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The effect of titanium oxide microparticles on mechanical properties, absorption and solubility processes of a glass ionomer cement

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ABSTRACT

Glass Ionomer Cement (GIC) is one of the important dental temporary filing materials. The aim of this study is to evaluate the effect of adding 3, 5 and 7 wt. % of TiO_2 microparticles to conventional GIC powder (Riva Self Cure) on mechanical properties and its effect on absorption and solubility processes. TiO_2 particles additives improved compressive strength and biaxial flexural strength, where the compressive strength increased with increasing in the added ratio, while the highest value of the biaxial flexural strength was at 3 wt.%. The addition of TiO_2 particles improved the surface Vickers microhardness values, with highest value at 5 wt. %. On other hand TiO_2 addition improves the wear resistance as additives increased. The most acidic beverages (the lowest pH value) were the most effective in increasing the absorption and solubility percentage of samples. Orange juice was more effective followed by cola and then coffee and tea were less effective. Finally, its recommended that patients should reduce drinking these acidic beverages because its harmful effect on dental fillings.

Keywords: Riva Self Cure, Titanium Oxide, Mechanical Properties, Absorption, Solubility.

تأثير الجسيمات المايكروية لأكسيد التيتانيوم على الخواص الميكانيكية وعمليات الامتصاص و الذوبان في مادة العزل المزججة الاسمنتية

الخلاصة

تعتبر مادة العزل المزججة الاسمنتية Glass Ionomer Cement (GIC) احد المواد المهمة في حشوات الاسنان المؤقتة. الهدف من هذه الدراسة هو تقييم تأثير إضافة (3 و 5 و 7)وزن % للجزئيات الدقيقة TiO_2 إلى مادة الـGIC التقليدية (Riva self cure) على الخواص الميكانيكية وتأثيرها على عمليات الامتصاص والذوبان. أدت إضافات الجسيمات TiO_2 إلى تحسين قوة الانضغاط وقوة الانثناء ثنائية المحور ، حيث زادت قوة الانضغاط مع الزيادة في النسبة المضافة ، في حين كانت أعلى قيمة لقوة الانحناء ثنائية المحور عند 3 وزن ٪. أدت إضافة جزيئات TiO_2 إلى تحسين قيم صلادة فيكرز للسطح حيث كانت أعلى قيمة عند 5 وزن ٪. لقد أدت إضافة TiO_2 إلى تحسين مقاومة التآكل مع زيادة نسبة الإضافة. كانت معظم المشروبات الحمضية (أقل قيمة من الرقم الهيدروجيني) هي الأكثر تأثير في زيادة نسبة الامتصاص والذوبان لعينات TiO_2. كان عصير البرتقال أكثر تأثير يليه الكولا ثم القهوة والشاي أقل تأثير. لذا يوصي الباحثين بتقليل هذه المشروبات الحمضية لأن لها تأثير ضار على حشوات الأسنان.

الكلمات الرئيسية: العلاج الذاتي ريفا ، أكسيد التيتانيوم ، الخصائص الميكانيكية ، الامتصاص ، الذوبان.

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1. INTRODUCTION

The glass ionomer cement (GIC) may be defined as water-based material which sets through an acid-base chemical reaction between the basic powder of the fluoroaluminosilicate glass and the polyacrylic acid solution **(Singh, T.R. Mahesh; .Suresh, P; .Sandhyarani, J; .Sravanthi, J, 2011).** GIC possess certain unique advantages like adhesion to the dental structure and base metal, good biocompatibility, low toxicity, as well as release of fluoride for a long period of time, coefficient of thermal expansion match to tooth structure and ease of use (**Bortoletto, C. C.; Junior, W. G. M.; Bussadori, L. J. M.; S. Kalil, 2013**). However, GIC has low mechanical strength and fracture toughness which limit their clinical application as filling materials in high stress bearing sites **(M. G. Lyapina; M. Tzekova; M. Dencheva; A Krasteva; M. Yaneva-Deliverska; A. Kisselova, 2016)**. There have been many attempts to overcome these problems by adding material to enhance GIC such as adding metal particles, ceramic, or glass fiber to GIC powder **(Azam, T. Y. T.; Ying, Q. X.; Rahman, I. A.; Masudi, S. M.; Luddin, N.; Rashid, R. A., 2013).** TiO_2 is inorganic oxide possess promising properties such as high chemical stability, biocompatibility and nontoxic (**Elsaka, et al., 2011)**. Therefore, the aim of this study was to investigate the effect of adding 3, 5 and 7 wt. % of TiO_2 microparticles powder to GICon the mechanical properties and its effect on absorption and solubility processes**.**

2. EXPERIMENTAL WORK

2.1 Materials

Conventional glass ionomer cement (Riva self cure, shade A2, SDI limited, Australia) which is in powder-liquid form (23.739 μm average particle size) was used in this work as a base material, and TiO_2 microparticles powder (51.311 μm average particle size and 99% purity) was used as an additive material.

2.2 Sample preparation

Prior to samples preparation, X-ray diffraction analysis for TiO_2 microparticles powder was performed by X-ray diffractometer (D2, PHASER, Bruker) with diffraction angles between 20° and 80°. The result of XRD analysis of micro TiO_2 powder was shown in **Fig.1** which displayed that TiO_2 was pure and this results matches with (**Armstrong, A. R.; Armstrong, G.; Canales, J.; García, R.; Bruce, P. G., 2005; Thamaphat, K.; Limsuwan, P.; Ngotawornchai, B., 2008**) . Four different samples were prepared in total, one representing the base material without any addition, while the other representing the base material with the addition of 3, 5, and 7 wt. % TiO_2 microparticles powder. A calibrated electronic balance with four digits accuracy (Sartorius, BL210S) was used to prepare the mixed powders (and where ever needed in this work). The mixed powders then kept in glass tubes to prevent particles from stacking on the walls of the tube. Then, the powders mixture were mixed using a tube roller mixer machine with rotation speed 60 rpm for 2 hours in order to obtain homogenous and uniform powders mixture. The powder mixture was mixed with the aqueous solution (hardener liquid) with a ratio of 1/1 (one scoop of powder /one drop of the liquid) in accordance to the manufacturer's instructions (**SDI, 2013**). The resulting paste was immediately inserted into a mold made of stainless steel, and then covered from both sides with glass slides for a relatively flat and smooth surface. The cement was clamped for couple of minutes and left it in an incubator at 37 ℃ for 5 minutes for set and harden. The sample was then removed from the mold and stored in the room condition until the examination **(Gjorgievska, E.; Tendeloo, G. V.; Nicholson, J. W.; Coleman, N. J.; Slipper, I. J.; Booth, S., 2015).**

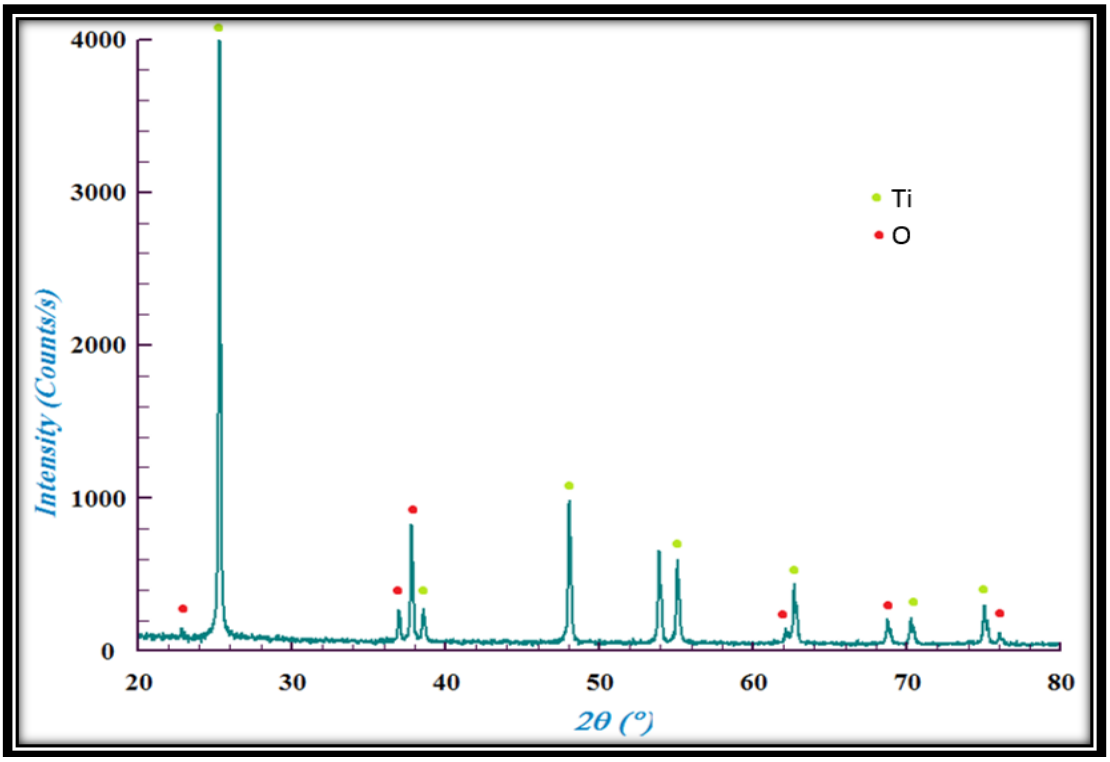

Figure 1. XRD pattern for TiO_2 microparticles powder.

2.3 Compressive strength test

Cylindrical stainless-steel mold (with 5 mm internal diameter and 10 mm height) was used to prepare the specimens. Four different samples with 3 three repetitions each (12 sample in total) were prepared for compression test following the procedures outlined in ISO 9917-1 and ASTM C1424-15 (**ISO, 2003; ASTM, 2015**). The compression test was done using the testometric machine (AX M500-capacity load 25kN) computerized system with a crosshead speed of 0.5 mm/min (**ISO, 2003**).

2.4 Microhardness test

Cylindrical stainless-steel mold (with 5 mm internal diameter and 10 mm height) was used to prepare the specimens for Vickers surface microhardness test. After 24 hr., Vickers surface microhardness test was done by using the device Digital Micro-hardness tester (Q-Time, TH-715). A 50 g load indenter and dwell time of 10 s was applied on each specimen's surface **(Alobiedy, et al., 2019)**. Three indentations were done on each specimen's surface at different locations and the mean value of them was determined.

2.5 Biaxial flexural strength test

The biaxial flexural strength (BFS) was determined with piston on three ball technique using the Testometric AX M500-25kN computerized system. Three stainless steel balls of 3.2 mm diameter that were equidistant from each other were placed on a circle with a diameter of 10 mm surrounded by a ring of 14 mm diameter and 2 mm height to prevent sample movement. Disk stainless steel mold (with 14 mm internal diameter and 1.25 mm thickness) was used to prepare specimens for biaxial flexural strength test. The mixed paste was inserted in the mold until set then left in room condition. After 24 hr., the specimen was centered and supported on steel balls and the load was applied vertically on the center of specimen by a 1.2 mm diameter flat tip of piston with a cross head speed of 0.1 mm/min as shown in **Fig.2**. The fracture load for each specimen was recorded by the system software and then the BFS value was determined using the following equation **(Wille, S.; Hölken, I.; Haidarschin, G.; Adelung, R.; Kern, M., 2016**):

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

While X and Y were determined as following:

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

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

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), $\mathbf{r_1}$ radius of the support circle, $\mathbf{r_2}$ radius of the loaded area and $\mathbf{r_3}$ specimen radius.

Figure 2. The biaxial flexural strength test

2.6 Wear rate losses determination

Cylindrical stainless steel mold (with 5 mm internal diameter and 10 mm height) was used to prepare the specimens forwear test. The wear test was determined with a pin on disc apparatus. The apparatus was designed to produce continuous sliding contact between the materials studied and stainless steel disk. Prior to test, the samples were weighed using same system above, and then the sample was held in contact with the disk surface and loaded with a vertical load of one kilogram. The operation time was 90 seconds and the disk was rotate with a constant rotational speed of 51rad/sec. The length of each sliding stroke was approximately 30 cm. The normal saline liquid was used as lubrication that was dropped continuously during operation period by distillation device connected to the specimen holder to avoid frictionheating effects **(Munshi, et al., 2015)**, and simulate the wet environment in the oral cavity **(Cha, H. S.; Lee, Y. K.; Lim, B. S; Rhee, S. H.; Yang, H. C, 2004**). After each trial the surface of steel disk was cleaned and the sample was taken, cleaned, wiped up by drying paper and weighed again. Then wear rate is calculated using the following equation (**Hussein & Muhammad, 2014; Ahmed, 2017**):

$$W.R. = \Delta M/\ \omega\ r\ t \quad \ldots. (4)$$

2.7 Absorption test

Disk stainless steel mold (with 8.5 mm internal diameter and 1.9 mm thickness) was used to prepare the specimens for each group of materials. The samples were then weighed and kept in individual closed glass containers in distilled water, tea, coffee, orange juice and coca cola at room temperature, where each beverage was renewed every day **(Ayatollahi, M.R.; Yahya, M. Y.;**

Karimzadeh, A.; Nikkhooyifar, M.; Ayob, A., 2015; Aliping-McKenzie, et al., 2004). **Table.1** shows the pH value of the used beverage before immersing the samples. For all samples, after the storage process, the samples were rinsed with distilled water before weighing to remove the layers deposited on their surface from the beverages. The surface of each sample was then dried by placed it on drying paper for increasing the accuracy of the results. Then samples weighed after 1- and 2-days intervals. The weight difference of the sample before and after immersion in the solution represents the gained weight. The absorption percentage for each sample was calculated using the following formula:

$$\text{Absorption percentage}=\frac{W2-W1}{W1}X100\% \quad \ldots.(5)$$

Where **W1**: weight of specimen before immersion in distilled water and various beverages and **W2:** weight of specimen after storage process in distilled water and various beverages

Table1. The pH level of beverages.

Beverage	Distilled water	Tea	Coffee	Orange juice	Coca cola
pH value	7	6	5.9	3.4	2.9

2.8 Solubility test

Samples were prepared and stored in beverages for 7 days intervals using the same method used for absorption testing. After 7 days of storing, the samples were taken from the solutions and rinsed with distilled water to remove any stuck particles on the surface, then dried for 30 minutes using an electrical furnace at 70 ^{o}C (**Goenka, et al., 2012)**. Finally, samples were weighed and equation 5 was used to calculate the solubility ratio.

3.RESULTS AND DISCUSSION

3.1 Compressive strength test

The addition of TiO_2microparticlesincreases the compression strength proportionally to the addition ratios as shown in **Fig.3**. This is may be described due to the increase of particles joining between the materials, in addition to the fact that the compressive strength of TiO_2 particles is higher than compressive strength of glass particles, therefore the resistance of material to compression will increased, as well as the strength of the bonds formed between the particles of TiO_2 and GIC (**Garoushi, et al., 2018**). The maximum compression strength was 14.735% higher than GIC for 7 wt. % TiO_2 while the minimum value was 1.794% higher for 3 wt. % TiO_2.

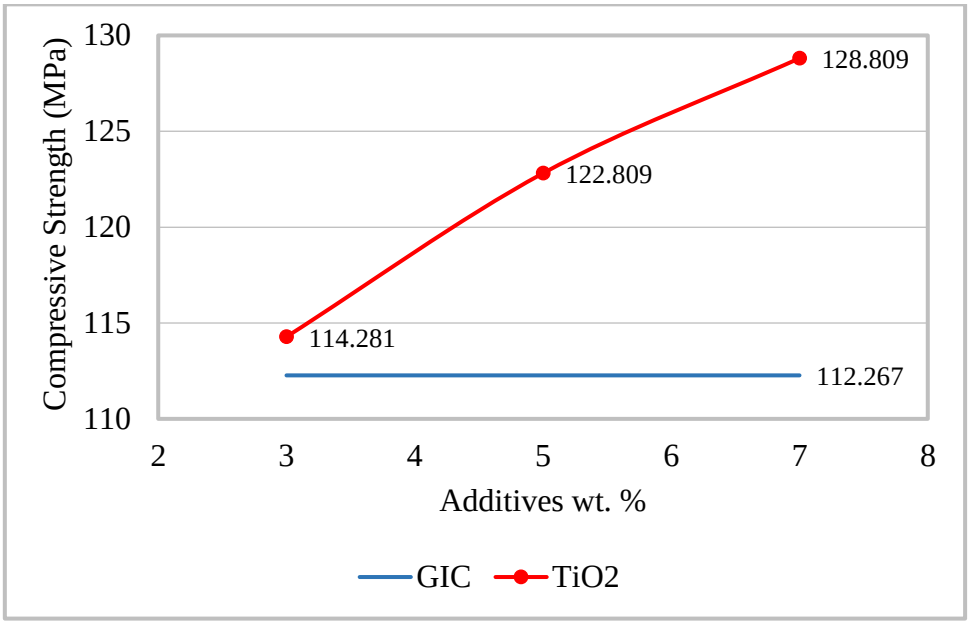

Figure 3. Therelation between compressive strength(mean value) and TiO_2 additives wt. %

3.2 Microhardness test

The addition of TiO_2 microparticles increased the surface microhardness values with the increasing the addition ratio till 5 wt. %, after which the microhardness decreased with the ratio increases, but remains higher than the GIC material as shown in **Fig. 4**. The reason for increased the surface microhardness values with increased the ratios of added TiO_2 due to the fact that the hardness of TiO_2 is greater than the hardness of the basic material, in addition to the strength of the bonds formed between the particles of additive TiO_2 and GIC material. All these reasons will lead to increase the strength of surface resistance to plastic deformation. When increasing the added ratios after 5 wt. %, the surface microhardness values start to decrease while maintaining their values higher than the basic material and this may be due to the large particle size of additives which is 51.311 µmas compared to the particle size of GIC which is 23.739 µm and this may cause reduction in the strength of cohesion the material, and will therefore provide areas of weak and easy of spread the cracks and then their resistance to permanent deformation will decrease. The maximum variation percentage was 55.044% at 5 wt. % TiO_2, where the minimum variation percentage was 10.295% at 7 wt. % TiO_2.

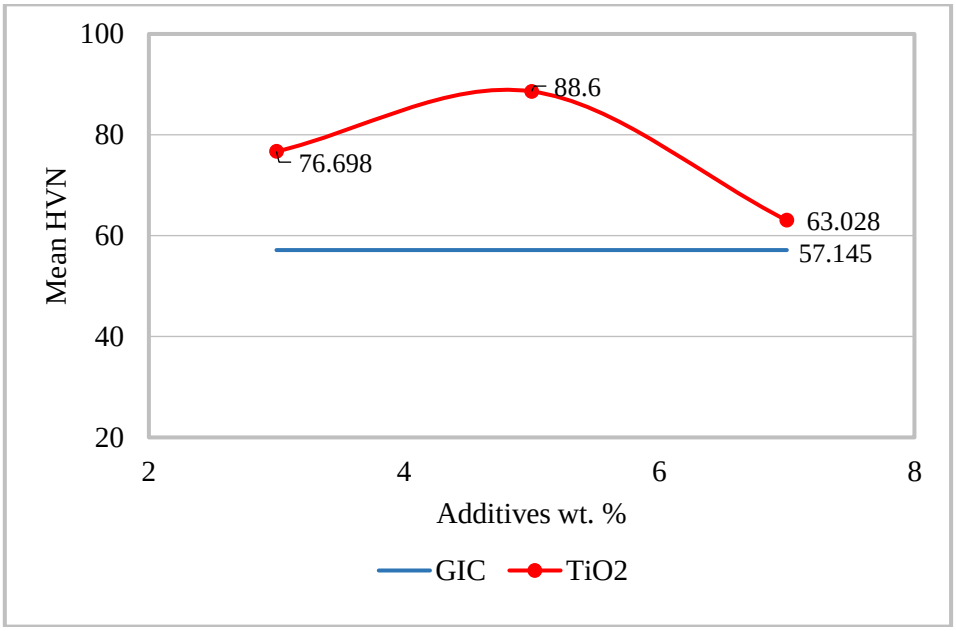

Figure 4. The relation between Vickers microhardness number (mean value) and TiO_2 additives wt%

3.3 Biaxial flexural strength test

The BFS has improved with the addition of TiO_2 microparticles (**Fig.5**). This is due to the formation of strong bonds between the particles of the additive and GIC (**Garoushi, et al., 2018**), therefore will need more force to break the bonds and greater strength to spread the cracks that leads to failure of the material. When increasing the ratio of the additive after 3 wt. %, a decrease in BFS was observed because of the increase speed reaction of particles with liquid acid and thus may cause a lack of homogeneity of the material. This will reduce the strength of cohesion of the material, provide weak areas and reduce resistant of material to the growth and formation of cracks and will lead to faster failure of samples. The maximum value was 84.411 % higher at 3 wt. % TiO_2, while the minimum value was 28.628% higher at 7 wt. % TiO_2.

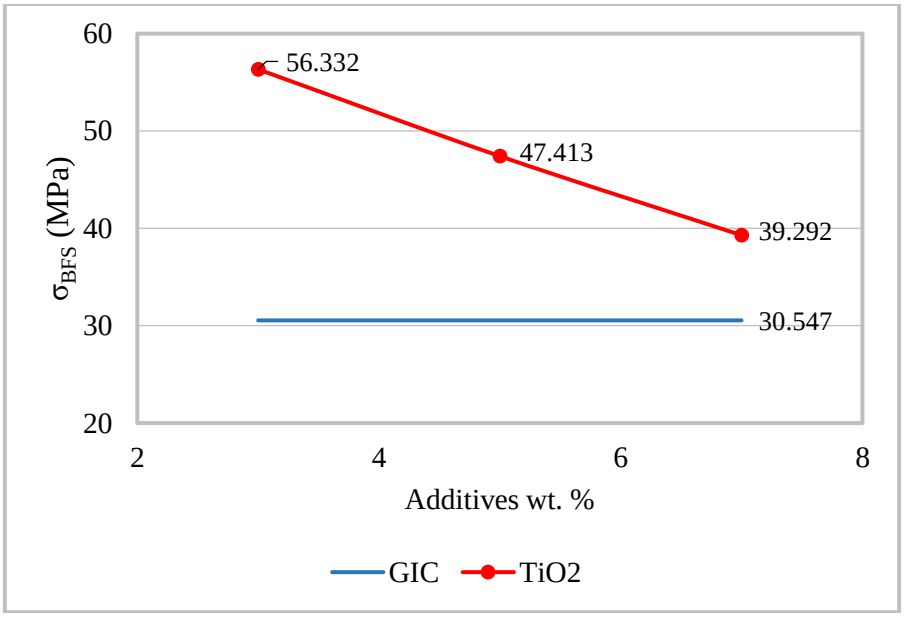

Figure 5. The relation between biaxial flexural strength(mean value) and TiO_2additives wt. %

3.4 Wear rate losses determination

The addition of TiO_2 microparticles decreased the wear rate losses with increasing additives as shown in **Fig.6**. The reason for this is due to the fact that TiO_2 particles is harder than GIC's powder, in addition to the strength of bonds formed between TiO_2 and GIC particles. Therefore, the wear resistance of material will be increased and those decreased the number of missing layers from the friction surface of the sample and the stainless steel. All these reasons will reduce the weight losses and hence reduced the wear rate losses. The minimum wear rate (which gives maximum enhancement percent of 28.564%) at 7 wt. % TiO_2, while the maximum wear rate (which gives minimum variation percentage of -14.287%) at 3 wt. % TiO_2.

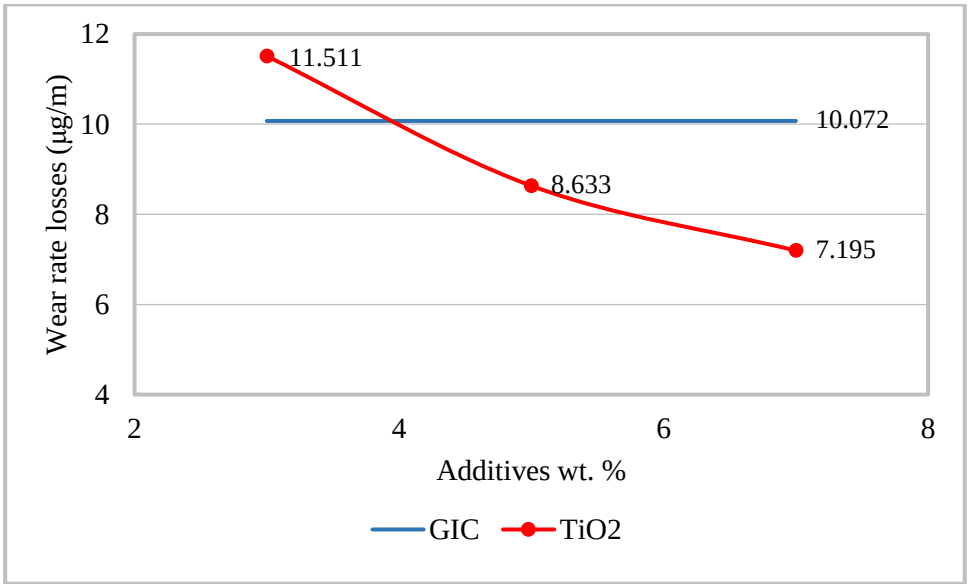

Figure 6. The relationship between Wear rate losses and TiO_2 additives wt. %.

3.5 Absorption test

As shown in **Fig.7**, addition of TiO_2 particles decreased the distilled water absorptivity as additive ratio increased. The TiO_2 particles fill the voids in between GIC particles and decrease the porosity. For both days the maximum absorption was for 3 wt. % TiO_2 while minimum absorption was recorded for 7 wt. % TiO_2.

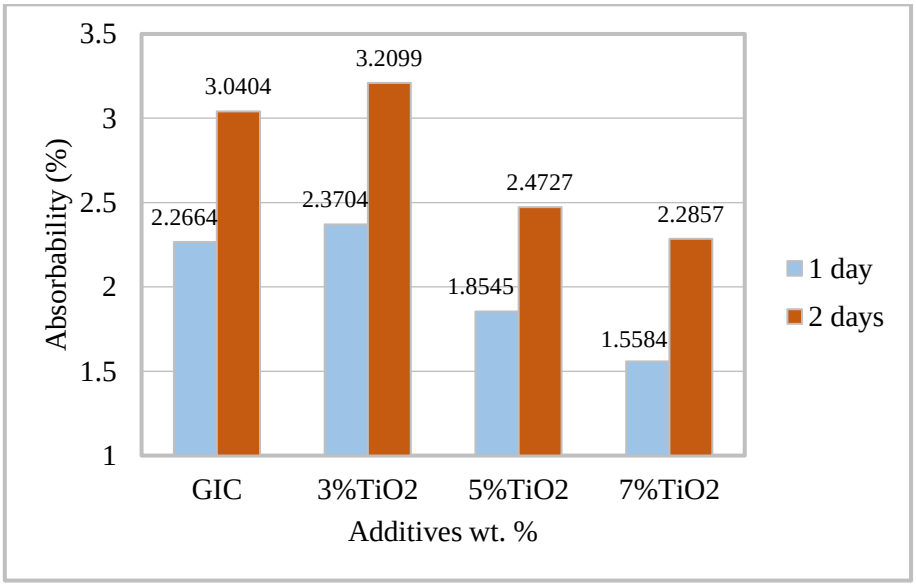

Figure 7. The results of distilled water absorption after 1 and 2 days for TiO_2 additives .

Also, the tea absorption rate was reduced with addition of TiO_2 (**Fig.8)**. However, the absorption rate increased with the additions increasing but it still lower than pure GIC sample. This may be because the materials in the tea began to decompose the particles in the sample and penetrates within it and thus increase porosity and absorption rate.

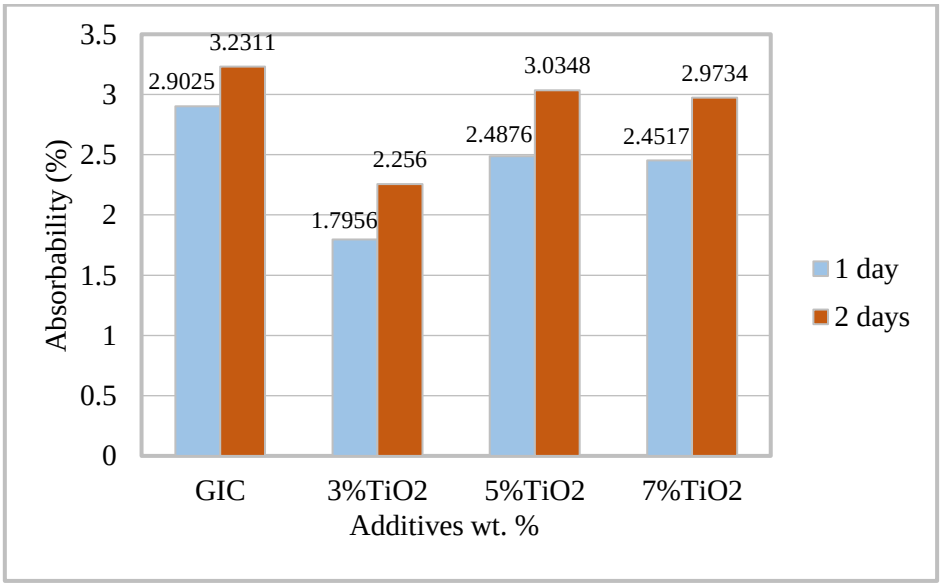

Figure 8. The results of tea absorption after 1 and 2 days for TiO_2 additives.

Deferent behaviour was appearing with coffee (**Fig.9)** because coffee is a more acidic drink. Its components began to dissolve particles in TiO_2 samples and penetrate the sample, thereby increasing sample porosity and absorption rate.

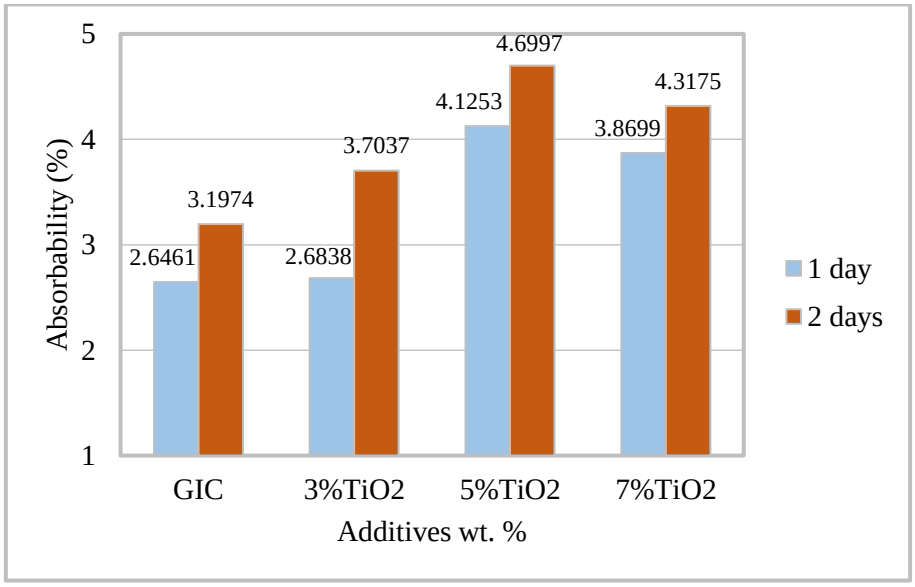

Figure 9. The results of coffee absorption after 1 and 2 days for TiO_2 additives

As **Fig.10** shows the addition of TiO_2 particles increases the absorption rate in orange juice. This due to low pH of orange juice, so the components of this juice began to dissolve the particles in the samples and penetrated into the sample and thus increase porosity of sample and absorption rate.

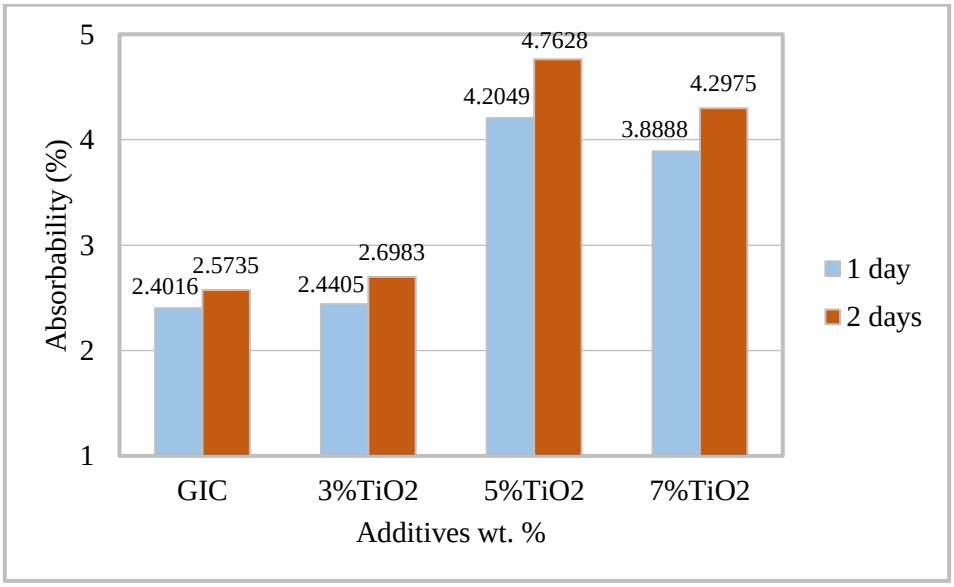

Figure 10. The results of absorption after 1 and 2 days for TiO_2 additives by orange effect

Unlike other beverages 7wt.% of TiO_2 gives lower absorptivity than other addition percent (**Fig. 11**), this may be due to the fact that cola has high acidic value, so its components begin to decompose the particles into the sample and penetrate into it, thus increasing the porosity of the sample and its absorbability.

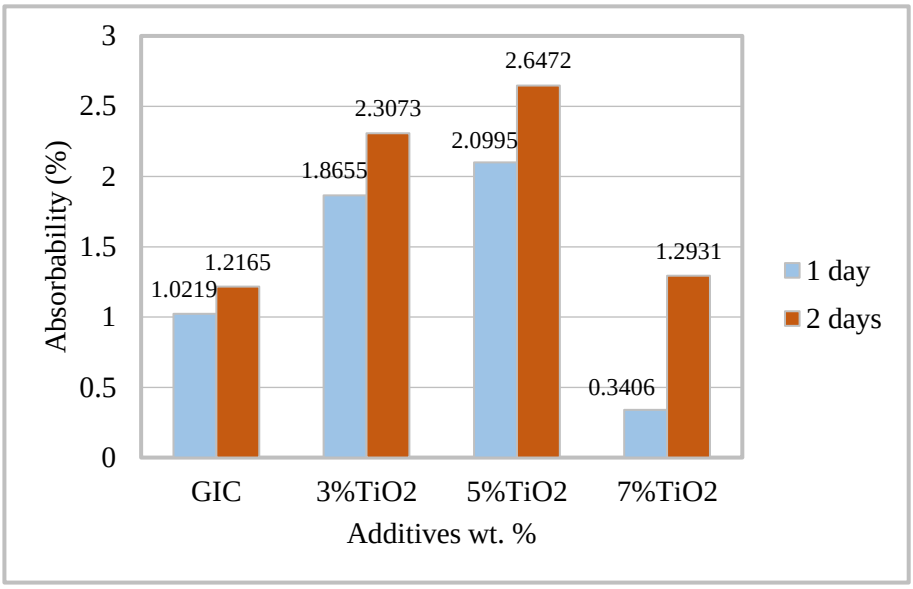

Figure 11. The results of absorption after 1 and 2 days for TiO_2 additives by cola effect.

3.6 Solubility test

TiO_2 samples are less solubility with distilled water compared to pure GIC sample as shown in **Fig.12.** This is due to the fact that the addition of TiO_2 makes the material more resistance to decompose in distilled water.

The solubility in both tea and coffee was decreased as addition increased and this may be due to the fact that TiO_2 particles are more degrade in acidic medium.

Unlikely, Orange juice increased the solubility of TiO_2 samples as compared with pure GIC due to the fact that is have the lowest pH as compared with other beaverages.

Finally, the addition of TiO_2 particles increased the solubility percentage in cola, but the solubility decreased with increasing the added ratios where become less than pure GIC at 7 wt. % due to the fact that the materials become more resistance to degradation in acidic medium.

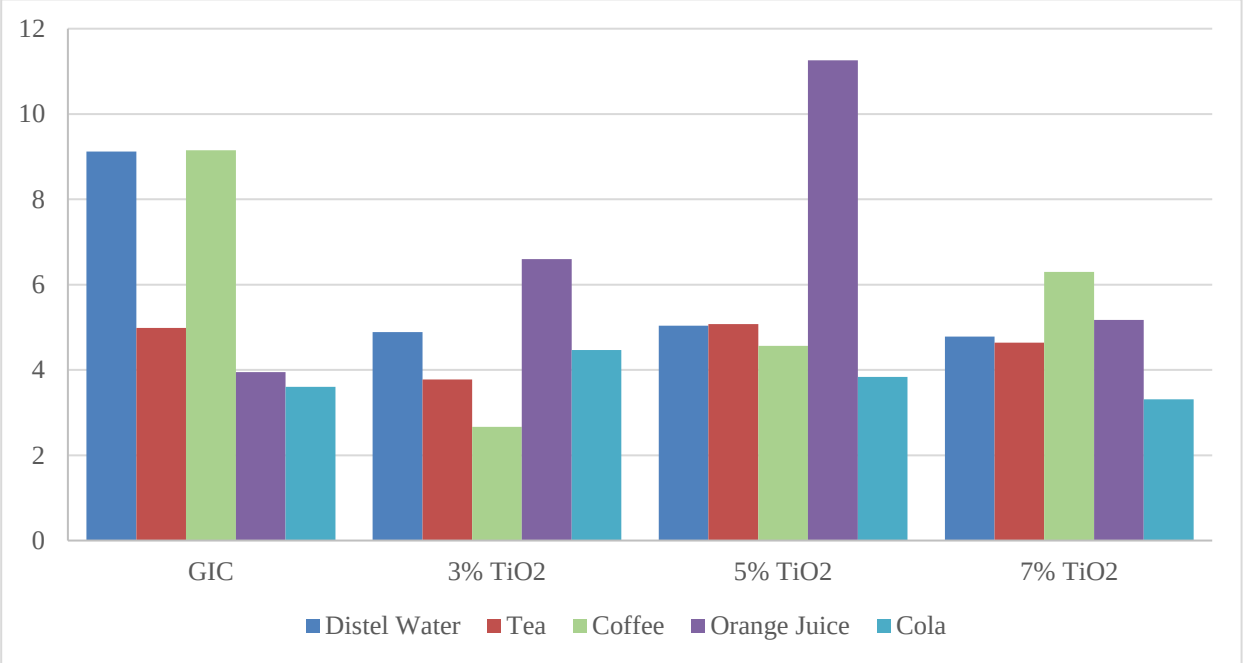

Figure 12. The results of solubility after 7 days for TiO_2 additives for all beavareges.

4. CONCLUSIONS

TiO_2 particles additives improved compressive strength and biaxial flexural strength, where the compressive strength increased with increasing in the added ratio, while the highest value of the biaxial flexural strength was at 3 wt.%. The addition of TiO_2 particles improved the surface Vickers microhardness values where the highest value was at 5 wt. %. Adding TiO_2 has improved the wear resistance with increased adding ratio. The most acidic beverages (the lowest value of pH) were the most effective in increasing the absorption and solubility percentage TiO_2 samples. Orange juice effect followed by cola then coffee and tea were less effective. The authors recommend patients to reduce these acidic beverages because it has a harmful effect on dental fillings.

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NOMENCLATURE

GIC = Glass Ionomer Cement
Cs = Compressive strength, MPa.
VHN = Vickers Microhardness numbers, kgf/mm^2
BFS = Biaxial Flexural Strength, MPa.
WR=wear rate losses, µg/m
ΔM= mass losses, µg
ω = disk rotating speed, rad/sec
r = disk radius, m
t = slipping time, sec