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Physicomechanical properties of a zinc-reinforced glass ionomer restorative material

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Abstract: We compared a zinc-reinforced glass ionomer restorative material (ChemFil Rock) with three commercially available glass ionomer cements (GICs), namely, Fuji IX GP Extra, Ketac Molar Quick Aplicap, and EQUIA Fil, with respect to fracture toughness, microhardness, roughness, and abrasive wear. Fracture toughness (K_{IC}) was tested according to ISO 13586 ($n = 10$). Hardness, roughness, and abrasive wear were also tested ($n = 9$). Data were analyzed using the Wilcoxon rank-sum test with adjustment for multiple comparisons ($\alpha = 0.05$). As compared with the other GICs ChemFil Rock exhibited a greater increase in surface roughness ($P < 0.05$) and lower microhardness ($P < 0.01$). The wear resistance of ChemFil Rock was comparable to that of the other GICs ($P > 0.05$). ChemFil Rock had significantly lower fracture toughness as compared with EQUIA Fil ($P = 0.01$) and significantly higher fracture toughness as compared with the other GICs ($P < 0.02$). In conclusion, as compared with the three

other commercially available GICs, ChemFil Rock had intermediate fracture toughness, the lowest microhardness, and the greatest change in surface roughness. (J Oral Sci 56, 11-16, 2014)

Keywords: glass ionomer; wear; hardness; fracture toughness.

Introduction

Glass ionomer cements (GICs) were first launched in Europe in 1975 (1) and first marketed in the United States in 1977. Since then, the composition of GICs has been modified to improve their mechanical properties, resulting in the many GIC materials available today. Conventional GICs are used by dentists because of their biocompatibility, low cytotoxicity (2), fluoride release, and limited microleakage (3). However, they also have less-desirable physical and mechanical properties such as poor polishability, susceptibility to dehydration and moisture contamination during initial setting (4), and low fracture toughness and flexural strength (5).

GICs are recommended in situations such as Class I, II, III, and V restorations in primary teeth, Class III and V restorations in permanent teeth, interim therapeutic restorations, and in atraumatic restorative technique.

A zinc-reinforced glass ionomer (ZRGI) restorative

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Table 1 Materials, codes, descriptions, manufacturers, mixing and setting times, and batch numbers of materials tested

Material	Code	Description	Manufacturer	Mixing time (s)	Setting time (min)	Batch
ChemFil Rock	CFR	Zinc-reinforced glass ionomer	Dentsply	15	6:00	1105000887/1106000636
Fuji IX GP Extra	FIX	Packable glass ionomers	GC America	10	2:30	1112101
Ketac Molar Quick Aplicap	KM	Packable glass ionomers	ESPE	10	3:30	471469
EQUIA Fil	EF	Resin-coated glass ionomer cement	GC America	10	2:30	1204241
Premise Composite	PC	Nanofilled hybrid composite resin	Kerr	N/A	N/A	4442265

Table 2 Mean values and standard deviations for Knoop hardness, surface loss, roughness, and fracture toughness

Material	Knoop hardness (KHN, kg/mm ²)	Surface loss (μm)	Roughness change (R_a , μm)	Fracture toughness (K_{IC} , MPa·m ^{1/2})
ChemFil Rock	52.39 \pm 2.67 ^c	4.69 \pm 1.23 ^{a,b}	0.79 \pm 0.14 ^a	0.99 \pm 0.07 ^b
Fuji IX GP Extra	66.86 \pm 5.36 ^a	5.21 \pm 1.48 ^a	0.10 \pm 0.98 ^{a,b}	0.80 \pm 0.04 ^c
Ketac Molar Quick Aplicap	62.53 \pm 2.91 ^{a,b}	3.79 \pm 2.82 ^{ab}	0.62 \pm 0.60 ^{a,b}	0.85 \pm 0.09 ^c
EQUIA Fil	58.64 \pm 2.01 ^b	5.72 \pm 1.04 ^a	0.14 \pm 0.46 ^b	1.21 \pm 0.23 ^a
Premise Composite	45.44 \pm 2.87 ^d	3.07 \pm 0.93 ^b	0.68 \pm 0.97 ^{ab}	N/A

*Values with the same superscript letters are not significantly different ($P > 0.05$).

material (ChemFil Rock, Dentsply Caulk) was recently introduced to improve flexural strength (6), hardness, wear resistance, and fracture toughness. However, few studies have evaluated these properties. The aim of this study was therefore to compare a ZRGI restorative material with three commercially available GICs in relation to fracture toughness, microhardness, surface roughness, and abrasive wear.

Materials and Methods

Experimental design

This study investigated restorative material as the single experimental factor, using a completely randomized design in two independent phases. In phase 1 (surface properties), specimens ($n = 9$) of ChemFil Rock (CFR) and three other commercially available conventional GICs—Fuji IX GP Extra (FIX), Ketac Molar Quick Aplicap (KM), and EQUIA Fil (EF)—and a resin-based composite (control), Premise (PC), were tested for surface roughness (R_a), surface loss (μm), and microhardness (KHN) (Table 1). In phase 2 (fracture toughness), GIC specimens ($n = 10$) were prepared and tested for fracture toughness (MPa·m^{1/2}). Color shade A2 was selected for all materials.

Phase 1 (test of surface characteristics)

Specimen preparation

The sequence of specimen ($n = 9$) preparation and testing followed a previously determined randomization schedule. Each material was mixed according to the manufacturers' instructions, injected into circular metal molds ($\phi = 5$ mm; height 2 mm), covered with a

Mylar strip and a glass slide, and allowed to set for the recommended time (Table 1). Resin surface sealant was applied to EF specimens and light-cured for 20 s. PC was syringed into metal molds and covered with a Mylar strip, after which the resin was polymerized using a Demi light-curing unit (Kerr, Danbury, CT, USA) with a light output of 625 mW/cm² for 40 s. The power of the curing unit was measured using a radiometer (Demetron; Kerr) to ensure that light output exceeded 400 mW/cm².

Specimens were maintained in 100% relative humidity at 37°C for 20 min (7,8) and embedded in acrylic resin (Varidur; High Performance Mounting Kit; Buehler, Lake Bluff, IL, USA) to facilitate mounting in the testing devices. Specimen surfaces were wet-polished using a sequence of 500, 1,200, 2,400, and 4,000 grit silicon carbide paper (9), then immersed in distilled water at 37°C for 24 h (6,8-10).

Measurement of baseline surface roughness and surface loss

The specimens were scanned by a 3-D optical profilometer (Proscan 2000, Scantron Industrial Products Ltd., Taunton, UK) using the S5/03 chromatic sensor. Two areas were scanned. First, roughness was measured in a square (0.5×0.5 mm) located at the center of the specimen (step size, 0.01×0.01 ; number of steps, 100). Then the entire specimen surface (3×3 mm) was scanned to measure wear (step size, 0.1×0.1 ; number of steps, 60). All scanning was completed at a frequency of 100 Hz with full sensor speed (100%).

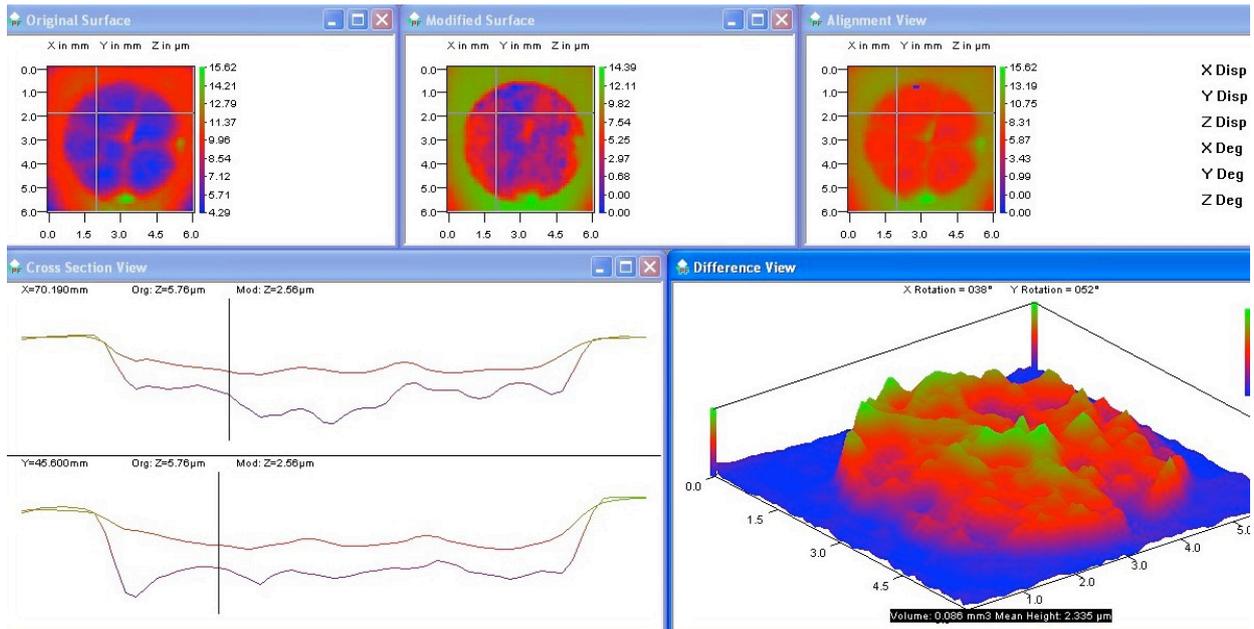


Fig. 1 Representative images of material surface loss as determined by image subtraction (profilometer) using the Ketac Molar Quick Aplicap. The upper three screens represent pre-surface treatment (left), post-surface treatment (middle), and superimposition of pre- and post-surface treatment images (right). In the lower left screen, the upper and lower lines represent pre- and post-surface treatment morphology, respectively. The lower right screen shows final surface loss.

Microhardness measurement

Three indentations were made at the surface periphery of each specimen with a Knoop diamond indenter under a 0.245-N load and a dwell time of 30 s (11-13), using a hardness tester (LECO Corporation, St. Joseph, MI, USA). The average of the three measurements was recorded as the microhardness value for the specimen.

Toothbrushing abrasion test

Next, each specimen was mounted on a custom-made toothbrushing machine and brushed using a straight, soft toothbrush (Oral-B 40, Procter & Gamble, Cincinnati, OH, USA) with a load of 1.96 N (14-16) at a speed of 175 cycles/min for 20,000 double strokes (7,17,18). This is reported to simulate roughly 2 years of brushing (18). A new toothbrush was used with each specimen. A dentifrice (Crest Cavity Protection, Procter & Gamble) was used as an abrasive slurry with a paste to water weight ratio of 1:1 (17,18). Each specimen was brushed with 80 g of slurry, and a fresh slurry was used for each specimen. After toothbrushing, specimens were rinsed with tap water and gently air-dried.

Surface measurements after abrasion

Specimen surfaces were rescanned with the Proscan profilometer to determine surface loss and roughness after toothbrush abrasion. Specimens were seated on a

custom jig that ensured identical positioning of specimens for all measurements. Measurements of surface loss and roughness were calculated by image subtraction, based on differences between pre- and post-treatment profiles. Dedicated software (Proform software, version 2.0.17, Scantron Industrial Products Ltd.) was used for the calculations (Fig. 1).

Phase 2 (test of fracture toughness, K_{Ic})

Forty rectangular-bar specimens ($n = 10$) were fabricated by injecting each material into a stainless steel mold ($25 \times 2.5 \times 5$ mm), which produced a 2-mm notch in the specimens. Specimens were maintained in 100% relative humidity for 1 h (19), then in distilled water at 37°C for 24 h before testing (8,20).

Before testing, the height and width of each specimen was re-measured with a digital caliper (Mitutoyo Corporation, Kawasaki, Japan). The values were entered into the software (TestWorks 4.0, MTS Systems Co., Eden Prairie, MN, USA) for the screw-driven universal testing machine (MTS Sintech Renew 1123). A 3-point bending test device and a crosshead speed of 0.2 mm/min were used. Fracture toughness (K_{Ic}) was calculated using the following equation:

$$K_{Ic} = f(a/w)(F/h\sqrt{w})$$

where K_{Ic} is fracture toughness ($\text{MPa}\cdot\text{m}^{1/2}$), F is force at

the beginning of crack propagation (N), a is crack length (mm), h is specimen thickness (mm), w is specimen width (mm), and $f(a/w)$ is the fracture geometry factor, calculated as:

$$6\alpha^{1/2} [1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)] / [(1+2\alpha)(1-\alpha)^{3/2}]$$

Statistical methods

The Wilcoxon rank-sum test was used to compare groups for differences in Knoop hardness, abrasive wear, surface roughness (R_a), and fracture toughness (K_{Ic}). The Sidak adjustment was used to control for multiple pair-wise group comparisons. All statistical analyses were carried out at a 5% significance level.

Results

Phase 1

Knoop hardness

Among the tested materials, FIX had the highest hardness value (66.86 KHN) and PC had the lowest value (45.44 KHN). The hardness value was significantly higher for FIX than for EF ($P = 0.004$). The hardness was significantly lower for CFR than for EF, KM, and FIX ($P \leq 0.008$). The hardness of PC was significantly lower than that of the other groups ($P = 0.006$ CFR, $P = 0.004$ FIX, KM and EF).

Toothbrush abrasion

The mean average surface loss for EF, FIX, CFR, KM, and PC was 5.72 μm , 5.21 μm , 4.69 μm , 3.79 μm , and 3.7 μm , respectively. The values for EF and FIX were significantly higher than that for PC ($P = 0.004$ and $P = 0.046$, respectively). However, there was no significant difference between KM and the other groups ($P > 0.05$).

Roughness

Mean change in R_a in CFR, PC, KM, EF, and FIX was 0.79 μm , 0.68 μm , 0.62 μm , 0.14 μm , and 0.10 μm , respectively. Values for roughness change were significantly or marginally higher for CFR than for EF and FIX ($P = 0.006$ and $P = 0.060$, respectively). However, there was no significant difference between PC and the other tested materials ($P > 0.05$).

Phase 2

Fracture toughness

The K_{Ic} for EF was the highest among the tested GICs and was significantly higher than the values for CFR, FIX, and KM ($P = 0.013$, $P = 0.001$, $P = 0.001$, respectively). The K_{Ic} for CFR was significantly higher than the values for FIX and KM ($P = 0.001$ and $P = 0.022$, respectively).

However, there was no significant difference between FIX and KM ($P > 0.05$).

Discussion

The addition of zinc oxide particles is the essential modification of CFR, which also contains a high-molecular-weight acrylic acid polymer. The manufacturer maintains that inclusion of zinc oxide enhances the setting reaction and increases strength, while retaining similar methods of clinical application and working time as compared with regular GICs.

A nanofilled hybrid composite (Premise) was included as a control group in the Phase 1 study (microhardness, wear, and surface roughness testing) because there were no obvious differences between the various GIC groups in our preliminary pilot study. No significant difference was found in the hardness values of FIX and KM, which were harder than the other materials tested. These findings are consistent with those of previous studies (6,12). EF was significantly harder than CFR, as was noted in another study (6). Microhardness was significantly lower for PC than for the GICs, which accords with the findings of previous studies (21,22). The relatively low hardness of CFR in this study could be due to filler size and morphology (11) or to insufficient dispersion of zinc and glass particles.

Abrasive wear did not significantly differ among the GICs tested in the present study, which confirms the findings of previous investigations (15,23). However, the abrasive wear of all GICs was significantly or marginally greater than the values noted in the tested nanofilled hybrid composite (Premise). Due to their composition, GICs exhibit more wear than composites. Their acid-base reaction results in a matrix comprising an ionically cross-linked polyalkenoate network, which is weaker than the matrix of composites strengthened by fillers and 2-hydroxyethyl methacrylate polymer chains (24).

Attraction of dental plaque to roughened restorations is a concern since it may increase the risk of secondary caries (18). The surfaces of carefully polished dental restorations can be compromised by subsequent home care, including toothbrushing. Most studies of the effects of toothbrushing and polishing on dental restorations concluded that restoration surfaces are smoother before polishing or toothbrushing and tend to increase in roughness afterward (10,15,25,26).

In the current study, CFR exhibited the greatest change in surface roughness among GICs, perhaps due to differences in composition. Previous studies concluded that the surface roughness of GICs is affected by filler size, shape, distribution, and number of particles in the matrix

(10).

In this study, the fracture toughness of GICs was tested after 24 h, to ensure assessment at the peak strength of the materials (27). Fracture toughness was significantly greater for EF than for the other GICs; CFR had the second highest fracture toughness. The high fracture toughness of EF may be due to the resin coat applied to the surface. In addition, as compared with CFR, EF has a wider ranges of clinical uses in high-stress-bearing areas and cases requiring buildup. The relative high fracture toughness of CFR could be due to formation of zinc-polycarboxylate complexes during the setting reaction (6). Additionally, the incorporation of itaconic acid as a comonomer in CFR might increase the flexural and tensile strength of GIC (28). Another explanation for the relative high fracture toughness of CFR is its small mean particle size as compared with other conventional GICs (11). Our data on the fracture toughness of KM are consistent with those from a previous study (29). In summary, we conclude that, as compared with the other tested GICs, CFR had intermediate fracture toughness and comparable abrasive wear but inferior surface roughness and hardness characteristics.

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References

- Nicholson JW (1998) Chemistry of glass-ionomer cements: a review. *Biomaterials* 19, 485-494.
- Costa CA, Ribeiro AP, Giro EM, Randall RC, Hebling J (2011) Pulp response after application of two resin modified glass ionomer cements (RMGICs) in deep cavities of prepared human teeth. *Dent Mater* 27, e158-170.
- Cehreli SB, Tirali RE, Yalcinkaya Z, Cehreli ZC (2013) Microleakage of newly developed glass carbomer cement in primary teeth. *Eur J Dent* 7, 15-21.
- McCabe JF, Jones PA, Wilson HJ (1979) Some properties of a glass ionomer cement. *Br Dent J* 146, 279-281.
- Topbasi B, Öveçoglu ML, Türkmen C (2003) Flexural strength and fracture surface characterization of glass-ionomer cements stored in water. *Oral Health Dent Manag* 2, 18-26.
- Zoergiebel J, Ilie N (2013) Evaluation of a conventional glass ionomer cement with new zinc formulation: effect of coating, aging and storage agents. *Clin Oral Investig* 17, 619-626.
- Momoi Y, Hirotsaki K, Kohno A, McCabe JF (1997) In vitro toothbrush-dentifrice abrasion of resin-modified glass ionomers. *Dent Mater* 13, 82-88.
- Zhao J, Weng Y, Xie D (2009) In vitro wear and fracture toughness of an experimental light-cured glass-ionomer cement. *Dent Mater* 25, 526-534.
- Heintze SD, Forjanic M, Ohmiti K, Rousson V (2010) Surface deterioration of dental materials after simulated toothbrushing in relation to brushing time and load. *Dent Mater* 26, 306-319.
- Bala O, Arisu HD, Yikilgan I, Arslan S, Gullu A (2012) Evaluation of surface roughness and hardness of different glass ionomer cements. *Eur J Dent* 6, 79-86.
- Xie D, Brantley WA, Culbertson BM, Wang G (2000) Mechanical properties and microstructures of glass-ionomer cements. *Dent Mater* 16, 129-138.
- Bonifácio CC, Kleverlaan CJ, Raggio DP, Werner A, de Carvalho RC, van Amerongen WE (2009) Physical-mechanical properties of glass ionomer cements indicated for atraumatic restorative treatment. *Aust Dent J* 54, 233-237.
- Raggio DP, Bonifácio CC, Bönecker M, Imparato JC, Gee AJ, Amerongen WE (2010) Effect of insertion method on knoop hardness of high viscous glass ionomer cements. *Braz Dent J* 21, 439-445.
- Frazier KB, Rueggeberg FA, Mettenburg DJ (1998) Comparison of wear-resistance of Class V restorative materials. *J Esthet Dent* 10, 309-314.
- Rios D, Honório HM, de Araújo PA, Machado MA (2002) Wear and superficial roughness of glass ionomer cements used as sealants, after simulated toothbrushing. *Pesqui Odontol Bras* 16, 343-348.
- de Paula AB, Fucio SB, Ambrosano GM, Alonso RC, Sardi JC, Puppim-Rontani RM (2011) Biodegradation and abrasive wear of nano restorative materials. *Oper Dent* 36, 670-677.
- Tanoue N, Matsumura H, Atsuta M (2000) Analysis of composite type and different sources of polymerization light on in vitro toothbrush/dentifrice abrasion resistance. *J Dent* 28, 355-359.
- Tanoue N, Matsumura H, Atsuta M (2000) Wear and surface roughness of current prosthetic composites after toothbrush/dentifrice abrasion. *J Prosthet Dent* 84, 93-97.
- Yamazaki T, Schricker SR, Brantley WA, Culbertson BM, Johnston W (2006) Viscoelastic behavior and fracture toughness of six glass-ionomer cements. *J Prosthet Dent* 96, 266-272.
- Moshaverinia A, Brantley WA, Chee WW, Rohpour N, Ansari S, Zheng F et al. (2010) Measure of microhardness, fracture toughness and flexural strength of N-vinylcaprolactam (NVC)-containing glass-ionomer dental cements. *Dent Mater* 26, 1137-1143.
- Kim KH, Ong JL, Okuno O (2002) The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 87, 642-649.
- Blackham JT, Vandewalle KS, Lien W (2009) Properties of hybrid resin composite systems containing prepolymerized filler particles. *Oper Dent* 34, 697-702.
- Peutzfeldt A, Garcia-Godoy F, Asmussen E (1997) Surface hardness and wear of glass ionomers and compomers. *Am J Dent* 10, 15-17.
- Davidson CL (2006) Advances in glass-ionomer cements. *J Appl Oral Sci* 14, Suppl, 3-9.
- Carvalho FG, Fucio SB, Paula AB, Correr GM, Sinhoreti

- MA, Puppin-Rontani RM (2008) Child toothbrush abrasion effect on ionomeric materials. *J Dent Child* 75, 112-116.
26. Kakuta K, Wonglamsam A, Goto S, Ogura H (2012) Surface textures of composite resins after combined wear test simulating both occlusal wear and brushing wear. *Dent Mater J* 31, 61-67.
27. Lucas ME, Arita K, Nishino M (2003) Toughness, bonding and fluoride-release properties of hydroxyapatite-added glass ionomer cement. *Biomaterials* 24, 3787-3794.
28. Moshaverinia A, Roohpour N, Darr JA, Rehman IU (2009) Synthesis and characterization of a novel N-vinylcaprolactam-containing acrylic acid terpolymer for applications in glass-ionomer dental cements. *Acta Biomater* 5, 2101-2108.
29. Bonilla ED, Mardirossian G, Caputo AA (2000) Fracture toughness of various core build-up materials. *J Prosthodont* 9, 14-18.