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Research Article

# Fracture Toughness, Hardness, and Microstructure of Ceramic Modified Versus Resin Modified Glass Ionomer Restorative Materials

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**Received date: February 16, 2022; Accepted date: March 25, 2022; Published date: March 30, 2022**

**Citation:** Ibrahim M. Hamouda, and Noha N. Mohamed**.** (2022). Fracture Toughness, Hardness, and Microstructure of Ceramic Modified Versus Resin Modified Glass Ionomer Restorative Materials."J. Biomedical Research and Clinical Reviews. 6(5); DOI: 10.31579/2692-9406/110



## Abstract

The aim of this study was evaluation and comparison of fracture toughness, hardness and microstructure of ceramic-modified versus resin-modified glass ionomer restorative materials. For fracture toughness, 20 specimens from both materials were fabricated in a stainless-steel split mold (25mm length, 2.5mm thickness and 5mm width). Fracture toughness was measured using Lloyd universal testing machine with cross head speed 2 mm/min. For hardness testing, 20 specimens from both materials were fabricated in a disc-shaped stainless-steel mold (5mm diameter and 2mm height). All specimens were stored in distilled water at 37°C for 24 hours. Hardness was measured using Vickers micro hardness tester with 50 g load. The microstructure of both materials was examined using scanning electron microscopy. The results of this study showed there wasn't significant difference between both materials in fracture toughness. The hardness of ceramic-modified glass ionomer was higher than that of the resin-modified type. The addition of ceramic filler to the glass ionomer didn't improve its fracture toughness. The microstructure of resin-modified glass ionomer showed tight packing between the matrix and the resin filler, while the ceramic-modified glass ionomer showed separated matrix from the ceramic filler.

**Keywords:** cermics- resins; glass ionomers; hardness; fracture toughness; restorations

## Introduction

Glass ionomer was developed in 1960s by Allan Wilson to replace dental silicate cement that were used for restoration of anterior teeth. The conventional glass ionomer cements (GICs) have several attractive properties, such as fluoride releasing, bacteriostatic action, chemical bonding to enamel and dentine, similar to tooth color and high biocompatibility. Also, they have low coefficient of thermal expansion similar to the tooth structure. On the other hand, they have several drawbacks such as high-water solubility, slow setting, low fracture toughness and poor resistance to wear and dehydration during setting [1,3].

Because of low tensile strength, brittleness, and fracture toughness of GICs, a several modifications have been done to improve their mechanical properties. These modifiers were addition of resin materials, ceramics, glass fibers, metal particles, palladium or glasses [3]. In the late 1980´s, the addition of polymerizable hydrophilic resins to conventional glass ionomer cements resulted, in the development of resin-modified (RMGICs). RMGICs have been introduced to improve the strength and hardness of the conventional GICs. These materials have good wear resistance, high fracture toughness, higher resistance to moisture, and a longer working-time. Although of the previous improvements still they have high polymerization shrinkage and low wear resistance [4,5].

Recently, a new ceramic-reinforced glass ionomer has been introduced to the dental market. This tooth-colored material is produced by the manufacturer to combine the high strength ceramic materials and the aesthetics of GICs. It can be concluded that ceramic-reinforced glass ionomer can be used as an effective restorative material due to its higher mechanical properties and fluoride releasing capacities [6]. Nanotechnology plays an important role in the modification of GICs such as nano-bioceramic, nano-sized hydroxyapatite, and nano-filled resin to improve the mechanical properties of GICs [7].

Regarding the continuous development of GICs restorative materials, the null hypothesis of this study was there were no significant difference between the ceramic modified or resin modified GICs in the mechanical or structural properties.

## Materials and methods

The materials used in this study were, resin-modified glass ionomer (Photac fil, 3M ESPE AG Dental Products, D-82229 seefeld, Germany); and ceramic-modified glass ionomer (Amalgomer CR, Advanced Healthcare Ltd., Tonbridge , TN11 8JU , UK).

### Fracture toughness test:

A total of 20 specimens were prepared from both materials, 10 specimens each in a specially designed stainless-steel split mold of 25mm length, 2.5mm thickness and 5mm width. The mold contained a V-shaped elevation to produce a notch in the specimen with a standard geometry (0.5mm width and 2.5mm depth). The mold was first wiped with a separating agent using a cotton pellet to facilitate the separation of the cured specimens. Photac-fil specimens were prepared using the capsule form of the material which activated for 3 seconds using its special activator. The activated capsule was mixed in amalgamator for 15 seconds according to the manufacturer's instructions. The mix was dispensed into the mold using its special applicator and condensed in the mold using plastic condenser wiped with a separating medium. A celluloid strip and a glass slab were used to compress the material in the mold under pressure of about 0.5 Kg producing smooth surface. Each specimen was cured from both sides using light curing unit (Litex 680 A, Dentamerica, California, USA) for 30 seconds for each side according to the manufacturer's instructions. Each specimen was cured in three overlapped parts corresponding to the area covered by the tip of the light curing unit with a light intensity 450 mW/cm$^2$. Light intensity was monitored via a radiometer at 450 mW/cm$^2$. Light intensity was monitored by a radiometer (Demetron, Kerr, USA). Specimens were removed from the mold after curing and stored in distilled water at 37˚ C for 24 hours.

Amalgomer CR were prepared by mixing of the powder with the liquid according to the manufacturer's instructions on a glass slab using plastic spatula. The mixed material was condensed into the mold and left for 3 minutes 30 seconds setting time. Specimens were stored in distilled water at 37˚ C for 24 hours. All specimens were subjected to three-point bending test using Instron universal testing machine (Model 2006; Instron Corp., 5500 R). Each specimen was fixed in the lower part of the machine with the notch facing down. A cylindrical aluminum block with tapered end was fixed to the upper part of the machine by locking screws. This tapered block applies central load (just above the notch) with a cross head speed of 2 mm/min until fracture occur. The fracture load was recorded in Kg (which was transformed into Newton according to the formula 1 Newton = 0.102 Kilograms). Stress intensity factor $K_{IC}$ (MPa.m$^{0.5}$) was obtained from the peak load and the specimen configuration by the following equation [8].

$$K_{IC} = [ (P_Q . S) / (B.W^{1.5})] F (a/w).$$

Where $P_Q$ is the peak load (N), S is the span length (m), B is the specimen thickness (m), a is the crack length (m), W is the width of the specimen (m) and (a/w = 0.5).

$$F(a/w) = \frac{3(a/w)^{0.5} [1.99-(a/w)(1-a/w)(2.15-3.93(a/w)+2.7(a/w)^2)]}{2(1+2a/w)(1-a/w)^{1.5}}$$

### Hardness test:

A total of 20 specimens were prepared from both materials, 10 specimens each in a specially designed stainless-steel split mold to produce disc-shaped specimens with 5mm diameter and 2mm thickness. Specimens were prepared as mentioned before. The hardness was measured using a microhardness tester (Digital Vicker's Microhardness tester (FM-7) Japan) with 50 g loaded diamond indenter. Vickers hardness number (VHN) of each indent was automatically calculated by the aid of micro-computer within the tester. The results were recorded in Kg/mm$^2$.

### Microstructure test:

Two representative polished specimens from both materials were used for Scanning Electron Microscopy (SEM) examination (Electron probe micro-analyzer operating at 30KV Jeol type). A gold layer of 3 µm thickness was spattered over the specimen's polished surface using argon sputter coater (S150 A sputter coater, Edward, England) for 2 minutes. A high vacuum was used for dehydration of the coated specimens before SEM analysis.The microstructure of the coated specimens was analyzed at magnifications X1700 and X4000.

### Statistical analysis

The mean values of fracture toughness and hardness were subjected to statistical analysis using t-test to assure their significance.

## Results

Mean fracture toughness for Photac-fil and Amalgomer CR glass ionomers are presented in Table 1.

| Materials | Mean + SD | t-zalue | p-vlue |
|---|---|---|---|
| Resin-modified glass ionomer (Photac-fil) | 54.3 + 3.1 | 10.31 | <0.001 |
| Ceramic-modified glass ionomer (Amalgomer CR) | 69.1+ 3.3 | | |

**Table 1:** *Mean and standard deviation for fracture toughness values of both materials*

The results showed that resin-modified glass ionomer (Photac-fil) had slightly higher fracture toughness than that of ceramic-modified glass ionomer (Amalgomer CR). The statistical analysis of the results showed that there was no significant difference between both materials in fracture toughness (P>0.05).

Mean hardness values for both materials are presented in Table 2.

| Materials | Mean + SD | Mean + SD | p-value |
|---|---|---|---|
| Resin-modified glass ionomer (Photac-fil) | 54.3 + 3.1 | 10.31 | <0.001 |
| Ceramic-modified glass ionomer (Amalgomer CR) | 69.1+ 3.3 | | |

**Table 2:** *Mean and standard deviation for hardness of both materials*

The statistical analysis of the results showed significantly higher hardness values for ceramic-modified glass ionomer (Amalgomer CR) than that of the resin-modified glass ionomer (Photac-fil) ($P<0.05$).

The microstructure of Photac-fil showed that there was tight packing between the matrix and the resin filler particles with the presence of little porosity (Figure1). EDEX showed the elemental structure of resin-modified glass ionomer (Photac-fil ) showed that the main elements constructing the glass part of the glass ionomer (calcium, aluminuim and silica) Figure 2. While in Amalgomer CR, under the same magnifications X4000, the material showed slight separation between the matrix and the ceramic filler with the presence of flaws indicating the brittle nature of the filler (Figure 3). EDEX of the ceramic-modified glass ionomer (Amalgomer CR) showed that the elemental structure has a peak for zirconia in addition to the main elements of glass ionomer (calcium, aluminuim and silica) Figure 4.

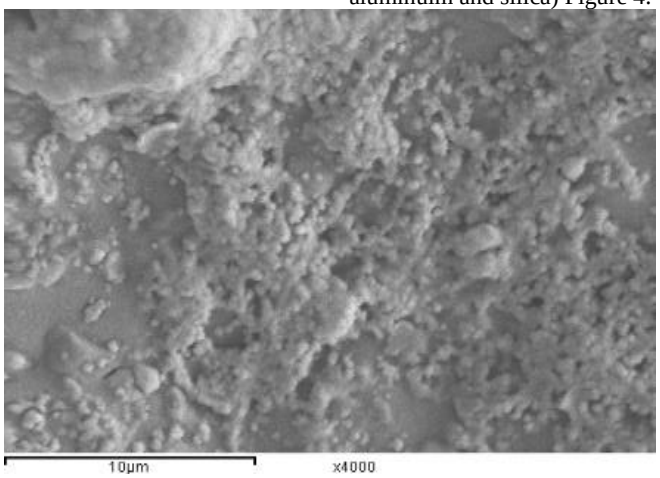


**Figure 1:** *Scanning electron micrograph showing microstructure of photac- fil (X4000 )*

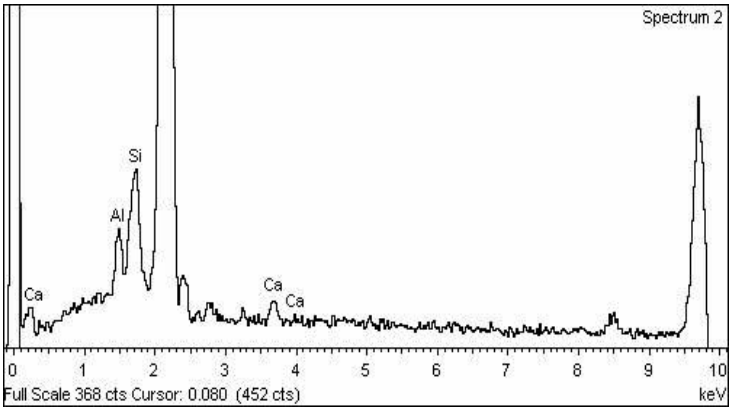


**Figure 2:** *EDEX showing elemental structure of photac-fil*

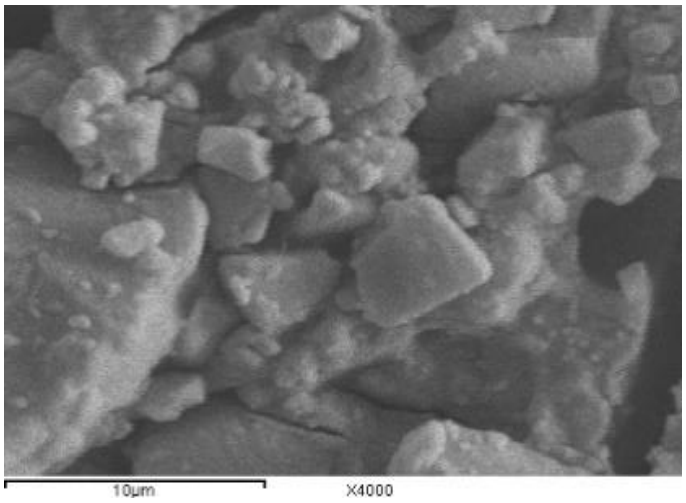


**Figure 3:** *Scanning electron micrograph showing microstructure of Amalgomer CR (X4000)*

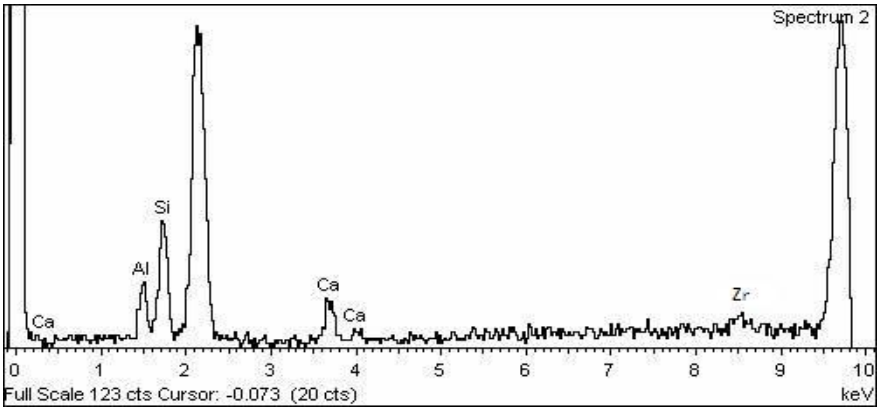


**Figure 4:** *EDEX showing elemental structure of Amalgomer CR*

## Discussion

Glass ionomer restorative materials are widely used in restorative dentistry because of their unique attractive properties. Glass ionomer cements are being used for several applications in dentistry. In order to overcome its low mechanical properties, several modifications have been introduced to the conventional GICs. The basic modifications for the conventional GICs included the incorporation of auto-cured or photo-cured resin systems to produce resin-modified glass ionomer cements. Resin modified GICs is improved by addition of nano-sized fillers to RMGICs, reducing the size of the glass particles, and producing nano-sized bioceramics to the glass powder [9.10].

The constant development in the field of restorative dentistry yield the production of a ceramic-modified glass ionomers which was supposed by the manufacture to have very good properties as it combined the chemical adhesion and fluoride release of conventional glass ionomers with excellent esthetics and packing capacity like that of amalgam which make it used as a posterior restorative material. There were many attempts to add bio inert ceramics such as zirconia powder to the conventional GICs to its mechanical properties [11].

Fracture toughness is a stress intensity parameter that resists propagation of a surface flaw or a pre-existing crack through the material. Fracture toughness of different dental materials have been conducted by a various method such as the single-edge notched beam method [12].

Resin-modified glass ionomers has higher fracture toughness than that of the conventional glass ionomer. This improvement in fracture toughness was due to the resin parts which is hydroxyethyl methacrylate (HEMA) or Bis GMA in the liquid cause's improvement of the physical properties. Also, smaller powder particle size of up to 10 mm produced smoother surface, which causes higher fracture toughness of resin-modified glass ionomers. The high fracture toughness values of the resin-modified glass ionomer may be one of the contributing factors in the clinical success in high stress-bearing areas [13].

Amalgomer CR is ceramic modified GIC, which complies with the international standards of GIC and with the standard for amalgams. The ceramic part contributed in excellent erosion and wear resistance and also improves the radiopacity and improves the strength of the cement [6]. Amalgomer CR setting is a conventional chemical acid–base reaction, while the resin-modified GICs depend on light curing. The results of this study showed higher fracture toughness and hardness for the Amalgomer CR than that of Photac-fil due to the presence of the zirconia as a ceramic material of high hardness. Zirconia is known to be an excellent material for strengthening and toughening of any restoration because of its peculiar character of a phase transformation from tetragonal to monoclinic under stress. This transformation inhibits crack propagation and increases the fracture toughness [3].

Ceramic-modified glass ionomer restorative materials have high mechanical properties comparable to dental amalgam. Zirconia-modified GICs are sensitive to moisture. Storage in artificial saliva has a detrimental effect on the failure load of ceramic-modified GICs that may indicate long-term deterioration in service [3,14]. Amalgomer CR could be used as a permanent restorative material because of its higher compressive strength and comparable antimicrobial efficacy to the conventional GICs [14]. The reason may be due to the filler type which is ceramic particles in Amalgomer CR and according to the manufacturer the ceramic filler is able to react partially with the matrix, which may produce some bonding and thus increases the overall strength of the restoration while in Photac-fil glass ionomer cement no such reinforcement of filler particles.

The results of the present study showed no significant difference in the fracture toughness of resin-modified glass ionomer (Photac-fil) and ceramic-modified glass ionomer (Amalgomer CR). This may be attributed to the brittle nature of the ceramic filler which in turn don't contribute in increasing the fracture toughness of the material. Fracture toughness of ceramic-modified glass ionomer was approximately near to that of resin-modified glass ionomers and this can be explained on the basis that the reinforcement with zirconia which undergo phase transformation under stress with increase in volume 4% causing local compressive area stopping crack propagation and in turn increase fracture toughness [3].

The results in the present study showed that the Amalgomer CR, recorded higher Vicker's hardness number than Photac-fil. These results may be explained on the basis that the resin-modified glass ionomer contains a resin component on the surface of the specimen and the filler particles migrate toward the bulk of the material. This resin-rich layer often remains only partially polymerized due to the oxygen inhibition of polymerization, this effect doesn't occur with the ceramic-modified glass ionomer [15,16] The higher vicker's hardness numbers for the ceramic-modified glass ionomer are explained on the basis of the hard nature of the zirconia reinforcement [17].

Scanning Electron microscopic evaluation in the present study showed that, Photac-fil had tight packing between the matrix and the resin filler particles with the presence of little porosity. While Amalgomer CR had slight separation between the matrix and the ceramic filler with the presence of flaws indicating the brittle nature of the filler. These results were in agreement with another study which reported that the SEM photomicrograph of amalgomer CR revealing the matrix of the material with glass and zirconia fillers. After 6 months of water aging, the surface showed zirconia fillers not bonded to the matrix, surface irregularities,

and microporosities [18]. The examined specimens showed the presence of voids which may be due to the air bubbles trapped within the material during specimens' preparation [17].

## Conclusions

Based on the results of this research, the following conclusions were observed:

1. Resin-modified glass ionomer (Photac-fil) showed slightly higher fracture toughness than that of the ceramic modified glas ionomer (AmalgomerCR).
2. Resin-modified glass ionomer (Photac-fil) showed significantly lower hardness than that of the ceramic modified glas ionomer (AmalgomerCR).
3. The microstructure examination revealed there was higher integrity between matrix and filler in the resin-modified glass ionomer (Photac-fil) more than ceramic-modified glass ionomer (AmalgomerCR).

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**DOI:** 10.31579/2692-9406/110