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GLASS IONOMER CEMENT MECHANICAL PROPERTIES ENHANCEMENT USING HYDROXYAPATITE MICRO AND NANO PARTICLES

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ABSTRACT

The aim of this study was to evaluate the effect of adding micro and nano particles of hydroxyapatite (HA) to commercially glass ionomer cement self-cure GIC restorative powder. Compressive strength, biaxial flexural strength, Microhardness and wear rate were investigated for the prepared samples. These additions enhance the mechanical properties of the GIC except the wear rate value. The maximum compression strength was 110 MPa with 7wt% HA micro particle, while 5wt% nanoparticles gives highest Microhardness and biaxial flexural strength, 76.84 VHN and 37.096MPa respectively. On other hand the wear rate were increased when HA particles were added.

Keywords: ionomer, microhardness, biaxial strength, self-cure glass.

1. INTRODUCTION

The glass ionomer cement (GIC) is water-based materials that set by an acid-powder ratio of special composition where action between apolyalkenoic acid and a fluroalumino silicate glass to form cements when mixed together, putty-like pastes are formed which set to hard translucent substances within 2-10 minutes. GIC were developed logically from the dental silicate cement which has been widely used in clinical applications since its invention in the early years of this century [1, 2]. Nevertheless, GIC are brittle and have poor mechanical properties, such as low fracture toughness, fracture strength and wear resistance. These are the main disadvantages, which limit their range of use as a filling material in dentistry [3]. Numbers of researches were tried to enhance its mechanical properties by making various aspects. The main field was adding components that are in the forcing phase such as metal particles, fibers and ceramics [4, 5]. The exceptional biological behavior of hydroxyapatite and the crystal structure similarity to human dental structure, make the researchers tried study the effect of adding HA on the GIC behavior [6, 7]. The purpose of the present study was to evaluate the effects of adding (3, 5, 7) wt. % of HA (micro and nano particles size) into conventional GIC and study its effect on the mechanical properties.

2. EXPERIMENTAL WORK

2.1 Materials

A commercially Riva self-cure glass ionomer cement shade A1 from SDI limited (Australia) was used as a base material and Hydroxyapatite ($HCa_5O_{13}P$) micro and Nano particles were used as an additive materials. The average diameters were 24µm, 26µm and 40 nm for GIC and additive micro and nano particles respectively. Both additives purity were 99%.

2.2 Sample preparation

Before preparation of samples, the GIC and micro particles powder were examined by EDX (Sputter Coater S150A, Japan) analysis was used to characterize the elements composition of used powders (Table-1).

Table-1. EDX analysis of powders.

GIC			HA micro particles		HA nanoparticles	
Element	**Compound**	**wt.%**	**Element**	**wt.%**	**Element**	**wt.%**
Silicon	SiO_2	55.99	Calcium	75.98	calcium	72.18
Aluminum	Al_2O_3	31.33	Phosphorus	19.95	Phosphorus	21.88
Phosphorus	P_2O_5	7.70	Carbon	2.12	Carbon	4.01
Fluorine	F	3.61	Oxygen	1.14	Oxygen	1.93
Sodium	Na_2O	1.35				
Oxygen	O_2	2.02x				
Total		100	Total	100		100

Seven different samples in total were prepared; one represents the as it is material while the others represent the base materials with the additions of (3, 5 and 7) wt. % from both micro and nano particles HA.

A B303, monobloc inside, weighing technology balance with three digits accuracy was used to prepare the mixed powders. Then these mixtures were stored in glass cuvettes to prevent particles stacking on the wall of the tube. Later a Tube Roller Mixer machine was used for homogenous powders mixing the process was done with 60 rpm for two hours. The combination powder was mixed with the aqueous solution (hardener liquid) in a ratio of one powder plastic spatula to one drop of hardener liquid according to the manufacturer instructions. The paste was placed into stainless steel molds and then covered with glass slides form both sides to obtain a relatively flat and smooth surface. Clamped the glass for couple of minutes and left it in an incubator at 37 °C for five minutes to set and harden. Finally the samples were removed from the mold and stored at room condition until the samples investigated [8, 9].

2.3 Compression strength and Microhardness test samples

Three cylindrical stainless steel mold specimens (5 mm dia. and 10 mm height) were prepared for each group of materials according to ISO 9917-1. The compressive strength (MPa), Cs, of the specimens was characterized using Testometric AX M500-25kN computerized system with a crosshead speed of 0.25 mm/min (Figure-1). Other same shape specimens were used for Microhardness characterizations using Q-Time digital Microhardness tester.

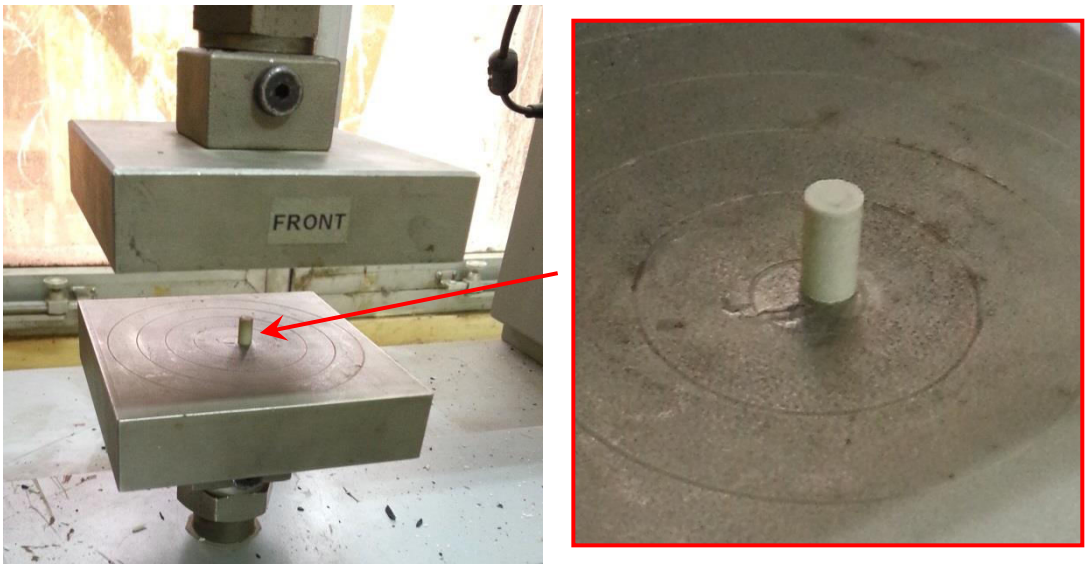

Figure-1. The compression sample on the testometric holder and a magnified image for the sample.

2.4 Biaxial flexural strength test samples

The biaxial flexural strength (BFS) test was performed by using the piston-on-three ball technique in Testometric AX M500-25kN computerized system. A holder with three stainless steel balls of 3.2 mm dia. were placed on a 10 mm dia. circle with equidistant from each other surrounded by a ring of 14 mm dia. and 2 mm height to prevent sample movement. Three disks specimens were prepared from each sample in a 14 mm inner dia. stainless steel ring and 1.25 mm height. The mixed paste was placed in the ring till set then placed in room condition. Day after, the specimen were placed on the holder and the load was applied vertically by a 1.2 mm diameter flat surface piston with a crosshead speed of 0.1 mm/min, Figure-2. The system software record each specimen fracture load continually then the following equations were used to calculate the BFS value [10]:

$$S=\frac{-0.2387(X-Y)}{d^2} \quad (1)$$

X and Y were determined as following:

$$X = (1+v)ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{1-v}{2}\right]\left(\frac{r_2}{r_3}\right)^2 \quad (2)$$

$$Y = (1+v)[1+ln\left(\frac{r_1}{r_3}\right)^2] + (1-v)\left(\frac{r_1}{r_3}\right)^2 \quad (3)$$

Where S is biaxial flexural strength (MPa), P fracture load (N),d specimen disk thickness at fracture origin (mm), v Poisson's ratio (0.25), $r_1$radius of the support circle, $r_2$radius of the loaded area and $r_3$specimenradius.

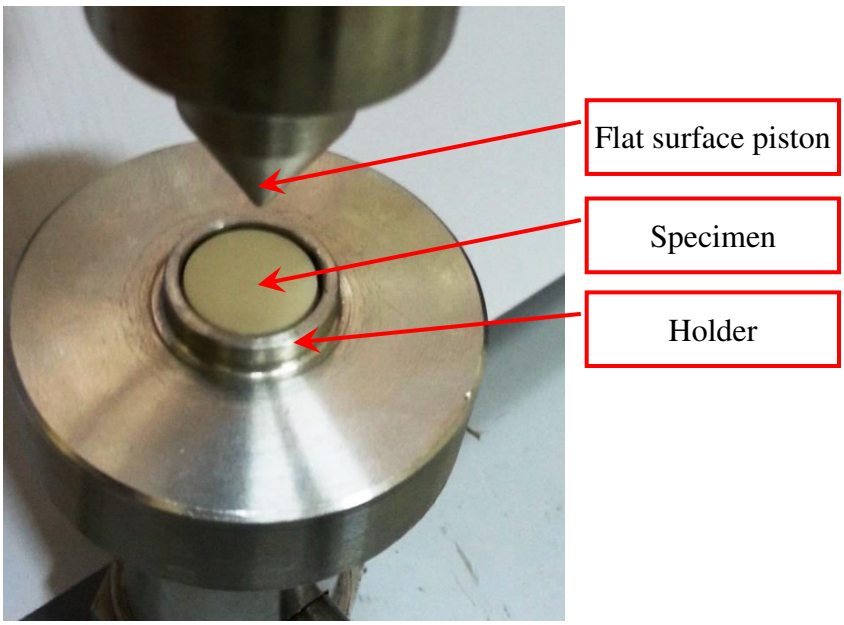

Figure-2. The BFS sample, holder and psiton.

2.5 Wear rate losses determination

Pin on disc technique were used to measure the materials wear rate (WR) according to ASTM G99 [11]. The apparatus was designed to provide continuous sliding contact between the sample and a stainless steel disk. The specimen was held in contact with the substrate surface using one kilogram load rotated at constant rate of 480 rpm. The length and duration of each sliding stroke was approximately 9.5 cm and 0.125s. The total number of revolutions for each WR test was 720 cycles and this was counted digitally. The disk surface was cleaned before and after each sample investigation to remove any contaminations. Three specimens of each material were tested to obtain the mean value. WR in average losses weight was determined according to[12]:

$$W.R. = (M_1-M_2)/\omega rt \quad (4)$$

Where: W.R. Wear rate (g/m), M_1-M_2 mass losses, ω disk rotating speed (rpm), r sample radius and t slipping time (min)

3. RESULTS AND DISCUSSIONS

3.1 Compression strength test

Generally the addition of HA micro particles increased the compressive strength significantly as compared with the control samples, as Figure-3 shows, while the adding of more than 4 wt.% of HA nanoparticles decreases the compression strength.

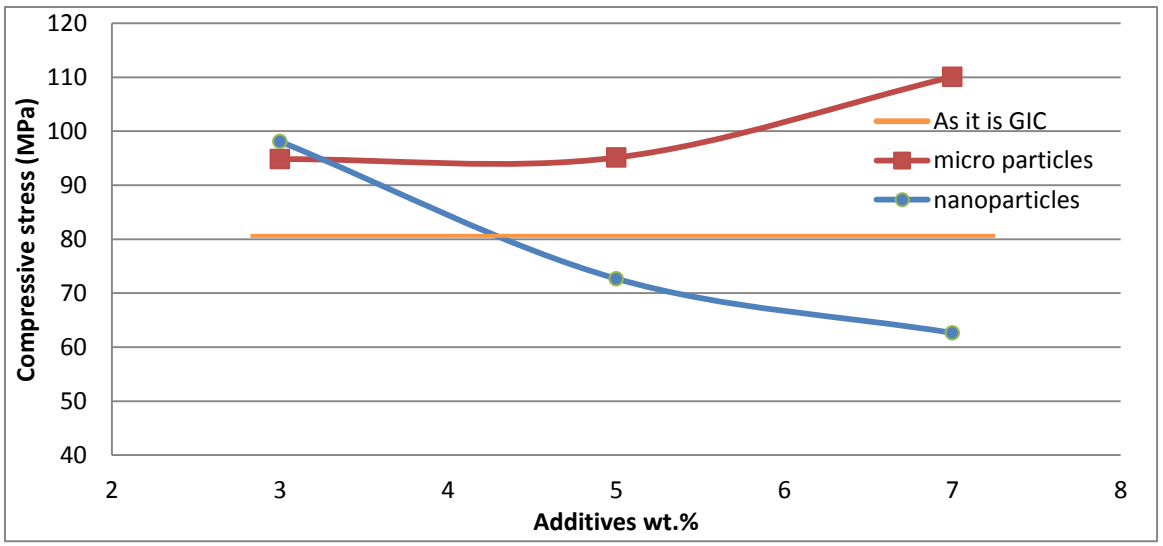

Figure-3. The relation between compressive stress (mean value) and HA additive wt%.

3.2. Surface microhardness

Both micro and nano particles of HA enhance the surface microhardness of the GIC (Figure-4), the best values were with 5%wt. nano and micro particles addition with 78 and 65 VH respectively. The more and less addition of 5% gives less hardness values.

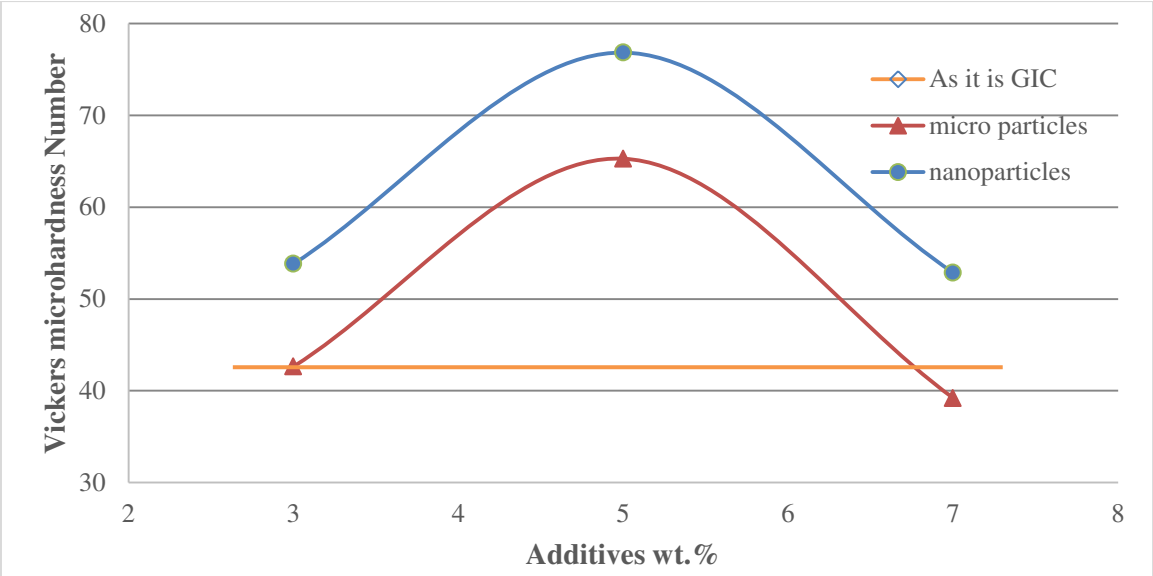

Figure-4. The relation between Vickers microhardness number (mean value) and HA additives wt%.

3.3 Biaxial flexural strength results test

The nanoparticles increased the BFS more than the micro particles of HA (Figure-5), but also the best values were for the addition of 5wt%as comparing each particles size separately. On other hand nanoparticles gives higher results than micro.

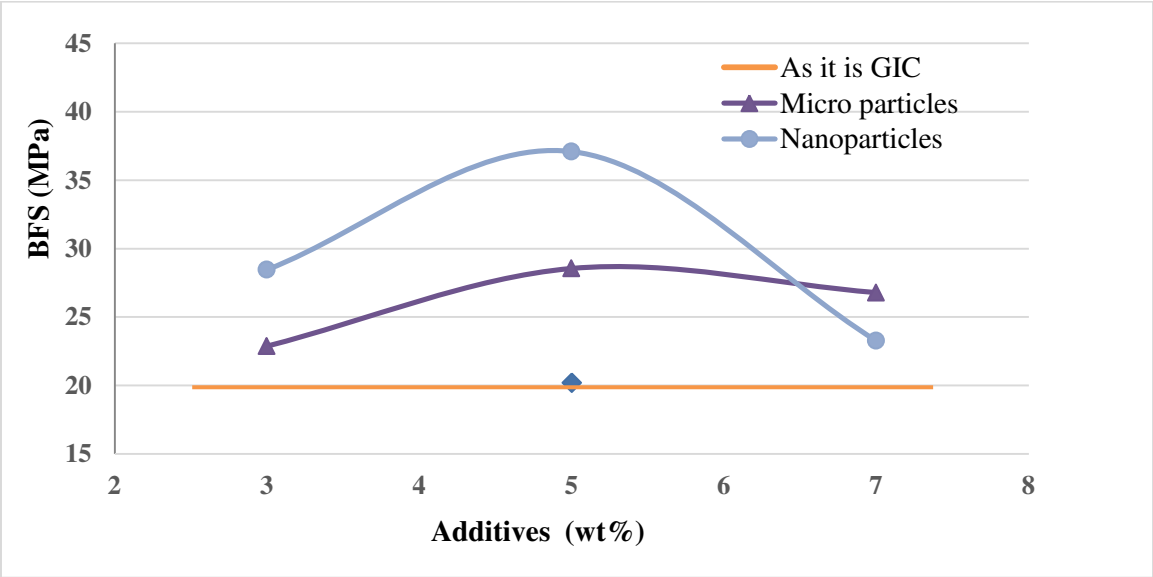

Figure-5. The relation between Biaxial flexural strength (mean value) and HA additives wt%.

3.4 Wear rate

On the contrary the addition of HA particles increase the wear rate losses (Figure-6), which means worse results than the pure GIC samples. The 5wt% of micro particles gives the less value of wear rate (0.001080 mg/m) as compared with (0.0009 mg/m) for the control sample.

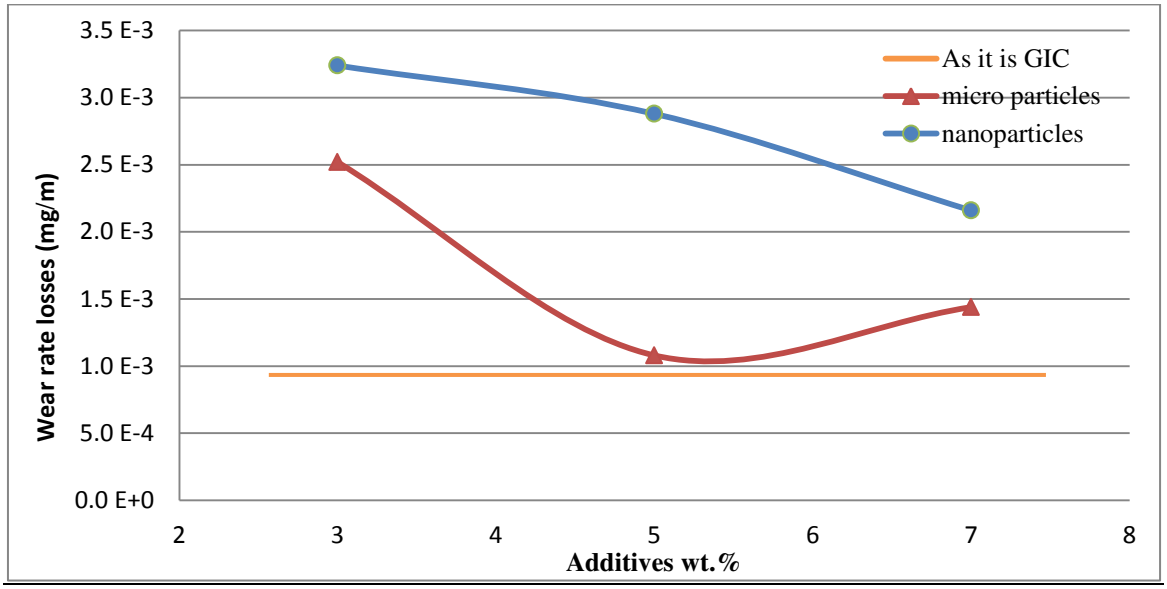

Figure-6. The relation between Wear rate losses (mean value) and HA additives wt%.

4. CONCLUSIONS

In general the addition of HA particles enhance the mechanical properties of the GIC, except the wear rate. These results were expected due to the ceramic behavior of HA as compared with the glass of GIC. The 5wt% gives the best ratio of additions among the range of (3 -7) wt%. On other hand Nanoparticles HA give better results than micro particles for both BFS and Microhardness while the results were reversed with compression strength and wear rate losses.

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