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Development of New Al_2O_3/TiO_2 Reinforced Glass-Ionomer Cements (GICs) Nano-Composites

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ABSTRACT

Titania and alumina nano particles were used for the reinforcement of commercial glass ionomer cement. The mechanical properties of TiO_2–GICs were investigated with different titania concentrations. GC Fuji II LC (Improved) (GC America) acted as a standard for comparison. Results showed that the mechanical properties generally increased with increasing amount of TiO_2 nano particles until 5 wt.% and then decrease when further amount of titania concentrations from 5 to 10 wt.% was applied. Furthermore, by using 5 wt.% Al_2O_3 nano powders no significant differences in hardness and mechanical strengths were observed in comparison with 5 wt.% TiO_2-GICs. The compressive and diametral tensile strengths of Al_2O_3/TiO_2–GICs was, however, significantly greater than another GICs due to the better particle size distribution and interfacial bonding between the particles and the matrix.
KEYWORDS: Glass ionomer cements; Al_2O_3; TiO_2; Nano powders; Mechanical properties.

1- INTRODUCTION

Glass-ionomer cement (GIC) was initially emerged as a restorative material in early 1970s, by Wilson et al. [1]. These materials can be derived from the introduction of a powder (usually a fluoroaluminosilicate glass) into a liquid (polyalkenoic acid such as polyacrylic acid), combined to form a plastic mass, and then followed by a transformation to a stiff solid, namely a specific type of cement, due to an acid-base reaction between the components [2].

These kinds of cements present some great advantages such as translucent, good chemical bonding to the tooth structure, biocompatibility, anticariogenic action (due to fluoride release), low value of thermal expansion coefficient and stability in an aqueous environment; which ascertain these materials as promising alternatives for dentistry, artificial ear ossicles, and bone substitute plates for craniofacial reconstruction [3]. Nevertheless, the brittle nature and inferior mechanical strength of these materials are recognized as the most significant shortcoming of conventional GICs [4].

Since the invention of GIC, noteworthy efforts have been made to further improvements and enhancements of their physical and mechanical properties. For instance, addition of amalgam alloys and stainless steel powders as well as different fillers, such as silver-cermets, carbon and alumino-silicate fibers and, incorporation of hydroxyapatite into glass-polyalkenoate have been taken into account to overcome the aforementioned drawbacks of GICs [5-7]. More recently, scientists have come up with the idea of introducing nano particles such as TiO_2 nano tubes, nano hydroxyapatite, nano-fluoroapatite into the GIC matrix to enhance their mechanical strength due to their high surface area to volume ratio which enhances their interfacial interaction with the cement matrix [8-11].

In the present investigation, both nano alumina and titania powders have been utilized for reinforcement of GIC bodies. The aim of this study was to investigate the mechanical properties (microhardness, compressive and diametral tensile strength) of new nano alumina and/or titania reinforced GICs. Fuji II LC samples have been applied as standard references for comparison.

2- Experimental procedure

TiO_2 nanopowder (P25, Degussa Co., Frankfurt, Germany) with a particle size ranging from 11 to 27 nm as well as Al_2O_3 nanocrystalline powder (Taimicron TMDAR, Japan) with primary particle size around 100 nm were used as feed-stocks. Commercial grade of glass powder and liquid (Fuji II LC GC America) have been utilized in the experiments to prepare GIC samples.

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Titania nanopowders were firstly added to Fuji II LC powder in different concentrations from 1 to 10 wt.% The mixture was subsequently introduced to the liquid and then poured into the molds. Dimensions of the applied molds for compressive and diametral tensile strengths were 8mm and 3mm in height and 4mm and 6mm in diameter, respectively. The specimen surfaces were then irradiated with a visible-light curing unit (Palm Light TM, Cao Group, USA), after which, the specimens were removed from the molds and subjected to mechanical measurement tests. The compression and diametral-compression tests were carried out in accordance with British standards 6039–1981 for dental GICs [12]. An Instron 4206 tester was used with a 100 kN load cell and a cross-head speed of 1.0 mm/min. Vickers microhardness was measured by indentation at load of 100 g and dwell time of 20 s. Vickers impressions were conducted on the surface of the previously polished samples. After obtaining the optimum concentration of TiO_2 powder, Al_2O_3 and Al_2O_3/TiO_2 GIC samples were prepared in 5wt.% and tested by aforementioned procedure. At least five specimens, for each data point, were applied to calculate the mean value. The fracture surfaces of GICs were observed using a scanning electron microscopy (SEM, JEOL 5410, Japan).

3- RESULTS AND DISCUSSION

Table 1 demonstrated the mean compressive strength, diametrical tensile strength and microhardness values of different GIC samples. Mechanical properties of Fuji II LC samples are also included in table 1, as a comparison. The addition of 1 %wt. of TiO_2 concentration has not been observed to result in significant variation in the value of compressive strength neither for titania added specimens nor Fuji II LC samples. While increasing the amount of TiO_2 powder from 1 to 3 wt.% has led to a moderate increment of compressive strength, the specimens containing 5 wt.% TiO_2 have demonstrated more highlighted enhancement in the value of compressive strength as well as a higher diametrical tensile strength and microhardness values compared to the others. Likewise, one can hardly find any noteworthy improvement in microhardness and diametrical tensile strength of GIC samples, resulted from the addition of titania fraction from 1 to 3 wt.%; though, these parameters display an obvious ascsent in 5 wt.% TiO_2-GIC specimens.

Table 1. Compressive strength, diametral tensile strength and microhardness of different glass ionomer cements.

Samples	Compressive strength (MPa)		Diametral tensile strength (MPa)		Microhardness (VHN)	
	amount	SD*	amount	SD	amount	SD
Fuji II LC	117.43	±13.16	23.72	±2.69	58.03	±6.36
1wt.% TiO_2	125.63	±20.39	25.75	±1.83	69.85	±2.85
3wt.% TiO_2	147.93	±22.88	24.21	±3.57	74.16	±2.54
5wt.% TiO_2	185.17	±18.14	31.01	±2.66	83.16	±1.45
10wt.% TiO_2	140.93	±21.07	26.13	±2.96	103.26	±7.42
5wt.% Al_2O_3	190.57	±22.94	31.24	±1.32	96.23	±4.79
5wt.% TiO_2/ Al_2O_3	202.56	±12.78	33.31	±0.92	109.61	±3.26

In contrast, the increasing trend for the values of the above mechanical properties was not observed to be sustained trough increasing the amount of TiO_2 powder to 10wt.%, while compressive and diametrical tensile strengths of these samples were found to have undergone a degrading tendency unlike their microhardness values which are still higher than the specimens with lower fraction of TiO_2. The low compressive and diametrical tensile strengths values of 10 wt.% titania samples are due to the poor interfacial bonding between TiO_2 and the matrix. In other words, because of the very small particle size and large surface area of the nano sized TiO_2; there may be inadequate ionomer to hold the large amount of TiO_2 nano particles (10 wt.%) effectively [9]. Though, as titania nano particle is the harder phase compared to GIC’s powder and polymer, increasing titania powder to 10wt.% has led to the enhancement of microhardness values. Khaled et al. [9] reported that the cement reinforced with 1 wt.% functionalized TiO_2 nano tubes demonstrated considerably superior K_{IC} values, about 73% higher than that of control cement. However, there is a decreasing trend of K_{IC} values at loading over 1 wt.% TiO_2 nano tubes. Therefore, the increase in the powder concentration from 5 to 10 wt.% would not only expose no positive contribution to generate improved mechanical properties, but also has pose detrimental effects to degrade mechanical properties of the specimens. As a result, the optimum as well as the maximum amount for the introduction of Al_2O_3 and Al_2O_3/TiO_2 nano powders were determined to be 5wt.% concentration.

What can be postulated from table 1 is the absence of any significant difference in the compressive and diametrical tensile strength for the composites prepared by 5 wt.% Al_2O_3 compared to those of fabricated by TiO_2 nano powders. On the other hand, The Al_2O_3-GIC composites demonstrated a fairly higher microhardness values than TiO_2-GIC composites, stemming from the attributes presented by alumina for deriving higher microhardness as well as mechanical strength as a harder phase compared to titania particles [9]. Furthermore, significant differences in mechanical properties of Al_2O_3/TiO_2-GICs can be observed, and the composites fabricated by both Al_2O_3 and TiO_2 nano powders have displayed the highest mechanical strength as well as microhardness values. The microhardness of the Al_2O_3/TiO_2-GICs is about 88% higher than that of Fuji II LC. In addition, the compressive and diametrical tensile strengths of Al_2O_3/TiO_2-GICs are near 73 and 44% higher than those of Fuji II LC, respectively.

The mechanical strength is found to be dependent upon the particle size and particle size distribution of composites [13]. As a result, the composition of glass particles with a larger particle size and Al_2O_3/TiO_2 particles with nano size particles has led to a wide distribution of the particle size. This would allow a high packing density of the mixed particles within the glass ionomer matrix [13]. On the other hand, the wide distribution of nano sized particles within the larger glass particles can generate a high packing density of glass ionomer cement with an enhanced mechanical strength.

One can observe the cross-sectional view of Fuji II LC samples in Fig. 1(a). Presence of some large voids in Fuji II LC samples must be associated with the air bubbles trapped inside GIC through transferring of the mixed cement into the mold cavity. The presence of cracks and voids on the fracture surface of GICs shed light on this fact that internal defects and feebleness of the cements have been probably owing to the weakness at the glass particle-matrix interface, resulting in the poor mechanical properties of the GICs [2, 13].

Fig. 1(b) illustrates the fracture surface of 5 wt.% TiO_2 contained samples. As can be observed in the figure, the specimens entail an adequate mix of both large and small sized particles within the matrix. Very low amount of voids due to sample preparation are found along the cross-section in comparison with Fuji II LC sample (Figure 1(a)). This suggests that TiO_2 nano particles improve bonding effects between the particles and the matrix. Likewise, Fig. 1(c) and (d) show fracture surface of 10 wt.% TiO_2 and 5 wt.% Al_2O_3/TiO_2 GICs, respectively. As can be seen, presence of some voids in the fracture surface of 10 wt.% TiO_2-GIC confirms the poor mechanical properties of this cement. However, lower amount of voids as well as good distribution of nano powders in Al_2O_3/TiO_2-GIC can result in the highest amount of mechanical properties.

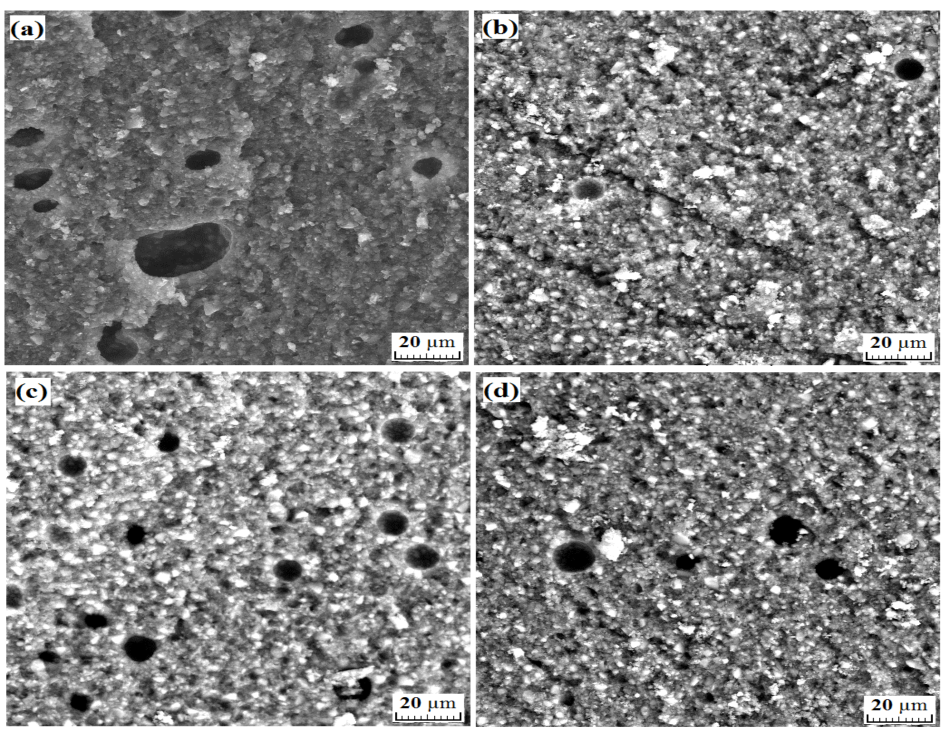

Figure 1. SEM micrograph from fracture surface of GIC samples: (a) Fuji II LC, (b) 5 wt.% Tio_2, (c) 10 wt.% TiO_2 and (d) 5 wt.% Al_2O_3/TiO_2 (in back scatter mode).

In addition to particle size and particle size distribution, the cross-linking formation during setting plays an important role on the final mechanical properties of the cements [5, 13]. An acid-base reaction occurs during the setting procedure and forms a salt hydrogel, acting as the binding element in matrix within which the glass plays a reinforcing role. Releasing metal ions can take place upon the introduction of acid into the powders and the released metal ions operate as cross-linking species, allowing the formation of stable cement. Gu et al. [5] reported that the low strength values of Miracle Mix (MM) samples can be attributed to the metal ions which cannot be leached from the amalgam alloy particles to form the cross-linking. They believe that poor interfacial bonding between the amalgam alloy particles and the matrix could be proposed as a promising evidence to verify the aforementioned phenomenon. They also state that better interfacial bonding between the particles and the matrix; propose that the aluminum and zirconium ions may have reacted with the Polyacrylic acid, forming the cross-linking in the YSZ-GIC samples [5]. Similarly, it seems that good interfacial bonding as well as cross-linking between Al_2O_3/TiO_2 nano particles and the cement matrix, resulted from the formation of aluminum salt bridges among trivalent aluminum ions in the glass as well as aluminum and titanium ions from the nano powders, provides the final strength of Al_2O_3/TiO_2-GICs. Thereby, this may present another reason for the increase of mechanical properties in GICs as a result of applying Al_2O_3/TiO_2 nano powders. However, ionic interactions and ionic bonding between the reinforcing particles (Al_2O_3 and/or TiO_2) as well as the polyacrylic acid seem to be in need of additional investigations.

4- Conclusion

As reflected in the above experimental results, Al_2O_3 and TiO_2 nano powders can be employed to manufacture advanced GIC composites, since a small loading of these nano particles is capable of deriving a significant enhancement in mechanical properties of the resulting composites, as well as maintaining biocompatibility and aesthetic properties of the base cement.

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