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*Original Research Article*

# Effect of heat on the hardness of glass carbomer cement used in atraumatic restorative treatment

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## Abstract

**Introduction and Objective:** This study aimed to evaluate the effect of heat treatment on the microhardness of two glass ionomer cements: EQUIA Forte (GC Corporation) and GCP Glass Fill (GCP-Dental). **Material and methods:** Twenty specimens of the two glass ionomer cements were prepared following the manufacturer's instructions, and 10 specimens of each material received heat treatment with a light curing unit (Carbo LED lamp, GCP-Dental). After seven days of storage in distilled water at room temperature (23°C), the 40 specimens were submitted for Vickers microhardness test (microhardness HMV 2T). Five indentations were performed on each specimen with a load of 100g, with a 10 second penetration time. **Results:** For both materials, the average microhardness value were higher in the groups that did not undergo heat treatment, the group presenting with the highest microhardness value was EQUIA Forte (GC Corporation) without heat treatment (125.3), and the lowest value was found for GCP Glass Fill (GCP-Dental) with heat treatment (72.9). **Conclusion:** The heat treatment had no influence on the microhardness of the glass ionomer cements tested.

## Introduction

Conventional glass ionomer cement has been used as a restorative material in clinical practice because of its excellent characteristics such as biocompatibility [14], chemical adhesion to enamel and dentin [18], adhesion to wet surfaces [3], coefficient of linear thermal expansion very close to that of the tooth [5], and continuous fluoride release [8] helping in the remineralization of the dental structure.

The advantages of glass ionomer cement have made it the material of choice for atraumatic restorative treatment (ART) [15]. ART is a minimal intervention approach that seeks to preserve as much dental structure as possible and entails the partial removal of decayed tissue and infected dentin with manual cutting instruments. Cotton rollers are used to prevent contamination of the site. Glass ionomer cement is applied after cleaning the cavity [4, 6, 7].

Conventional glass ionomer cements have some drawbacks such as a slow setting time, which makes it easier to have their water content altered either by dehydration or absorption of water from saliva [3]. The setting reaction of these cements is divided into two phases and begins immediately after mixing the powder containing the glass particles with the acidic liquid [9]. The first stage starts 10 minutes after mixing, and this material is susceptible to water contamination [9] and even dehydration in the second stage [9].

In an attempt to improve the mechanical characteristics of glass ionomer cements, some studies [3, 9] suggest the application of heat and the use of ultrasound during the setting reaction of these cements to reduce water sensitivity, which results in surface softening and thereby low wear resistance [9].

Another alternative was the development of a new glass carbomer restorative material. This developed material was self-curing and chemically similar to conventional glass ionomer cement [11]. Glass carbomer cements differ from conventional glass ionomer cements in their nanometric dimensions of the powder particles and fluorapatite in their composition [1]. Moreover, as indicated by the manufacturer, the glass carbomer cement requires energy (heat) to be applied from a light source of 1,400 mW/cm² for 60-90 seconds. The Carbo LED lamp (GCP-Dental, Ridderkerk, the Netherlands) is recommended by the manufacturer to accelerate setting reaction and achieve excellence in the properties of this material.

Therefore, the aim of this study was to evaluate the effect of heat treatment on the hardness of glass ionomer cements. The null hypothesis tested was as follows: (1) glass ionomer cements will have the highest microhardness values when subjected to heat treatment; (2) the glass carbomer cement will have a higher microhardness value when subjected to heat treatment.

## Material and methods

Two encapsulated glass ionomer cements were tested in this study: the GCP Glass Fill (GCP-Dental, Ridderkerk, the Netherlands) and EQUIA Forte glass carbomer (GC Corporation, Tokyo, Japan), combined with a heat source (Carbo LED lamp, GCP-Dental, Ridderkerk, the Netherlands) (table I).

**Table I** – Glass ionomer cements used in the study

| Commercial name | Composition | Lot |
|---|---|---|
| EQUIA Forte (GC Corporation, Tokyo, Japan) | Aluminum and silicate glass, polyacrylic acid, and distilled water | 7311044 |
| GCP Glass Fill (GCP-Dental, Ridderkerk, The Netherlands) | Fluoride glass, aluminum and silicate, apatite, and polyacids | 1506081 |

* According to the manufacturer's information

### Sample preparation

Twenty specimens (5mm diameter and 2mm thickness) were made for each glass ionomer cement, and 10 specimens of each material were heat-treated (Carbo LED lamp, GCP-Dental, Ridderkerk, the Netherlands). The total number of specimens prepared was 40.

Glass ionomer cements were prepared following the guidelines of their manufacturers. The contents of the capsules after rupture of the inner seal by hand pressure were immediately homogenized in an Ultramat 2 double elliptical capsule mixer (SDI, Victoria, Australia). After the mix homogenization time (10 seconds), the capsules were adapted to the applicator (Riva Applicator 2, SDI, Victoria, Australia). The matrix base was placed on a glass plate that had Vaseline applied (solid Vaseline, Quimidrol, Joinville, Brazil) to facilitate the removal of the specimens. Then, a polyester strip (Maquira Dental Products, Maringá, Brazil) was placed over the matrix base. The glass ionomer cements were then applied inside the matrix with the aid of a spatula (Millennium, São Caetano do Sul, Brazil).

After this procedure, a new polyester strip (Maquira Dental Products, Maringá, Brazil) was placed over the specimen, and with the upper metallic part of the matrix, the material was compressed with a weight of 2 kg for 30 seconds to allow overflow and obtain a smooth surface [10].

Ten specimens of each material, GCP Glass Fill (GCP-Dental) and EQUIA Forte (GC Corporation), received heat treatment with the Carbo LED lamp (GCP-Dental Carbo LED lamp, GCP-Dental, Ridderkerk, the Netherlands) at 1,200 mW/cm$^2$ for 60 seconds, as indicated by the manufacturer of GCP Glass Fill (GCP-Dental). The light curing unit had its power measured by a Demetron analog radiometer (Kerr, Orange, USA) before the preparation of each specimen to ensure full capacity with regard to light intensity.

After completion of the elaboration processing of each specimen, the matrix was opened and the specimen was removed, and its thickness was confirmed by a specimeter (Golgran, São Caetano do Sul, Brazil).

The upper surfaces of the specimens were identified using an ink point pen in the northern region by referring to the position of the operator with a blue pen (BIC, Clichy Cedex, France) to guide the microhardness test.

At the end of these steps, the specimens were placed in amber colored plastic containers (EMBALEVE, Joinville, Brazil), previously identified and marked kept for 24 hours in a 100% humidity environment (Waterbath, Kottermann Labortechnick, Germany) to complete the setting reaction of the glass ionomer cements. Then, each specimen was immersed in 20 ml of distilled water at room temperature (23°C), for seven days.

### Microhardness test

The specimens were submitted for the Vickers microhardness test using the HMV 2T Micro Hardness Tester (Shimadzu Corp., Kyoto, Japan).

The specimens were placed on the microdurometer table and the adjustment was performed until focus was obtained. The surface was analyzed for smoothness without the presence of flaws or bubbles. Then, five indentations were made in each specimen, positioned to the north, center, south, east, and west, respectively. When pacing the indentation at the extremities, at distance of 1 mm from the margin of the specimen was respected in order to guarantee the result without weakening the material [10].

The test was performed under a load of 100g, with a penetration time of 10 seconds [10]. The microhardness tests were read with the Vickers diamond tip which produces a square shaped indentation. Once the start/end point of each indentation line was determined, the mean reading of the two formed diagonals ($\mu$m) was converted to Vickers Hardness (HV) values by the device itself.

### Statistical analyses

For each specimen, an average of the five microhardness data collected was calculated. These data were subjected to a one-way analysis of variance through the ANOVA statistical test at a significance level of 5%. Tukey test was applied for contrast of the means. Values of $p<0.05$ were considered significant.

## Results

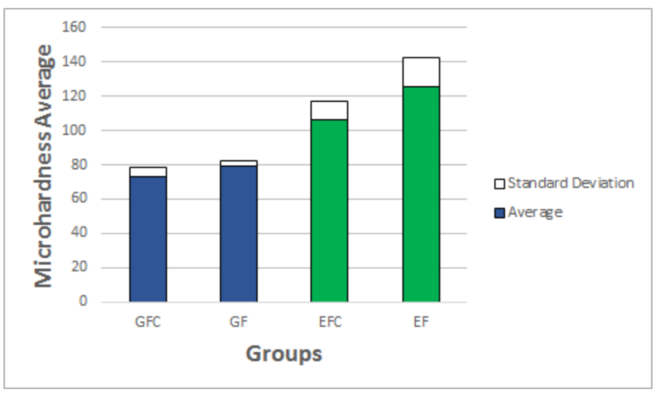
The highest microhardness average readings were obtained for the non-heat-treated EQUIA Forte (GC Corporation) group, followed by the heat-treated EQUIA Forte (GC Corporation) group and the heat-treated GCP Glass Fill; the lowest average was for the GCP Glass Fill group without heat treatment (table II).

**Table II** – Microhardness averages for each group and respective standard deviations

| | **GCP Glass Fill with heat treatment** | **GCP Glass Fill without heat treatment** | **EQUIA Forte with heat treatment** | **EQUIA Forte without heat treatment** |
|---|---|---|---|---|
| **Average** | 72.9560 | 78.9710 | 106.0230 | 125.3600 |
| **Standard deviation** | 5.14 | 3.44 | 10.58 | 17.13 |
| **Sample variance** | 26.48 | 11.85 | 112.04 | 293.61 |

The microhardness mean values of the materials were higher in the non-heat-treated groups for both GCP Glass Fill (GCP-Dental) and EQUIA Forte (GC Corporation). Analysis of variance (ANOVA) showed significant differences ($p<0.05$) between the groups.

Through the analysis of the microhardness averages obtained from each experimental group with the respective standard deviations, it was observed that the groups of EQUIA Forte glass ionomer cement (GC Corporation) with and without heat treatment presented statistical differences among themselves and that their microhardness values were higher than that of the GCP Glass Fill (GCP-Dental) glass carbomer groups. Microhardness values of GCP Glass Fill (GCP-Dental) glass carbomer groups with and without heat treatment were statistically similar to each other. The microhardness of heat-treated GCP Glass Fill (GCP-Dental) material showed statistical difference when compared to that of EQUIA Forte (GC Corporation) with and without heat treatment. The microhardness of GCP Glass Fill (GCP-Dental) material without heat treatment showed statistical difference when compared to EQUIA Forte (GC Corporation) with and without heat treatment.




**Graph 1** – Microhardness average for each group and the respective standard deviations

## Discussion

Microhardness studies are appropriate to evaluate the behavior of materials when exposed to occlusal trauma. On analyzing the results of the present study and comparing them with the literature, we found divergent results regarding the influence of heat on the hardness of glass ionomer cements.

The heat-treated EQUIA Forte glass ionomer cement (GC Corporation) tested in the present study had an average Vickers microharness value of 125.3 that is significantly higher than that found in the literature [2] that showed that the highest Vickers microhardness value of conventional glass ionomer cement Ionofil Molar (Voco, Cuxhaven, Germany) was 74.2. In one study [2], among all the tested materials, the highest microhardness value of 85.1 was found for the Argion Molar silver reinforced glass ionomer cement (Voco, Cuxhaven, Germany) probably because silver amalgam powder is physically more rigid. However, in a previous study [16], the highest Vickers microhardness value found was 97.7 for Fuji IX glass ionomer cement (GC Corporation) without surface protector. This is a cement with high viscosity, which means that it has a higher powder/liquid ratio which leads to a shorter setting reaction time and, consequently, a better performance in the microhardness test. In another study [12], the highest Vickers microhardness value found was 105.1 for the conventional glass ionomer cement Riva Self Cure (SDI) without heat treatment, after one week of storage. The setting reaction and mechanical properties of glass ionomer cements are influenced by the size of their filler particles and their distribution in the matrix because small particle size increases the surface area and provides greater space for acid etching, thereby reducing setting time.

The non-heat-treated glass carbomer, GCP Glass Fill (GCP-Dental), had a microhardness value of 78.9, which is close to the value found in another study [11], where the authors evaluated the same GCP Glass Fill (GCP Dental) without heat treatment and the Vickers microhardness value found was 67.4. In the present study, the microhardness of GCP Glass Fill (GCP-Dental) without heat treatment showed lower values when compared to EQUIA Forte glass ionomer cement (GC Corporation). In another study [11], the glass ionomer cements, resin modified Fuji II LC (GC America) and Photac Fil (3M ESPE), showed lower microhardness values (63.4 and 48.6, respectively) when compared to that found for glass carbomer cement (67.4). This fact may be explained by the difference in the chemical composition of these glass ionomer cements and the authors of the study [11]. The presence of smaller glass particles in the composition of the glass carbomer cement studied results in the greater microhardness value of this material.

Heat-treated EQUIA Forte glass ionomer cement (GC Corporation) had a microhardness value of 106.0 and the glass carbomer GCP Glass Fill (GCP-Dental) had a microhardness value of 72.9, a similar result to that found in another study [12] that revealed that the highest Vickers microhardness value was 106.2 for heat-treated glass ionomer cement Fuji IX GP Fast (GC Europe). According to these authors [12], heat treatment only accelerated the setting reaction without significantly influencing the final mechanical characteristics of the materials. In contrast to the work of two other authors in different researches [13, 17], they found a way to improve the final microhardness of glass ionomer cements (Ketac Fill Plus Aplicap – 3M/ESPE; Ketac Universal Aplicap – 3M/ESPE; EQUIA Fill – GC-Dental and Ketac Molar – 3MESPE) through heat treatment and found higher microhardness values (35.4, 81.6, 47.1, and 55.0, respectively) compared to specimens of the same materials not subjected to heat treatment (11.0, 36.9, 39.9, and 53.0, respectively). According to these authors [13, 17], heat energy transfer from the light source to the material leads to increased internal ion mobility of materials in the early stages of the setting reaction, resulting in increased viscosity and decreased clutter of molecules, which leads to an acceleration of the setting reaction and consequent improvement in surface hardness of these materials.

Another study [10] showed a higher microhardness value of 60.4 (± 5.9) for glass carbomer (GCP-Dental) activated with the Carbo Led lamp curing unit (GCP-Dental) while the present study found a Vickers microhardness value of 72.9 for glass carbomer GCP Glass Fill (GCP-Dental) activated with the same heat source. In agreement with this research [10], heat treatment did not influence the microhardness nor improve the mechanical properties of the glass carbomer. According to the authors [10, 11], the factors responsible for improving this property were its nanoparticle composition and microstructure, which increased the reactive surface of the glass carbomer, thus leading to a higher microhardness.

A similar study [1] presented a microhardness test with the same materials used in the present study (EQUIA Forte glass ionomer cement (GC Corporation) and carbomer cement GCP Glass Fill (GCP-Dental)), both heat treated. The results revealed a Vickers microhardness value of 87.0 for EQUIA Forte ionomer cement (GC Corporation) and 61.2 for GCP Glass Fill (GCP-Dental) [1], similar to the values found in the present study. Heat treated glass ionomer cement EQUIA Forte (GC-Corporation) showed a higher Vickers microhardness value (106.0) compared to the heat-treated GCP Glass Fill (GCP-Dental) (72.9). The Vickers microhardness results were higher for the EQUIA Forte material (GC-Corporation), probably due to the high powder/liquid ratio and particles size [1].

Therefore, the setting reaction and subsequent mechanical properties of glass ionomer cements are influenced by particle sizes and their distribution in the matrix and not by heat treatment.

## Conclusion

From the results found, it can be concluded that heat treatment did not influence the microhardness of glass ionomer cements or increase the microhardness value of glass carbomer cement.

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