

Physical Property Investigation of Contemporary Glass Ionomer and Resin Modified Glass Ionomer Restorative Materials

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Abstract

Objective: To investigate selected physical properties of nine contemporary and recently-marketed glass-ionomer cement (GIC) and four resin-modified glass-ionomer cement (RMGIC) dental restorative materials.

Methods: Specimens (n=12) were fabricated for fracture toughness and flexure strength using standardized, stainless steel molds. Testing was completed on a universal testing machine until failure. Knoop Hardness was obtained using failed fracture toughness specimens on a microhardness tester, while both flexural modulus and flexural toughness was obtained by analysis of the flexure strength results data. Testing was completed at 1 hour, 24 hours, 1 week, and then at 1, 3, 6, and 12 months. Mean data was analyzed with Kruskal-Wallis and Mann Whitney (p = 0.05)

Results: Consultation with the tables within will reveal that physical properties results were material dependent. Physical properties of the GIC and RMGI products were inferior at hour compared to that at 24 hours. Some improvement in selected physical properties were noted over time, but most materials reached stability by one week. A few materials demonstrated improved physical properties over the course of the evaluation.

Conclusions: Under the conditions of this study,

1. GIC and RMGI physical property performance over time was material dependent.

 Although differences in GIC physical properties were noted, the divergences were of such small magnitude that it is unlikely that it would be of clinical significance.

3. Modest increases in some GIC physical properties were noted in time, especially flexural modulus and hardness, which lends support to reports of a maturing silicate matrix.

4. Overall, GIC product physical properties were more stable than RMGI, provides credence to reports of RMGI matrix degradation due to imbibition of water.

5. Globally, RMGI products demonstrated higher values of flexure strength, flexural toughness, and fracture toughness.

Manuscript

Introduction

The optimal dental restorative material would be biocompatible, demonstrate adhesion to tooth material, cariostatic, and serve as a dentin and enamel replacement with appropriate strength, wear resistance, flexure, and esthetics. ¹ The ideal dental restorative material may have yet to be discovered, but as advances in technologies progress it is hoped that future materials can be developed that address caries, function, biocompatibility, and minimal environmental impact. ^{2,3}

Glass-ionomer cements were invented and developed by Wilson and Kent in the early 1970's as an attempt to overcome shortcomings associated with silicate restorative cements. ^{4,5} Glass ionomer cements generally consist of a mixture of various polyalkenoate (polyacrylic) acids and tartaric acid that react with a fluoroaluminosilcate glass. The setting reaction occurs via an acid-base reaction between the acid and the glass surface which liberates metal cations that serve as a source of crosslinking between the polyalkenoate chains. ^{6,7} Additional maturation beyond 24 hours continues consisting of polymer cross linking and formation of a silica gel phase. ⁸⁻¹⁰

GICs are self-adhesive materials that bond to tooth hard tissues through chemical bonding to dental hard tissues, ¹¹ as the ionic bond between the polyalkenoate acid carboxyl groups and hydroxyapatite in enamel and dentin is responsible for the GIC adhesion capability. ^{11,12} The early GIC restorative materials have been improved with changes in formulations in attempts to improve their physical properties and clinical handling characteristics. ^{7,13,14} These modifications have included the use of alternative polyacids, ^{7,15,16} water-activated dehydrated polyacid powders, ^{7,15,17} cermets, ¹⁸ metal additions, ¹⁹⁻²¹ smaller glass particle size, ²² antibacterial agents, ^{23,24} different glass compositions, ^{15,25} and most recently aluminum-free glasses. ²⁶

Resin-modified GICs (RMGICs) were first developed in the latter 1980's for use as bases and liners but were developed further to serve as direct restorative materials, hopefully to overcome early moisture sensitivity and lower mechanical properties associated with the GIC restoratives of that era. 27-29 Similar to GIC materials, RMGICs cure via an acid-base reaction but additionally with a free-radical polymerization of the material's resin content. 28,30,31 The resin content is added either by a direct addition of a resin monomer such as 2-hydroxyethyl methacrylate (HEMA) or is uniquely grafted on the polyalkenoate acid chain. These monomers polymerize either by external photo activation or by an internal chemical reaction. 28,29 RMGI materials also demonstrate bonding to tooth structure, as X-ray photoelectron spectroscopy and infrared spectroscopy have demonstrated chemical bonding to tooth material as well as having the ability to form hybrid layer micromechanical interlocking similar to, but not to the same extent as resin adhesive systems. 32-36 Although the exact composition of each GIC and RMGI materials is usually proprietary, the polymerized resin composition in the early RMGI products was estimated to be approximately 4.5-6%.³⁷ The resin and polyalkenoate setting reactions compete with each other as the resin content retards the polyalkenoate reaction with stereo chemical distortion of the polyalkenoate acid chains making reaction sites less available. Furthermore, RMGIs contain less water for the polyalkenoate reaction to proceed as resin is added at the expense of water. 11,38-40

The setting reaction of glass ionomer materials has been a subject of interest in the scientific community. Glass ionomer products have been investigated by several different methods to include infrared spectroscopy, ⁴¹ Raman spectroscopy, ⁴² NMR spectroscopy, ⁴³ pH measurements, ⁴⁴ rheology, ^{45,46} dielectric spectroscopy, ⁴⁷⁻⁵⁰ inductively coupled plasma optical emission spectroscopy (ICP-OES), ⁵¹ and thermal analysis techniques. ⁵² Although some laboratory novel GIC formulations have shown the capacity for continuation of the acid-base reaction, ³⁰ it has been established by different analysis methods that the GIC polyalkenoate acid-base reaction is essentially complete by 24 hours. ^{30,41,43,53,54} However, the continued changes in both the organic and inorganic GIC matrix past 24 hours remains a

source of interest as some GIC products continue to display an increase in physical properties with time. ⁵⁵⁻⁵⁸ The initial setting reaction and subsequent silica matrix formation is a multifaceted phenomenon and Nicholson ⁷ relates the possibility of an intermediary phase that may play a role in the timing of these intertwining processes. Evidence of such a phase has been recently reported by Dickey et al ⁵⁹ who also observed a complex phase, particular to the glasses used, that delayed the crosslinking of the GIC matrix. After the acid-base reaction completion, both Stamboulis et al ⁴³ and Zainuddin et al ³⁰ reported using different analysis techniques the formation of a silicate network and a hydrated silica gel phase, as reported earlier of consisting of either a pure silicate or a mixed silicate phosphate matrix that forms and whose maturation contributes to the improvement of physical properties. ⁶⁰

Both GIC and RMGI materials have been shown to display excellent clinical performance with atraumatic restoration treatment (ART), ⁶¹⁻⁶⁶ as well as definitive restorations in both primary and permanent teeth. ⁶⁷⁻⁷³ A number of both GIC and RMGI restorative products have been recently marketed but lack independent research of these materials' physical properties over time. The purpose of this study was to evaluate selected physical properties, namely flexural strength, flexural modulus, Knoop hardness, fracture toughness, and the flexural toughness of newer GIC and RMGI restorative materials compared with materials that have enjoyed marketing tenure. The null hypotheses is that there will be no difference in the physical properties between the tested restorative materials.

Materials and Methods

The restorative materials used in this study were comprised of four RMGI products and nine GIC products and are listed in Tables 1 and 2, respectively.

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Material	Manufacturer	Powder/Liquid Ratio (g/g)	Powder Content	Liquid Content
Fuji II LC Capsules	GC America (Alsip, IL, USA)	0.33/.010	Trade Secret	HEMA 25-50% Polybasic Carboxylic Acid 5-10% UDMA 1-5% Dimethacrylate 1-5%
Ketac Nano Quick Mix Capsules	3M ESPE (St. Paul, MN, USA)	N/A	PASTE A: Silane Treated Glass 0- 55% Silane Treated Zirconia 0- 30% PEGDMA 5-15% Silane Treated Silica 5-15% HEMA 1-15% BISGMA <5% TEGDMA <1 %	PASTE B: Silane Treated Ceramic 40-60% Copolymer of Acrylic and Itaconic Acids 20-30% Water 10-20% HEMA 1-10%
Riva LC Capsules	SDI Limited (Bayswater, Victoria, AUS)	0.42/0.14	Fluoroaluminosilicate glass powder 95-100%	Polyacrylic Acid 15-25% Tartaric Acid 1-5% HEMA 20-30% Dimethacrylate Cross-linker 10- 25% Acidic Monomer 10-20%
Riva LC HV Capsules	SDI Limited	0.47/0.14	Fluoroaluminosilicate glass powder 95-100%	Polyacrylic Acid 15-25% Tartaric Acid 1-5% HEMA 15-25% Dimethacrylate Cross-linker 10- 25% Acidic Monomer 10%

Content information obtained from manufacturer information

Material	Material Manufacturer		Powder Content	Liquid Content		
Chemfil Rock Capsules	Dentsply International, (York, PA, USA)	entsply ternational, * Polycarboxylic acid 10-25% ork, PA, 5A)		* Polycarboxylic acid 10-25%		Polycarboxylic acid 10-25% Tartaric Acid 2.5- 10%
Fuji IX GP EXTRA Capsules	GC America (Alsip, IL, USA)	0.40/0.12	Trade Secret	Trade Secret		
Fuji Triage Capsules	GC America	0.30/0.15	Trade Secret	Trade Secret		
Ketac Molar Quick Aplicap	ap Oxide Glass Chemicals (non-fibrous) SM ESPE (St. Paul, MN, USA) Oxide Glass Chemicals (non-fibrous) 85-95% Copolymer of Acrylic Acid-Maleic Acid 1-5% Dichlorodimethylsilane Reaction Product with Silica <2%		Water 60-65% Copolymer of Acrylic Acid-Maleic Acid 30-40% Tartaric Acid 10%			
Ketac Silver Aplicap	3M ESPE	*	Silver 45-55% Oxide Glass Chemicals 40-50% Titanium Dioxide 1-5% Copper <0.01	Water 40-60% Copolymer of Acrylic Acid-Maleic Acid 30-50% Tartaric Acid 5-15%		
Riva Protect Fast Capsules	SDI Limited, (Bayswater, Victoria, AUS)	0.34/0.19	Fluoro Aluminosilicate Glass 90% Polyacrylic Acid 10%	Polyacrylic Acid 25% Tartaric Acid 10%		
Riva Self Cure Fast Capsules	SDI Limited	0.40/0.15	Fluoro Aluminosilicate glass 90-95% Polyacrylic Acid 5-10%	Polyacrylic Acid 20-30% Tartaric acid 10-15%		
Riva Self Cure High Viscosity Capsules	SDI Limited	0.50/0.13	Fluoro Aluminosilicate glass 90-95% Polyacrylic Acid 5-10%	Polyacrylic Acid 20-30% Tartaric acid 10-15%		
Riva Silver Capsules	SDI Limited	0.72/0.14	Fluoro Aluminosilicate Powder 40-60% Polyacrylic Acid <10% Alloy Powder 30-50%	Polyacrylic Acid 30% Tartaric acid 10% Balance ingredient 60%		

Twelve specimens were fabricated for each test. Fracture toughness and flexural strength specimens were fabricated using standardized, stainless-steel molds (Sabri Dental Enterprises, Downers Grove, IL,

USA). Materials were placed into respective molds on a mylar-strip-covered glass slab with a second mylar strip placed on top of the filled mold. A glass microscope slide was then placed with digital pressure to form a uniformly flat surface with GIC materials were allowed to set for the manufacturer recommended setting time in an oven at 35 °C. RMGI materials were polymerized using a light-emittingdiode (LED) visible-light-curing (VLC) unit (Bluephase G2, Ivoclar-Vivadent, Amherst, NY, USA) for 20 seconds in an overlapping fashion on both sides. The performance of the VLC unit was periodically assessed using a laboratory-grade laser power meter (10A-V1, Ophir-Spiricon, North Logan, UT, USA). Specimens were then removed from their respective molds and refined as needed using surgical scalpels removing any flash material from the edges. Completed specimens were stored under dark conditions in (0.2M) physiologic phosphate buffered saline solution at 37 °C and 98 ± 1 percent humidity until the appointed time of testing. Testing occurred at one hour, 24 hours, one week, and then at 1, 6, and 12 months after fabrication.

Flexural Strength: Specimens were formed in a 2 x 2 x 25 mm mold (Figure 1) and were tested on a three



point bend apparatus mounted on a universal testing machine (Alliance RT/5, MTS Corporation, Eden Prairie, MN, USA). Specimens were stressed using a cross head speed of 0.5mm/min until failure with the maximum force obtained recorded. Flexure strength results was determined using the formula



Flexural Modulus: Modulus was determined by the slope of the linear portion of the flexure strength stress-strain curve (Figure 2). Mean results were determined and recorded.



Flexural Toughness: Toughness was determined by integrating the area under the flexural testing stress/strain curve (Figure 3). Mean values were recorded.



Fracture Toughness: Specimens were fabricated in accordance with the single edge notch beam method as described in ASTM Standard E399 (Figure 4).



The specimens were fabricated as described earlier using a custom stainless steel mold as seen in Figure 5. Each specimen's dimensions were measured in three equally-spaced positions along the specimen with the mean recorded.



As with flexure strength testing, specimens were tested in 3-point bending apparatus with a crosshead

speed of 0.5mm/min in a universal testing machine (Alliance RT/5) until failure (Figure 6).



After failure, the true notch length was measured with digital measuring microscope (Hirox 4400, Hirox USA, Hackensack, NJ, USA). The fracture toughness calculation was accomplished using the following equation:



Micro Hardness: Surface hardness was determined using specimens obtained from the fracture toughness testing samples. The microhardness testing machine parameters can be seen in Figure 7.

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LOUGI D BAGE 36		15 25	mm.			
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Distance betwee	in indents:	0 525	mm			
No of indents		11	_			

Eleven Knoop Hardness (KH) indentations were made taken in an alternating fashion over a 5.25 millimeter distance with 0.5 millimeter spacing between indentations using a pyramidal Knoop indenter (OmniMet MHT, Buehler Manufacturing, Inc., Lake Bluff, IL, USA) using a 100-gram load and a dwell time of 10 seconds (Figure 8).



The width of the indentations were directly measured at 50X magnification (Figure 9).

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Knoop hardness (KHN) was computed based on the equation

$HK = P/Cl^2$

P = Applied load (kg) C = indenter-related constant (0.07028) (Compensates for projected area of indentation) (Square of the indenter long diagonal) I = measured length of the indentation

The mean of the eleven measurements was calculated as the representative mean hardness for each sample. A total of ten specimens were tested for each material, with the mean determined.

Statistical Analysis: The Shaprio-Wilk Test and Bartlett's Test identified irregularities in both the distribution and variance of the mean data. Therefore, the data was analyzed using non-parametric Kruskal-Wallis with Mann-Whitney post hoc testing was performed with a 95 percent level of confidence (p = 0.05). Statistical analysis was performed using SPSS 21 (IBM/SPSS, Chicago, IL, USA).

Results

The mean results for the physical property testing of the GIC restorative materials are listed in the following Tables 3-7.

	R MARINE S	Fle	exural Stren	igth (MPa)					
	Conventional Glass Ionomers								
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months		
Ch	16.59 (4.6)	33.62 (6.4)	27.45 (3.1)	34.88 (6.3)	33.88 (6.3)	38.66 (5.9)	32.1 (5.7)		
Chemfil	Ва	E cd	Bb	C cd	Ccd	Dd	BC bc		
-	17.43 (5.7)	26.81 (4.7)	19.83 (5.07)	27.16 (3.4)	31.48 (5.3)	24.0 (3.0)	30.7 (5.9)		
FUJITXEXTRA	Ba	CD ab	A ab	AB ab	BCb	Aa	ABC b		
	10.38 (5.3)	19.88 (4.9)	26.4 (6.0)	24.38 (4.3)	24.57 (5.0)	23.23 (4.3)	26.1 (6.0)		
Fuji Triage	Aa	ABb	Bc	A bc	A bc	A bc	ABc		
	18.6 (5.4)	21.65 (7.6)	29.65 (6.6)	32.3 (6.7)	31.48 (5.2)	30.5 (6.7)	30.3 (3.5)		
Ketac Molar Quick	Ва	ABCa	BCb	Cb	BCb	BCb	ABC b		
Kata Oliver	9.78 (2.1)	26.26 (5.4)	25.42 (5.7)	31.2 (3.3)	30.08 (3.3)	26.37 (3.0)	29.52 (5.3)		
Ketac Silver	Aa	BCD bc	ABb	BC d	BC cd	AB bc	ABC bcd		
Dive Destant	8.8 (4.2)	18.04 (4.4)	19.43 (5.4)	24.38 (3.6)	23.41 (4.5)	24.17 (4.1)	24.67 (6.0)		
Riva Protect	Aa	Ab	A bc	Ad	A cd	A cd	Ad		
	17.7 (4.2)	28.81 (4.0)	33.78 (5.0)	31.63 (4.4)	28.28 (4.2)	32.7 (6.0)	33.2 (3.3)		
Riva Self Cure Fast	Ba	DE bc	CD d	BC bcd	AB b	C bcd	C cd		
	19.7 (4.4)	25.78 (6.3)	37.59 (5.5)	33.83 (3.3)	32.58 (3.2)	29,67 (3.5)	28.11 (4.6)		
Riva Self Cure HV	Ba	BCD b	De	C de	BC cd	BC bcd	ABC bc		
Dive Cilves	9.46 (3.3)	28.9 (6.7)	33.7 (4.0)	33.6 (3.0)	34.3 (4.3)	32.7 (4.2)	30.9 (4.8)		
Riva Sliver	Aa	DE b	CD c	Cc	Cc	C bc	ABC c		

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Table 4

	Modulus (GPa)								
Conventional Glass Ionomers									
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months		
Chamfil	4.8 (0.4)	9.02 (0.5)	7.68 (1.05)	8.45 (1.1)	8.53 (1.1)	10.48 (1.8)	9.9 (1.3)		
Cnemm	BC a	CD cd	ABb	B bc	A bc	BC d	A cd		
ENU IN ENTRA	4.8 (1.2)	9.7 (1.5)	7.5 (1.1)	10.9 (1.9)	14.67 (1.2)	10.3 (1.1)	12.2 (2.2)		
FUILIA EATRA	Ba	Dc	AB b	CDE c	Dd	BCc	B cd		
Cull Trings	3.1 (0.6)	7.8 (0.5)	8.4 (1.4)	9.0 (0.9)	10.0 (2.0)	10.2 (1.0)	10.2 (1.6)		
Fuji Inage	Aa	Вb	BCb	AB bc	ABc	Bc	Ac		
Katas Malas Quick	6.9 (0.8)	11.1 (0.8)	12.5 (1.7)	10.2 (1.7)	12.2 (1.7)	11.4 (1.2)	11.4 (1.4)		
Retac molar Quick	Da	Eb	D cd	BC bc	C bc	CD bc	AB bc		
Katas Silves	5.6 (0.6)	8.0 (1.7)	9.5 (1.1)	10.6 (0.6)	9.8 (1.1)	8.4 (0.9)	10.9 (2.4)		
Retac Silver	Ca	BCb	C cd	CD d	A cd	A bc	AB d		
Dive Destant	2.9 (0.5)	5.6 (0.9)	6.8 (1.0)	8.1 (0.9)	8.8 (0.9)	8.1 (0.9)	9.5 (0.1)		
Riva Protect	Aa	Ab	Ac	Ad	A de	Ad	Ae		
	6.5 (0.6)	9.3 (0.8)	11.5 (1.1)	12.2 (0.6)	11.4 (1.7)	11.9 (0.8)	12.2 (1.1)		
Riva Self Cure Fast	Da	Db	Dc	Ec	BCc	Dc	Bc		
	8.2 (0.9)	11.1 (0.9)	12.7 (1.1)	12.2 (1.5)	12.3 (1.2)	13.6 (0.8)	12.2 (1.5)		
Riva Self Cure HV	Da	Eb	D cd	E bc	C bc	Ed	B bc		
Di	5.5 (0.9)	9.9 (.7)	11.9 (1.0)	11.6 (1.0)	11.6 (1.2)	10.0 (0.7)	10.0 (0.5)		
Riva Silver	BC a	Db	Dc	DE c	BCc	Bb	Ab		

n=12; Capital letters annotate statistically similar groups per column, lower case letters annotate statistically similar groups per row (p = 0.05)

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	Conventional Glass Ionomers								
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months		
Chamfil	0.034 (0.02)	0.065 (0.02)	0.065 (.03)	0.078 (0.02)	0.072 (0.02)	0.075 (0.03)	0.054 (0.02)		
Chemin	BC a	C bc	C bc	Cc	C bc	C bc	C ab		
E.U.IV EVIDA	0.039 (0.02)	0.041 (0.02)	0.03 (0.01)	0.036 (0.01)	0.036 (0.02)	0.030 (0.01) A	0.042 (0.01)		
FUJI IX EXTRA	Ca	AB a	Aa	Aa	AB a	а	ABC a		
Full Trians	0.022 (0.02)	0.021 (0.01)	0.042 (0.01)	0.035 (0.01)	0.033 (0.02)	0.029 (0.02)	0.036 (0.01)		
Fuji Inage	ABC a	ABb	ABCc	A bc	A bc	A bc	ABc		
Katas Malas Outst	0.030 (0.01)	0.022 (0.01)	0.039 (0.02)	0.055 (0.02)	0.047 (0.02)	0.047 (0.03)	0.044 (0.01)		
Ketac Molar Quick	BC ab	Aa	AB abc	Bc	AB bc	AB bc	ABC bc		
Kata - Ciluar	0.01(0.001)	0.044 (0.01)	0.036 (0.02)	0.047 (0.005)	0.050 (0.01)	0.043 (0.01)	0.044 (0.01)		
Retac Silver	Aa	B bc	ABb	ABc	ABc	AB bc	ABC bc		
Dive Destant	0.019 (0.01)	0.032 (0.01)	0.046 (0.04)	0.039 (0.01)	0.034 (0.01)	0.038 (0.009)	0.036 (0.01)		
Riva Protect	AB a	ABb	ABC b	AB ab	AB ab	AB ab	AB ab		
Dive Calf Cure Fast	0.026 (0.01)	0.044 (0.01)	0.056 (0.01)	0.048 (0.01)	0.037 (0.01)	0.047 (0.02)	0.051 (0.01)		
Riva Self Cure Fast	ABC a	B bc	BC c	AB bc	AB ab	AB bc	BC bc		
Dive Calf Cure LD	0.024 (0.01)	0.030 (0.01)	0.059 (0.02)	0.052 (0.01)	0.048 (0.01)	0.03 (0.006)	0.033 (0.01)		
Riva Self Cure HV	ABC a	ABa	BCb	Bb	ABb	Aa	Aa		
Dius Siluss	0.012 (0.006)	0.044 (0.02)	0.054 (0.01)	0.050 (0.01)	0.053 (0.01)	0.055 (0.02)	0.054 (0.01)		
Riva Silver	A a	Bb	ABC c	ABc	Bc	Bc	Cc		

Table 6

Knoop Hardness									
Conventional Glass ionomers									
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months		
Chamfil	12.6 (3.0)	37.4 (5.4)	31.0 (5.3) A	39.6 (3.2)	43.5 (7.0)	43.7 (7.7)	41.4 (2.5)		
Chemin	Aa	Bc	b	A cd	Ad	AB d	A cd		
ENIL IN EXTRA	18.7 (5.0)	50.3 (5.7)	41.5 (5.4) B	43.7 (7.7)	52.2 (5.5) BCD	53.6 (5.3)	53.2 (4.9)		
FUJI IN EN IRA	AB a	Cc	b	AB bc	С	BCc	Cc		
Euli Triana	17.7 (3.3)	29.6 (8.1)	40.3 (3.5) B	47.4 (4.3)	52.4 (5.4) BCD	48.1 (6.7)	44.4 (2.2)		
Fuji Inage	Aa	Ab	С	ABC de	е	ABC de	AB cd		
Katao Malao Oulah	19.6 (6.2)	53.3 (1.8)	57.7 (11.7)	46.8 (11.6)	53.7 (5.2)	53.6 (8.5)	59.8 (2.4)		
Retac Molar Quick	AB a	C bc	Cbc	ABC b	CD bc	C bc	Dd		
Kata a Ciluar	18.2 (6.0)	33.8 (3.1)	30.4 (3.9) A	41.5 (8.0)	46.9 (4.3)	48.2 (5.0)	48.4 (4.7)		
Retac Silver	AB a	ABb	b	Ac	AB cd	ABC d	BC d		
Dive Destant	14.0 (3.3)	29.0 (2.9)	39.4 (2.4) B	42.8 (4.0)	42.1 (5.6)	44.9 (5.9)	41.2 (3.7)		
Riva Protect	Aa	Ab	c	Ac	Ac	ABC c	Ac		
Dive Ball Come Fast	25.5 (8.0)	52.6 (7.0)	61.4 (3.3) C	54.4 (5.3)	56.3 (5.2)	45.9 (9.4)	52.4 (5.1)		
Riva Self Cure Fast	Ba	C bc	d	C cd	D cd	ABC b	C bc		
Dia 0.40	25.5 (6.3)	52.1 (7.5)	46.2 (5.7) B	52.1 (4.5)	50.8 (4.1) BCD	47.1 (4.0)	48.5 (4.1)		
Riva Self Cure HV	Ba	Cb	b	BCb	b	ABC b	BC b		
Dive Cilver	18.6 (6.3)	46.7 (6.4)	40.4 (12.0)	42.5 (7.2)	47.8 (3.5) ABC	40.4 (2.1)	42.3 (1.4)		
Riva Silver	AB a	Cb	Bb	Ab	b	Ab	Ab		

n=12; Capital letters annotate statistically similar groups per column, lower case letters annotate statistically similar groups per row (p = 0.05)

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		F	racture To	ughness	A Constanting	NULLAWS	Star In Star
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months
	0.26 (0.02)	0.3 (0.03)	0.3 (0.04)	0.28 (0.05)	0.27 (0.02)	0.26 (0.03)	0.28 (0.03)
Chemfil	BC a	CD a	D a	DE a	Ba	Ba	CD a
	0.22 (0.04)	0.20 (0.04) A	0.22 (0.03)	0.2 (0.02)	0.2 (0.04)	0.22 (0.03)	0.22 (0.03)
Fuji IX EXTRA	Ва	а	Aa	Aa	Aa	Aa	Aa
	0.15 (0.02)	0.18 (0.02) A	0.21 (0.01)	0.22 (0.02)	0.21 (0.01)	0.22 (0.02)	0.23 (0.02)
Fuji Triage	Aa	b	Ac	AB cd	A cd	A cd	AB d
	0.26 (0.03)	0.27 (0.04)	0.26 (0.03)	0.24 (0.03)	0.21 (0.05)	0.22 (0.03)	0.26 (0.03)
Ketac Molar Quick	BCb	BCb	BCD b	C ab	Aa	Aa	ABC a
Kata Cilura	0.23 (0.04)	0.26 (0.02) B	0.27 (0.03)	0.29 (0.03)	0.28 (0.02)	0.27 (0.03)	0.31 (0.03)
Retac Silver	Ba	b	CD b	CD cd	BC bc	B bc	Dd
Dive Desta at	0.13 (0.04)	0.2 (0.02)	0.23 (0.02)	0.22 (0.02)	0.23 (0.03)	0.22 (0.01)	0.23 (0.02)
Riva Protect	Aa	Ab	ABc	AB bc	A bc	A bc	AB bc
Dive Calf Cure Fast	0.25 (0.07)	0.27 (0.03)	0.28 (0.02)	0.27 (0.02)	0.31 (0.02)	0.29 (0.02)	0.27 (0.08)
Riva Self Cure Fast	BC a	BC ab	CD ab	CD ab	СЬ	BC ab	BCD ab
Dive Salf Cure HV	0.28 (0.07)	0.3 (0.02)	0.27 (0.04)	0.3 (0.02)	0.3 (0.03)	0.31 (0.02)	0.28 (0.05)
Riva Self Cule HV	Ca	Da	CD a	Da	BCa	Ca	CD a
Pius Silus	0.21 (0.06)	0.28 (0.04)	0.26 (0.03)	0.26 (0.03)	0.27 (0.03)	0.35 (0.04)	0.32 (0.04)
Riva Sliver	Ва	BCD b	BC b	CD b	Bb	Dc	D bc

The mean physical property results for the RMGI restorative resins are displayed in the following Tables 8-12.

		Table	28			
	Fle	exural Stren	ngth (MPa)		AND READS	E.S. State
	Resi	n Modified G	lass lonomers			
1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months
52.08 (6.0)	76.2 (5.4)	72.5 (8.3)	76.7 (7.7)	63.2 (3.8)	59.3 (2.5)	61.5 (9.4)
Ba	Cc	B bc	Cc	Bb	Ab	Bb
32.0 (6.8)	49.3 (7.1)	61.3 (10.5)	48.5 (12.7)	49.2 (13.7)	64.7 (7.5)	46.3 (10.1)
Aa	Ab	Ac	Ab	Ab	Ac	Ab
29.1 (3.8)	55.5 (7.4)	59.2 (5.4)	60.3 (6.5)	60.8 (8.4)	64.4 (6.1)	62.4 (4.5)
Aa	Bb	A bc	B bc	B bc	Ac	Bc
27.8 (4.7)	53.1 (4.4)	59.6 (7.8)	62.8 (4.2)	54.4 (10.3)	64.5 (4.6)	59.3 (5.1)
Aa	ABb	A cd	Bd	AB bc	Ad	B bcd
	1 hour 52.08 (6.0) B a 32.0 (6.8) A a 29.1 (3.8) A a 27.8 (4.7) A a	Fie Resi 1 hour 24 hrs 52.08 (6.0) 76.2 (5.4) B a C c 32.0 (6.8) 49.3 (7.1) A a A b 29.1 (3.8) 55.5 (7.4) A a B b 27.8 (4.7) 53.1 (4.4) A a AB b	Table Flexural Strett Resin Modified G 1 hour 24 hrs 1 week 52.08 (6.0) 76.2 (5.4) 72.5 (8.3) B a C c B bc 32.0 (6.8) 49.3 (7.1) 61.3 (10.5) A a A b A c 29.1 (3.8) 55.5 (7.4) 59.2 (5.4) A a B b A bc 27.8 (4.7) 53.1 (4.4) 59.6 (7.8) A a AB b A cd	Table 8 Flexural Strength (MPa) Resin Modified Glass Ionomers 1 hour 24 hrs 1 week 1 month 52.08 (6.0) 76.2 (5.4) 72.5 (8.3) 76.7 (7.7) B a C c B bc C c 32.0 (6.8) 49.3 (7.1) 61.3 (10.5) 48.5 (12.7) A a A b A c A b 29.1 (3.8) 55.5 (7.4) 59.2 (5.4) 60.3 (6.5) A a B b A bc B bc 27.8 (4.7) 53.1 (4.4) 59.6 (7.8) 62.8 (4.2) A a AB b A cd B d	Table 8 Flexural Strength (MPa) Resin Modified Glass lonomers 1 hour 24 hrs 1 week 1 month 3 months 52.08 (6.0) 76.2 (5.4) 72.5 (8.3) 76.7 (7.7) 63.2 (3.8) B a C c B bc C c B b 32.0 (6.8) 49.3 (7.1) 61.3 (10.5) 48.5 (12.7) 49.2 (13.7) A a A b A c A b A b 29.1 (3.8) 55.5 (7.4) 59.2 (5.4) 60.3 (6.5) 60.8 (8.4) A a B b A bc B bc B bc 27.8 (4.7) 53.1 (4.4) 59.6 (7.8) 62.8 (4.2) 54.4 (10.3) A a A B b A cd B d A B bc A B bc	Table 8 Flexural Strength (MPa) Resin Modified Glass Ionomers 1 hour 24 hrs 1 week 1 month 3 months 6 months 1 hour 24 hrs 1 week 1 month 3 months 6 months 52.08 (6.0) 76.2 (5.4) 72.5 (8.3) 76.7 (7.7) 63.2 (3.8) 59.3 (2.5) B a C B b A b A b 32.0 (6.8) 49.3 (7.1) 61.3 (10.5) 48.5 (12.7) 49.2 (13.7) 64.7 (7.5) A a A b A b A c 29.1 (3.8) 55.5 (7.4) 59.2 (5.4) 60.3 (6.5) 60.8 (8.4) 64.4 (6.1) A a B b A c 27.8 (4.7) 53.1 (4.4) 59.6 (7.8)

n=12; Capital letters annotate statistically similar groups per column, lower case letters annotate statistically similar groups per row (p = 0.05)

			Table	9			
			Modulus	(GPa)			
		Resi	n Modified G	lass lonomers	3	Store and	
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months
	5.0 (0.5)	7.2 (0.9)	6.1 (0.3)	8.1 (0.8)	6.8 (0.6)	5.7 (0.5)	7.6 (0.8)
Fuji II LC	Ca	C cd	B bc	Ce	B cd	B ab	C de
	2.8 (0.3)	4.0 (0.1)	4.6 (0.4)	4.4 (0.4)	5.0 (0.5)	4.7 (0.2)	5.1 (0.4)
Ketac Nano	Aa	Ab	Ac	Ac	A de	A cd	Ae
	3.9 (0.1)	6.1 (0.5)	6.5 (0.3)	6.4 (0.6)	7.1 (0.7)	6.5 (0.6)	6.5 (0.9)
Riva LC	Ba	Bb	Bb	Bb	Bc	Cb	Bb
	4.0 (0.2)	6.9 (0.7)	9.0 (0.8)	8.5 (0.7)	8.9 (0.9)	9.7 (0.7)	10.4 (1.0)
Riva LC HV	Ва	Cb	C cd	Cc	C cd	D de	De

			Table	10			
		F	lexural To	ughness	ALC: NOT STREET	CHARTER AND	Stand days
		Resir	Modified G	lass lonomers			
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months
F-0.010	0.46 (0.2)	0.055 (0.07)	0.53 (0.1)	0.45 (0.1)	0.38 (0.05)	0.4 (0.04)	0.29 (0.1)
FUITILC	B ab	Cb	Bb	Bb	Ca	Ва	BC a
Kata a Maraa	0.24 (0.1)	0.37 (0.1)	0.53 (0.2)	0.39 (0.2)	0.28 (0.1)	0.54 (0.1)	0.25 (0.12)
Ketac Nano	Aa	B ab	Bc	AB ab	Ba	Cc	AB a
Di	0.15 (0.05)	0.31 (0.08)	0.30 (0.06)	0.33 (0.07)	0.29 (0.08)	0.38 (0.09)	0.36 (0.04)
Riva LC	Aa	ABb	Ab	AB bc	BCb	Bc	Cc
-	0.13 (0.05)	0.24 (0.06)	0.21 (0.05)	0.26 (0.04)	0.19 (0.07)	0.23 (0.04)	0.18 (0.04)
RIVA LC HV	Aa	A ab	A ab	Ab	Aa	A ab	Aa

n=12; Capital letters annotate statistically similar groups per column, lower case letters annotate statistically similar groups per row (p = 0.05)

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	A CONTRACTOR	- Isaaa ahaa	Knoop Har	aness			A. Conditional
		Res	sin Modified Gl	ass lonomers		Provide Providence	ALL PLANE IN
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months
	30.5 (3.5)	29.2 (2.6)	28.6 (2.3) A	27.7 (2.8)	26.6 (1.5)	25.1 (0.8)	24.4 (1.2)
Fuji II LC	Cd	B cd	cd	A bcd	A abc	A ab	Aa
Martin a Maria	26.2 (2.1)	26.3 (1.1)	27.7 (1.5) A	27.3 (5.0)	27.1 (1.2)	25.9 (2.1)	27.6 (1.4)
Ketac Nano	Ва	Aa	а	Aa	Aa	Aa	Ba
	23.1 (1.0)	26.1 (1.7)	27.0 (0.9) A	28.4 (2.1)	28.4 (2.1)	25.7 (2.0)	27.2 (2.2)
Riva LC	Aa	Ab	bc	Ac	Ac	Ab	B bc
	24.0(1.03)	32.1 (3.5)	C 33.2 (1.8) B	32.1 (1.2)	32.6 (2.3)	37.8 (2.8)	39.3 (2.1)
RIVA LC HV	AB a	b	b	Bb	Bb	Bc	Cc

n=12; Capital letters annotate statistically similar groups per column, lower case letters annotate statistically similar groups per row {p = 0.05}

			Table	12			
	No. 12 Contraction	F	racture To	ughness			
		Resir	Modified G	lass lonomers	and Republication	And the second second	
	1 hour	24 hrs	1 week	1 month	3 months	6 months	12 Months
	0.74 (0.1)	0.67 (0.05)	0.66 (0.04)	0.65 (0.04)	0.62 (0.04)	0.58 (0.04)	0.68 (0.04)
Fuji II LC	Cc	Bb	СЬ	Bb	B ab	Ca	Db
	0.59 (0.06)	0.51 (0.03) A	0.45 (0.1)	0.51 (0.07)	0.46 (0.07)	0.46 (0.06)	0.48 (0.03)
Ketac Nano	Bb	а	Aa	Aa	Aa	Aa	Ва
	0.47 (0.03)	0.51 (0.04) A	0.54 (0.03)	0.5 (0.06)	0.49 (0.09)	0.53 (0.02)	0.53 (.004)
Riva LC	Aa	ab	Bb	A ab	A ab	Bb	Cb
	0.43 (0.02)	0.49 (0.03)	0.48 (0.07)	0.47 (0.06)	0.43 (0.1)	0.47 (0.05)	0.42 (0.03)
RIVA LG HV	Aa	Aa	Aa	Aa	Aa	Aa	Aa

Discussion

In this evaluation nine GIC and four RMGI restorative products were evaluated over a period up to 12 months. This study not only included products that have had market tenure but also included newer materials, some of which have not been previously reported in the scientific literature. Only precapsulated materials were chosen for this study to eliminate variables that have been suggested by hand-mixed preparation of glass-ionomer materials.^{74,75}

This study attempted a different approach in physical property evaluation as compared to that observed by manufacturers as well as in other evaluations. One difference is that this study used 0.2M phosphate-buffered saline solution for storage, as this media represents a more physiologic as well as a basic storage media that can be thought of as slowly aggressive over time. Also, the physical properties were evaluated starting at one hour after specimen preparation, as the authors maintain that this would better represent when materials might be first subjected to oral forces. Each material was evaluated for flexure strength, flexural modulus, flexural toughness, Knoop hardness, and fracture toughness. Flexure testing encompasses both compressive and tensile components (Figure 10) and is considered a relatively important in vitro test that may possess clinical relevance. ⁷⁶



The graphical display of the flexural strength results for the GIC and RMGI materials over the 12 months of this evaluation are shown in Figures 11 and Figure 12, respectively.



The GIC materials exhibited inferior flexure strength values at one hour compared to that observed at 24 hours. At 24 hours, Chemfil Rock, Riva Silver, and Riva Self Cure fast demonstrated greater flexure strength than Ketac Molar Quick, Fuji Triage, and Riva Protect, with the other products being intermediary. Between 24 hours and one week physical property development ascribed to maturation of the silicate matrix seemingly contributed to significantly improve flexure strength for Fuji Triage, Ketac Molar Quick, Riva Self Cure Fast, Riva Self Cure HV, and Riva Silver. Interestingly, Fuji IX EXTRA and Chemfil Rock did not demonstrate further flexure strength improvement after 24 hours and while demonstrating some variation remained stable for the remained of the evaluation. After one week, there was no significant change in flexure strength with Fuji Triage, Ketac Molar Quick, Ketac Silver. Only Riva Protect demonstrated further significant flexure strength improvement that peaked at one month and remained stable thereafter.

The flexure strength results in this study were similar for that reported by Hu et al, ⁷⁷ but had higher values for Fuji IX Extra, Chemfil Rock, and Riva Self Cure than that from Zoergibel & Ilie, ⁷⁸ who used a

methodology with parameters different from this evaluation. Flexure strength values in this study were similar to that reported for Riva Self Cure and Fuji IX Extra, but lower for Ketac Molar Quick as that stated by Bonifácio et al. ⁷⁹ Values for Ketac Molar were similar as that reported by Yamazaki et al ⁸⁰ and results for Ketac Molar and Ketac Silver were almost identical to that reported by Xie et al. ⁸¹ A rather rare study reporting one hour glass-ionomer material physical properties by Lucksanasombool et al ⁸² reported flexural values that were higher than that found in this study, but those authors' method was different than the one used in this evaluation.



All RMGI products significantly increased in flexure strength between one and 24 hours, with Fuji II LC demonstrating significantly greater flexure strength than the other materials. After 24 hours, the RMGI products continued to demonstrate significant increases in flexure strength, with Ketac Nano displaying

its maxium flexure strength at 1 week but declining thereafter. Interestingly, although Fuji II LC demonstrated its highest flexure strength at one month, it then demonstrated a slow flexure strength decline for the remainder of the evaluation. Riva LC HV had significantly greater flexure strength at one and six months but declined at three months. Only Riva LC continued to develop flexure strength that peaked at 6 months and then remained stable until 12 months.

The flexure strength value trend noted in this evaluation is similar to that reported by Azillah et al ⁵⁷ who likewise reported a decline in Fuji II LC flexure strength after 100 days. The flexure strength values reported by those authors are somewhat comparable, but the results are not directly comparable as Azillah et al had a different methodology, using a four-point-bend testing apparatus. ⁵⁷ Furthermore, flexure strength values found during this evaluation were greater than reported by Xie et al ⁸³ and Weng et al, ⁸⁴ who used a higher loading rate than this evaluation. The RMGI flexure strength values in this study are similar to that reported by Yamazaki et al ⁸⁵ and nearly identical to that reported for Fuji II LC by Xie et al. ⁸¹

The GIC product mean modulus results are depicted in Figure 13.



Flexural modulus is in part a flexure strength function as modulus is determined from the linear portion of the flexure strength stress-strain curve. All GIC products had significant modulus increase between one and 24 hours, of which most products demonstrated a slow modulus increase up to one month, which lends support of a silicate matrix maturation process. ⁶⁰ The only exceptions to this were Ketac Molar Quick, Riva Self Cure Fast, Riva Self Cure HV, and Riva Silver, whose modulus was essentially complete and stable after one week, which agrees with an observation made by Wren et al. ⁵⁶ Riva Protect was the only GIC product that demonstrated modulus increase all throughout the 12-month evaluation.



Similar to the GIC products, RMGI materials demonstrated a significant modulus increase between one and 24 hours. However, any modulus results beyond 24 hours was material specific. For instance, Fuji II LC demonstrated its highest modulus values at one month, and although modulus declined at three and six months, displayed 12 month modulus values similar to that observed at one month. Ketac Nano demonstrated a slow modulus development that at 12 months was significantly greater than that observed at one month. Likewise, Riva LC HV likewise slowly increased in modulus, that was significantly greatest at six months and stable thereafter. Of the RMGI products only Riva LC largely maintained the modulus established at 24 hours throughout the remainder of the evaluation. RMGI modulus development may deserve additional thought. It is well established that the resin content in RMGI impedes the polyalkenoate reaction, as the resin presence renders the polyalkenoate acid less able to interact with the glass component due to distortion of the polyalkenoate acid polymer chain, as well as contain less water as compared to the GIC products. 38-40 In an infrared spectroscopy study involving Fuji II LC, Young 41 reported that the resin free radical polymerization reaction was essentially complete five minutes after VLC exposure. Thereafter, spectral changes associated with the polyalkenoate acid-base reaction occurred largely supported by water absorption. Although the initial RMGI polyalkenoate acid-base reaction product was approximately 17 times less than a conventional GIC, the slow progression of the polyalkenoate reaction due to water absorption reduced this difference by approximately 75 percent by four days. ⁴¹ Additionally, Wan et al ⁵⁴ with infrared spectroscopy also found evidence that the acid-base reaction continued for 96 hours for Fuji II LC. Therefore, any potential polyalkenoate reaction after resin component polymerization is dependent upon water absorption through the set resin matrix. It seems plausible to offer the explanation is that as water is absorbed over time, sufficient resin matrix plasticization may ensue to allow both to allow polyalkenoate components to interface and supply the needed water for a reduced acid-base reaction to occur.

Flexural toughness is a rarely-reported result in the dental scientific literature. Referring back to Figure 3, flexural toughness is defined as integrating the area under the total stress-strain curve. Hence, toughness can be thought to represent a material's ability to resist total catastrophic rupture, and may reflect the level of matrix organization. Toughness can be reasoned as both a function of a material's flexural strength and modulus, as both aspects affect the both the length and slope of the stress-strain curve and hence the area underneath. The toughness determinations for the GIC materials can be observed in Figure 15.



As flexural toughness is a function of flexure strength, it is logical to expect that the GIC materials had significant increase in toughness between one and 24 hours. Curiously, Chemfil Rock toughness at 24 hours was significantly greater than the other materials, which it maintained for the majority of the evaluation except at one week and 12 months. Fuji IX Extra, Ketac Silver, Riva Protect, Riva Self Cure Fast, and Riva Self Cure HV did not demonstrate flexural toughness increase beyond that observed at one week. Under the conditions of this study, flexure strength was found to influence flexural toughness more than modulus. Accordingly, as presented in Table 13, correlation analysis at 12 months found that the flexural toughness values were strongly correlated with the flexure strength of Chemfil Rock, Fuji IX EXTRA, and Riva SC HV, while Fuji Triage and Riva Self Cure Fast displayed a moderate correlation.

Material	Flexure Strength (r ² ; p value)	Flexural Modulus (r ² ; p value)	
Chemfil Rock	0.906; p < 0.001	0.042; p = 0.897	
Fuji IX EXTRA	0.829; p < 0.001	0.251; p = 0.251	
Fuji Triage	0.765; p = 0.001	0.198; p = 0.095	
Ketac Molar Quick	0.381; p = 0.408	0.351; p = 0.623	
Ketac Silver	0.204; p = 0.140	0.021; p = 0.948	
Riva Protect	0.234; p = 0.114	0.132; p = 0.245	
Riva SC Fast	0.657; p = 0.001	0.042; p = 0.897	
Riva SC HV	0.854; p < 0.001	0.049; p = 0.88	
Riva Silver	0.126; p = 0.696	0.362; p = 0.384	

Table 13. GIC Flexural Toughness Correlation with Flexural



The RMGI flexural toughness values for Riva LC essentially mirrored its flexural strength performance, largely demonstrating a significant flexural toughness increase throughout the evaluation. Ketac Nano and Riva LC HV also performed analogous to their flexure strength results as well, but the 12 month results were similar to that observed at one hour. Curiously, Fuji II LC exhibited a progressive decline in flexural toughness past 24 hours that resulted in its 12 month results being at levels below that observed at one hour.

As exhibited with some GIC materials, both Fuji II LC and Ketac Nano flexure strength was found to exhibit a strong correlation with flexural toughness (Table 14).

able 14. RMGI Fle Streng	xural Toughness Corr th and Modulus (Spea	elation with Flexur arman's)
Material	Flexure Strength (r ² ; p value)	Flexural Modulus (r ² ; p value)
Fuji II LC	0.839; p < 0.001	0.028; p = 0.931
Ketac Nano	0.848; p < 0.001	0.323; p = 0.054
Riva LC	0.175; p = 0586	0.044; p = 0.891
Riva LC HV	0.713; p = 0.009	0.028; p = 0.931

Review of the literature reveals little research reporting GIC and RMGI flexural toughness results, as this study may be the first to report 12-month results for these materials.

Surface hardness is the resistance of a material's localized surface to an indentation force, and results in surface deformation of which the magnitude is largely related to physical properties of the underlying material matrix. ⁵¹ To minimize any RMGI potential surface microhardness irregularities due to the polywave LED light curing unit used ⁸⁶ 11 Knoop hardness indentations spaced 0.5 millimeter apart in alternating fashion apart were made and read on each specimen. The mean of the 11 hardness values

was then recorded as that specimen's representative KHN value. This was accomplished for a total of ten specimens. The 12-month results of the hardness testing are shown in Figures, 17 and 18.



GIC materials demonstrated a significant increase in Knoop hardness between one and 24 hours with Fuji IX EXTRA, Ketac Molar Quick, Riva Self Cure Fast, HV and Silver demonstrating significantly greater hardness than the other materials. After 24 hours, hardness continued to significantly increase for Chemfil Rock, Fuji Triage, Ketac Molar Quick and Ketac Silver. In contrast, Fuji IX EXTRA, Riva Protect, Riva Self Cure Fast, Riva Self Cure HV, and Riva Silver all essentially demonstrated no change in hardness beyond 24 hours. At 12 months, Ketac Molar Quick hardness was significantly greater than any of the other materials, with Fuji IX EXTRA, Ketac Silver, Riva Self Cure Fast, and Riva Self Cure HV all being similar and greater than the remaining materials. The hardness results found during this study were lower for Chemfil Rock than that reported by Al-Angari et al ⁸⁷ but comparable for Fuji IX EXTRA and Ketac Molar Quick. Riva SC demonstrated same behavior as that Shiozawa et al ⁵⁵ who likewise reported a significant KHN increase after 24 hours to one week. Lastly, Ketac Silver hardness values were very similar to that reported by De Moor and Verbeeck. 88

As discussed earlier, hardness values reflects resistance of the underlying material matrix to deformation. ⁵¹ Continued increases in hardness for Chemfil Rock, Fuji Triage, Ketac Molar Quick and Ketac Silver indicates increases in matrix physical properties, thus further indicating a maturation process of the silicate matrix.



With the RMGI materials only Riva LC HV manifested a slow but significant increase in Knoop hardness over the 12 month evaluation. Riva LC hardness values established at 24 hours did not change for the duration of the evaluation. Ketac Nano did not exhibit any hardness increase beyond that observed at one hour, but Fuji II LC mysteriously demonstrated its highest hardness values at one hour after which began a steady decline resulting in a significantly less hardness at 12 months. Riva LC HV portrayed the most interesting result in after initial stability between 24 hours and three months demonstrated a deferred significant hardness increase at 6 months that was maintained at 12 months. As previously discussed, some RMGI products have been reported to exhibit a delayed acid-base that may slowly progress up to 96 hours after preparation. ^{54,81} Although this reported delay may provide some rationale for hardness improvement between 24 hours and one week, however no RMGI materials demonstrated a Knoop hardness increase for this time period. Realistically, any improvement would be due to the continued maturation of the silicate matrix, but explanation for the late Riva LC HV hardness increase is presently unclear. The only compositional difference between Riva LC HV and Riva LC is that Riva LC HV contains a higher powder/liquid ratio and may contain a slightly smaller amount of HEMA.

Fracture toughness has been described by Ferracane as an in vitro test that may correlate with clinical performance, as fracture toughness testing determines a material's ability to resist crack propagation from a pre-existing flaw. ⁸⁹ To wit, Tyas reported a correlation between fracture toughness and the clinical performance of resin composites used in Class IV preparations, ⁹⁰ while Ferracane et al demonstrated a correlation between marginal deterioration and fracture toughness in a clinical study involving resin composites placed in denture teeth. Fracture toughness during this evaluation were testing using the single edge notched beam method in accordance with ASTM Standard E399 (Figure 19).

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The results of the fracture toughness testing for the GIC and RMGI products are shown in Figures 20 and

21, respectively.



For the GIC materials, fracture toughness peformance was material specific. Only Fuji Triage, Ketac Silver, Riva Protect, and Riva Silver displayed any significant increase in fracture toughness over the evaluation. Neither Chemfil, Fuji IX EXTRA, Ketac Molar Quick, Riva Self Cure Fast, nor Riva Self Cure HV demonstrated any significant fracture toughness increase over the 12 month evaluation period. However, the fracture toughness testing resulted in very low variability, so sensibly any significant changes are unlikely to be of clinical significance, and essentially GIC fracture toughness values changed very little during this evaluation. The reason for this these results are somewhat perplexing, as the flexure toughness results did not follow the same trend evidenced by the other GIC physical property results. As fracture toughness is a material's ability to prevent crack propagation through its matrix, fracture toughness could be thought as the material's microstructure organization and internal ability to blunt internal defect continuation. 92 It is interesting that the two cermets evaluated in this study demonstrated higher fracture toughness values than the other GIC products. It could be construed perhaps the cermet microstructure involving intefaces of the included metals may afford some crack deflection ability. Nonetheless this rationale is presently conjecture and cannot be confirmed under the conditions of this study. The results of this study can serve to reinforce a recent report by Baig et al 93 that after evaluating different GIC powder/liquid ratio has questioned fracture toughness testing's discriminatory value in physical property evaluation. Under the conditions of this study, the fracture toughness results for Ketac Molar Quick were similar to that values reported by Yamazaki et al. 85 Contrastingly, this study's results were lower than that reported for Chemfil Rock , Fuji IX EXTRA, and Ketac Molar Quick Al-Angari and colleagues, 87 lower for Ketac Silver and Ketac Molar Quick reported by Ilie et al, 94 as well as the one-hour results reported for Fuji IX reported by Lucksanasombool et al. ⁸² All of these studies used different methodologies than the present study that could account for these differences.



With the RMGI materials, it was puzzling to to observe that both Fuji II LC and Ketac Nano displayed significant decreases in fracture toughness at one hour after preparation that continued for one week with Ketac Nano but persisted for Fuji II LC up to six months. The reason for this surprising fracture toughness deminuation is presently unknown, but at first though could possibly be due to irregularities between the resin and conventional polyalkenoate domains and/or matrix plasticization due to water absroption tendency that has been reported for RMGI materials. ⁹⁵ Hoewever, tt may not be plausible to

account for this difference to water absorption alone, especially for the rapid decrease between one and 24 hours. Riva LC HV did not experience any fracture toughness changes from that observed at one hour for the evaluation duration. Riva LC was the only RMGI material to demonstrate significantly greater fracture toughness, but again the variance was small and any statistically significant changes are unlikely to be of clinical relevance. RMGI frature toughness reports are sparsely reported in the dental literature. Findings for Fuji II LC and Ketac Molar were similar to that reported by Yamazaki et al, ⁸⁵ whereas this study's findings were lower to values for Fuji II LC, Ketac Molar, and Ketac Silver as reported by Ilie and colleagues. ⁹⁴

Upon review of the physical properties displayed in the study, it should be readily observed that the GIC restorative materials exhibited more physical property stability than some of the RMGI products. Although the means of the RMGI physical properties when globally considered below may represent a somewhat stable material, individual materials at each observation time demonstrated significant declining physical property changes of flexural toughness and modulus, fracture toughness, and hardness. Some of these physical property variations have been discussed as being a result of the hydrophilic nature of the contained RMGI resins and the resultant water absorption process. ⁹⁵⁻⁹⁷ Accordingly, Kanachanavasita et al ⁹⁵ reported that RMGI products absorbed water twice to that observed with GIC materials and storage in artificial saliva caused exponential absorption increase after 30 days which was thought to be due to RMGI matrix changes. ⁹⁵ Small and colleagues ⁹⁶ observed that RMGI materials showed dramatic increase in water absorption as compared to resin composites while Versluis et al ⁹⁷ reported that the water absorption characteristics of some RMGI products produce an overall expansion of the matrix. Hence, the plasticization of the matrix along with structural changes may be thought as one of the main causes of RMGI loss of physical properties over time.

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Global comparison of the GIC and RMGI materials was undertaken to show material property comparisons between the two classes of products as well as identify general physical property trends. The mean results of all of the GIC and RMGI individual physical properties were calculated. Understandably, due to the marked disparity between the samples sizes as well as the different nature between the two materials, the authors chose not to pursue statistical analysis. The global comparison in flexure strength is shown in Figure 22.



The RMG restorative materials demonstrated higher flexure strength values than the GIC products. It could be reasonable to assume based on this singular comparison that a RMGI product might be the choice material for restorations exposed to functional forces. Realistically, as discussed earlier GIC performance in functional areas in the ART technique can serve their suitability for functional areas as well. ⁶¹⁻⁶⁷ The GIC products overall demonstrated an increasing flexure strength trend up to one month,

whereas the RMGI products were noticed to display a decreasing flexure strength trend between one week and three months.



In contrast to flexure strength, GIC products were observed to display higher modulus values than the RMGI products. Furthermore, the GIC products displayed an increasing modulus trend more so than the RMGI materials, which further supports a silicate network maturation. ^{9,10} Additionally noteworthy, both material types demonstrated a decrease in modulus at six months, which can be conjectured as some change in the polyalkenoate network within both materials. Yamazaki and colleagues ⁸⁵ reports that RMGI products could present lower modulus due to the viscoelastic behavior of the included resin matrix network.

When flexural toughness global results are reviewed (Figure 24) it can be seen that GIC



products are observed to have a lower flexural toughness which illustrates the brittle nature of these materials. Furthermore, GIC products as a whole displayed little change in toughness properties past one week. The declining values for the RMGI products between one week and three months could be thought due to water absorption matrix disruption, however such does not provide explanation of the RMGI toughness increase at 6 months.

GIC products displayed higher Knoop hardness values as compared to the RMGI materials, as seen in Figure 25.



GIC products also revealed a slight increasing trend up to three months, which again can be thought to reinforce concepts of a the maturing silicate matrix. The lower values for RMGI values may be thought to be due to the viscoelastic behavior afforded by the resin content, which produces substantial material creep under load with resultant stress relief and recovery.⁸⁵ The RMGI products appear to demonstrate stability after one week and did not demonstrate a hardness decline after six months observed by Shiozawa et al.⁵⁵

As can be seen in Figure 26, RMGI products as a group demonstrated higher fracture toughness values than the GIC restorative materials. Again this difference demonstrates the brittle nature of the GIC materials that affords little internal means for crack deflection, whereas crack propagation through the viscoelastic and different internal RMGI phases will require more energy for crack propagation.



This global comparison depicts that RMGI products demonstrate higher flexure strength, flexural toughness, and fracture toughness than the GIC counterparts. Hence, some may reach conclusions that RMGI products should afford superior performance in functional areas. Realistically, clinicians should again not disregard the evidence of GIC material success and survival in the ART technique, made even so more relevant when one considers the austere conditions that these GIC materials are placed. ⁶¹⁻⁶⁶ Furthermore, clinical studies have recently been reported in which a GIC materials has performed well in an adult functional environments. ⁹⁸

Conclusions

This study evaluated the physical properties of nine GIC and four RMGI restorative materials over 12 months. Under the conditions of this study:

1. GIC and RMGI physical property performance over time was material dependent.

 Although differences in GIC physical properties were noted, the divergences were of such small magnitude that it is unlikely that it would be of clinical significance.

 Modest increases in some GIC physical properties were noted in time, especially flexural modulus and hardness, which lends support to reports of a maturing silicate matrix.

4. Overall, GIC product physical properties were more stable than RMGI, which could lend support to reports of RMGI matrix degradation due to imbibition of water.

5. Globally, RMGI products demonstrated higher values of flexure strength, flexural toughness, and fracture toughness.

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