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Effect of Resin-modified Glass Ionomer Cement Dispensing/Mixing Methods on Mechanical Properties

TA Sulaiman • AA Abdulmajeed • A Altitinchi • SN Ahmed • TE Donovan

Clinical Relevance

Automix resin-modified glass ionomer cements (RMGIs) may be easier and more efficient than a hand mix version of the same RMGI, but they may not have the same favorable mechanical properties, which could potentially affect the longevity of the cement.

SUMMARY

Statement of the Problem: Resin-modified glass ionomer cements (RMGIs) are often used for luting indirect restorations. Hand-mixing traditional cements demands significant time and may be technique sensitive. Efforts have been made by manufacturers to introduce the same cement using different dispensing/mixing methods. It is not known what effects these changes may have on the mechanical properties of the dental cement.

Purpose: The purpose of this study was to evaluate the mechanical properties (diametral tensile strength [DTS], compressive strength [CS], and fracture toughness [FT]) of RMGIs with different dispensing/mixing systems.

Methods and Materials: The RMGI specimens (n=14)—RelyX Luting (hand mix), RelyX Luting Plus (clicker–hand mix), RelyX Luting Plus (automix) (3M ESPE), GC Fuji PLUS (capsule-automix), and GC FujiCEM 2 (automix) (GC)—were prepared for each mechanical test and examined after thermocycling (n=7/subgroup) for 20,000 cycles to the following: DTS, CS (ISO 9917-1) and FT (ISO standard 6872; Single-edge V-notched beam method). Specimens were mounted and loaded with a universal testing machine until failure occurred. Two-/one-way analysis of variance followed by Tukey honestly significantly different post hoc test was used to analyze data for statistical significance ($p<0.05$).

Results: The interaction effect of both dispensing/mixing method and thermocycling was significant only for the CS test of the GC group ($p<0.05$). The different dispensing/mixing methods had no effect on the DTS of the tested cements. The CS of GC Fuji PLUS was significantly higher than that of the automix version

*Taiseer A Sulaiman (Taiseer), BDS, PhD, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Awab A Abdulmajeed, DDS, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Ali Altitinchi, BDS, MSc, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Sumitha N Ahmed, DDS, MS, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

Terrence E Donovan, DDS, University of North Carolina, Department of Operative Dentistry, Chapel Hill, NC, USA

*Corresponding author: 5405H Koury Oral Health Science Building, Chapel Hill, NC 27599, USA; e-mail: sulaiman@unc.edu

DOI: 10.2341/17-166-L

($p < 0.05$). The FT decreased significantly when switching from RelyX (hand mix) to RelyX Luting Plus (clicker–hand mix) and to RelyX Luting Plus (automix) ($p < 0.05$). Except in the case of the DTS of the GC group and the CS of GC Fuji PLUS, thermocycling had a significant effect reducing the mechanical properties of the RMGI cements ($p < 0.05$).

Conclusions: Introducing alternative dispensing/mixing methods for mixing RMGIs to reduce time and technique sensitivity may affect mechanical properties and is brand dependent.

INTRODUCTION

Dental cements have been used to retain indirect restorations. The success and longevity of these restorations are highly dependent on the durability of the cements. Zinc phosphate cement was the traditional cement in use since 1879.[1] Zinc phosphate cement was provided as a powder and liquid that required a special mixing technique to achieve optimum success.[2] Over the past 40 years, other cements have been introduced with variable degrees of success. These cements are polycarboxylate cement, glass ionomer and resin-modified glass ionomer cements (RMGIs), and, finally, resin-based cement in its self-adhesive or adhesive versions.[3-5]

Extensive research has been conducted over the years, and most of the newer versions of these cements lack proper clinical evaluation. *In vitro* tests provide useful information concerning the material itself; however, caution should be taken before concluding any clinical significance from these studies, as many of these tests do not relate directly to the potential clinical performance of the cement. In regard to the retentive function of cements, it has been concluded[6,7] that several variables interact with each other, such as the resistance and retention form of the preparation, surface characteristics of the tooth and the intaglio surface of the restoration, and, finally, variables pertaining to the cement itself.

Dental cements are provided with different dispensing/mixing methods. Some are hand mixed from a powder-liquid formulation, while others are supplied as a paste-paste consistency that is either hand mixed (in a "clicker" device) or equipped with an automixing tip that dispenses and mixes the cement. Clinicians likely assume that cements of the same brand with two different forms of mixing method have the same properties. However, for the cements to be used with different dispensing/mixing devices, the basic formulations must be modified physically and chemically. These modifications may affect the resultant mechanical properties of the cements.

Johnson and others[8] made the remarkable discovery that the powder-liquid version of some RMGI cements was more retentive than the paste-paste counterpart. Retention ability of the cements was the primary focus in their study. No evaluation of specific mechanical properties was done in that study. It is therefore important to further investigate the possible consequences of different dispensing/mixing methods on the diametral tensile strength (DTS), compressive strength (CS), and fracture toughness of RMGI cements. The research hypothesis was that changing the dispensing method of the cement has no effect on its mechanical properties.

METHODS AND MATERIALS

The RMGI cements used for this study and their dispensing methods are listed in Table 1. With regard to the DTS, 14 cylindrical-shaped specimens (6.0±0.1 mm in diameter and 3.0±0.1 mm in height) of each RMGI were prepared at room temperature (23°C±1°C), according to specification No. 27 of the American National Standards Institute/American Dental Association,[9] slightly overfilling the stainless-steel molds that rested on a glass slide with the mixed cement; two transparent ethylene films were used to confine the cement on either side of the mold to minimize oxygen exposure from the atmosphere. A second glass slide was placed on top of the mold and pressed by slight finger pressure to allow extrusion of excess cement. After each RMGI cement setting time, the specimens were then carefully removed from the molds; any specimen with an obvious void was excluded from testing, and a new specimen was made. A No. 15 blade was used to remove excess cement from the edges of the specimen; an 800-grit silicon-carbide abrasive paper (MicroCut™, Buehler, Lake Bluff, IL, USA) was used for final smoothing. The final dimensions of each specimen were calculated by taking the mean of two measurements at right angles to each other, made to an accuracy of 0.01 mm using a digital micrometer (Digimatic Micrometer, Mitutoyo Corporation, Aurora, IL, USA). Specimens were then stored in water at 37°C for 24 hours to achieve maximum maturity and were then mounted diametrically, and the DTS was calculated for the nonthermocycled specimens (n=7) and after thermocycling (n=7) (20,000 cycles at 5°C-55°C and a 15-second dwell time; TC-8, SD Mechatronik,

Table 1: *Resin-modified Glass Ionomer Cements Used for the Study and Their Dispensing/Mixing Method*

Resin-modified Glass Ionomer	Manufacturer	Dispensing Method	Composition	Lot No.
RelyX Luting	3M ESPE	Powder:liquid Hand mix	Powder: fluoroaluminosilicate glass, potassium persulfate, ascorbic acid, opacifying agent	N768842
			Liquid: methacrylated polycarboxylic acid, water, HEMA, tartaric acid	
RelyX Luting Plus	3M ESPE	Paste:paste clicker–Hand mix	Paste A: fluoroaluminosilicate glass, proprietary reducing agent, HEMA, water, opacifying agent	N748797
			Paste B: methacrylated polycarboxylic acid, Bis-GMA, HEMA, water, potassium persulfate, zirconia silica filler	
RelyX Luting Plus Automix	3M ESPE	Paste:paste automix	Paste A: radiopaque fluoroaluminosilicate glass, opacifying agent, HEMA, water, dispersion aid, reducing agent	N769817
			Paste B: nonreactive zirconia silica filler, methacrylated polycarboxylic acid, HEMA, resin monomers, water, potassium persulfate, photoinitiator	
GC Fuji PLUS	GC Corporation	Powder:liquid Capsule-automix	Powder: fluoroaluminosilicate glass, initiator, pigment	1602101
			Liquid: 4-META, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer 65%-70 %wt	
GC FujiCEM 2	GC Corporation	Paste:paste automix	Paste A: fluoroaluminosilicate glass, initiator, UDMA, dimethacrylate, pigment, silicon dioxide, inhibitor	1602082
			Paste B: silicon dioxide, UDMA, dimethacrylate, initiator, inhibitor	

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacryalate; 4-META, 4-methacryloxyethyl trimellitate anhydride; HEMA, hydroxyethyl methacrylate; UDMA, urethane dimethacrylate.

Feldkirchen-Westerham, Germany). The maximum force (N) at failure was recorded using a universal testing machine (Instron 4411, Instron, Norwood, MA, USA) at a cross-head speed of 1.0 mm/min, according to the following formula:

$$DTS = 2F/\pi dt,$$

where F indicates maximum force applied in newtons; d indicates the diameter of the specimen; and t indicates the thickness of the specimen.

For the compressive strength test, 14 cylindrical-shaped specimens (4.0±0.1 mm in diameter and 6.0±0.1 mm in height) (ISO standard 9917-1)[10] for each RMGI were prepared at room temperature (23°C±1°C) and mixed according to the manufacturers' instructions, and specimen preparation was handled similarly to that of the DTS test previously explained.

Specimens were then stored in water at 37°C for 24 hours. CS was calculated for half of the specimens before thermocycling, and the other half were thermocycled for 20,000 cycles at 5°C-55°C with a 15-second dwell time prior to CS calculation. Measurement of the force (N) at which failure occurred was recorded using a universal testing machine at a cross-head speed of 1.0 mm/min. CS was calculated according to the following formula:

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

where P indicates maximum force applied (in newtons) and d indicates the measured diameter of the specimen (in millimeters).

Concerning the FT test, the single edge V-notched beam method, according to ISO standard 6872, was used to measure the FT.[11] Beam-shaped specimens (n=14/group) measuring 21.0 ± 0.1 mm in length and with a rectangular cross section of 4.0 ± 0.2 mm in thickness and 3.0 ± 0.2 mm in depth, as seen in Figure 1A and B, were prepared for each RMGI cement at room temperature (23°C±1°C) and mixed according to the manufacturer's instructions. A notch depth of approximately 0.5 mm was machined into the bar specimen using a 300-μm-thick diamond blade. Diamond polishing paste (3.5 μm, Kent Supplies, Quebec, Canada) was placed into the notch tip, and a razor blade was inserted into the notch along with light force (5-10 N) that was applied using gentle back-and-forth motion and as straight as possible.

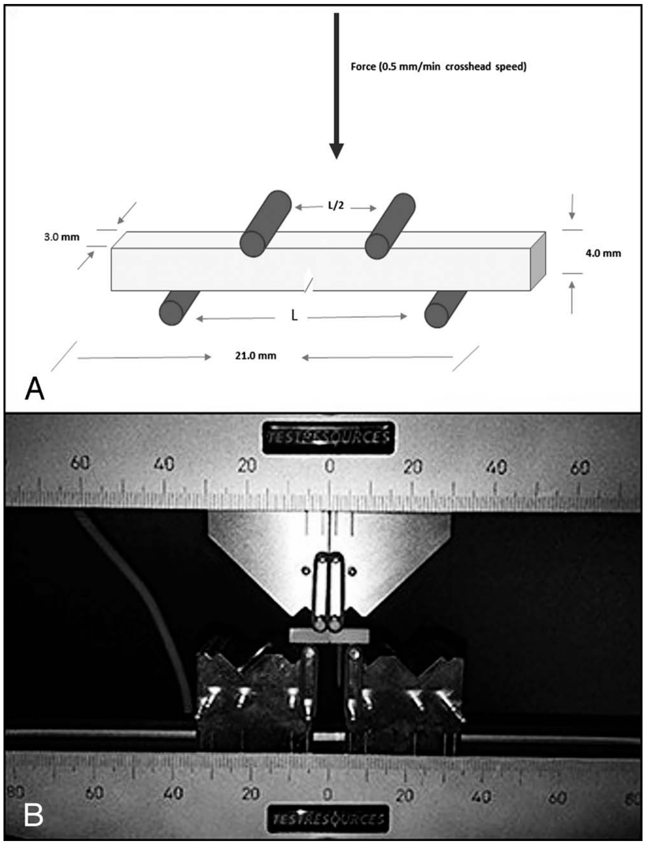

Figure 1. *(A) Schematic illustration of fracture toughness specimen. L is the distance between the rollers in the four-point bending fixture. (B) Specimen placed on four-point bending fixture.*

A calibrated light microscope was used to examine both ends of the V-notch and to measure the depths of the V-notches with magnification power of >50× to three significant figures. The width (b) and thickness (w) of each specimen were recorded using a digital micrometer capable of measurements to ±1-μm accuracy (QuantuMike Micrometer, Mitutoyo Corporation). The specimens were then cleaned with acetone in an ultrasonic bath and were then dried by heating to 110°C for 10 minutes. The drying process time was modified from the original ISO standard 6872 of one hour intended for ceramic materials to avoid damage to the RMGI specimens. In addition to drying the specimens after ultrasonic cleaning, this heating process likely aided the polymerization reaction in reaching its maximum potential. Specimens were then immersed in water at 37°C for 24 hours prior to testing. The FT test was carried out before (n=7) and after thermocycling for 20,000 cycles at 5°C-55°C with a 15-second dwell time (n=7), using a four-point bending fixture. The V-notch was placed facing down (tensile side). The specimens were loaded on a universal testing machine with a crosshead speed of 0.5 mm/min. FT K_{Ic} (MPa×m$^{1/2}$) was calculated using the following equation:

$$K_{Ic} = \frac{F}{b\sqrt{w}} \cdot \frac{S1 - S2}{w} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} \cdot Y$$

$$Y = 1.9887 - 1.326\alpha - \frac{(3.49 - .68\alpha + 1.35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2}$$

where K_{Ic} = fracture toughness; α = relative notch depth (m); Y = stress intensity shape factor; F = fracture load (MN); b = specimen width (m); w = specimen thickness (m); $S1$ = support span (m); and $S2$ = inner four-point span (m).

Two- and one-way analyses of variance (ANOVAs) were conducted for each RMGI cement group with the main variables of "hand mix" and "automix," depending on the method of dispensing. The post hoc multiple-comparison Tukey test was used for pair-wise comparisons between the group means. A significance level of $\alpha = 0.05$ was used for all tests. All statistical analysis was performed with Statistical Package for Social Sciences (SPSS version 24, SPSS Inc, Armonk, NY, USA).

RESULTS

The combined effect of both independent parameters (dispensing/mixing method and thermocycling) was significant only for the CS of the GC group ($p<0.05$) according to two-way ANOVA.

According to one-way ANOVA, when comparing the effect of different dispensing/mixing methods on the mechanical properties tested (Table 2), there were no statistically significant differences in DTS within the RelyX and GC groups. The mean CS of GC Fuji Plus (capsule-automix) was significantly greater than that of GC FujiCem 2 (automix) ($p<0.05$). The mean FT of RelyX Luting (hand mix, 0.50 MPa×m$^{1/2}$) was significantly greater than that of RelyX Luting Plus (clicker–hand mix, 0.36 MPa×m$^{1/2}$), which was also significantly greater than that of RelyX Luting Plus (automix, 0.24 MPa×m$^{1/2}$). There was no significant difference in FT between GC Fuji Plus (capsule-automix) and GC FujiCem 2 (automix). The percentage of change in mechanical properties according to the dispensing/mixing method can be seen in Table 3.

When determining the effect of the thermocycling on mechanical properties (Table 2), there was a significant reduction DTS of the RelyX cements after

Table 2: *Mean and Standard Deviation (SD) of Mechanical Properties of Resin-modified Glass Ionomer (RMGI) Cements Before and After Thermocycling (TC)[a]*

Resin-based Cements		Diametral Tensile Strength, MPa (Mean±SD)	Compressive Strength, MPa (Mean±SD)	Fracture Toughness, MPa × $m^{1/2}$ (Mean±SD)
RelyX Luting (hand mix)	Before TC	20.93 ± 2.99*	104.24 ± 12.98*	0.80 ± 0.12*
	After TC	13.51 ± 1.91	65.07 ± 5.49	0.50 ± 0.06A
RelyX Luting Plus (clicker–hand mix)	Before TC	21.85 ± 4.17*	108.14 ± 6.27*	0.67 ± 0.11*
	After TC	11.79 ± 3.24	61.68 ± 5.11	0.36 ± 0.04B
RelyX Luting Plus (automix)	Before TC	26.44 ± 5.45*	114.37 ± 6.16*	0.55 ± 0.02*
	After TC	18.55 ± 9.84	68.24 ± 8.86	0.24 ± 0.01C
GC Fuji CEM Plus (capsule-automix)	Before TC	18.08 ± 4.10	91.81 ± 10.63	0.63 ± 0.07*
	After TC	12.61 ± 2.97	81.08 ± 7.75A	0.29 ± 0.05
GC FujiCEM 2 (automix)	Before TC	14.91 ± 3.46	85.22 ± 11.04*	0.50 ± 0.09*
	After TC	14.55 ± 3.11	46.86 ± 4.01B	0.28 ± 0.07

[a] ** Indicates statistical difference* $p < 0.05$ *between the same RMGI cement brand before and after TC. Different letters indicate statistical difference within the same cement group after TC;* $p < 0.05$.

thermocycling ($p<0.05$), with no significant effect on the DTS of the GC cements. The CS of all RMGI cements reduced significantly ($p<0.05$) after thermocycling, except in the case of GC Fuji Plus. FT values decreased significantly for all RMGI cements after thermocycling ($p<0.05$).

DISCUSSION

Based on the results of this *in vitro* study, it is evident that changing the dispensing/mixing method of the cement may translate into unfavorable effects on its mechanical properties. However, as in many other *in vitro* studies, caution should be taken before concluding any clinical relevance related to these changes. How this change in mechanical properties can affect the clinical longevity of the retention ability of RMGI cements is yet to be determined.

The research hypothesis for this study was that there was no effect of changing the dispensing/mixing method of the RMGI on the resultant mechanical properties. This hypothesis was rejected. Changing the dispensing method altered at least one or more of the mechanical properties of each cement significantly. It is not exactly clear what modifications to the composition of the RMGI cements were made by the manufacturers to accommodate for the change in dispensing/mixing method. Further research should be done to examine in detail the changes in the cements' composition and their correlation to the changes in mechanical properties.

In the study of Johnson and others,[8] the automix (paste:paste) RMGI cement was less retentive than the hand-mix (powder:liquid) counterpart. They acknowledged the fact that the two versions of the same cement are not the same and that the manufacturer modified the chemical composition of the cements. They speculate that the manufacturer may have formulated a new aqueous base paste to allow the automix reaction. Furthermore, the traditional chemical initiators are sensitive to water, and in order to control setting times and stability of the automix version of the cement, new initiators were

Table 3: *Percentage Change in Mechanical Properties When Changing from Hand Mix Dispensing/Mixing Method of Each Resin-modified Glass Ionomer Cement Type*

	Diametral Tensile Strength, MPa	Compressive Strength, MPa	Fracture Toughness, MPa × $m^{1/2}$
RelyX Luting (hand mix) / RelyX Luting Plus (clicker)	↓ 12.7%	↓ 5.2%	↓ 28%
RelyX Luting (hand mix) / RelyX Luting Plus (automix)	↑ 37.3%	↑ 4.8%	↓ 52%
RelyX Luting Plus (clicker) / RelyX Luting Plus (automix)	↑ 57.3%	↑ 10.6%	↓ 33.3%
GC Fuji PLUS (capsule- automix) / GC FujiCEM 2 (automix)	↑ 15.3%	↓ 42.2%	↓ 3.4%

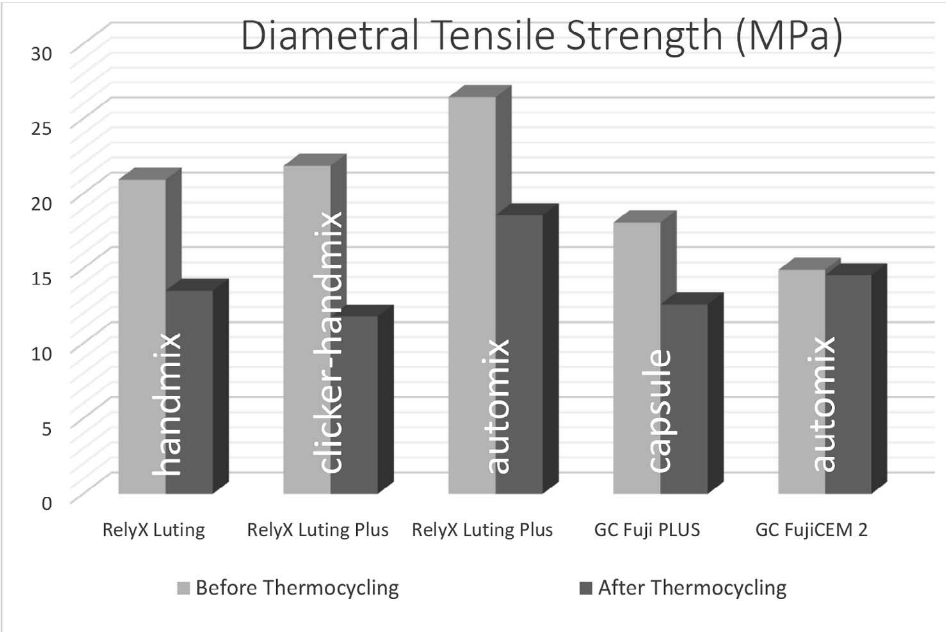

Figure 2. *Mean diametral tensile strength of resin-modified glass ionomer cements before and after thermocycling.*

developed. Therefore, this study investigated the changes in mechanical properties that resulted between the different versions of the same cement. This information may be important for the profession, as most clinicians likely assume equivalence in the performance of the same cement brand with different dispensing/mixing methods.

Based on the results of this study, there was no significant effect in DTS values of the RMGI cements with different dispensing methods (Figure 2), despite the 37.3% increase in DTS value from the hand-mix (powder:liquid) to the automix (paste:paste) version of the RelyX RMGI cements. The CS values showed a significant effect with one type of RMGI cement (GC Fuji PLUS), with a 42.2% drop in its CS value noted when changing to the automix version (Figure 3). The FT value of the RelyX Luting (hand mix) dropped around 28% compared to the (clicker–hand mix) version and further dropped to 52% when changing to the automix version (Figure 4). Clearly, there is no equivalence in mechanical properties for

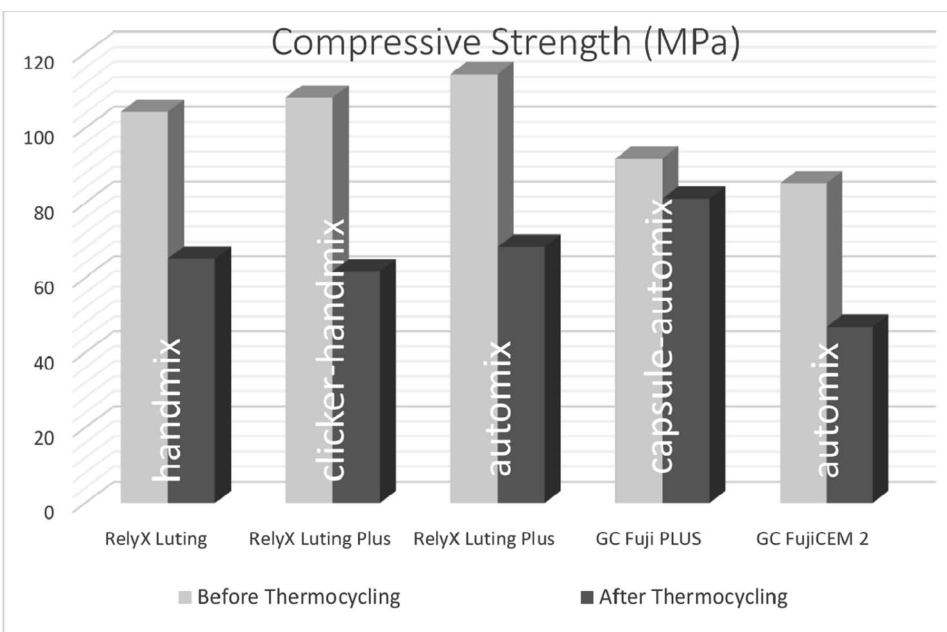

Figure 3. *Mean compressive strength of resin-modified glass ionomer cements before and after thermocycling.*

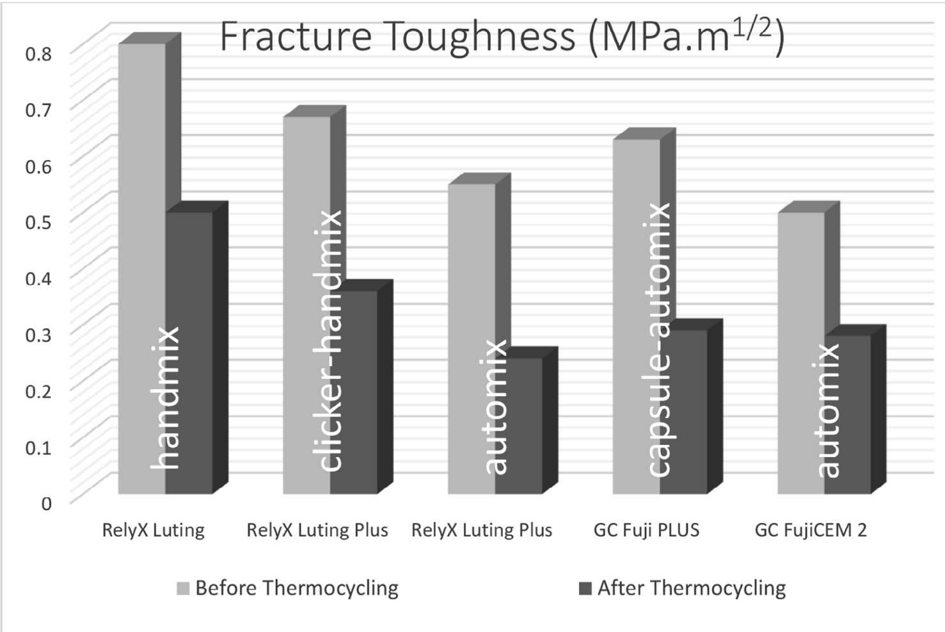

Figure 4. *Mean fracture toughness of resin-modified glass ionomer cements before and after thermocycling.*

the same cement with different dispensing/mixing methods.

Thermocycling (artificially aging) the RMGI cements added to this study in that it allowed us to simulate the physiological aging experienced by the cements in the oral environment. It has been reported[12] that TC for 10,000 cycles at 5°C-55°C with a dwell time of 15 seconds may approximately correspond to one year of clinical function. In this study, a period of 20,000 cycles was adopted to further age the cements to a two-year clinical function. Indeed, TC reduced the mechanical properties of all RMGI cements evaluated in this study, some more significantly than others.

It is very important to translate these findings with caution when searching for clinical recommendations; as one cement brand's mechanical properties reduce in value when switching from a powder-liquid mix to a paste-paste mix, other properties may be enhanced. The bottom line is that "easier may not always be better."

CONCLUSIONS

Within the limitations of this study, the following may be concluded:

1. The mechanical properties of different brands of RMGI cements are not the same, and certain properties may change when switching the dispensing/mixing method.
2. The mechanical properties of all RMGI cements are affected by thermocycling. This process may have an effect on the durability and function of the cement.
3. Clinical relevance from this study should be interpreted with caution. It is not clear how changing the dispensing/mixing method of the same cement brand affects the cements' durability, but the differences in mechanical properties are quite interesting.

Acknowledgement

The authors are grateful to the manufacturers for providing the materials included in this project.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 28 August 2017)

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