Flexural Strength and Flexural Fatigue Properties of Resin-Modified Glass Ionomers

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Abstract

- **Objective:** To determine the physical properties of several resin-modified glass ionomers (RMGIs) by means of flexural strength and flexural fatigue testing, and to compare them to conventional glass ionomer cements (GICs) and flowable composite resins.
- **Methods:** RMGI samples were fabricated according to ISO 4049 standard. Rectangular specimens were produced using a polytetrafluoroethylene (PTFE) mold with dimensions of 2 x 2 x 25 mm. Flexural strength and flexural fatigue were measured by means of the 3-point bending tests using an Instron universal testing machine at 0.75mm/min and 0.03Hz for 100 cycles, respectively. Flexural stress, load, and displacement were recorded for all tests. Data were statistically compared (ANOVA, SNK, p < 0.05). Statistical data analysis for flexural fatigue was achieved through the least frequent events method (failures versus non-failures). The following RMGIs, flowable composites, and GICs were tested: 1) Activa Bioactive-Restorative; 2) Activa Bioactive-Base/Liner; 3) Tetric EvoFlow; 4) Beautifil Flow Plus; 5) Geristore; 6) Fuji Filling LC; 7) Fuji Lining LC; 8) Ketac Nano; 9) Fuji Triarc; 10) Ketac Nano; and 11) Vitrebond Plus.
- **Results:** The flexural strength of Activa-enhanced RMGIs was statistically significantly greater than all other RMGIs and GICs (p < 0.001). The flexural fatigue of Activa-enhanced RMGIs and flowable composites was significantly greater than all other materials (p < 0.001). The flexural fatigue of the Activa-enhanced RMGIs was comparable to the two flowable composites tested.
- **Conclusion:** The Activa-enhanced RMGIs demonstrated comparable flexural strength and flexural fatigue to flowable composites. Activa-enhanced RMGIs and flowable composites demonstrated flexural strength and flexural fatigue significantly greater than all other tested materials.

(J Clin Dent 2015;26:23–27)

Introduction

Flexural strength and flexural fatigue behavior are significant physical properties of dental restorative materials. This point is underscored by the fact that the International Standards Organization (ISO) testing protocol for polymer-based dental restorative materials (ISO 4049, Type 1) utilizes flexural strength as a specified test method for acceptance of polymer-based restorative materials claimed by the manufacturer as suitable for restorations involving occlusal surfaces. This research report compares the flexural strength behavior of various resin-modified glass ionomer materials (RMGIs; including recently introduced RMGIs) to flowable composite resins and conventional glass ionomers in order to better assess the relative *in vitro* strength properties of these dental materials.

RMGIs are used as luting agents, liners, and restorative materials. A large body of research has been published reporting their physical properties, fluoride release, biocompatibility, flexural strength, bonding properties, and the clinical success.

The use of fluoride-containing materials in dentistry is a well-established fact. Fluoride release, weight loss, *i.e.*, dimensional change and water sorption, the effects of incorporation of nano-fluorapatite, and the recharge behavior of nano-filled RMGIs have been reported by several investigators. Nevertheless, conventional glass ionomer cements (GICs) generally release more fluoride than RMGIs. While the benefits of fluoride release are considered important, biocompatibility is an equally important prerequisite for any dental restorative or luting material. RMGIs and, for that matter, all resin-based materials cannot be considered to have the same level of biocompatibility as conventional GICs. In another study, cellular biocompatibility after 72 hours of exposure showed that
Materials and Methods

Specimen Preparation

Rectangular specimens were produced using a PTFE mold with dimensions of 2 x 2 x 25 mm according to the ISO 4049 standard. All specimen materials were prepared, mixed, and dispensed according to the manufacturers’ instructions. The material was placed into the mold and covered with a glass cover slip. Specimens were cured utilizing an Elipar S10 curing light (3M ESPE, St. Paul MN, USA). The illumination cycle was set for 20 seconds per cycle with a minimum of three curing cycles on each exposed side of the specimens. The resulting specimen’s extraneous flanges were dressed with SiC paper (1200 grit) at a 45° angle. Specimens were stored at 37°C for 24 to 48 hours before testing. Table I lists the materials tested.

Flexural strength and flexural fatigue testing were carried out with an Instron ElectroPuls E1000 (Instron, Norwood, MA, USA) and a three-point bending test. Flexural strength was tested at 0.75 mm/min until catastrophic failure occurred. Flexural fatigue was tested at 0.03 Hz for 100 cycles. An R-value (min. load/max. load) of 0.1 was used for the fatigue cycles (i.e., minimum load was set to be 10% of maximum load). Flexural fatigue values were determined through the least frequent events method (failures versus non-failures). Using this method, tests were conducted sequentially, with the maximum applied stress in each succeeding test being increased or decreased by a fixed increment of stress, according to whether the previous test resulted in failure or not. The test was stopped, and the fatigue value recorded at a level below which no further failure occurred. Flexural stress, load, and displacement were recorded for all tests. Flexural strength data were statistically compared using an ANOVA with Student-Newman-Keuls and a significance factor of p < 0.05. Statistical data analysis for flexural fatigue was achieved by applying ANOVA and Student-Newman-Keuls statistical methods (significance factor of p < 0.05) to the data derived from the least frequent events method (failures versus non-failures).

Results

As noted in Table II, the flexural strength of the various materials tested demonstrated values ranging from approximately 20 megapascals (MPa) to 115 MPa. Analysis for flexural strength revealed Groups 1–4 were statistically different from and outperformed Groups 5–11 (p < 0.001). Differences occurred between Group 5 and all groups (p < 0.001). Flexural fatigue statistics demonstrated Groups 1–5 were statistically different from Groups 6–11 (p < 0.001).

The conventional RMGI cements failed the flexural fatigue test at 0.3 mm of deflection. Most specimen failures occurred during the start or first cycle of testing. One GC Fuji Lining specimen achieved approximately three cycles of flexural fatigue before failure during a test restart. One Ketac Nano specimen achieved approximately 20 cycles of flexural fatigue testing before failure. Activa Bioactive-Base/Liner enhanced RMGI achieved four flexural fatigue run-outs to 100 cycles at 0.3 mm (R = 10) without specimen failure. Activa Bioactive-Restorative enhanced RMGI achieved seven flexural fatigue run-outs to 100 cycles at 0.3 mm (R = 10). One Activa Bioactive-Restorative specimen broke during the test setup procedure. Statistical analysis (ANOVA and Student-Newman-Keuls) for the fatigue tests demonstrated that Groups 1–5 were statistically different from Groups 6–11 (p < 0.002).

Discussion

The present study compared both the flexural strength and flexural fatigue limit of various flowable resin composites, enhanced RMGIs, conventional RMGIs, and GIC dental restorative materials. The three-point flexural strength test is designed as the primary strength test for resin-containing dental restorative materials under the international testing standard (ISO 4049) and, as such, was an appropriate test method to compare strength properties for the materials included in this study. The lowest flexural strength value was measured for the GIC material, Fuji Triage, while the highest value was demonstrated for the flowable composite resin, Tetrigi EvoFlow. This finding was not unexpected, as prior studies have demonstrated that GICs are brittle materials with low flexural strength, while composite resin materials have previously demonstrated high-
er strength values than either conventional RMGs or GICs. The unexpected finding of this study was the range of flexural strengths demonstrated by the various RMGI materials tested. The mean values for the flexural strength for the two Activa Bioactive RMGI materials were not statistically different (p > 0.05) from either of the composite resin type flowable materials, Tetric EvoFlow and Beautiful Flow Plus. Nevertheless, the mean flexural strength values for this group of four materials, both the two composite resins and two Activa enhanced-RMGI materials, were statistically significantly different from the remaining six conventional RMGI and GIC materials tested. By way of comparison to prior flexural strength values reported in the literature, flexural strength values

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Lot Number &amp; Expiration</th>
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</thead>
<tbody>
<tr>
<td>Activa Bioactive- Restorative (RMGI); Pulpdent Corporation, Watertown, MA USA</td>
<td>Blend of diurethane and other methacrylates with modified polyacrylic acid (44.6%) Amorphous silica (6.7%) Sodium fluoride (0.75%)</td>
<td>120710 Exp. 02/2015 130212 Exp. 02/2015</td>
</tr>
<tr>
<td>Activa Bioactive-Base/Liner (RMGI); Pulpdent Corporation, Watertown, MA USA</td>
<td>Blend of diurethane and other methacrylates with modified polyacrylic acid (53.2%) Amorphous silica (3.0%) Sodium fluoride (0.90%)</td>
<td>121212 Exp. 01/2015 130130 Exp. 01/2015</td>
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<tr>
<td>Tetric EvoFlow (FC); Ivoclar Vivadent AG, Schaan, Lichtenstein</td>
<td>Bis-GMA (10-25%) Urethane dimethacrylate (10-25%) Decamethylene dimethacrylate (2.5-10%)</td>
<td>R53827 Exp. 06/2016</td>
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<tr>
<td>Beautiful Flow Plus (FC); Shofu Inc, Kyoto Japan</td>
<td>Bis-GMA (15-25%) Triethylenglycol dimethacrylate (12-14%) Al₂O₃ (1-2%) DL-Camphorquinone Other ingredients</td>
<td>031317 Exp. 02/2016 121215 Exp. 11/2015</td>
</tr>
<tr>
<td>Geristore (RMG); Den-Mat, Santa Maria CA USA</td>
<td>Resin-based Fluoro Alumina Silica Glass</td>
<td>P281010002 Exp. 04/2014 R9030100010 Exp. 10/2014</td>
</tr>
<tr>
<td>Fuji Filling LC (RMGI); GC America Inc. Alsip, IL USA</td>
<td>Paste A: Alumino-fluoro-silicate glass (amorphous) (75-85%) 2-Hydroxyethyl methacrylate (10-12%) Urethanedimethacrylate (2-5%) Paste B: Distilled water (20-30%) Polyacrylic acid (20-30%) Urethanedimethacrylate (12-15%) Silicon dioxide (fumed/amorphous) (10-15%)</td>
<td>1301151 Exp. 01/2015 1302061 Exp. 02/2015 1304011 Exp. 04/2015</td>
</tr>
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<td>Fuji Lining LC (RMGI); GC America Inc. Alsip, IL USA</td>
<td>Powder: Alumino-fluoro-silicate glass (amorphous) (70-80%) 2-Hydroxyethyl methacrylate (10-15%) Urethane Dimethacrylate (5-10%) Liquid: Polyacryl acid (65-75%) 2-Hydroxyethyl methacrylate (8-10%) Proprietary Ingredient (5-15%)</td>
<td>1207121 Exp. 07/2014 1301091 Exp. 01/2015</td>
</tr>
<tr>
<td>Fuji IX GP Extra (GI); GC America Inc. Alsip, IL USA</td>
<td>Powder: Fluoro Alumino-silicate glass (amorphous) (90-100%) Polyacrylic acid (5-10%) Liquid: Polyacryl acid (65-75%) 2-Hydroxyethyl methacrylate (8-10%) Proprietary Ingredient (5-15%)</td>
<td>1301191 Exp. 01/2015</td>
</tr>
<tr>
<td>Fuji Triage (GI); GC America Inc. Alsip, IL USA</td>
<td>Powder: Alumino-fluoro-silicate glass (amorphous) (90-100%) Liquid: Poly acryl acid (30-40%) Proprietary Ingredient (10-15%)</td>
<td>1207031 Exp. 07/2014 1303121 Exp. 03/2015 1303111 Exp. 03/2015</td>
</tr>
<tr>
<td>Ketac Nano (RMGI); 3M ESPE Dental Products St. Paul, MN USA</td>
<td>Silane treated glass (40-55%) Silane treated zirconia (20-30%) Polyethylene glycol dimethacrylate (PEGDMA) (5-15%) Silane treated silica (5-15%) 2-Hydroxyethyl methacrylate (HEMA) (1-15%) Glass powder (&lt;5%) Bisphenol A diglycidyl ether dimethacrylate (BISGMA) (&lt;5%) Triethylene glycol dimethacrylate (TEGDMA) (&lt;1%)</td>
<td>N428630 Exp. 03/2014 N473044 Exp. 08/2014 N462190 Exp. 05/2014</td>
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<tr>
<td>Vitrebond Plus (RMGI); 3M ESPE Dental Products St. Paul, MN USA</td>
<td>Copolymer of acrylic and itaconic acids (35-45%) Water (30-40%) 2-Hydroxyethyl methacrylate (HEMA) (20-30%)</td>
<td>N438478 Exp. 08/2014 N481263 Exp.01/2015 N435204 Exp. 08/2014 N486813 Exp. 01/2015</td>
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of conventional RMGI materials range from 42 to 66 MPa, 25 to 60 MPa, and 16.9 to 59 MPa in other reported studies.\textsuperscript{21}

The data reported in the present study are in agreement with others\textsuperscript{11,22} which also observed that conventional RMGIs have significantly higher flexural strength than the GICs tested. The minimum requirement of ISO 4049 for occlusal restorations is 80 MPa.\textsuperscript{21}

The mean flexural strengths of flowable composites and Activa Bioactive RMGIs in Groups 1–4 (Table II) were above this minimum requirement of ISO 4049 for occlusal restorations (80 MPa), indicating that these materials are appropriate in some stress-bearing areas such as certain occlusal restorations. The mean flexural strength of Groups 5–11 did not meet the minimum requirement of ISO 4049 for occlusal restorations (80 MPa); therefore, their use in stress-bearing areas should be viewed with caution or even contraindicated.

Based on these findings, judicious use of certain RMGIs for Class I, II, III, and V restorations may be warranted. In particular, Activa Bioactive-Restorative, which the manufacturer reports to contain a proprietary, resilient resin matrix with energy-absorbing elastomeric components (a blend of diurethane and methacylates with modified polyacrylic acid and polybutadiene modified diurethane dimethacrylate), may be more suitable in higher stress (bearing) areas that are contraindicated for GICs.\textsuperscript{24}

With the introduction of RMGIs, many reports have appeared in the literature describing incorporation of micro- and nano-hydroxyapatite,\textsuperscript{26} use of a nano-filled resin-modified glass-ionomer material to reduce surface roughness changes upon mechanical brushing,\textsuperscript{27} spherical silica particles,\textsuperscript{28} and bioglass\textsuperscript{23,24,25} in pursuit of better physical properties and to render these materials bioactive. One could question these efforts as RMGIs already contain reactive, ion-releasing glasses, which in the broadest sense render this class of dental biomaterials “bioactive.” The active release of calcium, phosphate, and fluoride ions from the Activa Bioactive materials and their interaction with dentin and enamel could benefit the longevity of the restoration. Furthermore, the addition of energy-absorbing properties through the use of proprietary resins may present an added benefit by rendering the material more resilient against impact forces, as evidenced by the deflection at break values reported in this study. A well-established benefit of RMGIs is their reduced sensitivity to water loss compared to conventional GICs.\textsuperscript{27} Laboratory findings indicate that at room temperature, the equilibrium water uptake values for Fuji II LC varied from 2.47 to 2.78%. The percentages for the Activa materials, 1.9% for the auto-cure product and 1.7% the light cure material, compare favorably with Fuji II LC.\textsuperscript{28}

### Conclusion

Both Activa Bioactive-enhanced RMGIs (Groups 1–2) demonstrated comparable flexural strength and flexural fatigue to flowable composites (Groups 3–4) and significantly greater flexural strength and flexural fatigue compared to conventional RMGIs and GICs within the present testing limits. The results of this in vitro study suggest that the Activa Bioactive RMGI materials tested demonstrate flexural strength properties in conformity with and in excess of the minimum requirement of ISO 4049 for occlusal restorations (80 MPa). Nevertheless, controlled clinical studies are recommended to confirm the clinical performance of this dental restorative material.

### Acknowledgment

This study was supported in part by Pulpdent Corporation, Watertown, MA. Dr. Cornelis Pameje serves as a consultant to this company. The other co-authors have no financial interest in this company, which manufactures some of the products evaluated in this laboratory study.

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### References

14. Chadwick BL, Evans DJ. Restoration of class II cavities in primary molar teeth with conventional and resin-modified glass ionomer cements: a system-


