

Evaluation of Flexural Strength and Microhardness of Different Type Glass Ionomer Cements

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Abstract

Purpose: The aim of this study was to evaluate and compare flexural strength and microhardness of different types of glass ionomer cements (conventional, resin-modified and glass hybrid glass ionomer cement).

Materials and Methods: A total of 30 samples (n = 10) were prepared for microhardness test, and an additional total of 30 samples (n = 10) were prepared for the flexural strength test. Customized stainless steel molds (25×2×2 mm³) were produced for the flexural strength test, and 10 mm and 2 mm plexiglass molds were produced for the microhardness test. The prepared samples were stored in distilled water in a dark bottle at 37 °C (±1 °C) for 24 hours. A three-point bending test was performed using a universal testing device, and the maximum force values required to fracture the samples were determined by the device in Newton. Scanning electron microscopy was used to evaluate the surface properties of the samples subjected to three-point bending testing. For the Vickers microhardness test measurements were made from different areas of the upper surfaces of each sample and the average of five measurements were calculated in MPa.

Results: When comparing the flexural strength values, EQUIA Forte Fil group observed the highest flexural strength value, while Riva LC HV group had lowest flexural strength value. When comparing the microhardness values, EQUIA Forte Fil group showed the highest microhardness value, whereas Riva LC HV group observed the lowest microhardness value.

Conclusions: The use of reinforced glass ionomer cements such as EQUIA Forte Fil, which has the highest microhardness and flexural strength values, can increase clinical success.

Key words: flexural strength; glass ionomer cements; microhardness

Introduction

Glass ionomer cement (GICs) can be considered fundamental restorative materials; they are suitable, cost-effective, and easy options for long-term use. They typically self-cure without the need for an adhesive system and do not require complex dental equipment.¹

GICs are used mainly as alternatives to other materials in dentistry due to their properties such as chemical adhesion to tooth structures, fluoride release, bactericidal abilities, and potential to promote remineralization, biocompatibility, thermal compatibility with enamel, and low toxicity.² However, GICs have some disadvantages, such as poor polishability, susceptibility to moisture contamination, low microhardness, fracture toughness, and flexural strength (FS). Therefore, they are preferred for surfaces that are not exposed to high stress levels.³

To overcome these disadvantages, resin monomers were added

to GICs, and it was aimed to improve the mechanical properties and wear resistance of GICs.⁴ Resin-modified GICs (RMGICs) have essentially the same clinical applications as conventional GICs. They can also be used as lining and base material, especially in the primary dentition, in Class V restorations, as fissure sealants and as bonding agents for orthodontic brackets.^{5,6} RMGICs have improved adhesion, aesthetics, moisture sensitivity and good mechanical properties, and are also advantageous due to their light cure and ease of application.⁷ In addition, easy-to-use RMGICs have been produced that are supplied in capsules that allow for an ideal powder/liquid ratio and automatic mixing, facilitating manipulation and optimizing properties of the materials.^{8,9}

EQUIA Forte (EQ), a glass hybrid GIC, is among the materials developed to enhance the mechanical properties of GICs. It has been claimed by the manufacturer that the glass particles contained in EQ powder are equally distributed, ultra-fine and highly reactive, and that the molecular weight of polyacrylic acid is increased,

Table 1. Technical profiles of used materials

Materials	Manufacturer	Type	Composition	p/l Ratio
Riva LC HV	SDI, Victoria, Australia	RMGIC	Polyacrylic acid, tartaric acid, hydroxyethyl-methacrylate, Fluoro-alumino-silicate glass	4.7/1.4
Fuji IX GP® Fast	GC, Tokyo, Japan	Conventional glass-ionomer cement	Alumino-fluoro-silicate glass, polyacrylic acid, distilled water, polybasic carboxylic acid	0.4/0.12
EQUIA Forte Fil	GC, Tokyo, Japan	High-viscosity glass-ionomer cement (HVGIC)	Polyacrylic acid, Polybasic carboxylic acid, Alumino-fluoro-silicate glass, iron oxide	0.4/0.13
EQUIA Forte Coat	GC, Tokyo, Japan	Resin coating	Light-Cured Self-Adhesive Wear Resistant Resin Coat	-

strengthening the mechanical properties of the material.¹⁰ This material represents a new combination of the advantageous properties of GIC and resin coating. The resin coating serves to safeguard the GIC from water contamination during the initial setting phase, while also sealing surface cracks and porosities. Consequently, it enhances wear resistance and toughness, along with improving translucency and achieving a superior marginal seal.¹¹

The durability of dental restorative materials against functional forces, which are indispensable for long-term clinical success, is important.¹² Evaluating mechanical properties like FS and Vickers microhardness (VHN) of restorative materials is a useful and practical method to assess their mechanical behavior.¹³ Therefore, the aim of this study was to evaluate and compare the FS and VHN of different GICs type (Fuji IX GP® Fast, Riva LC HV, EQUIA Forte Fil). The null hypothesis of this study was that there would be no statistical difference in the FS and VHN values of different GICs type.

Material and Methods

The power analysis of the study was performed to determine the sample size, it was decided to take 10 specimens to each group and the power of the test was found to be $p = .89680$. In this study, three different types of GICs were evaluated: glass hybrid GIC (EQUIA Forte Fil), conventional GIC (Fuji IX GP® Fast) and resin-modified GIC (Riva LC HV). The technical profiles of these GIC materials are presented in Table 1.

Sample Preparation

A total of 30 samples ($n = 10$) were prepared for VHN test, and an additional total of 30 samples ($n = 10$) were prepared for the FS test. Customized stainless steel molds ($25 \times 2 \times 2 \text{ mm}^3$) were fabricated in accordance with ISO 4049 specifications for the FS testing, while 10 mm and 2 mm plexiglass molds were produced for the VHN testing. After mixing the GICs in the capsule mixer for 10 seconds, they were filled into the molds to slightly overflow, and a mylar strip was placed on both surfaces. Pressure was applied with a glass layer to prevent air bubble formation during curing and to obtain a smooth surface. Subsequently, for the EQ samples, a layer of EQ Forte Coat was applied to the cement surface using a micro brush and polymerized with a LED light curing unit (Elipar S10, 3M ESPE) that emits light at a wavelength of 430–480 nm for 20 seconds. As for the Riva LC HV samples were polymerized with the same LED light curing unit for 20 seconds at the same wavelength. Finally, the samples were stored in distilled water inside a dark bottle at $37^\circ\text{C} (\pm 1^\circ\text{C})$ for 24 hours.

Flexural Strength Test

A total of 30 samples ($n=10$) were subjected to a three-point bending test at a speed of 1 mm/min using the universal testing device LF Plus (LLYOD Instruments, Amatek Inc., England). The maximum force values required to fracture the samples were determined by the device in Newton (N). The FS of the samples were calculated in MPa using the formula $\sigma = 3PI/2bd^2$. (σ : bending strength, I: distance between support points, b: sample width, d: sample thickness and P: maximum load amount during fracture)

Microhardness Test

Total 30 samples ($n=10$); a load of 300 g was applied through the Vickers indentation for 15 seconds with a digital microhardness tester (Shimadzu HMV-M3, Kyoto, Japan). Measurements were made from different areas of the upper surfaces of each sample, and the average of five measurements were calculated in MPa.

Scanning Electron Microscopy (SEM) Analysis

SEM (LEO-EVO 40/Cambridge-UK) was used to evaluate the surface properties of the samples subjected to FS testing. Samples (BAL-TEC SCD 050 (Liechtenstein)) were gold coated before evaluation by SEM. The entire surface of the sample was scanned and representative areas showing fracture surfaces were photographed at a magnification of $\times 1,000$ with an accelerating voltage of 20 kV.

Statistical Analysis

The data obtained from this study were evaluated using SPSS 23.0 (Statistical Package for Social Science Version: 23). One-way ANOVA analysis was performed to evaluate the data, and the Tukey test was used to find groups that differed as a result of the analysis. p values less than or equal to .05 were considered statistically significant.

Results

The mean and standard deviation values obtained from the FS and VHN tests of the GICs are presented in Table 2. When comparing the FS values, the EQ group observed the highest FS value, while the Riva LC HV group had the lowest FS value. There were statistically significant differences among all materials. ($p < 0.05$). When comparing the microhardness values, the EQ group showed the highest VHN value, whereas the Riva LC HV group observed the lowest VHN value. There were statistically significant differences among all materials. ($p < 0.05$).

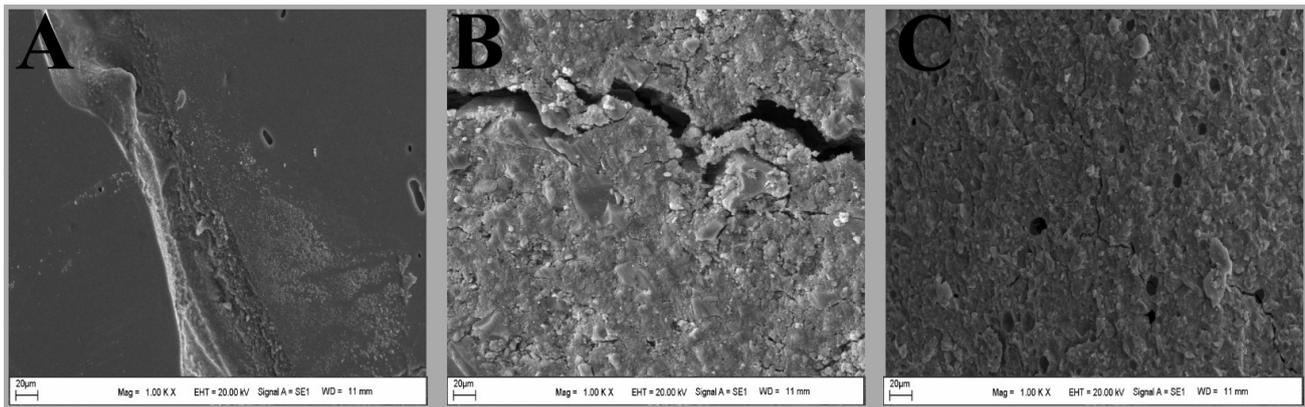


Figure 1. SEM images of samples after FS test: (A) Riva LC HV, (B) Fuji IX GP® Fast, (C) EQUIA Forte Fil

Table 2. Mean and standard deviation (\pm Sd) values of FS and VHN

Materials	Flexural Strength	Microhardness
	Mean \pm Sd	Mean \pm Sd
Riva LC HV	24.77 \pm 2.01A	56.38 \pm 2.90A
Fuji IX GP® Fast	27.65 \pm 2.1B	65.99 \pm 3.71B
EQUIA Forte Fil	44.34 \pm 1.91C	74.97 \pm 2.35C
p value	0.000*	0.001*

Different capital letters in the columns indicate statistical difference.

*: $p < 0.05$ was accepted as the significance level.

SEM Examination

In our study, following the FS test, a randomly selected sample from the fracture surfaces of all groups was subjected to SEM analysis at a magnification at x1,000 (Figure 1). When the SEM images of the materials were examined, a homogeneous surface was observed in the Riva LC HV group with resin content, while different levels of surface irregularities and large gaps caused by broken particles were observed in the Fuji IX GP® Fast and EQUIA Forte Fil groups.

Discussion

The null hypothesis was rejected based on the observed statistical differences in both the FS and VHN values among the GICs with distinct properties used in this study. The use of GICs in dentistry continues to evolve with changes in their mechanical properties and material composition. The physical and mechanical properties of these materials are determined by various factors, including the chemical composition of the polycarboxylic acid, glass structure, its concentration and molecular weight, setting reaction, and powder/liquid ratio.¹⁴ Therefore, in this study, we evaluated the mechanical properties of different GICs, which included a reinforced GIC (EQ Forte Fil), a self-curing conventional GIC (Fuji IX Fast), and a light-curing resin-modified GIC (Riva LC HV), each exhibiting distinctive setting reactions.

Various tests, including microhardness, compressive, diametral tensile, and FS tests, are employed to assess the physical and mechanical properties of dental materials.^{15–18} Microhardness is one of the most important physical properties of dental materials, and the microhardness value can reflect the setting type and reaction of a dental material. FS is one of the important parameters characterizing the ability to withstand chewing loads in stress-bearing areas. The three-point bending test is designed as the primary strength test for testing restorative materials under the international testing standard (ISO 4049).¹⁹

The manufacturer has claimed that the glass particles contained in EQ powder are equally distributed, ultra-fine, and highly reac-

tive and that the molecular weight of polyacrylic acid is increased, strengthening the material's mechanical properties. Moreover, the manufacturer suggests that this material can be used in Class II restorations. This material has also been reported to have high FS and high resistance to abrasion.¹⁰

Moshaverinia et al.²⁰ in their study, evaluated the physical properties of EQ, Fuji IX, and ChemFil Rock glass ionomers, they found the FS and VHN values of the EQ group to be higher than the Fuji IX group. The results of our study are also consistent with this study. Due to the optimized molecular weight of polyacrylic acid, more carboxylic acid groups are used in the acid-base reaction. Thus, polysalt bridge formation and cross-linking in the structure of the cement increases, and the material shows mechanically stronger properties. The presence of highly reactive glass will also strengthen the surface hardness of the cured cement.

In addition, the coating material, a nano-filled resin applied to the EQ surface, may have contributed significantly to the increased resistance of the material against mechanical forces. In research, the observation of higher FS and VHN values after coating glass ionomers with nano-filled resin may support this situation.^{21,22} In addition, metal oxide nanoparticles are used to provide antibacterial properties to restorative materials and improve their mechanical properties.^{23,24} The EQ we used in our study contains iron oxide and therefore may have shown the highest FS and VHN values.

A higher filler volume fraction is expected to lead to increased surface hardness.²⁵ Thus, the hardness of most GICs can be improved by increasing the p/l ratio and reducing the particle size of fillers, which can lead to an increase in the microhardness of these materials.²⁶ Although Riva LC HV had the highest p/l ratio, it was found to have the lowest microhardness among the materials. Due to the plasticizing effect of HEMA, a hydrophilic monomer present in Riva, it may have exhibited lower VHN values compared to other GICs. In their study evaluating light-cured and self-cured glass ionomers in terms of microhardness, Prabhakar et al.²⁷ found that light-cured glass ionomers exhibited lower microhardness values. In our study, the light-cured Riva LC HV group also exhibited lower VHN values than the self-cured Fuji IX group.

It can be speculated that the FS values of the tested materials may be related to the materials' chemical compositions and the hydration of the hardened matrix. When evaluating the results of our study, the highest FS values were observed in self-cured GICs. This may be attributed to the chemical reactions occurring during hardening, which form a highly dense complex. In a study assessing the physical properties of resin-modified and conventional GICs, Moberg et al.²⁸ found FS values for Riva LC HV similar to those in our study. In a study conducted by Bonifacio et al.²⁹ assessing the physical and mechanical properties of different GICs, the FS and microhardness values for Fuji IX were similar to our study's findings.

Conclusion

The use of EQ, characterized by its superior VHN and FS values, along with similarly reinforced GICs, has the potential to enhance clinical success. A limitation of this study is that the oral environment was not simulated. Longer-term in vitro and in vivo studies are required to comprehensively evaluate the biological effects as well as the various physical and mechanical properties of the materials used.

Author Contributions

Idea/Concept: KB, EMA; Design: İİ; Control/Supervision: KB; Data Collection and/or Processing: KB, EMA, İİ; Analysis and/or Interpretation: İİ; Literature Review: KB, EMA; Writing the Article: KB, EMA, İİ; Critical Review: KB; References and Fundings: KB, İİ; Materials: KB, EMA, İİ.

Conflict of Interest

The authors declare that they have no conflicts of interest.

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