Evaluation of microhardness of newly developed glass carbomer-based dental filling material

Zeki Arslanoğlu¹, Ediz Kale², Ayşe Meşe³, Halenur Altan⁴

¹ Private Dental Practice, Hatay, Turkey.
² Private Dental Practice, Bursa, Turkey.
³ Dicle University, Faculty of Dentistry, Department of Prosthodontics, Diyarbakır, Turkey.
⁴ Gaziosmanpaşa University, Faculty of Dentistry, Department of Pediatric Dentistry, Tokat, Turkey

Abstract

Aim: This study measured the microhardness of a newly developed glass carbomer dental filling material within the first 24 hours after setting and compared it against that of two different glass ionomers, with and without heating during setting, and a resin-modified glass ionomer.

Methodology: Six cavities were prepared on acrylic resin discs. PMMA blocks were randomly divided into six groups of six cylinders and each cavity was filled with one of the tested materials according to their group. The groups were: Group 1, Fuji Triage™ with heat; Group 2, GCP Glass Fill™; Group 3, Equia™ with heat; Group 4, Riva LC™; Group 5, Fuji Triage™; and Group 6, Equia™. Microhardness was measured by the Vickers hardness scale using a microindentation hardness tester at 2, 4, 6, 12 and 24 hours after initial setting of the materials.

Results: Fuji Triage™ with heat applied during setting (Group 1) proved to be the hardest material, while Riva LC™ (Group 4) and GCP Glass Fill™ (Group 2) were the softest. Heating the Fuji Triage™ during the initial setting period significantly increased its physical strength after 24 hours.

Conclusions: It was seen that microhardness of materials which used in our study was effected both time and heat.

Keywords: Glass carbomer, heat application, mechanical behavior, glass ionomer, Equia

Introduction

Having been used for many years for restoration of posterior teeth, amalgams are now being phased out and replaced by composite materials, ceramic inlays and onlays and, most recently, glass-ionomer fillings. Amalgam fillings often fail to meet aesthetic expectations and contain mercury, leading modern patients to prefer biomimetic restorations (1). In biomimetic dentistry, glass-ionomer fillings represent a significant advancement over composite or ceramic restorations because they require less tissue removal, ensure remineralization, chemically bond to the tooth, and exhibit a thermal expansion coefficient similar to that of natural teeth (2).

Despite these positive features, the first glass ionomer cements (GICs), introduced by Wilson and Kent in the beginning of 1970s, were of limited use. These
early materials suffered from undesirable physiomechanical features, long hardening times, and moisture sensitivity during hardening (3,4). Hybrid restorative materials, such as resin-modified glass ionomer cements (RMGICs) and polyacid-modified composite resins (5,6), were developed to combine the advantages of GICs with the aesthetic and physiomechanical properties of composite resins. Although the mechanical characteristics of these hybrid materials were an improvement over those of traditional GICs, resin-containing GICs were not suitable for all applications involving permanent teeth. The failure of these fillings to meet expected mechanical criteria is often cited as the reason for their limited use (7).

The pursuit of an ideal dental restorative material continued with the development of high-viscosity GICs and, most recently, glass carbomers. The mechanical characteristics of high-viscosity GICs were enhanced by adjusting their powder/liquid ratios, particle size and distribution, and by removal of excess calcium ions on the surface of the glass particles. The hardening mechanism of these newly developed, high-viscosity cements was the same as that of traditional GICs, but the resulting fillings exhibited improved wear resistance, increased surface hardness and flexural and compression resistance, and decreased resolution. Several studies have reported that the hardening reaction of high-viscosity GICs is faster than that of traditional GICs. Thus, exposure to moisture in the early stages of setting does not negatively affect the physical properties of the material (1,8,9,10).

Recently, the effects of heat applied to GICs during setting have been evaluated. While some researchers have suggested that heating during setting increases the mechanical properties of GICs (11), other reports have contradicted these findings (12).

Glass carbomer cement (GCC), developed by Willem Van Den Bosch and Raimond Nicolaas Van Duinen in 2004, boasts excellent biomimetic and physiomechanical properties. Glass carbomer fillings harden via a chemical reaction similar to that of high-viscosity GICs. The liquid component of GCCs is polyacrylic acid, with the powder component being comprised of a nanofiller and fluorapatite (13).

This study examines the microhardness of a new GCC-based dental filling material during the first 24 hours after setting and compares these data against those of two GICs, with and without heating during the initial setting period, and an RMGIC filling material.

Materials and Methods

The current study evaluated four different dental filling materials in six groups, with each group containing six specimens. The groups, detailed in Table 1, are as follows: Group 1, Fuji Triage™ with heating during setting; Group 2, GCP Glass Fill™; Group 3, Equia™ with heating during setting; Group 4, Riva LC™; Group 5, Fuji Triage™; and Group 6, Equia™ (Table 1). Thirty-six polymethyl methacrylate (PMMA) cylinders (10 mm thick and 50 mm in diameter) were fabricated. A circular cavity (2 mm deep and 10 mm in diameter) was machined into the center of one base of each cylinder and a cyanoacrylate adhesive was applied along the edge of the cavity.

The PMMA cylinders were randomly divided into six groups of six cylinders each and each cavity was filled with one of the tested dental filling materials. Note that all of the materials were mixed for 10 s using a capsule mixer (400M; Linea TAC, Asti, Italy) prior to their application. Where applied, heat during chemical setting was supplied by a 1,400 mW lamp (GCP Carboled Lamp; GCP Dental, Ridderkerk, Netherlands). Group 1 cavities were filled with Fuji Triage™ using a Fuji Applicator® and heated for 60 s during setting. Group 2 cavities were filled with GCP Glass Fill™ using a GCP carboCAP Applicator®. The specimens were then coated with a layer of GCP Gloss™ (GCP Dental) and heated for 60 s. Group 3 cavities were filled with Equia™ using a Fuji Applicator® and heated for 60 s. Group 4 cavities were filled with Riva LC™ using a RIVA Applicator® and heated for 20 s during curing. Group 5 cavities were filled with Fuji Triage™ using a Fuji Applicator®. These were allowed to set at room temperature. Group 6 cavities were filled with Equia™ using a Fuji Applicator® and allowed to set at room temperature.

Ninety minutes after application, surface irregularities in each filling material were mechanically smoothed with 1,200 and 2,400 grit SiC sandpaper and polished consecutively on 6-, 3- and 1-μm diamond lap wheels.

Indentation microhardness measurements were acquired at room temperature with an FM 700™ (Future-Tech Corp, Kanagawa, Japan) microindentation hardness tester. Each specimen was subjected to triplicate Vickers hardness tests at 2, 4, 6, 12 and 24 hours after application of the filling material. The Vickers indenter was applied with a load of 9.8 N and loading time of 15 s. The Vickers microindentation hardness (Hv) of each specimen was calculated as

\[ H_v = 1.8544 \frac{P}{d^2} \]

where P is the applied test load in Newtons, d is the average of two indentation diagonal lengths in micrometers, and 1.8544 is a geometrical constant of the diamond pyramid tip.
Table 1. Material brand names, specifications, compositions and manufacturers

<table>
<thead>
<tr>
<th>Material Brand Name</th>
<th>Specification</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuji Triage</td>
<td>Glass Ionomer</td>
<td>Aluminofluorosilicate glass, polyacrylic acid, distile water, pigment, polybase carboxylic acid</td>
<td>GC, Japan</td>
</tr>
<tr>
<td>GCP Glass Fill</td>
<td>Glass Carbomer</td>
<td>Nanofluoro, hydroxyapatite, liquid silica</td>
<td>GCP, Netherland</td>
</tr>
<tr>
<td>Equia</td>
<td>High-Viscosity Glass Ionomer</td>
<td>Polyacrilic acid, aluminosilica glass, distile water</td>
<td>GC, Japan</td>
</tr>
<tr>
<td>Riva LC</td>
<td>Resin-Modified Glass Ionomer</td>
<td>2-hydroxyethyl methacrylate, acrylic acid homopolymer, tartaric acid</td>
<td>Riva, SDI, Bayswater, Australia</td>
</tr>
</tbody>
</table>

**Statistical Analysis**

One-way analysis of variance (ANOVA) was used to statistically analyze the data at the p < 0.05 level of significance. Sample distributions were analyzed using the Kolmogorov-Smirnov Z test. Analyses were conducted using SPSS statistical software (SPSS Inc., Chicago, IL, USA).

**Results**

The data in Table 2 and Figure 1 show that, 2 hours after application, Triage with (TRIAGE+) or without (TRIAGE−) heat was the hardest of the evaluated dental filling materials, while Riva LC (RIVA) was the softest. However, after 24 hours, Triage with heat (TRIAGE+) proved to be the hardest dental filling material, with RIVA and GCP Glass Fill (GCP) being the softest. Although Equia without heat (EQUIA−) was seemingly harder than Equia with heat (EQUIA+) at both the 2- and 24-hour timepoints, the difference was not statistically significant (p>0.05). After 24 hours, TRIAGE+ was significantly harder than TRIAGE− (p=0.002). Changes in the microhardness levels of GCP and RIVA were similar throughout the experiment, and no significant difference in hardness was observed (p>0.05).

Table 2. Microhardness variation of dental filling materials as a function of time after application

<table>
<thead>
<tr>
<th>Time</th>
<th>HV (MPa)</th>
<th>TRIAGE+ (with heat)</th>
<th>GCP</th>
<th>EQUIA+ (with heat)</th>
<th>RIVA</th>
<th>TRIAGE− (without heat)</th>
<th>EQUIA− (without heat)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 hour</td>
<td>650.45 ± 43.30</td>
<td>277.52 ± 31.54</td>
<td>580.60 ± 63.54</td>
<td>247.22 ± 6.70</td>
<td>712.81 ± 19.19</td>
<td>656.09 ± 38.80</td>
<td></td>
</tr>
<tr>
<td>4 hour</td>
<td>623.49 ± 19.18</td>
<td>378.78 ± 27.38</td>
<td>554.62 ± 8.42</td>
<td>355.98 ± 21.57</td>
<td>647.69 ± 51.95</td>
<td>740.39 ± 20.79</td>
<td></td>
</tr>
<tr>
<td>6 hour</td>
<td>613.57 ± 18.40</td>
<td>373.97 ± 9.24</td>
<td>584.85 ± 19.73</td>
<td>318.84 ± 16.55</td>
<td>641.02 ± 38.24</td>
<td>469.05 ± 13.22</td>
<td></td>
</tr>
<tr>
<td>12 hour</td>
<td>577.62 ± 7.65</td>
<td>352.16 ± 8.52</td>
<td>664.91 ± 5.02</td>
<td>296.53 ± 29.60</td>
<td>675.73 ± 8.24</td>
<td>551.82 ± 1.67</td>
<td></td>
</tr>
<tr>
<td>24 hour</td>
<td>782.45 ± 77.98</td>
<td>496.63 ± 13.95</td>
<td>603.54 ± 35.63</td>
<td>499.63 ± 6.68</td>
<td>597.53 ± 15.58</td>
<td>658.80 ± 54.87</td>
<td></td>
</tr>
</tbody>
</table>

TRIAGE+ (with heat) | GCP | EQUIA+ (with heat) | RIVA | TRIAGE− (without heat) | EQUIA− (without heat) |
Discussion

While both Vickers and Knoop microhardness methods are frequently used to determine the hardness of dental filling materials, Poskus et al. (14) found the two methods to be strongly correlated with no obvious advantages of one over the other. Therefore, the current study makes use of Vickers microhardness measurements.

Kuter et al. measured the hardness of various GICs at 24 hours post-application and reported that the application of heat during setting can positively affect their mechanical properties (11). Conversely, Ulrike et al. reported that heating during setting does not have a significant effect on the mechanical properties of GICs. In the current study, the application of heat to Equia™, a high-viscosity GIC, had no significant effect on its hardness after 24 hours (p > 0.05) (12). In contrast, heating during setting did have a significant positive effect on the microhardness of Fuji Triage™ fillings (p < 0.05).

In many studies, 24 hours were allowed for the filling materials to reach their maximum hardness levels. In glass-ionomer filling materials, the hardening mechanism continues for 24 hours after mixing (7, 15). Mobarak et al. examined the hardness of various light-cured, tooth-colored dental materials at 15 minutes, 24 hours, and 7 days after their application (7). Despite a study suggesting that hardness measurements on such materials cannot be trusted so soon after setting (16), Mobarak’s team reported that the notches obtained during hardness measurements at 15 minutes were accurate and easily readable (7). The original intent of the current study was to measure hardness beginning at 15 minutes post-application. However, the chemical setting kinetics of Fuji Triage™ did not allow for such early measurements and resulted in material breakage or detachment from the cavity. Therefore, the earliest reliable measurements of microhardness were obtained at 2 hours post-application.

Sample storage conditions following material application can influence the physical properties of many dental filling materials. In some studies, samples were stored under wet conditions immediately after application (17) while other studies maintained dry conditions during the early stages of material curing (7). Mobarak et al. stored their samples in dry conditions and observed higher hardness values than those obtained in similar studies (7). They suggested that samples stored in wet environments experience hydrolytic plasticization and softening of the material surface. In the present study, we found that storing Fuji Triage™ and Equia™ specimens in distilled water did not allow for hardness measurements due to sample surface dissolution, especially after 2 hours post-application. Therefore, our samples were kept dry prior to hardness measurements.

Very few articles describe changes in the microhardness of glass-ionomer dental restorative materials within the first 24 hours after application. The current study is the first to examine these changes at 2, 4, 6, 12, and 24 hours. We found that the GCC, on which there are very few studies, did not exhibit particularly high mechanical properties in the first 24 hours after application. Changes in the microhardness of GCC were compared with those of Equia™. While both materials are used in similar applications, Equia™ was a significantly harder material after 24 hours (p<0.05).
Microhardness of Newly Developed Glass Carbomer

Conclusions

Within the limitations of this study; Heat treatment of Equia™ during setting did not change the physical strength of the cured material. Heat treatment of Triage™ improved the physical strength of the cured material after 24 hours. Changes in hardness during the first 24 hours post-application were similar between GCP Glass Fill™ and Riva LC™.

It is important to emphasize that the results of the present study are valid for the laboratory conditions used. Laboratory data may provideand insight into clinical performance, however, a direct relationship between laboratory and clinical performance cannot be assumed.

Ethical Approval: Ethics committee approval was received for this study from Şanlıurfa University.

Peer-review: Externally peer-reviewed.


Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study has received no financial support.

Acknowledgments: The authors wish to express sincere appreciation to Dr. Osman ŞAHİN for his kind cooperation and help.

References

1. Faridia MA, Khabeeb A, Haroonc S. Flexural strength of glass carbomer cement and conventional glass ionomer cement stored in different storage media over time. Med Princ Pract 2018;27:372-7. (Crossref)