INVESTIGATIONS ON MECHANICAL PROPERTIES OF TITANIUM REINFORCED GLASS IONOMER CEMENT (GiC) - CERAMIC COMPOSITES SUITABLE FOR DENTAL IMPLANT APPLICATIONS

R. B. DURAIRAJ[a*], S. SIVASARAVANAN[b], D. K. SHARMA[c]
S. RAMACHANDRAN[d], A. HEBOYAN[e]

[ab] School of Mechanical Engineering, Sathyabama Institute of Science and Technology, Chennai
[c] Department of Mathematics , Jaypee University of Engineering and Technology, Guna (M.P), India.
[d] Department of Mechanical Engineering , CMR Institute of Technology , Bengaluru , India.
[e] Department of prosthodontics , Yerevan state medical University, Armenia

Reinforcement of high strength, corrosion resistant materials to conventional ceramic composites effects on improvement of wear resistance, hardness and other mechanical behavior of the ceramics. In this paper it has been discussed about the effect of Titanium (Ti) metal powder (280 Mesh) is Reinforced with Commercially available Glass Ionomer Cement (GiC) to form a ceramic composites in the standard ratios of 8% and 16%. These composites samples were prepared in laboratory at room temperature and capsule dies were used for the preparation of the samples. All the samples were taken for wear, Scanning electron Microscope, Surface hardness, Diametrial tensile test, and Raman Spectroscopy. It is observed that the wear resistances of the Ceramic composites were improved in increasing the addition of Ti percentage, and greater hardness values were achieved on 8% of Ti addition to GiC ceramic composites. In SEM Images of the samples we have observed many cracks on the inner region because of setting time, from these, it is observed that settling time of these ceramic composites have greater role in the achievement of mechanic properties because, the inner region of the ceramic composites required more time to settle (Dry) when compared to the outer surface.

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1. Introduction

Dental cement or filler materials were employed to fill in the oral environment which is stable and to give resistance towards the wear and tear of the food particles chewing by the human being. Biag MS and Fleming GJP (2015) reported that the GI powder has undergone various modifications from its earlier GI powder formulation (G200) to improve their reaction with polyacid liquid. It has been improved by manufacturers in terms of chemical composition, molecular weight for ease handling in the oral region [1]. Wilson AD (1996) The Glass Ionomer (GI) is the Major Composition of Silica, Aluminium oxide, Calcium fluoride and sodium fluoride which is the usable GI cement after mixed with 50% of poly acrylic acid solution clinically[2]. Imran alammoheet (2018) et al were experimented on addition on nano hydroxyapatite powder with silica to GIC. It was reported that the drastic increase on Vickers hardness (64.5 to 68.7) and Compressive strength of 143.2 to 13.94 Mpa, which is due to the addition on 10% of Hydroxyapatite nano powder with silica to the conventional GIC[3]. Li sun et al (2018) were experimented on addition of Fluorinated graphene with conventional GIC in 0.5%, 1%, 2% and

[*]Corresponding author: rbdurairajmech@gmail.com

4% of weights. During this addition of Fluorinated graphene, it was observed that the increase and hardness and compressive strength and also the antibacterial rate of s.mutans is increased up to 85% during the 4% addition of Fluorinated graphene[4]. Ghadirrajabzadeh et al (2014) were experimented on development of Hydroxyapatite / yattria stabilized zirconia (HAP/YSZ) reinforced GIC. It shows that the addition of 20% Hydroxyapatite / 80% Yattria stabilized zirconia with conventional GIC was found increased in compressive strength of (1857 – 245 MPa) and higher diametral tensile strength of (11 – 14 MPa) , Microhardness of (104 – 106 Mpa) when compared to pure GIC of Compressive strength (65-88Mpa) , Diametral tensile strength (5-9.5 MPa) and microhardness (70-89Mpa)[5].Kamila RosamiliaKantovitz et al (2020) we worked on addition of TiO_2 nanotubes to conventional GIC. They were observed that the addition of TiO2 nanotubes does not have much impact on the adhesiveness. But it has a greater role in improvement of compressive strength and microhardness and also the weigh reduction of the surface after the wear examination when compared to conventional GIC[6]. Sufyangaroushi, pekkavallittu, lippolassila (2017) were reinforced hollow and solid discontinuous glass fibers with pure GIC. The addition of 10% weight of hollow glass fibers on GIC were improved the fracture toughness, flexural strength when compared to unreinforced materials[7]. I.Paiva et al (2017) were worked on addition of silver nano particles with GIC in a polyacrylate solution. It is reported that higher concentration of silver were results in increase of settling time up to 32%. It also induced the growth of E-coli growth distribution which indicating the diffusion of silver nano particles over the material surroundings. The metabolic activity of s.mutans was improved with higher concentrations of silver[8].Hanan Al Zraikat et al (2011) were undergone an experiments on reinforcement of casein phosphopeptide–amorphous calcium phosphate with GICupto 5% of weight. This addition significantly decrease the fluoride release rate and higher calcium and inorganic phosphate release were found. The addition of 3% casein phosphopeptide–amorphous calcium phosphates with GIC were improved the anticariogenic ability without affecting the mechanical properties of the GIC[9]. Ismail Ab Rahman, et al (2017) were performed the addition of nanozirconia with Hydroxyapatitte and silica nano powder to GIC. The highest hardness values were achieved in 5% addition of the Mixture with GIC , which is about 54% (79.38 HV) in reference to the non reinforced GIC (40-50 HV)[10]. In this research work the addition of Titanium powder has greater impact on improvement of the mechanical behavior of the materials. In addition to this the effect of setting time has greater impact on further enhancement of the composites in terms of its mechanical behavior.

Table 1. Reinforcement materials and its effects on Glass ionomer cement.

Reinforcement Materials	**Authors**	**Discussions**	**Year**
Nano – Hydroxyapatite + Silica	Imran alamMoheet et al	Highest value of Vickers hardness and Compressive strength were recorded in addition of HAP Nano powders to silica in comparison with conventional GIC	2018
Fluorinated Graphene	Li sun et al	Addition of Fluorinated Graphene to the Conventional GIC will not only improve the Vickers hardness and Compressive strength , it also improves the antibacterial activity	2018
hydroxyapatite/yttria-stabilized zirconia (HA/YSZ)	Ghadirrajabzadeh	After settling time of 1 to 7 days the (20% Hydroxyapatite / 80% Yttria stabilized zirconia) + GIC combination shows higher compressive strength of 11- 14 MPa	2014
TiO_2 Nanotubes	Kamila RosamiliaKantovitz et al	Addition of TiO_2 nanotubes with GIC has reduced the percentage of weight loss after the surface wear	2020
Hollow and solid discontinuous glass fibers	Sufyangaroushi, pekkavallittu , lippolassila	The addition of hollow and solid glass fibers to GIC were improved the flexural strength and toughening performance in comparison with pure GIC	2017

Reinforcement Materials	Authors	Discussions	Year
Silver nanoparticles	I.Paiva et al	Addition of silver nano particles has increased the setting time to 32%. it was also found the increase in metabolic activity s.mutan in higher concentration in increasing silver nano particles.	2017
casein phosphopeptide–amorphous calcium phosphate	Hanan Al Zraikat et al	The incorporation of casein phosphopeptide – amorphous calcium phosphate with GIC has improved the anticariogenic ability without affecting the mechanical properties of the GIC.	2011
Nano Zirconia + Hydroxyapatitte + Silica nano powder	Ismail Ab Rahman, et al	The Microhardness results were significantly improved up to 54% due to the addition of the nanozirconia + Hydroxyapatitte + silica nano powder to GIC.	2017
hydroxyapatite and fluoroapatitenanobioceramics	AlirezaMoshaverinia et al	After setting time of 1 – 7 days the cements showed good bonding strength, higher compressive strength (177 – 179MPa)	2008

2. Materials and methods

2.1. Materials

Commercially available Glass ionomer cement (GC FUJI IX Glass Ionomer Cements) were purchased from chennai market. Titanium metal powder of (250 mesh size) where purchased from (Gautam Dyes and Chemicals, Sowcarpet, Chennai.

2.2. Sample preparation

Samples of Pure GIC were transferred to circular glass plate which and mixed with titanium metal powder with calculated weight percentage of 8% and 16% with GIC. These samples were maintained in separate glass plates to avoid the errors during the sample preparations. The glue were added slowly to the powders and transferred to the circular capsules to get definite shapes which will be comfortable for testing purposes. All the samples were prepared under lab condition with temperature ranges from 36±1°C.

2.3. Setting time

All the prepared samples were taken for setting time as per the ISO standards[11]. The samples were tested at room temperature of 36±1°C with the relative humidity of 88% were maintained. The setting time was recorded by allowing in contact with intender vertically with the mass of 250g with diameter of 1 mm over the surface of the composites for the dwell time of 5s. the recorded setting time were listed in the table.

Table 2. Calculated setting of the composites.

SL No	Composition	Setting Time (s)
1	Pure GIC	243
2	8% Ti + GIC	325
3	16% Ti + GIC	356

2.4. Scanning Electron microscopy

The samples were shaped to 1cm X 1 Cm using wire cut EDM from the circular samples to study the surface morphology. These samples were tested in Carl ZEISS AG Brand Scanning electron Microscope made in Germany was available at International research center of sathyabama institute of science and Technology, Chennai.

2.5. Raman spectroscopy

The Reinforced samples were taken for raman spectroscopy (RENISHAW Lexel 95 – SHG TM) available at thin flim lab of international research center at sathyabama institute of science and technology. The test has been carried out under regular focus mode with spectra range of 100.7 to 3202, grating of 2400 l/mm (Vis) and exposure time of 20000 ms.

2.6. Linear reciprocating tribometer

The wear examination of the samples was taken in linear reciprocating tribometer of DUCOM Instruments. The samples were tested under the frequency range of 1-30 Hz, stroke length of 30mm, with frictional load varies from 0-10N with intender ball diameter of 6mm made up of Al_2O_3. This test was carried out in room temperature ranges 36±1°C.

2.7. Surface hardness

The surface hardness examination were done using Vickers Hard Wilson Wolpert – Germany make testing machine. The circular samples of 4mm diameter and 6mm height were used for the testing. Packs of three samples were maintained to produce the average results. Each pack includes a sample from pure GIC, 8%Titanium reinforced GIC and 16% Titanium reinforced GIC. The results were recorded and plotted with samples and hardness number.

3. Results

3.1. Scanning Electron Microscope

The scanning electron microscopic images were shown in Fig. 1 the reinforcement of GIC and Titanium powder was made properly with strong bonding. The titanium were able to notice over the surface in white color balls like structure and some of them were attached over the surface of GIC. It also observed some of the cracks over the surface of the dentin.

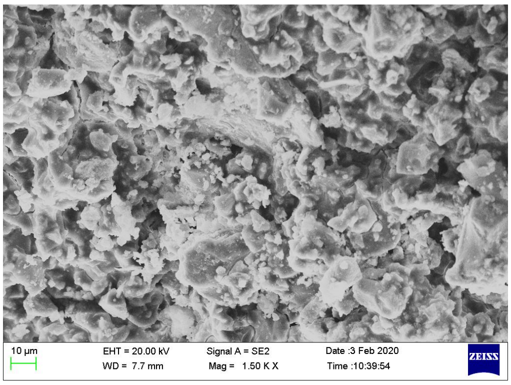

Fig. 1. Scanning Electron Microscopic image of Titanium reinforced GIC.

3.2. Raman spectroscopy

The Raman spectroscopic outcomes were plotted between Raman Shift/cm^{-1} and the Counts. The Peaks were seen on 482.21, 1462.14, and 2437.17 at center and height of 2180.5, 1120.86, and 667.47. It likewise watched the most extreme Peak height of 5783.58 with center of 2934.51 and width of 57.62. This peak values has been changed in examination with increase in addition of Titanium powder. At first, the reinforcement of 8% of Titanium to GIC shows the Peaks of 380, 823, 967, and 1021 which are smaller Peaks when contrasted with different compositions. If there should be an occurrence of 16% addition of titanium we have watched the Peaks ranges from 521, 642, 930, 981, 1110, and the most extreme Peak of 2935 which shows the holding of titanium with the GIC which shapes a solid fortification with the dentin.

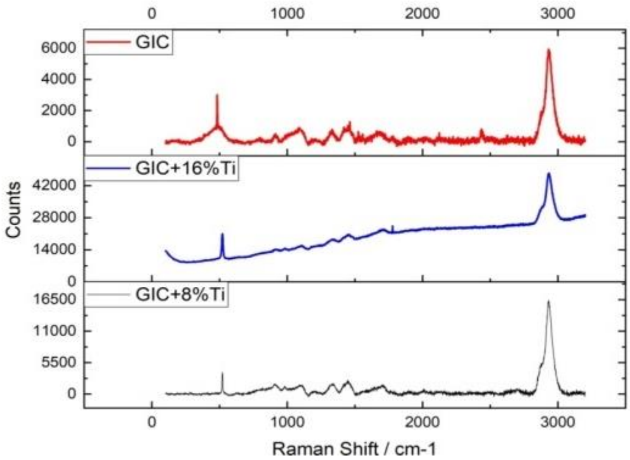

Fig. 2. Raman spectroscopic images of 8%, 16% Titanium Reinforced GIC and pure GIC.

3.3. Surface hardness

The surface hardness results were plotted for three trials with the recorded values. The results were shown in graph. It is observed that the surface hardness results of pure GIC ranges from (78.9 – 80.5), whereas the 8% of Titanium reinforced GIC shows the surface hardness of (30.2 – 38.7). in case of 16% reinforcement of Titanium with GIC shows greater improvement of surface hardness (56.5 – 67.2) this is about 90% of improvement on surface hardness values when compared to 8% addition of Titanium reinforcement. This is due to the impact of reinforcement in higher concentration with the GIC.

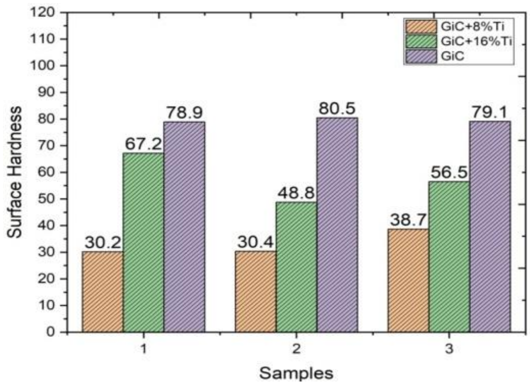

Fig. 3. Surface hardness results of Titanium reinforced samples.

3.4. Linear reciprocating tribometer

The Linear reciprocating tribometer results were plotted between co-efficient of friction and time (s). The test results shows that the addition of 8% Titanium reinforcement with GIC was subjected to more wear and tear in comparison with 16% addition of Titanium. The increase in concentration of titanium is acting as a protective layer of the dentin and reduces the wear rate. In continuation with the reduced concentration of titanium the percentage of reinforcement with GIC is poor and increasing linearly with the rate of wear.

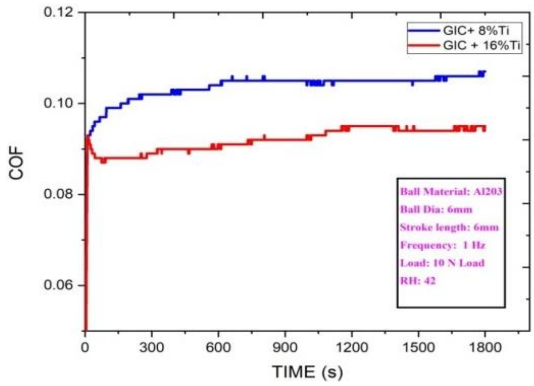

Fig. 4.Linear reciprocating tribometer results of Titanium reinforced GIC.

4. Discussions

The surface hardness value of 8% addition of Titanium to the GIC is not satisfactory even in comparison with the pure GIC. This shows the smaller addition of reinforcement materials will not have much influence; also it does not have any quantified improvement over the reinforcement with GIC (Prentice LH et al (2006)). In case of 16% addition of the Titanium reinforcement over GIC has shown about 90% improvement of results when compared to the previous one. This is due the formation of strong bonding with GIC and act as a thick protective layer which very suitable for the oral fillings. The surface hardness impact of 8% Titanium + GIC may also due to the setting time. Increase in setting time may results in formation of strong bonding with GIC which enable/ improve the strength of the reinforcement. The Presence of minute cracks on the surface area of Glass ionomer were due to dryness of the surfaces after the chemical reaction. These cracks may also occurs due to the shrinkage of resins with the powder and residual stresses between the organic matrix and fillers [19-20]. Some of the smaller pores were identified during the scanning electron microscopic examination. This is due to the air assimilate during the reinforcement with powder and the resin in hand mixture which may results in decreasing the polymer attainment due to impediment of setting time reaction of oxygen in the bubbles[21]. These pores will act as ainitial stage of cracks over the surface of the dentin which may affect the color and accumulate the oral fluids and biofilms which may finally cause dental restorations and gingival inflammations [23-24].

The Linear reciprocating tribometer results were plotted. These results revealed that the surface degradation of the materials will be more on less concentration of the reinforcements. The addition of 16% Titanium over the Glass ionomer will have greater resistance towards the wear. In case of 8% Titanium addition to the glass ionomer shows slight variations in the wear resistance. But, the conventional glass ionomer has observed with largest damage area in the reciprocating wear examination. Also it was found a huge material loss when compared to the titanium reinforced samples. Because of the presence of cracks over the surfaces of the glass ionomer, local stress concentrations were developed during the reciprocating wear examination [24 – 25].

5. Conclusions

The experimentation on Reinforcement of Titanium with Conventional Glass ionomer cement were performed. The following conclusions were made with this work. For 8% addition of titanium reinforcement with glass ionomer cement will not have greater effect in obtaining quantative improvements on the glass ionomer. This may reduce the rate of wear nearly about 25 to 35% during the linear reciprocating wear examination, but it is found the surface hardness values were very low when compare to pure Glass ionomer. For 16% addition of titanium to glass ionomer has some good improvements in terms of wear resistance, surface hardness results (64

Hv) which is greater than the 8% addition of Titanium. These substrates also found less cracks and very strong bonding with the glass ionomer.

Setting time has greater impact with the material properties. Factors like environmental conditions, mixing time, proportion of resin may influence directly in the mechanical properties of the materials.

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