THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Comparative evaluation of the physical properties of a reinforced glass ionomer dental restorative material

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ABSTRACT

Statement of problem. While glass ionomer cements have many unique properties and advantages, they still lack favorable mechanical properties. EQUIA Forte Fil is a newly developed glass ionomer cement (GIC) with improved mechanical strength. However, research and data on the physical properties of EQUIA Forte Fil are lacking.

Purpose. The purpose of this in vitro study was to evaluate and compare the compressive, diametral tensile, and flexural strengths of EQUIA Forte Fil with Fuji IX GP and ChemFil Rock, restorative GICs commonly used in dentistry. Moreover, fluoride-releasing properties and surface hardness of the GICs were also assessed.

Material and methods. Ten disk-shaped specimens of each GIC (EQUIA Forte Fil, Fuji IX GP, and ChemFil Rock) were fabricated for mechanical and surface hardness tests by using polydimethylsiloxane (PDMS) molds. The specimens were tested after 24 hours and 7 days of immersion in distilled water at 37 °C. By using a mechanical testing machine, the compressive, diametral tensile, and flexural strengths of each GIC were measured. Fluoride-releasing properties were also evaluated (10 specimens per group). A microhardness tester was used to measure the surface hardness. The mean data were analyzed by using 1- and 2-way ANOVA (α=.05).

Results. EQUIA Forte Fil glass ionomer cements exhibited significantly greater (P<.05) flexural strength and surface hardness than Fuji IX GIC specimens. However, no significant difference (P>.05) was observed between the compressive and diametral tensile strength of EQUIA Forte Fil and Fuji IX GIC specimens. ChemFil Rock exhibited higher flexural strength than EQUIA Forte Fil (P>.05) but significantly lower compressive strength and microhardness (P<.05). Tested GICs matured after 1 week of immersion in distilled water, demonstrating a significant improvement in their mechanical properties. All the examined glass ionomers exhibited comparable initial fluoride-releasing properties, whereas EQUIA Forte Fil exhibited significantly higher (P<.05) amounts of fluoride release from the bulk of the material after 4 weeks.

Conclusions. EQUIA Forte Fil is a promising restorative material with superior flexural strength and surface hardness compared with its predecessor, Fuji IX GP, or other commercially available glass ionomers. (J Prosthet Dent 2019;122:154-9)

In 1969, Wilson and Kent invented glass ionomer cements (GICs) by combining fluoroaluminosilicate glass particles and homopolymers or copolymers of polyacrylic acid.[1,2] GICs have unique biophysical properties such as fluoride ion release and recharging abilities, inhibiting bacterial acid metabolism and preventing enamel decalcification.[3-6] Other beneficial properties of GICs include a thermal expansion coefficient similar to that of dentin, biocompatibility, and adhesion to tooth structure without any pretreatment.[7,8] Because of these unique properties, GICs have a wide range of application in dentistry; for example, they are the materials of choice for atraumatic restorative treatment (ART).[9] They are also routinely used in pediatric dentistry.[6-9]

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Clinical Implications

Glass ionomer dental cements are well-studied dental materials with unique and advantageous properties. However, the poor mechanical properties of this type of dental material are still a shortcoming. EQUIA Forte Fil glass ionomer dental cement is an advantageous dental restorative material with excellent mechanical properties and surface hardness, which might expand its clinical application.

However, GICs have unfavorable characteristics that limit their clinical application. GICs exhibit modest mechanical properties, including brittleness, poor fracture toughness, and low flexural strength.[9-11] Additionally, in the early stages of setting, the material is sensitive to moisture and desiccation.[11-13] Therefore, additives have been used to enhance the mechanical properties of GIC and to expand its application.[3-5] For example, amalgam alloy powder has been added to its structure.[14] Alternatively, amalgam powders fused and sintered to basic glass particles (cermet cements) have been introduced as a reinforced dental restorative material.[15] Although multiple studies found improvements in GIC strength by using these approaches, other drawbacks including cement discoloration and toxicity of the incorporated material were found.[3-15]

To address these shortcomings, attempts have been made to improve the mechanical properties of GICs by other means. The compositions of GICs have the potential for biomaterial improvements and modifications to increase their mechanical strength and expand their application in restorative dentistry.[12] For instance, Moshaverinia et al[13-21] reported on additives that improved the mechanical strength and handling properties of GICs. These additives included methacryloyl (a proline derivative), N-vinylcaprolactam, N-vinylpyrrolidone modified acrylic acid copolymer, nanohydroxyapatite, and fluorapatite. Lucas et al[22] reported that when hydroxyapatite was added to a GIC, the material demonstrated a crystalline structure similar to that of human dental structures, enhancing the mechanical properties of the cement. Although these studies reported improvements in selective physical properties, the mechanical strength of GICs remains inferior to other restorative materials such as composite resin.

In attempts to enhance the mechanical properties of glass ionomer, glass ionomer particles have been reacted with a polyacid to form a set glass ionomer matrix structure. Then, the resulting set cement was added to the resin matrix. Studies have shown that this kind of modified glass ionomer, Giomer, showed improved mechanical properties and favorable fluoride-releasing and recharging characteristics.[23] Recently, a new type of glass ionomer, EQUIA Forte Fil (GC Corp), has been introduced. The mechanism of reinforcement of this GIC is based on the presence of evenly dispersed ultrafine and highly reactive glass particles and optimization (increase) of the molecular weight of the polyacrylic acid, leading to formation of a new class of restorative GIC with excellent mechanical properties.[24] The manufacturer claims that this new GIC material can be used in load-bearing class II restorations in addition to class I and V restorations. It has been reported that this new GIC exhibits increased flexural strength and resistance to wear and acid erosion.[24] However, the authors are unaware of articles reporting on the mechanical strength of this new restorative material in comparison with a well-established glass ionomer dental cement (Fuji IX GP; GC America). Therefore, the purpose of this in vitro study was to evaluate and compare the mechanical properties (compressive, diametral tensile, and flexural strengths), fluoride-releasing properties, and surface hardness of EQUIA Forte Fil with those of well-characterized and commonly used GICs: Fuji IX GP (GC America) and ChemFil Rock (Dentsply Sirona). The null hypothesis was that no difference would be found in the mean of physio-mechanical properties among EQUIA Forte Fil, ChemFil Rock, and Fuji IX GP glass ionomer cements.

MATERIAL AND METHODS

Glass ionomer cements (GICs) used were commercial-grade Fuji IX GP, ChemFil Rock, and Equia Forte Fil. Cylindrical and rectangular polydimethylsiloxane (PDMS) molds were cast from templates. Two cylindrical molds were fabricated, the first with dimensions of 4 mm in diameter (d) × 6 mm in height (h) and the second 6 mm d × 3 mm h. A single rectangular mold was fabricated with dimensions of 25 mm in length (l) × 2 mm h × 2 mm in width (w). GC Fuji IX GP, ChemFil Rock, and EQUIA Forte Fil capsules were used. GIC specimens were activated and mixed for 10 seconds in a GC capsule mixer Cm-II (GC Corp) and dispensed into the PDMS molds by using a GIC Applicator (ProMedica). Specimens were then fabricated and polymerized at room temperature per the manufacturers' instructions. Then, test molds were filled with GIC materials, flattened, and gently pressed by using a smooth plastic disk to shape the unpolymerized cement paste. After 30 minutes, the specimens were removed from the molds, and excess material was removed by hand. Subsequently, the fabricated specimens were conditioned in 20 mL of distilled water at 37 °C for 24 hours or 7 days. Ten specimens were processed per strength test (compressive, diametral, and flexural strengths) for each time point. The specimens for Vickers hardness (n=10 per group) were processed at 7 days only.

The compressive strength (CS), diametral tensile strength (DTS), and flexural strength (FS) tests were performed on a mechanical testing machine (Model 5564; Instron Corp) with a crosshead speed of 0.5 mm min^{-1}. Before testing, the mechanical testing machine was calibrated.

The CS was calculated from the data collected in Newtons (N) by using a modified version of the following equation:

$$CS=4p/\pi d^2,$$

where P is the load (N) at the fracture point and d is the diameter (mm) of the cylindrical specimen. This equation converts the load (N) to MPa.

The DTS specimens were tested by using a 1-kN load cell where data collected (N) were converted to MPa by using the following equation:

$$DTS=2P/\pi dh,$$

where P is the load (N) applied to the specimen, and d and h are the diameter and height of the specimen, respectively.

A 3-point bend test was used to study the flexural strength of each specimen. FS specimens were placed with their edges equidistant from the midline of the mechanical testing machine. The data from specimens subjected to FS were collected in terms of load (N) and converted into MPa. The MPa conversion was calculated by using the following equation:

$$FS=PI/2wh^2,$$

where P is the load, l is the distance between the supporting rollers, w is specimen width, and b is specimen height. In FS experiments, l was equal to 20 mm.

The Vickers hardness test (HV) was conducted on a microhardness tester (model 1600-4963; Buehler) based on previously reported protocols.[16] Before testing, the specimens were conditioned for 24 hours at 37 °C. Briefly, a 2.9-N force was applied to the specimens by using a diamond indenter for 15 seconds. The mechanical testing machine was calibrated before taking measurements. All results were generated and reported in HV units by using the microhardness tester. The Vickers hardness number (VHN) (kgf/mm^2) for each tested specimen was measured according to the following equation:

$$VHN=1.8544\times L/d^2,$$

where L=applied load (kgf) and d is mean diagonal length (mm).

The fluoride-releasing properties (such as the mean cumulative fluoride release, which is micrograms of fluoride ions released per unit surface area of each specimen) of each glass ionomer were analyzed based on the previously reported methods[16] by using a fluoride ion electrode (Ion Fluoride-Selective Electrode [ISE]; Thermo Scientific Corp). The electrodes were rinsed with distilled water before starting the analysis. Subsequently, the electrodes were calibrated. The meter and electrode combination was also calibrated according to published methods.[16] After continuous stirring of each specimen solution, a reading was recorded. Then, for each specimen, the cumulative fluoride release was calculated, and a cumulative release curve/time ($\mu g/mg\ cm^{-2}F^{-}$) was plotted for up to 4 weeks of analysis.

A scanning electron microscope (SEM, SUPRA 40/40 VP; Carl Zeiss SMT) was used to analyze the glass ionomer particles and compare the surface morphology of the specimens 1 week after setting (immersion in distilled water at 37 °C). For SEM analysis, 3 specimens per group were evaluated. In each specimen, 3 different locations were analyzed, and the number and size of cracks were measured and compared.

One- and 2-way ANOVA (factors: material×time) were used to determine whether there were significant differences among the values of the CS, DTS, FS, fluoride-releasing properties, and VHN of the tested glass ionomer specimens. One-way ANOVA was used for VHN, and 2-way analysis was used for the rest of mechanical evaluations where time×material were the tested factors (α=.05).

RESULTS

Figure 1 represents the compressive strength (CS) and diametral tensile strength (DTS) of the evaluated cements. Additionally, Fuji IX showed higher CS and DTS values in comparison with EQUIA Forte Fil after 1 and 7 days of storage in 37 °C distilled water; however, these values were not significantly higher. ChemFil Rock, on the contrary, showed lower CS and DTS values than EQUIA Forte Fil after 1 day and 1 week. Upon comparing the mechanical values of the tested specimens after 1 and 7 days, all the cements exhibited a significant increase (P<.05) in their CS and DTS after a week of immersion in distilled water at 37 °C.

Figure 2A demonstrates the results of the flexural strength (FS) measurements of the examined cements after 1 and 7 days of immersion in distilled water at 37 °C. The results showed a significant increase (P<.05) in the FS of EQUIA Forte Fil glass ionomer cements after 24 hours and 1 week in comparison with the Fuji IX glass ionomer group at the same storage times. Additionally, ChemFil Rock specimens showed higher flexural strength in comparison with EQUIA Forte Fil specimens. Moreover, all the tested glass ionomer cement specimens matured while immersed in 37 °C distilled water, displaying a significant increase in FS after 1 week compared with 24 hours (P<.05).

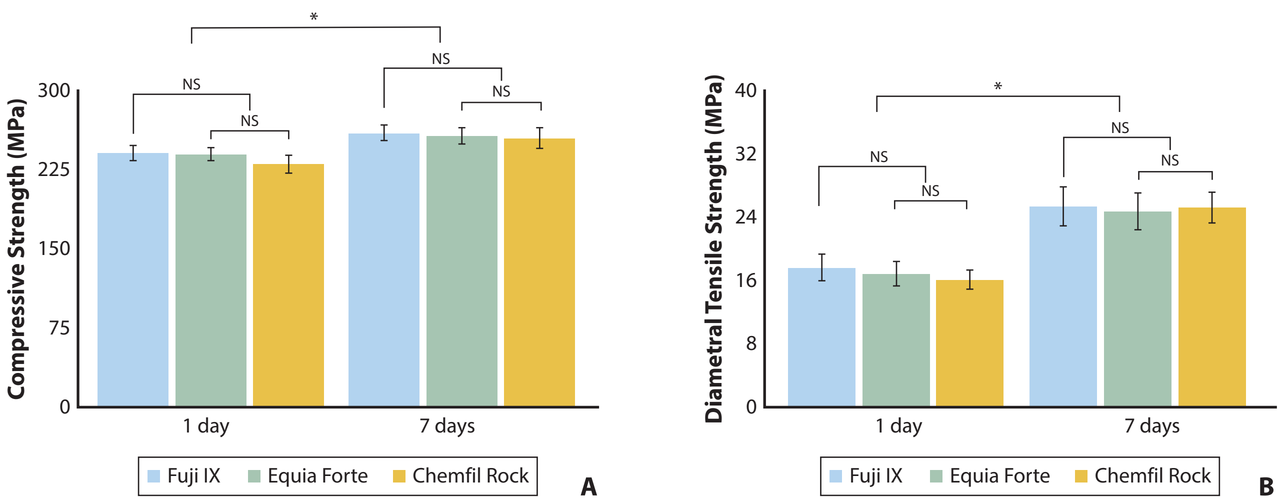

Figure 1. Results of GIC specimens after 1 and 7 days of storage in distilled water at 37 °C. A, Compressive strength. B, Diametral tensile strength. *P<.05. GIC, glass ionomer cement; NS, not significant.

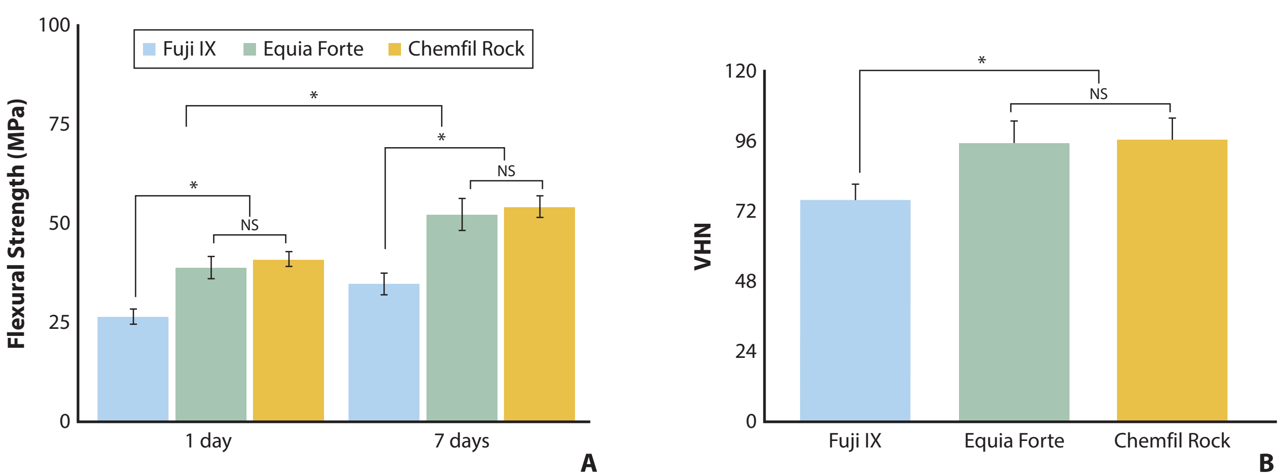

Figure 2. A, Flexural strength of GIC specimens after 1 and 7 days of storage in distilled water at 37 °C. B, Vickers hardness numbers (VHNs) of GIC specimens after 1 week of storage in distilled water at 37 °C. *P<.05. GIC, glass ionomer cement; NS, not significant.

The values of Vickers hardness are shown in Figure 2B. After 24 hours of storage in distilled water at 37 °C, EQUIA Forte Fil showed significantly greater surface hardness (P<.05) than Fuji IX. In addition, no significant difference was observed between the VHN values of EQUIA Forte Fil and ChemFil Rock.

Fluoride-releasing analysis results are shown in Figure 3 and demonstrate that EQUIA Forte Fil exhibited significantly increased (P<.05) fluoride release up to 8 weeks in comparison with ChemFil Rock and Fuji IX GIC.

The SEM micrographs of the surface of EQUIA Forte Fil, ChemFil Rock, and Fuji IX GIC (Fig. 4) show that the surface of EQUIA Forte Fil glass ionomer had comparable surface cracks to ChemFil Rock and fewer voids and cracks than Fuji IX.

DISCUSSION

The mechanical properties of glass ionomer cements (GICs) continue to improve through material modification and composition changes. In this study, the mechanical properties of EQUIA Forte Fil, ChemFil Rock, and Fuji IX GP were evaluated and compared. EQUIA Forte Fil is a newly formulated, improved GIC, whereas ChemFil Rock and Fuji IX are traditional GICs that are commonly used in dental practice. The null hypothesis that no statistically significant differences would be found in the mechanical properties of EQUIA Forte Fil, ChemFil rock, and Fuji IX GP was partially rejected, as EQUIA Forte Fil exhibited significantly higher flexural strength (FS) and Vickers hardness (HV) than Fuji IX. No significant difference was observed in the values of compressive strength (CS) and diametral tensile strength (DTS) among the tested GICs. Additionally, no difference was

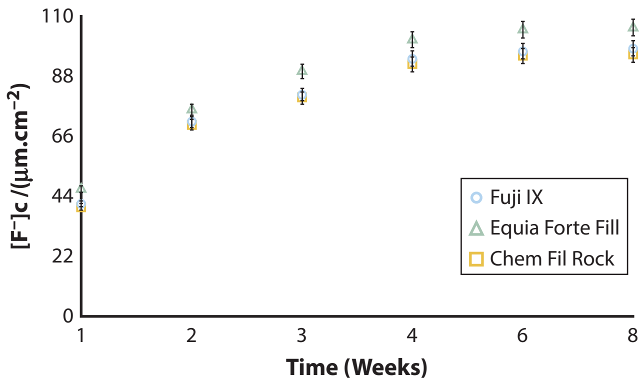

Figure 3. Cumulative analysis of fluoride-releasing properties of 3 tested glass ionomer cements.

observed in the fluoride-releasing properties of the 3 tested glass ionomer cements.

Glass ionomer dental cements are composed of an ion leaching fluoroaluminosilicate glass and homopolymers or copolymers of polyacrylic acid. To improve their mechanical properties, modifications have been made to their structure and composition.[3] For instance, metals and nonreactive fillers have been added to the structure of the glass particles to enhance the strength of GICs without compromising their handling or biological properties. Furthermore, modifications to the chemistry, concentration, and molecular weight of the polyacid have been attempted to strengthen the glass ionomer.[3,12]

In the newly available EQUIA Forte Fil glass ionomer, ultrafine and highly reactive glass particles have been dispersed evenly in the structure of the glass powder. In addition, the molecular weight of the polyacrylic acid has been optimized. Based on these modifications, this new type of GIC exhibits enhanced mechanical properties and improved wear and acid erosion resistance in comparison with the traditional GICs, Fuji IX GP, and ChemFil Rock.[24] Studies on the mechanical properties of EQUIA Forte Fil remain limited; the results obtained here for Fuji IX GP and ChemFil Rock correlated well with previously reported results on the CS, DTS, FS, and HV of this material.[25-29]

In the present study, a significant increase was observed in the flexural strength (FS) of EQUIA Forte Fil in comparison with Fuji IX GP, while the amount of increase in FS values was incremental in comparison with ChemFil Rock. This outcome might be attributed to the optimized molecular weight (Mw) of the polyacrylic acid, which increases the polysalt bridge formation and crosslinking in the structure of the set cement. An optimized Mw will lead to more availability of carboxylic acid groups for enhanced acid-base reaction. Moreover, the results showed a more pronounced maturation after 7 days of immersion in distilled water for EQUIA Forte Fil, which can be attributed to the availability of more carboxylic acid groups in the backbone of the polyacrylic acid, allowing reactions with Al^{3+} and Ca^{2+} ions to form the glass ionomer

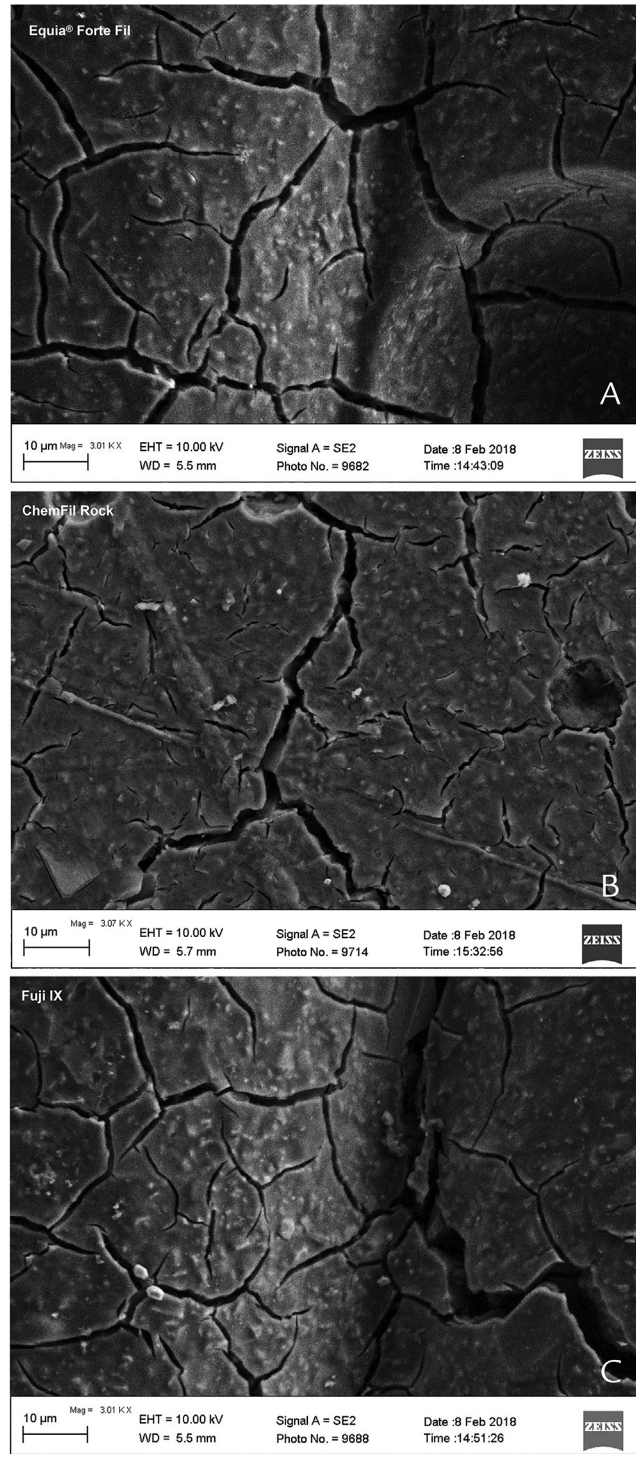

Figure 4. Scanning electron microscope images of surface of set glass ionomer cements after 24 hours. A, EQUIA Forte Fil. B, ChemFil Rock. C, Fuji IX. Original magnification ×3000.

particles. However, no significant difference was observed between the CS and DTS of EQUIA Forte Fil, ChemFil Rock, and Fuji IX GP. All the tested cements exhibited improved CS and DTS after 1 week of storage in distilled water relative to their performance after 1 day of storage.

Hardness represents the resistance of the surface of a material to indentation and deformation. The obtained results from the present study showed a significant increase in the hardness of EQUIA Forte Fil in comparison with Fuji IX GP. No difference was found in the VH values of EQUIA Forte Fil and ChemFil Rock. This finding confirms a more complete acid-base reaction in the bulk and on the surface of VH EQUIA Forte Fil material upon setting. This phenomenon is attributed to the presence of ultrafine and highly reactive glass particles evenly distributed throughout the structure. In addition to the optimized Mw, the presence of highly reactive glass will reinforce the surface properties of the set cement.

Altogether, the results of the present study suggest that EQUIA Forte Fil represents a significant improvement to a well-characterized GIC material (Fuji IX GP) and comparable properties to those of ChemFil Rock. In addition to maintaining CS and DTS values similar to those of Fuji IX GP, improvements to the EQUIA Forte Fil's flexural strength and Vickers hardness are promising for future applications of GICs in restorative dentistry. In the present study, only two other glass ionomers were evaluated for comparison. The other limitation of the present study is that only limited physical properties were analyzed. In future studies, more clinically relevant physical properties (such as wear resistance, fluoride release, and fracture toughness) should be studied. Comparative studies with other types of glass ionomer dental cements are also needed.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. EQUIA Forte Fil is a promising restorative material with superior flexural strength and surface hardness compared with its predecessor, Fuji IX GP or other commercially available glass ionomers.
2. This material might have a wide range of clinical applications in dental practice as a restorative dental material.

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https://doi.org/10.1016/j.prosdent.2019.03.012