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A Measure of Mechanical Properties, Microhardness, and Fluoride Releasing Properties of a New Glass Hybrid Restorative Dental Material

A thesis submitted in partial satisfaction of the requirements for the degree Master of Science in Oral Biology

by

Rana Mersal N. Alshammary

2019

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2019

ABSTRACT OF THE THESIS

A Measure of Mechanical Properties, Microhardness, and Fluoride Releasing Properties of a New Glass Hybrid Restorative Dental Material

by

Rana Mersal N. Alshammary

Master of Science in Oral Biology

University of California, Los Angles, 2019

Professor Alireza Moshaverinia, Chair

Equia® Forte Fil (EFF) is a new glass ionomer cement (GIC) with limited published data on its physical properties. This study aimed to evaluate and compare the mechanical Properties, microhardness, and fluoride releasing properties of EFF to two commonly used restorative GICs. Ten specimens of each GIC were tested. EFF exhibited significantly greater ($p < 0.05$) flexural strength and surface hardness than Fuji IX. However, no significant difference ($p > 0.05$) was observed between the compressive and diametral tensile strength of EFF and Fuji IX. ChemFil Rock revealed higher flexural strength than EFF ($P > 0.05$) but significantly lower compressive strength and microhardness ($P < 0.05$). All GICs exhibited almost the same fluoride burst and continued fluoride release from the bulk of the material. EFF has superior mechanical properties and surface hardness, which expands its clinical application to Class I and II restorations in stress bearing areas.

The thesis of Rana Mersal N. Alshammary is approved.

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Bo Yu

Alireza Moshaverinia, Committee Chair

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2019

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INTRODUCTION

The invention of glass ionomer cements (GICs), combining fluoroaluminosilicate glass particles and homo- or co-polymers of polyacrylic acid, by Wilson and Kent in 1969, ushered in a new era in dentistry that enabled advanced restorative care for adult and pediatric patients, alike. [1,2] GICs are formulated in powder and liquid, setting through an acid-base reaction between glass and acid.[34] Setting reaction of the glass polyalkenoate cement has been studied and is characterized by the two distinct phases: (1) Dissolution and (2) Gelation.

Dissolution Phase:

The surface layer of the glass reacts by the polyacid, causing some degradation of the glass with the release of calcium, aluminum and fluoride ions. The pH of the freshly mixed cement is approximately 2.6, an order of magnitude greater than that of phosphate bonded cement, but rises to 5.3 at 24 hrs.[35]

Gelation phase:

Once some calcium and aluminum ions are dissolved in the cement sol, the setting reaction begins with a formation of a salt hydrogel serving the binding matrix surrounded by water molecules which hydrate the formed metal polyalkenoate. Soluble metal ions in the forming matrix are attacked by aqueous fluids, resulting in a complex composite of glass particles enveloped by a siliceous hydrogel, bonded by a matrix of hydrated fluoridated calcium and aluminum polyacrylates.[36] The rate of the reaction is controlled by four interacting variables, namely:

1. Temperature[37]
2. Physical presentation of the powder, with finely ground glass particles being preferable[38]
3. Availability of the free fluoride ions [39]

4. Presence of tartaric acid.[40]

Light cured GICs also been employed to improve the setting reaction, with two commercially available products, the *Vitrebond** and *XR- ionomer*.[41, 42]

Properties:

Physical Properties: Four factors impact the physical properties of this material.[43-45] These factors are as follows:

1. Variation in the glass powder
2. Variation in the powder:liquid ratio
3. Hydration of the cement mass
4. Porosity of the matrix

The current ISO standard for GICs mandates a minimum compressive strength of 70 Mpa for application as liners or bases and 150 Mpa for use as direct restorative material.[46] Modern brands can meet and surpass these values, with strengths in the range 220-300 Mpa now available in many marketed cements.[47]

Adhesion:

GICs can bond naturally to the tooth surface, including both enamel and dentin, without any additional binding compounds required. Although initially, bond strengths are low compared with the bonded composite resins, typically 1.5-5.0 Mpa, durability is greater than them.[48] An ionic exchange between cement and tooth structure forms an ion-enriched layer in the cement where it interfaces with the structure of the tooth, and is likely responsible for the long-term durability of the cement's long-term adhesive bond.[49] The tooth surface is prepared clinically for bonding by conditioning, wherein the freshly cut tooth surface is treated with 37% aqueous

polyacrylic acid solution for up to 20 seconds, followed by rinsing, removing the smear layer, opening the dentinal tubules, and also somewhat demineralizing the surface of the tooth, increasing the surface area and facilitating micromechanical attachment.[50]

Fluoride release:

The glass powder's high fluoride content leads to high fluoride levels within the cement matrix. Unbound fluoride is available for elution, released into the mouth, and can locally inhibit caries akin to silicate cement, protecting the entire tooth.[51]

Biocompatibility:

GICs have substantial biocompatibility with the dental pulp under most clinical uses. In vivo and in-vitro have shown that glass ionomer cement can mildly irritate the dental pulp, with the inflammation proportional to the thickness of the residual dentin. [52] Glass polyalkenoate cement has a thermal diffusivity similar to that of dentine, which increases in proportion to the powder: liquid ratio.[53]

Abrasion resistance:

This group of materials is considerably less abrasion resistance than the composite resins and exhibits similar loss of material to silicate cement during testing in vitro.[54]

As derivatives of dental silicate and zinc polycarboxylate cement, GICs were first adhesive restorative material with a carcinostatic property. They were developed with the intent to blend the unique benefits of the two parent materials viz. ion-leachable glass powder and a poly (alkenoic acid) to produce an aesthetically suitable adhesive restorative material, and were primarily used to restore tooth erosion, root caries, and abrasion problems.

The original GIC was comprised of an aqueous solution of poly (acrylic acid) 45% which

reacted with calcium fluoroaluminosilicate glass powder resulting from the fusion of quartz, alumina, cryolite, fluorite, aluminum trifluoride and aluminum phosphate.[32] These initial materials were plagued by their sluggish setting, moisture sensitivity, and their opacity after setting. The incorporation inclusions that did not form a matrix into the glass was able to address many of the initial mechanical and aesthetic deficiencies of the set cement.[33] These inclusions are composed of both Metallic Inclusions (known as cermet cement) and Crystalline Inclusions (including corundum, rutile, aluminum titanate dispersed phase crystals). Since becoming available clinically, GICs have been under continual improvement. [30-31]

The unique and clinically useful biophysical properties of GIC cements, including fluoride ion release and recharging abilities, can inhibit bacterial acid metabolism and prevent further enamel decalcification. [4, 5, 6] Additionally, the thermal expansion coefficient of GICs is alike that of dentin, which enables adhesion to tooth structure and biocompatibility without the need for any pre-treatment.[7, 8] As a consequence of these unique and valuable properties, GICs are widely applicable in clinical dentistry, commonly utilized first line for atraumatic restorative treatment (ART), as well as in pediatric dentistry. [6-8]

Despite their initial appearance as a perfect material for clinical dentistry, GICs also suffer unfavorable properties that limit their routine clinical in dentistry. For example, GICs exhibit significant mechanical properties including brittleness, poor fracture toughness, and low flexural strength, which are undesirable under many clinical situations.[9, 10, 11] Moreover, in the initial stages of setting, GICs can become sensitive to desiccation and moisture.[11-13] This has necessitated the addition of reinforcing additives to GICs to enhance their mechanical properties and to broaden their clinical utility.[3-5] For instance, in order to improve their mechanical properties, amalgam alloy powder has been incorporated into them.[14] Additionally, others have

fused and sintered amalgam powders to basic glass particles to create cermet cements, which are now used as reinforced dental restorative materials.[15] Despite the many published studies that have found that these approaches and improve GIC strength, other shortcomings were discovered, including toxicity of the incorporated material and discoloration of the cement over time, which can be aesthetically undesirable. [3-15]

In response to these deficiencies, other means have been employed to enhance the mechanical properties of GICs by altering their composition further to address their physical and mechanical shortcomings, thereby improving their clinical utility in restorative dentistry. [12] For instance, Moshaverinia and colleagues demonstrated that a handful of specific chemical additives, including methacryloyl (a proline derivative), N-vinylcaprolactam, N-vinylpyrrolidone modified acrylic acid copolymer, nano-hydroxy- and fluroro-apatite, could substantially enhance GICs' handling properties and mechanical strength. [13-21] Similarly, Lucas and colleagues added hydroxyapatite was to the composition of a GIC, causing the material to adopt a crystalline structure mimicking human dental tissues, with substantially improved mechanical properties of the cement. [22] Despite these and other advances in improving selective physical properties of GIC cements, GICs remain inferior to other restorative materials such as resin composites with respect to their mechanical strength, again limiting their broader clinical utility.

Seeking to further improve the mechanical properties of glass ionomers, others have reacted glass ionomer particles with a polyacid to form a set glass–ionomer matrix structure, which was subsequently added to the resin matrix. It has been shown that these modified glass ionomers, known as Giomer, exhibit significantly more favorable mechanical properties and fluoride releasing/recharging features. [23] Lately, an innovative hybrid glass ionomer, known commercially as Equia® Forte Fil (EFF; GC Corp.), has been marketed to practicing dentists.

EFF's properties as a reinforced GIC are attributable to both the existence of evenly dispersed highly reactive and ultrafine glass particles, and improvement of the molecular weight of the polyacrylic acid, enabling the creation of an altogether new class of restorative GIC with superior mechanical properties and broader clinical utility.[24] EFF's manufacturer asserts that this novel GIC material is both safe and effective for use in load-bearing Class II restorations, as well as Class I and V restorations, because of its enhanced flexural strength, as well as its resistance to wear and acid erosion. [24] Despite the manufacturer's claims, a review of the literature has not found any articles demonstrating that the mechanical strength of EFF is either superior or non-inferior to a long-proven glass ionomer dental cement (Fuji IX GP). Therefore, this study aimed to evaluate and compare the surface hardness, mechanical properties (e.g., compressive, diametral tensile, and flexural strength), and fluoride releasing properties of EFF to clinically proven and regularly used GIC: Fuji IX GP (GC America) and ChemFil Rock (Dentsply). The null hypothesis was that there would be no statistical significance in the mechanical properties between EFF, Fuji IX GP, and ChemFil Rock glass ionomer cements.

MATERIALS AND METHODS

Specimen Preparation

GICs were commercial-grade Fuji IX GP (GC International, Tokyo, Japan), ChemFil Rock (Dentsply), and Equia® Forte Fil (GC America). Cylindrical and rectangular PDMS (polydimethylsiloxane) molds were cast from templates. Two cylindrical molds were manufactured, the first with dimensions of 4 mm diameter (d) x 6 mm height (h) and the second 6 mm d x 3 mm h. A single rectangular mold was manufactured with dimensions of 25 mm length (l) x 2 mm h x 2 mm width (w). GC Fuji IX GP, ChemFil Rock (Dentsply), and EQUIA®

Forte Fil capsules were used. GIC specimens were activated and mixed for 10 seconds in a GC capsule mixer Cm-II™ (GC Corporation) and dispensed into the PDMS molds using a GIC-applicator (Promedica, Germany). Specimens were then fabricated and cured at room temperature per the manufacturers' instructions. Next, test molds were filled with GIC materials, flattened, and gently pressed with a smooth plastic disc to shape the uncured cement paste. After 30 minutes, 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 strength) for each time point. HV specimens (n=10 per group) were processed at 7 days only.

Mechanical properties measurements

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

The CS was calculated from the data collected in units of Newtons (N) using a modified version of the 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 samples were tested using a 1kN lead cell where data collected (N) was converted to MPa by the equation

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

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

FS specimens were placed with their edges equidistant from the midline of the Instron. The data from specimens subjected to FS was collected in terms of load (N) and converted into MPa. The MPa conversion was calculated using the equation

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

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 20mm.

Vickers Hardness

The Vickers hardness tests (HV) were conducted on a microhardness tester (Model 1600-4963, Buehler, Lake Bluff, Illinois, USA) based on previously reported protocols [16, 29] Prior to testing, the specimens were conditioned for 24 hours at 37°C. Briefly, a 0.3 kilogram force (kgf) was applied to the specimens with a diamond indenter for 15 seconds. The mechanical testing machine was calibrated prior to taking measurements. All results were generated and reported in HV units by the microhardness tester.

Characterization of the Fluoride Releasing Properties

Fluoride releasing properties of each glass-ionomer were analyzed based on the previously reported methods [29] using an Fluoride ion electrode (Ion Fluoride-Selective Electrode (ISE),Thermo-Orion Ionplus, Thermo Scientific Corporation). The electrodes were rinsed with distilled water prior to start the analysis. Subsequently, the electrodes were calibrated. The meter/electrode combination was also calibrated according to the methods already in the literature.[29] After continuous stirring of each specimen solution, a reading was recorded. Next, for each specimen the cumulative fluoride release was calculated and a cumulative release curve/time (ug/ mg.cm^{-2} F^{-}) was plotted for up to four weeks of analysis.

SEM Analysis

Scanning Electron Microscopy (SEM, SUPRA 40/40 VP, Carl Zeiss SMT) was utilized to analyze the glass ionomer particles and compare the surface morphology of the specimens after setting.

Statistical Analysis

One-way and two-way ANOVA were used to determine if there were significant differences between the values of CS, DTS, FS, Fluoride releasing properties, and VHN of the tested glass ionomer specimens. A level of $\alpha < 0.05$ was used for statistical significance.

RESULTS

Figures 1A and 1B represent the compressive strength (CS) and diametral tensile strength (DTS) values of the evaluated cements, respectively. Interestingly, Fuji IX showed slightly higher ($p > 0.05$) CS and DTS values in comparison to Equia® Forte Fil after 1 and 7 days of storage in 37 °C distilled water. Chemfil Rock on the other hand, showed slightly lower ($p > 0.05$) CS and DTS values in comparison to Equia® Forte Fil after 1 day and 1 week. Upon comparing the mechanical values of the tested specimens after 1 and 7 days, it was found that all the cements exhibited a significant increase ($p < 0.05$) 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 both of the examined cements after 1 and 7 days of immersion in distilled water at 37°C. Our results showed a significant increase ($p < 0.05$) in FS of Equia® Forte Fil glass ionomer cements after the 24 h and 1 week in comparison to the Fuji IX glass ionomer group at the same storage times. Additionally, Chemfil Rock specimens showed higher flexural strength in comparison to Equia® Forte Fil specimens (P>.05). Moreover, all of the tested glass ionomer cement specimens matured while immersed in 37°C distilled water, displaying a significant increase in FS after 1 week compared to 24 h ($p < 0.05$).

In addition, the values of Vickers hardness are shown in Figure 2B. After 24 hours of storage in distilled water at 37ºC, Equia® Forte Fil showed significantly greater surface hardness ($p < 0.05$) than Fuji IX specimens and slightly higher VHN values (p>0.05) than Chemfil Rock specimens.

Our Fluoride releasing analysis results are shown in Figure 3 demonstrating that all the tested specimens released a similar ($p > 0.05$) amount of fluoride up to four weeks of analysis.

Our SEM analysis (Fig. 4 A and B) showed that Equia® Forte Fil and exhibited a smaller average particle size with a narrower particle size distribution (even dispersion of ultrafine particles) than other evaluated specimens. SEM micrographs of the surface of Equia® Forte Fil, Chemfil Rock, and Fuji IX GIC are shown in Figure 5, demonstrating that the surface of Equia® Forte Fil glass ionomer had comparable surface cracks as Chemfil Rock and fewer voids and cracks than that of Fuji IX.

DISCUSSION

Glass ionomer cements (GIC) continue to improve in mechanical properties through material modification and composition changes. In this study, we evaluated and compared the mechanical properties of two GICs, namely Equia® Forte Fil and Fuji IX GP; Equia® Forte Fil is a newly formulated, improved GIC, whereas Chemfil Rock and Fuji IX is already commonly used in dentistry. Our null hypothesis was no statistically significant differences would be found in the mechanical properties of Equia® Forte Fil, Chemfil rock, and Fuji IX GP. The null hypothesis was partially rejected, as flexural strength (FS) and Vickers hardness (HV) were significantly higher in Equia® Forte Fil than Fuji IX. Compressive strength (CS) and diametral tensile strength (DTS) showed no significant difference between the tested GICs. Moreover, no difference was observed in the fluoride releasing properties of the three different tested glass-ionomer cements.

Glass ionomer dental cements are composed of an ion leaching fluoroaluminosilicate glass and homo- or co-polymers of polyacrylic acid. In order to improve their mechanical properties, many modifications have been made to their structure and composition. For instance,

metals and non-reactive 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.

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 to the more established GIC, Fuji IX GP. [24] Studies on the mechanical properties of Equia® Forte Fil remain limited; the results obtained here for Fuji IX GP correlated well with previously reported results on CS, DTS, FS, and HV of this material. [25-29]

In the current study, a significant increase was observed in the flexural strength of Equia® Forte Fil in comparison to Fuji IX GP. This outcome might be attributed to the optimized molecular weight (Mw) of the polyacrylic acid, which leads to increase in 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, our 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 the carboxylic acid groups in the backbone of the polyacrylic acid, allowing reactions with Al^{+3} and Ca^{+2} ions to form the glass ionomer particles. However, no significant difference ($\alpha > 0.05$) was observed between the CS and DTS of Equia® Forte Fil and Fuji IX GP. Both cements exhibited improved CS and DTS after one week of storage in distilled water relative to their performance after one day of storage.

Hardness represents the resistance of the surface of a material to indentation and

deformation. The obtained results from the current study showed a significant increase ($\alpha < 0.05$) in the hardness of VH Equia® Forte Fil in comparison to Fuji IX GP. 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 with even distribution throughout the structure. In addition to the optimized Mw, the presence of highly reactive glass will reinforce the surface properties of the set cement.

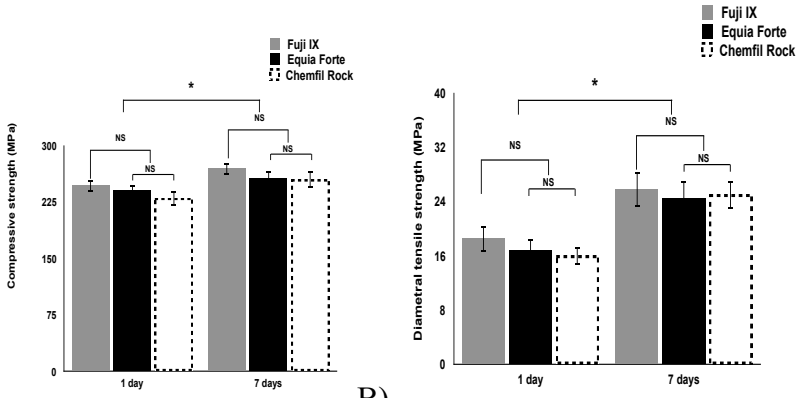
Altogether, the results of the current study suggest that Equia® Forte Fil represents a significant improvement to a well-characterized GIC material (Fuji IX GP). In addition to maintaining CS and DTS comparable to that of Fuji IX GP, improvements to the Equia® Forte Fil's flexural strength and Vickers hardness are promising for future expansion of the applications of GICs in restorative dentistry. In our future studies, more clinically relevant physical properties (e.g., wear resistance, fluoride release, and fracture toughness) will be studied. We will also perform comparative studies with other types of glass ionomer dental cements.

CONCLUSION

In this study, the physical properties (mechanical strength and fluoride releasing characteristics) of Equia® Forte Fil, Chemfil Rock, and Fuji IX GP, three glass ionomer dental cements, were evaluated and compared. Our results confirmed that Equia® Forte Fil is a promising dental restorative material with superior mechanical strength and surface hardness. This material can have a wide range of clinical applications in the everyday practice of pediatric dentistry and as a restorative material for Class I and II restorations in adults in stress-bearing areas.

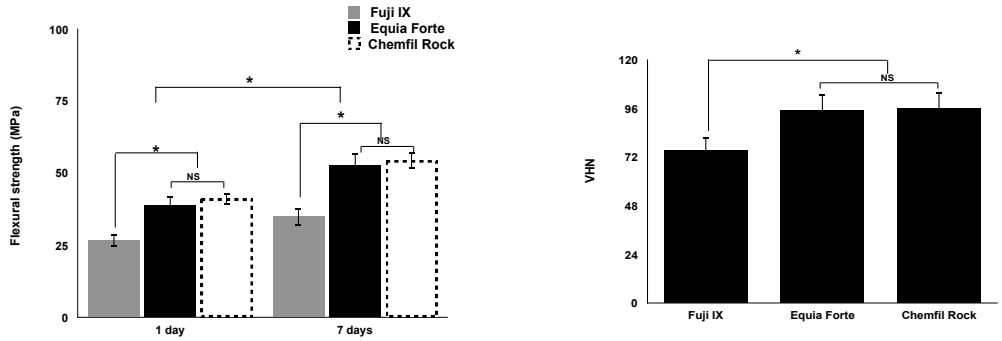
FIGURES

Figure 1. (A) Compressive and (B) diametral tensile strength results of the GIC specimens after 1 and 7 days of storage in distilled water at 37 °C.



A) B)

Figure 2. (A) Flexural strength of the GIC specimens after 1 and 7 days of storage in distilled water at 37 °C. (B) Vickers hardness numbers (VHN) of the GIC specimens after 1 week of storage in distilled water at 37°C.



A) B)

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

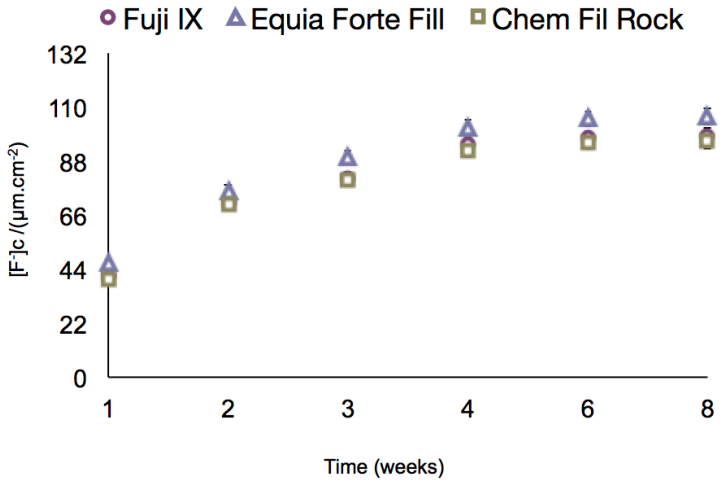


Figure 4. SEM images of (A) Equia® Forte Fil, (B) Chemfil Rock, and (C) Fuji IX glass ionomer particles.

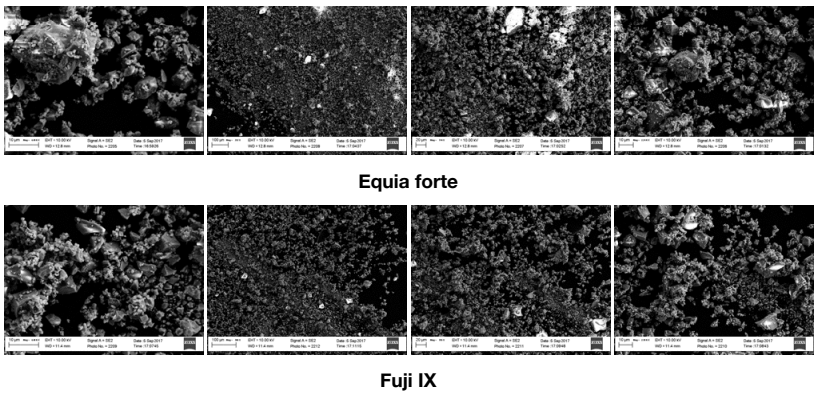
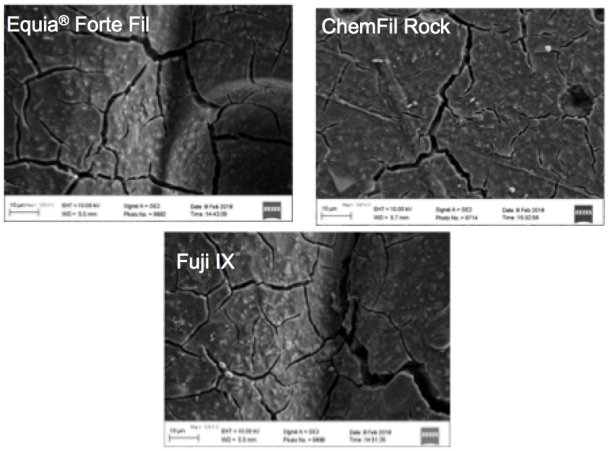


Figure 5. SEM images of surface of set (A) Equia® Forte Fil, (B) Chemfil Rock, and (B) Fuji IX glass ionomer cements after 24 hour of setting.



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