuhaa.jedy@@uokufa.edu.iq

Introduction

Glass ionomer cement (GIC) is widely used in restorative dentistry as a base under restoration, luting cement, repairing permanent and deciduous teeth and in minimally invasive restauration. This widespread use is due to its ability to form strong chemical bonds and self-adhere with ease, cost, lower thermal expansion co-efficient and great fluoride releasability [1]. However, its use is somewhat limited owing to its wear resistance, brittleness and poor resistance to crack propagation [1,2]. To improve these features, the incorporation of metallic particles, microfibers, or resin have been proposed [1-3]. These modifications have however yielded mixed results in terms of their effect on fluoride release and biocompatibility [1,2]. Nanotechnology has been introduced to enhance those properties of dental materials [1]. Incorporation of 2wt% reduced graphene silver nanoparticles into conventional GIC, greatly improving its antimicrobial activity but greatly compromised its surface hardness and mechanical quality [1]. The addition of zinc oxide nanoparticles to GIC decreased the microhardness but did not show significant enhancement in the antibacterial property [1]. Halloysite nanotubes increased the hardness and wear resistance of GIC, although the fluoride release was lower [6]. Conventional GIC modified with 1 to 4 wt% forsterite nanoparticles showed high compressive strength [4]. Depending on the application, metal oxide nanoparticles differ from one another because of their preferred physicochemical model, as well as biological properties. Also, Y2O3NP is used in technical applications due to its higher dielectric constant and thermal stability [6] and have been studied for their antibacterial [7] and antioxidant [8] properties. Y2O3 is chemically stable, displays unique optoelectronic properties [9], and has a high level of hardness [10].

Nanotechnology has been used as therapeutics for the treatment of diseases, including those affecting hepatic cells [11]. Y2O3 has a high free energy of oxide formation from elemental oxide [12]. Y2O3 NPs protect cells from ROS-mediated cell death [13,14]. Several studies investigated Y2O3 antioxidant effect on different cell types. An increase of concentration of Y2O3 NPs increases the generation of ROS in cells [15]. Yttrium oxide nanoparticles (YONP) have unusual properties and potential applications [16]. The aim of this study was to evaluate the effect of the addition of YONP on the microhardness of a GIC. Conventional glass ionomer cements represent the product of an acid-base reaction between fluoroaluminosilicate glass powder and polyacrylic acid [17]. A key aspect of this setting reaction is the setting of a cross-linked polyacrylate matrix, which endows the material with adhesive properties to dental substrates and allows for the continued release of fluoride ions [18]. While chemical properties look promising, the brittleness of the structure [19] may lead to failure of a glass ionomer under occlusal loads, resulting in clinical failure [20]. Without reinforcements, it will not be suitable for clinical dentistry [21]. One way to reinforce this polyacrylate cement would be to incorporate yttrium oxide nanoparticles throughout the cement fill [22], to optimize the filler polyacrylate cement matrix and reduce multiple avenues for crack propagation [23]. Additionally, owing to the strong affinity of yttrium oxide nanoparticles towards reactivity on their surface, inclusion of these species could potentially increase the density of the interfacial transition zone, thus improving the load-bearing capacity of the cement [24,25]. Hurd et al explored the potential use of yttrium oxide nanoparticles (Y2O3 NPs) in increasing the surface hardness and compression strength of a dental material [16]. These species can also serve as bioactive materials as free radical scavengers, which can help in minimizing oxidative damage at the restoration/tooth interface [17,18]. By combining yttrium oxide with a polyalkenoate matrix, it may be possible to achieve, if not improve, the high load bearing of the material while retaining the long-term strengths offered by metal oxide-based additive for tooth replacement structures [19].

Materials and Methods

The present in vitro study employed a conventional glass ionomer luting cement (Tokuyama Tokuso Ionomer GIC Luting Cement, Tokuyama Dental Corp., Japan). Yttrium oxide (Y2O3) nanoparticles were used for powder coating. The nanoparticles were of analytical grade and were used based on weight. All the materials were weighed using the aid of a solar-powered digital analytical balance (±0.001 g) to facilitate accurate proportions in accordance with the intended percentage of substitution. Forty-eight disc-shaped specimens were prepared (n = 12/group) and randomized into four groups, thus:

Group I (Control): Conventional GIC without substitutions.

Group II: 1 wt% Y2O3 nanoparticles substitution (i.e., 0.01 g Y2O3 + 0.99 g for GIC powder).

Group III: 3 wt% Y2O3 nanoparticles substitution (i.e., 0.03 g Y2O3 + 0.97 g for GIC powder).

Group IV: 5 wt% Y2O3 nanoparticles substitution (i.e., 0.05 g Y2O3 + 0.95 g for GIC powder).

The substitutions performed in these experimental groups made sure that an equivalent proportion by weight of yttrium oxide nanoparticles were substituted for the GIC powder to eliminate variation in total powder mass and preserved the powder–liquid ratio recommended by the manufacturer. The nanoparticle-modified powders were first dry-mixed to promote uniform distribution and subsequently blended using an amalgamator to enhance homogeneity and minimize particle agglomeration.

The cement was mixed in accordance with the manufacturer's instructions using two drops of liquid to each scoop of powder. The mixture was spatulated on a glass slab with a stainless-steel cement spatula to an even consistency. The mixture was placed in a 10 mm diameter, 2.5 mm thick cylindrical stainless-steel mold with a Mylar strip on the bottom and another on top of the mixture, and a glass slide was placed over the material with very gentle pressure to extrude excess material in order to form flat uniform surfaces, and excess cement was removed.

Specimens were allowed to gain initial set at room temperature for a period of 20 min before being demoulded. Immediately after demoulding, and prior to performing test, all surfaces were inspected and any small irregularities carefully removed using suitably fine-grit silicon carbide abrasive paper under controlled conditions.

All specimens were then stored at room temperature for 24 hours to allow completion of the early maturation phase prior to hardness testing.

Surface microhardness was measured with a Vickers microhardness testing machine (INDENTEC ZHF-, Indentec Hardness Testing, Brierley Hill, United Kingdom) that was previously calibrated. The specimen was placed perpendicular to the indenter and locked into a testing stage.

An excess weight of 200 g was placed centrally for 15 s to produce a diamond-shaped indentation in the specimen surface.

Three indentations were made for each specimen, at least 100 µm apart and from the margin of the specimen (to avoid stress interaction), and the diagonals of each indentation were measured using the integrated optical microscope of the testing unit.

The Vickers hardness number (VHN) was calculated automatically by the testing software according to standard methodology. The mean value of the three measurements was recorded as the final microhardness value for each specimen.

The statistical software package used was SPSS (Statistical Package for Social Sciences) version 18. One-way analysis of variance (ANOVA) test was used to compare normally distributed numeric variables by groups with Tukey's post hoc test. $P<0.05$ were considered as significant.

Results

The mean values and standard deviations for each experimental group are summarized in Table 1 and Figure 1. The highest mean microhardness was observed in the group of 5wt%. On the contrary, the control group demonstrated the lowest mean, with statistically significant difference between them (P=0.036). The microhardness values recorded direct proportion with the concentration of the added YONP. The microhardness values followed this trend: 5wt% > 3wt% > 1wt% > control.

Table 1. The mean and standard deviation of cement microhardness averages of each group.

Groups (n=12)	Mean (VHN)	Standard deviation
G1(control)	55.47	3.06
G2(1wt%)	56.81	3.95
G3(3wt%)	58.49	3.79
G4(5wt%)	59.73	3.99

Discussion

This study demonstrated a statistically significant enhancement in the Vickers microhardness of glass ionomer cement (GIC) following partial substitution of the conventional glass powder with 5 wt% yttrium oxide (Y_2O_3) nanoparticles. Although incremental increases were observed at 1% and 3% substitutions, statistical significance emerged only at 5% (p = 0.036), indicating a concentration-dependent reinforcement with a likely threshold effect.

The reason there was an increase in surface hardness could be due to the yttrium oxide (Y2O3) nanoparticles, which are ceramic metal oxides that exhibit strong bonding, high lattice

energy, and a stable cubic crystal structure [26, 27] and therefore have high stiffness or elastic modulus and resistance to plastic deformation [26]. When these rigid oxide nanoparticles exist in the dental matrix, they create overlaps and/or bridges between their respective particle surfaces that inhibit matrix deformation and assist with stress resistance. As a result, this behaviour of these individual hard nanophases contributes to an increase in surface hardness.

Yttrium oxide (Y_2O_3) is an inflexible ceramic that holds itself together with strong bonding, high lattice energy, and high elastic modulus [26,27]. When used in a glass ionomer cement (GIC) as a rigid reinforcing nano filler in the matrix of the cement, it helps to resist indentation and plastic deformation during hardness tests (Vickers, etc.). The load applied, is in part transferred from the relatively soft polysaccharide matrix to the relatively hard oxide particles, which enhances the hardness. Nanoparticles are very small (<100 nm) and may fit in between the individual fluoroaluminosilicate glass particles of GIC. This leads to a better packing density of individual particles; reduced presence of micro-voids and internal porosity; and production of a denser cement structure. Such a denser microstructure will resist indentation of its surface, resulting in increased hardness. The setting reaction occurs through an acid–base reaction between polyacrylic acid and the fluoroaluminosilicate glass, forming a cross-linked polysalt. If these nanoparticles are incorporated in a dental matrix (e.g., PMMA, composite, or GIC), it is expected that the rigid oxide particles will resist indentation and the load will be transferred through the softer matrix, thus increasing hardness.

Microhardness in conventional GIC is dependent on the integrity of the polyalkenoate salt matrix, how well the filler is packed, and the quality of the filler–matrix interface [28,29]. As these specimens were stored for 24 hours before measurement, these values pertain to an early post-maturation phase, and further ionic crosslinking would likely be served to help reinforce the matrix [29]. Thus, the improvements seen here at a 5% substitution are likely indicative not merely of a superficial change to the microstructure, but of a change in the load transfer properties of the cement itself.

The data indicates Y2O3 nanoparticles behaved as a mechanically efficient reinforcing phase replacing a part of the normal glass fraction. Being nanoparticles means that they can occupy nooks and crannies between larger glass particles, which is likely to increase packing and reduce microvoids. Greater packing is generally associated with increased hardness and resistance to plastic deformation in nano-modified GIC systems [30,31]. That 1% and 3% substitutions are not statistically significant indicates that the network of nanoparticles was not dense enough to constitute effective reinforcement. In particulate composition, mechanical modification does not tend to occur until a certain critical filler concentration is reached, one that allows effective stress through the matrix-particle-distributed interface [32]. Below this, it's as if the nanoparticles are 'lonely', as it were - like an inclusion that has a minimal bearing on the remainder of the bulk [33].

However, the substitution strategy also involves a degree of reduction of the amount of reactive glass per unit mass of formative GIC based on the requirement of that glass in the concurrent acid–base setting reaction of GIC [29], and in theory over-substitution could impair matrix formation, long term mechanical "set" [34]; in previous studies of teeth tracked into decay using nano-zirconia, nano hydroxypatite and bioactive glass, moderate levels of substitution by the nanoparticles was found to be synergistic, leading to increases in hardness, but more extensive substitution led in some cases to reduced rates of setting or mechanically defusing them into the GIC [34]. The present data suggest that 5% is a sort of threshold for transitional purposes, the benefits slight outweighing diminished content of reactive glass.

On a somewhat less optimistic note, however, increased surface microhardness may relate to improved wear resistance and surface degradation in conditions of stress for certain restorations in the clinical setting [28], whilst alone microhardness does not tell one anything about aspects of the material's compressive strength, flexural strength, toughness or resistance to fatigue and degradation with time by hydrolytic attack [35]. To then extrapolate findings to clinical superiority must be qualified until all mechanical and ageing data are in.

Conclusion

In this study, Y2O3 nanomaterials were mixed into traditional glass ionomer materials to examine the effects of varying Y2O3 ratios (up to 50 wt% mass replacement of the GIC) on surface hardness. Glass ionomers with increasing levels of Y2O3 produced a significantly increased surface microhardness compared to the control group at 24 hours with a replacement level of 5 wt%, but at lower replacement levels of Y2O3 (1 wt% and 3 wt%) did not produce significantly increased microhardness compared to the control group The liquid-to-powder ratios were held constant. However, the study used powders instead of adding powder to a liquid when calculating enhancements due to using nano-sized yttrium oxide as a filler. Thus, any enhancements seen with nano-sized yttrium oxide are not due to increased filler content. Therefore, the 5 wt% value may be a source of positive benefits prior to a detrimental effect on resin composition.

Though the microhardness of glass ionomer cement (GIC) improved due to the addition of Y_2O_3 NP, microhardness does not provide a complete picture of how well GIC performs in an environment similar to that of a filler material. Further investigations into GIC's flexural strength, fracture toughness, wear resistance, curing properties, and long-term stability are necessary to evaluate the clinical performance with the addition of Y_2O_3 NP.

In summary, within a specific concentration range, the addition of 5 wt% Y_2O_3 nanoparticles is a practical method for improving the early surface hardness of glass ionomer cement.

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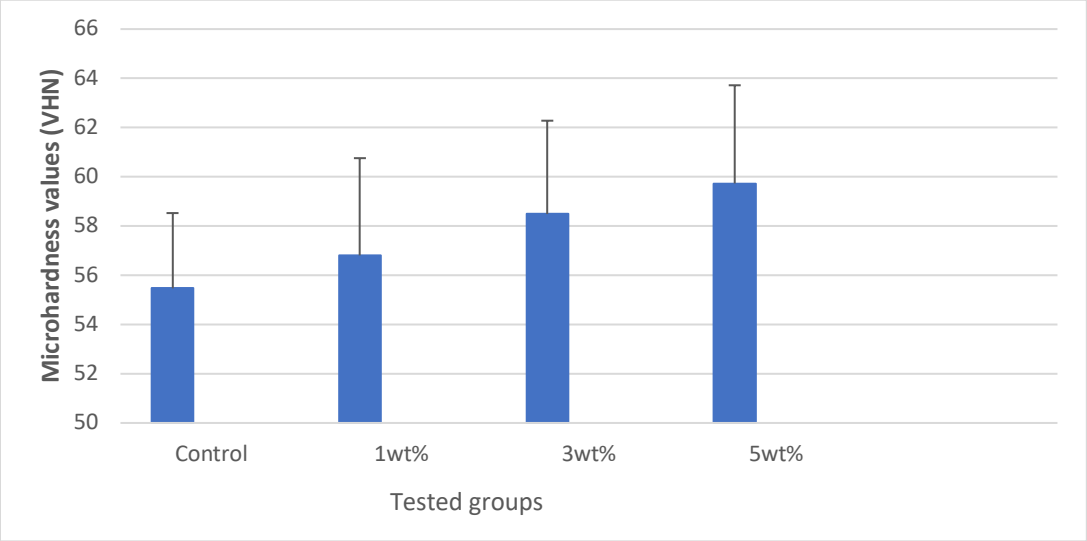

Figure 1. Mean microhardness values of the study groups.