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Mechanical Properties Enhancement of Conventional Glass Ionomer Cement by Adding Zirconium Oxide Micro and Nanoparticles

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ABSTRACT

The aim of this work is to enhance the mechanical properties of the glass ionomer cement GIC (dental materials) by adding Zirconium Oxide ZrO_2 in both micro and nano particles. GIC were mixed with (3, 5 and 7) wt% of both ZrO_2 micro and nanoparticles separately. Compressive strength (CS), biaxial flexural strength (BFS), Vickers Microhardness (VH) and wear rate losses (WR) were investigated. The maximum compression strength was 122.31 MPa with 5 wt. % ZrO_2 micro particle, while 3wt% nanoparticles give highest Microhardness and biaxial flexural strength of 88.8 VHN and 35.79 MPa respectively. The minimum wear rate losses were 3.776µg/m with 7 wt. % ZrO_2 nanoparticle. GIC-containing ZrO_2 micro and nanoparticles is a promising restorative material with improved mechanical properties expect wear rate losses.

Keywords: Glass ionomer cement, ZrO_2 micro and nanoparticles, Mechanical properties, Riva self-cure.

تحسين الخواص الميكانيكية لمادة العزل المزججة الاسمنتية التقليدية باضافة دقائق اوكسيد الزركنيوم المايكروية والنانوية

الخلاصة

ان الهدف من هذه الدراسة هو تحسين الخواص الميكانيكية لمادة العزل المزججة الاسمنتية (المستخدمة كحشوات للاسنان) من خلال اضافة دقائق اوكسيد الزركنيوم المايكروية و النانوية. تم خلط مادة الاسمنت الزجاجية مع دقائق اوكسيد الزركنيوم بالنسب (3 و5 و7)% وزنية ومن كل حجم على حدة. أجريت اختبارات قوة تحمل الضغط، قوة المرونة ثنائية محور الحمل، الصلادة السطحية المايكروية بالاضافة الى معدل تاكل المادة. اظهرت النتائج ان اعلى قيمة لقوة تحمل الضغط كانت 122.31 ميكاباسكال لنسبة ال 5% للحجم المايكروي بينما اعلى قيم لقوة المرونه ثنائية محور الحمل و الصلادة السطحية المايكروية 88.8 رقم صلادة فيكرز و 35.79 ميكاباسكال على التوالي للنسبة 3% النانوية. في حين ان اقل معدل للتاكل كان لنسبة ال 7% النانوية والتي كانت 3.776 مايكروغرام \ متر. هذه النتائج تبين ان استعمال مادة اوكسيد الزركنيوم حسنت من الخواص الميكانيكة للمادة الاساسية ماعدا نتائج اختبار معدل التاكل.

الكلمات الرئيسية: مادة العزل المزججة الاسمنتية ، اوكسيد الزركنيوم المايكروي والنانوي، الخواص الميكانيكية، مادة ريفا ذاتية الجفاف.

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1. INTRODUCTION

The glass ionomer cement (GIC) were invented from silicon cement material in the early years of this century where the first was introduced to the dental profession in 1972 by **Wilson, 1972**, This substance is composed of acid in addition to the powder, where they are mixed according to the manufacturer's instructions to form a mixture similar to the paste (cement) as it hardens within 2-10 minutes after the preparation process paste, **Nicholson, 1998** and **Wilson, 1972**. This material is characterized by excellent adhesion properties with the basic teeth structure and low fluoride release rate in addition to the coefficient of thermal expansion similar to the structure of the teeth with a decrease in toxicity and they are considered biocompatible led to their widespread use as luting materials, cavity liners and bases, and restorative materials, **Wilson, 1972**. One of the main reasons for reducing its use in the field of dental fillings is that it is a brittle material with rapid breakage when high stresses are applied in addition to its mechanical properties in general, for example, fracture resistance, the hardness of fracture and wear rate losses. All these reasons make use in the repair or fillings of dental front teeth which is applied stress less than the back part **Lohbauer, 2009** and **Wilson, 1972**. Therefore, many researchers worked on the development of mechanical properties through different techniques or methods to ensure the mechanical properties. The most prominent of these techniques or methods is the way to add some elements or compounds of the three types of materials such as metal and ceramic materials as well as polymers, **Ab Rahman, et al., 2014 and Sari, et al., 2014**. The materials among these attempts are added microparticles including; montmorillonite clay, zirconia, glass fibers, hydroxyapatite (HA), bioactive glass particles and casein phosphopeptide-amorphous calcium phosphate (CPP–ACP), **Moshaverinia, et al., 2011**. In addition, the use of nanoparticles has become a significant area of research in dentistry, such as added of HA and fluoroapatite nanobioceramics, ytterbium fluoride and barium sulphate nanoparticles, **Wilson, 1972**. Zirconium Oxide is characterized by good dimensional stability and toughness (in the same order as stainless steel alloys) have been used widely for the toughening and strengthening of brittle bioglasses material in biomedical applications. Furthermore, zirconium dioxide (ZrO_2) can be used in the fabrication of the high strength core for dental implants **Moshaverinia, et al., 2011**. The purpose of the present study was to evaluate the effects of adding (3, 5, 7) wt. % of ZrO_2 (micro and nanoparticles size) into conventional GIC and study its effect on the mechanical properties.

2. EXPERIMENTAL WORK:

2.1 Materials:

A commercially conventional self-cure GI restorative powder (Riva self-cure from SDI limited Australia company, shade A1) with a particle size of about 24 μm was blended in various proportions with ZrO_2 micro and nanoparticles with a particle size 65 μm and 20 nm respectively, both additives purity were 99%.

2.2 Sample Preparation

Before preparation of samples, the GIC and Zirconium Oxide micro and nanoparticles powder were examined by EDX (Sputter Coater S150A, Japan) to characterize the elements composition of used powders as shown in **Fig.1**. Seven different samples in total were prepared; one represents the control sample (the basic material without adding) while the others represent the basic materials with the addition of (3, 5 and 7) wt. % from both micro and nanoparticles ZrO_2.

A B303, monobloc inside, weighing technology balance with three digits accuracy was used to prepare the mixed powders. These mixtures were then stored in glass cuvettes to prevent particles from 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 suits for the required test and then covered with glass slides form both sides to obtain a relatively flat and smooth surface. Clamped the glass for a 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, **Gjorgievska, et al., 2015** and **Gu, et al., 2005**.

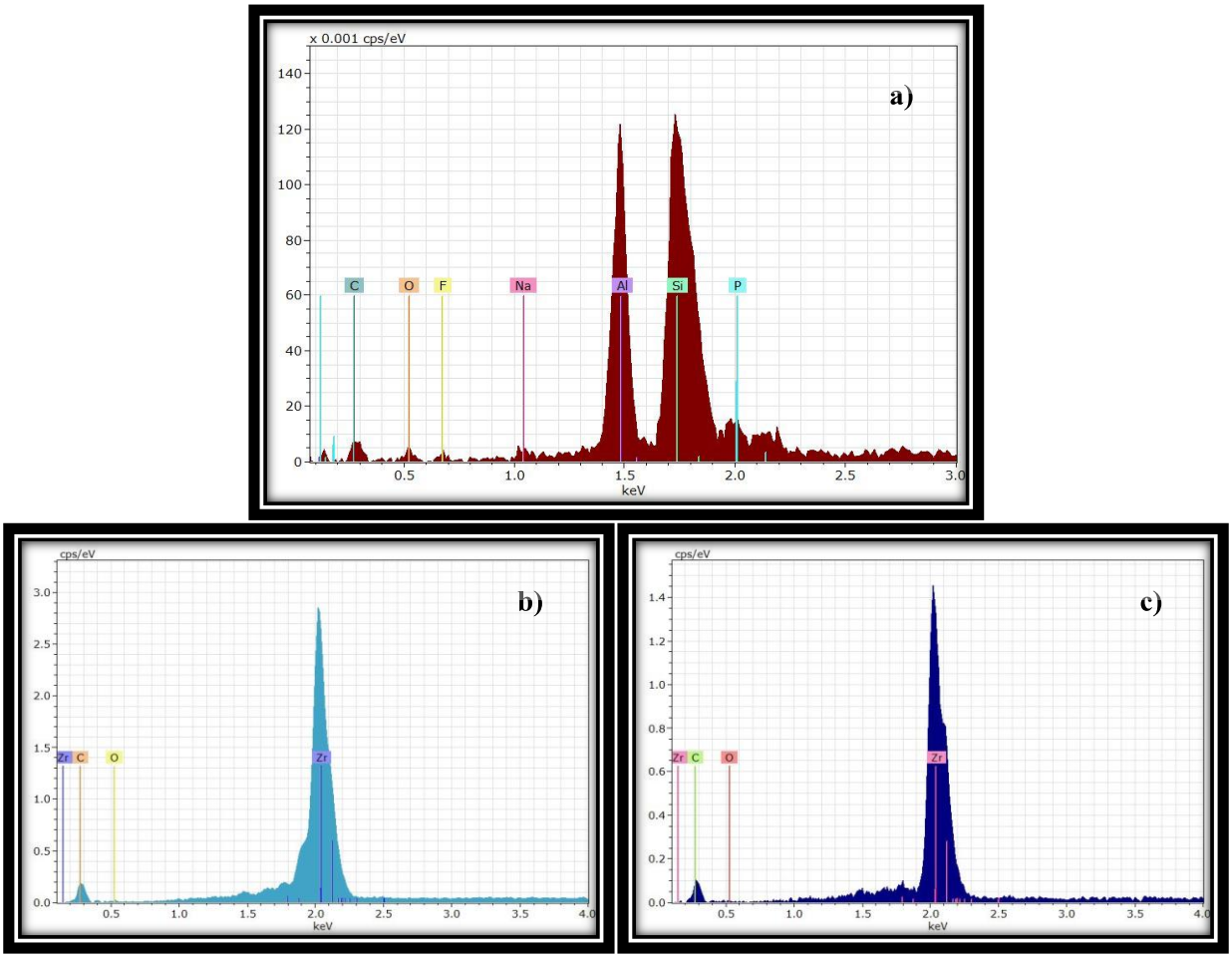

Figure 1. EDX examination results for GIC and ZrO_2 micro and Nano particles a) GIC, b) ZrO_2 micro and c) ZrO_2 nanoparticles.

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 (Dentistry-water-based cement part). The compressive strength (MPa), Cs, of the specimens was characterized using a Testometric AX M500-25kN computerized system with a crosshead speed of 0.25 mm/min as shown in **Fig.2**.

$$Cs = 4F/ (Pd^2) \quad \ldots\ldots (1)$$

Where F is the load (N) and d is the diameter of a specimen (mm)**, Wilson, 1972**.
Other same shape specimens were used for measuring surface microhardness using a Q-Time digital Microhardness Tester. A diamond indenter with 50 g load and a dwell time of 10 s were employed as shown in **Fig. 3**. The Vickers hardness (VHN) for each specimen was calculated using the following formula:

$$VHN=1.8544 \times L/d^2 \ldots\ldots (2)$$

Where L is the applied load (kg) and d is the mean indentations diagonal length (mm), **Wilson, 1972**.

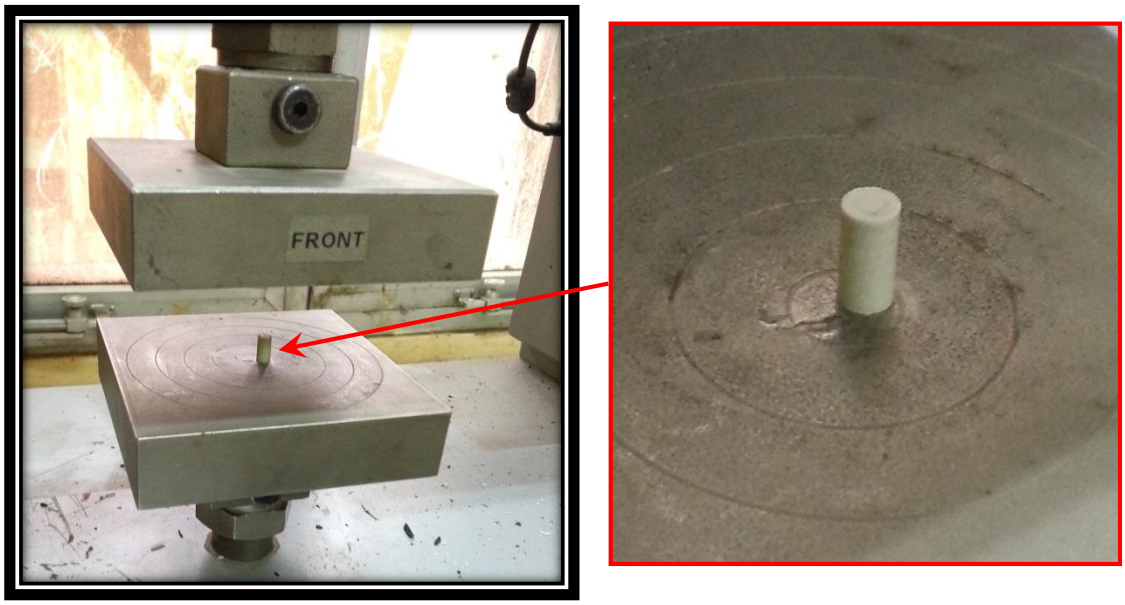

Figure 2. A compression sample on the testometric holder and magnified image for the sample on the right.

Figure 3. Q-Time digital Microhardness tester, the notch diameter calculated using the screen (the right image).

2.4 Biaxial flexural strength test samples:

The biaxial flexural strength (BFS) test was performed 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 diameter was placed on a 10 mm diameter circle with equidistant from each other surrounded by a ring of 14 mm diameter and 2 mm height to prevent sample movement. Three disks specimens were prepared from each sample in a 14 mm inner diameter stainless steel ring and 1.25 mm in height. The mixed paste was placed in the ring until set then placed in room condition. The day after, the specimen was 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, **Fig.4**. The system software record each specimen fracture load continually then the following equations were used to calculate the BFS value, **Wille, et al., 2016**:

$$S=\frac{-0.2387\ P\ (X-Y)}{d^2} \quad \ldots\ldots\ (3)$$

X and Y were determined as follows:

$$X = (1+v)\ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{1-v}{2}\right]\left(\frac{r_2}{r_3}\right)^2 \quad \ldots\ldots\ (4)$$

$$Y = (1+v)\left[1+\ln\left(\frac{r_1}{r_3}\right)^2\right] + (1-v)\left(\frac{r_1}{r_3}\right)^2 \quad \ldots\ldots\ (5)$$

Where S is a 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 specimen radius.

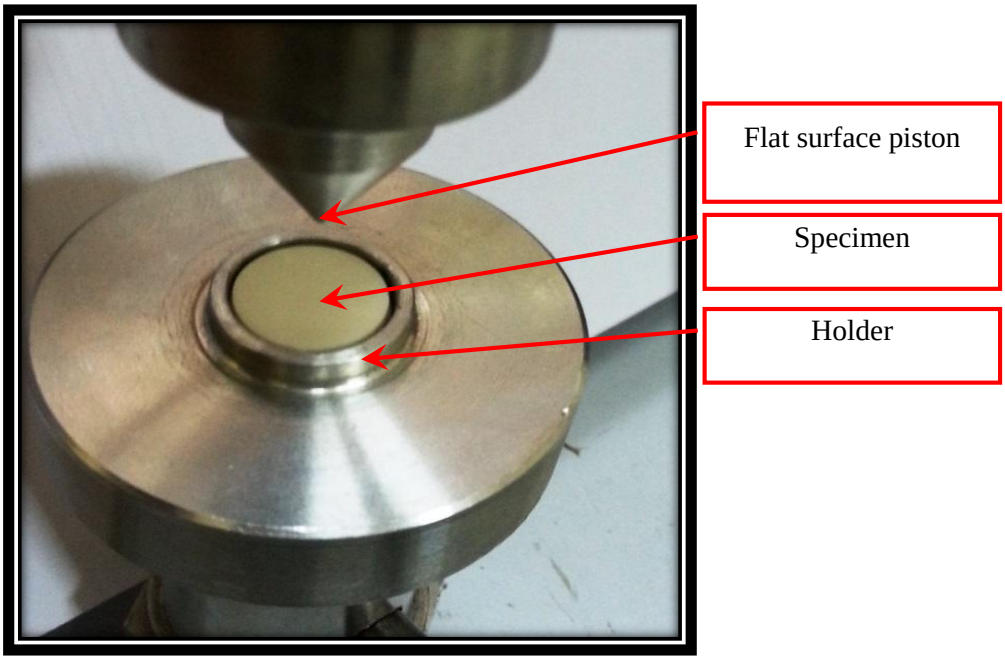

Figure 4. The BFS sample, holder and piston.

2.5 Wear rate losses determination

Pin on disc technique was used to measure the materials wear rate (WR) according to ASTM G99 (Standard 2000). 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 a one-kilogram load rotated at a constant rate of 480 rpm. The sample sliding stroke has a 95mm radius and 0.125s duration time. The total number of revolutions for each WR test was 720 cycles. 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 the following formula, **Bayer, 2004** and **Awham and Sadeer,2010**:

$$W.R. = \Delta M/\omega\, r\, t \quad \ldots\ldots (6)$$

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

3. RESULTS AND DISCUSSIONS:

3.1 Compression strength test

Generally, the addition of ZrO_2 micro and nanoparticles increased the compressive strength significantly as compared with the control samples, as **Fig. 5** shows. The Compression strength differs between the two particles types. The microparticles give the best result at 5wt% addition with 122.31 MPa (52.3% increasing) with semilinear results around this value for the 3 and 7 wt% addition, while adding of nanoparticles linearly decreased the compression strength from (104.35 to 85.63) MPa (29.9% to 6.61% higher) for (3 to 7) wt% respectively, which is also higher than the as it is sample (80.32MPa).

It is clear that the addition of ZrO_2 enhances the compression strength of the GIC. The reason of this compressive strength improvement after ZrO_2 addition can be explained due to the compression properties of this ceramic. On the other hand, the different effect between micro and nanoparticles addition is due to varies between there compressive properties.

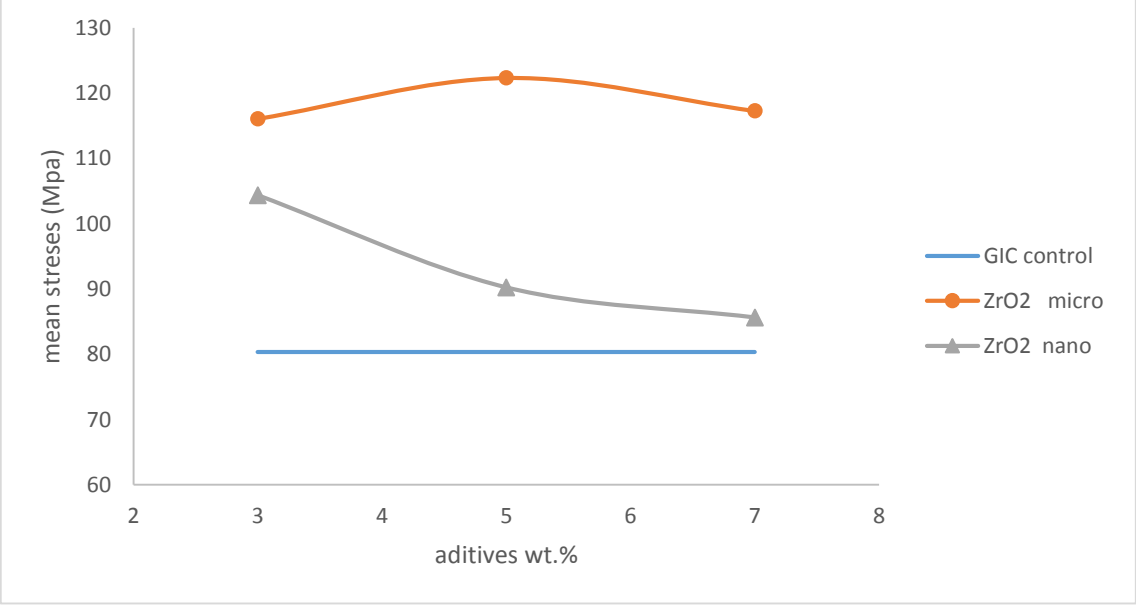

Figure 5. The relation between compressive stress (mean value) and ZrO_2 additives wt. %.

3.2 Surface Microhardness:

The addition of ZrO_2 micro and nanoparticles increased the microhardness significantly as compared with the control samples, **Fig. 6**. The nanoparticles give hardener surface than microparticles, but with inverse behavior. The higher hardness overall was 88.8 VHN at 3 wt.% nanoparticles which is 108.2% higher the value of GIC hardness 42.65 VHN, then the value was decreased with wt.% increasing, inversely the lowest hardness was 50.88 VHN at 3wt% ZrO_2 microparticles (19.3% higher than GIC) and increased as the wt.% increased. The nanoparticles behavior may be described due to the more wt% the higher particles agglomeration chance, and this will give low hardness area. On another hand, the hardness performance with more microparticles can be explained due to the high hardness of ZrO_2.

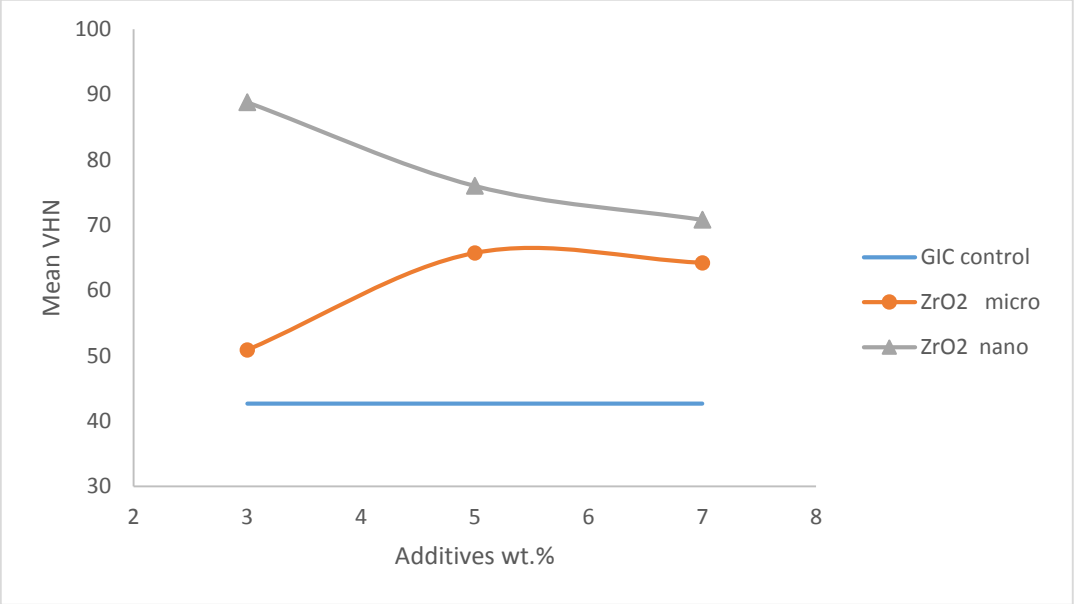

Figure 6. The relation between Vickers Microhardness numbers (mean value) and ZrO_2 additives wt. %.

3.3 Biaxial flexural strength results Test

Generally, the addition of ZrO_2 micro and nanoparticles increased the biaxial flexural strength significantly as compared with the control samples, except the 3wt% micro size which was less than the basic material, Fig. 7. The behavior between the two curves was different, the more addition of microparticles increases the strength value, while the addition of nanoparticles decreases it (but all nano values were higher than micro).

The addition of 3wt. % of ZrO_2 nanoparticles gives the best BFS results of 35.79 MPa (about 84% higher than the control sample), while the 5 wt. % microparticles give 24.06 MPa (about 23.6% more than the control sample) and as it is sample has 19.46 MPa BFS.

This difference between the micro and nanoparticles results may be explained due to the higher surface area of nanoparticles which made higher joining force between the particles and the hardener liquid than the microparticles made, which gives higher tensile strength. The more nanoparticles may cause kind of saturation that gives the linearly decreasing in BFS.

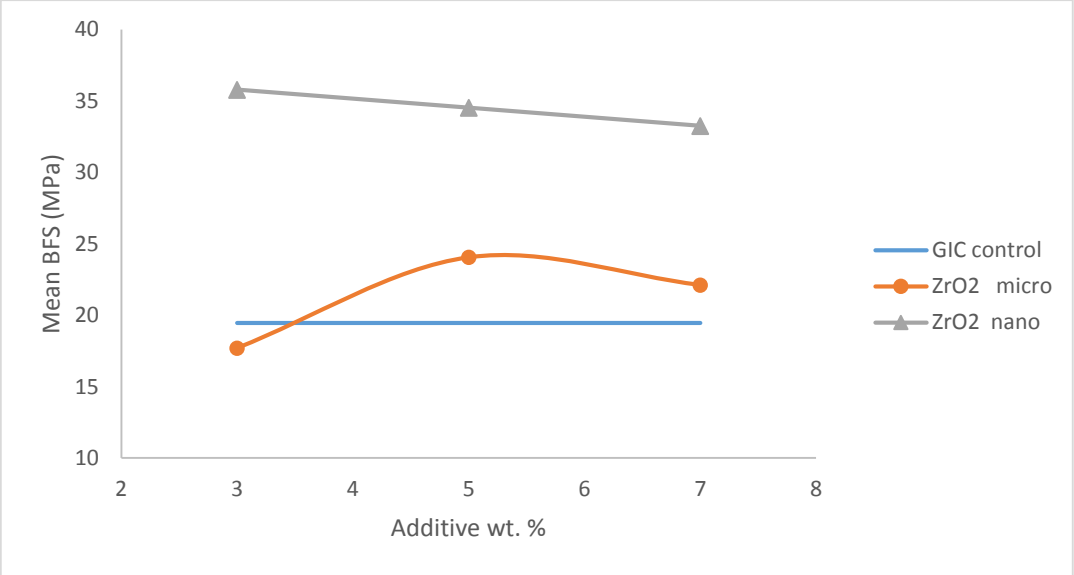

Figure 7. The relation between Biaxial flexural strength (mean value) and ZrO_2 additives wt. %.

3.5 Wear rate losses:

The addition of ZrO_2 micro and nanoparticles increased the wear rate losses significantly as compared with the control samples (which is undesirable), except in the case of the 7 wt% nanoparticle addition 3.776 µg/m which was less than the control value 4.720 µg/m, as shown in **Fig. 8**. Both particle sizes behave the same for each wt%, but with lower values for the nanoparticles, this may be explained as the more nanoparticles fills the decreased the surface roughness which decreasing the wear rate.

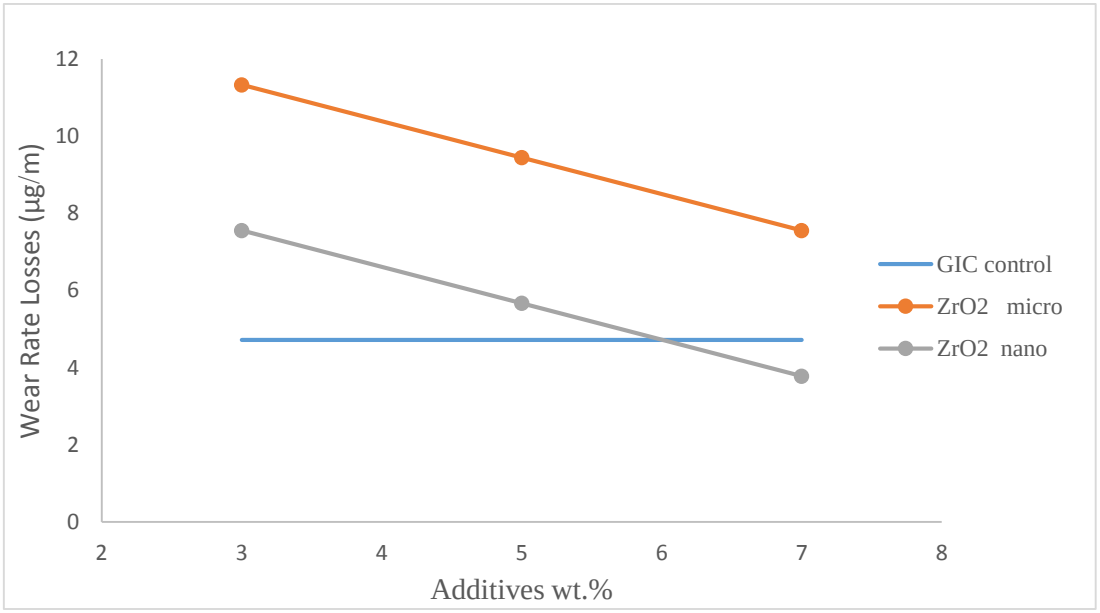

Figure 8. The relation between Wear rate losses (mean value) and ZrO_2 additives wt. %.

4. CONCLUSIONS

In general, the addition of ZrO_2 particles enhance the mechanical properties of the GIC, except the wear rate losses. These results were expected due to the ceramic behavior of ZrO_2 as compared with the glass of GIC. For microparticles, 5wt% gives the best ratio of additions among the range of (3 -7) wt. %, while the seven percent was the best in the wear rate losses.
The 3wt% gives the best ratio of additions among the range of (3 -7) wt. %, while the seven percent gives the best wear rate losses for nanoparticle size. The Nanoparticles ZrO_2 gives better results than microparticles for Microhardness, BFS and wear rate losses while the results were reversed for the compression strength.

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NOMENCLATURE

GIC = glass Ionomer Cement
Cs = compressive strength, MPa.
VHN = vickers Microhardness numbers, kg/mm^2
BFS = biaxial flexural strength, MPa.
WR= wear rate losses, µg/m