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Linear Coefficient of Thermal Expansion Evaluation of Glass Ionomer and Resin-Modified Glass Ionomer Restorative Materials

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Clinical Relevance

Conventional glass ionomer materials overall exhibit linear coefficient of thermal expansion (LCTE) similar to tooth structure, while some resin-modified glass ionomer materials have LCTE similar to that reported for resin restorative materials.

SUMMARY

Objective: The purpose of this evaluation was to evaluate the linear coefficient of thermal expansion (LCTE) of 12 conventional glass ionomer (GIC) and four resin-modified glass ionomer (RMGI) restorative materials.

Methods: GIC and RMGI specimens (2 mm × 5 mm × 5 mm) were fabricated (n=12) following manufacturer instructions and were placed in 0.2M phosphate-buffered saline and stored at 37°C and 98% humidity for one week. Specimens had LCTE determined with a thermomechanical analysis (TMA) unit using a 15°C-50°C heating cycle as well as a 50°C-15°C cooling cycle at a 5°C/min rate, using a 3-mm ball-point probe under 0.02 N probe pressure with all specimens kept saturated with PBS using a specially designed quartz container. Each specimen was tested three times, with the mean representing the specimen LCTE. Mean results between specimen heating and cooling were compared with paired Wilcoxon sign rank test, while results between materials were compared with Kruskal-Wallis/Dunn's (α=0.05).

Results: GIC LCTE ranged from approximately 5°C to 20°C ppm $°K^{-1}$, while the RMGI LCTE ranged from approximately 25°C to 47°C ppm $°K^{-1}$. With some exception, the LCTE during cooling displayed a greater trend.

Significance: Under moisture conditions similar to the oral cavity, GIC materials overall had LCTE values closer to that reported for tooth structure. RMGI materials displayed higher values, which was thought to be related to the amount of resin in the matrix. A generally greater LCTE trend with cooling for all materials was noted, but the small magnitude of the difference is presently thought to be of minor clinical significance.

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INTRODUCTION

The oral environment is subject to many challenges during daily function. Temperature changes fluctuate due to the ingestion of food and liquid, with one review reporting *in vivo* temperature ranges that varied as low as 0°C and as high as 70°C.[1] Restorative materials placed into this environment may show thermal expansion or contraction in response to these thermal changes. A high degree of difference in the thermal expansion characteristics between restorative materials and tooth structure may cause interfacial stress development, which has been implicated as one of the etiological factors in marginal deterioration and microleakage.[2-4]

Linear coefficient of thermal expansion (LCTE) determination methods have included thermochemical analysis,[5] thermomechanical analysis combined with moiré interferometry,[6] both beta radiation and theta dilatometry,[7,8] strain gauges,[9] and x-ray diffraction.[10] LCTE analysis of some conventional glass ionomer (GIC) and resin-modified glass ionomer (RMGI) restorative materials have been reported.[11-14] However, studies to date have investigated only a limited number of products, and evaluation conditions were not under intraoral humidity conditions. Furthermore, these investigations have not investigated material LCTE when subjected to a controlled cooling challenge. The purpose of this investigation was to investigate the thermomechanical properties of GIC and RMGI restorative materials using both heating and cooling challenges. The null hypothesis was that there would be no differences between materials and/or temperature challenge results.

METHODS AND MATERIALS

The GIC materials evaluated are listed in Table 1, with the RMGI products evaluated in Table 2.

Both GIC and RMGI specimens (2 mm × 5 mm × 5 mm) were fabricated using a polyvinylsiloxane mold. Materials were triturated/prepared and carefully injected into the mold and covered with a polyethylene strip and glass slide with digital pressure to prepare a flat surface. GIC materials were allowed to set for the recommended finishing time, while RMGI specimens were photoactivated for 20 seconds on both sides using a visible light curing unit (Bluephase G2, Ivoclar-Vivadent, Amherst, NY, USA). All manufacturer recommendations were followed. Specimens (n=12) were immediately placed in a 98% ± 2% 0.2M phosphate-buffered saline (PBS) environment at 37°C for one week under dark conditions. At the appointed testing time, specimens were placed in a thermomechanical analysis (TMA) unit (TMA841E, Mettler Toledo, Columbus, OH, USA) fitted with a 3-mm-diameter ball-point probe that maintained 0.02 N pressure against the specimen. During analysis, the specimens were maintained under PBS moisture using a specially designed quartz container (Figure 1) that was placed inside the TMA unit. The quartz container was subjected to the TMA thermal protocol (eg, "blank run") so that any effect from the container would be automatically subtracted from the TMA specimen results.

Figure 1. *Quartz container on TMA platform showing immersed sample.*

Specimens were subjected to a thermal challenge protocol that is graphically depicted in Figure 2.

The protocol consisted of an initial five-minute hold at 15°C to allow the sample to thermally equilibrate. The temperature was then raised to 50°C at a rate of 5°C/min, which was followed by a second five-minute hold to allow thermal stability. The specimen was then subjected to a cooling challenge back to 15°C at 5°C/min. LCTE was determined using the slope of each respective thermal challenge, with each specimen tested three times, with the mean representing the LCTE of each specimen. The Shapiro-Wilk and Bartlett's test identified discrepancies in both the data distribution and variance. Mean results between heating and cooling for each sample were compared with the Wilcoxon signed rank test, while results between materials were compared with Kruskal-Wallis and Dunn's post hoc test, with all analysis performed at a 95% level of confidence (α=0.05).

RESULTS

The LCTE results are listed in Table 3.

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Table 1: *GIC Restorative Products*

Material	Manufacturer	Powder/Liquid Ratio, g/g	Powder Content	Liquid Content[a]
Chemfil Rock Capsules	Dentsply, Sirona Int. (York, PA, USA)	[b]	Polycarboxylic acid 10%-25%	Polycarboxylic acid 10%-25% Tartaric Acid 2.5-10%
Equia	GC America (Alsip, IL, USA)	0.40/0.12	Trade secret	Trade secret
Equia Forte	GC America	0.40/0.13	Trade secret	Trade secret
Fuji Triage Capsules	GC America	0.30/0.15	Trade secret	Trade secret
Ketac Fil Plus	3M ESPE (St Paul, MN, USA)	[b]	Trade secret	Acrylic acid–maleic acid copolymer 35%-55% Tartaric acid 5%-10% Water 45%-55%
Ketac Molar Quick Aplicap	3M ESPE	[b]	Oxide glass chemicals (nonfibrous) 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	[b]	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%
Ketac Universal	3M ESPE	[b]	Glass, oxide, chemicals >95%	Acrylic acid–maleic acid copolymer 30%-50% Tartaric acid 1%-10% Benzoic acid <0.2%
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%

[a] *Remaining percent is assumed to be water*
[b] *Not available from manufacturer information; content information obtained from manufacturer information.*

Equia Forte demonstrated the lowest LCTE value, followed by Riva SC HV, Ketac Silver, Ketac Universal, Ketac Fil, and Fuji Triage. The RMGI products demonstrated higher LCTE values, with Ketac Nano having the highest LTCE value, followed by Riva LC, Riva LC HV, and Fuji II LC, all of which were statistically similar. In comparing heating and cooling LCTE, except for Riva Protect Fast, all materials demonstrated greater cooling LCTE. Chemfil Rock, Ketac Fil, Ketac Molar Quick, Ketac Silver, Riva SC Fast, and Riva Silver were the only materials that did not significantly differ from the heating challenge.

DISCUSSION

This study evaluated the thermomechanical characteristics of 11 GIC and four RMGI restorative materials. The determination of LCTE direct restorative materials is considered relevant as a large difference in the LCTE characteristics between interfaces may produce stress that could cause marginal deterioration and microleakage.[2-4] The LCTE of ceramic/metal-ceramic layers also must closely be matched to prevent interfacial stress development that may be the etiology of defects.[15-19]

Previous TMA studies for different materials have used protocols involving different ranges. Spierings

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Table 2: *RMGI Restorative Products*

Material	Manufacturer	Powder/Liquid Ratio, g/g	Powder Content	Liquid Content
Fuji II LC Capsules	GC America (Alsip, IL, USA)	0.33/0.010	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%

Abbreviations: BISGMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] Content information obtained from manufacturer information.

and others reported a 14°C to 56°C range[20]; Versluis and others, 26°C to 75°C[9]; Bullard and others, 5°C to 55°C[2]; Sideridou and others, 0°C to 80°C[12]; Sidhu and others 25°C to 50°C[11] Tezvergil and others, 23°C to 160°C[21]; Vaidyanathan and others, 30°C to 70°C[22]; Hashinger and Fairhurst, room temperature to 120°C[23]; and Kwon and others, 20°C to 80°C range.[24] Thermal conditions during intraoral function have been reported to occur over a wide range of temperatures, reported from −5°C to 76°C.[1,25,26] However, it has been suggested that the mean maximum intraoral temperature is approximately 46°C with fluids and 41°C with solid food.[27] As for the minimum range of intraoral functional temperatures, approximately 15°C was suggested by Youngson and Barclay,[28] and 0°C was reported by Palmer and others.[29] In addition, the reported TMA temperature change rate has also been variable, with values being reported of 10°C min^{-1},[3,10,13,20,24] 5C min^{-1},[11] 3°C min^{-1},[22] and 1°C min^{-1}.[23] The temperature range chosen for this study was 15°C to 50°C at a rate of 5°C min^{-1} and was based on the minimum temperature suggested by Youngson and Barclay[28] and the higher temperature slightly above the temperature suggested by Feuerstein and others.[27] The temperature change rate was chosen as a slow rate is recommended (1°C to 5°C min^{-1}) to allow sufficient heat penetration through the sample to prevent errors due to temperature lag. [30-32]

With restorative resins, LCTE has been reported to be influenced by the amount of filler contained in the resin.[21-23] Under the conditions of this study, the RMGI materials demonstrated higher LCTE values than the GIC materials. With the exception of Ketac Nano, the RMGI products do not contain fillers per se, and the LCTE in glass ionomer restorative materials may be influenced by the amount of resin contained within the matrix. Although tooth material LCTE have been accomplished with essentially 100% humidity[8] and GIC materials in a moist (65%) environment,[3] this study is the first, to the authors' knowledge, that has tested GIC materials totally immersed in PBS. Although this resulted in an environment with higher humidity than the range reported for the human oral cavity (78%-94%),[33] immersion in physiologic solution allowed a reproducible testing environment, as maintaining stable humidity values is difficult within the TMA device.

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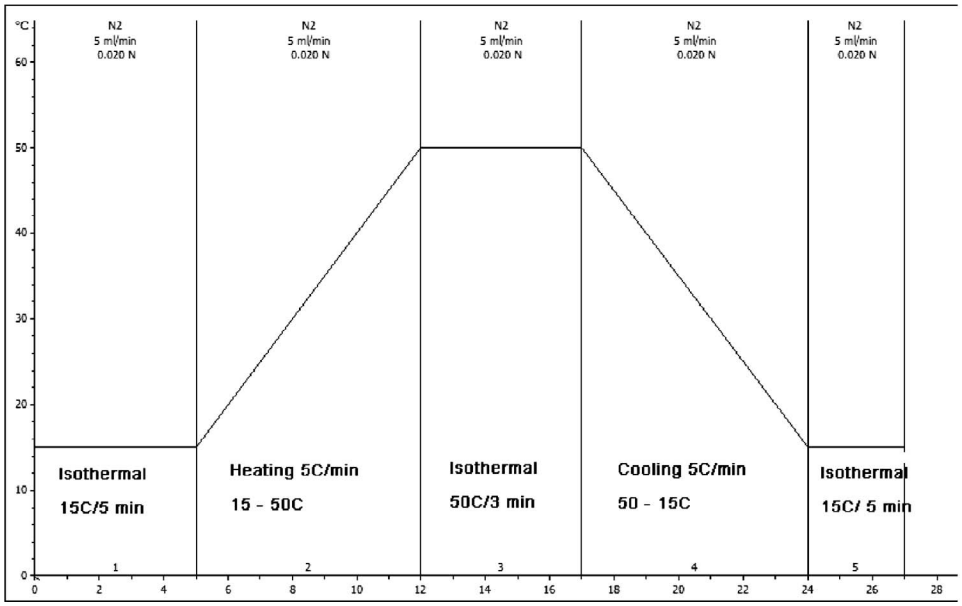

Figure 2. *TMA regimen.*

With the exception of Chemfil Rock and Riva Protect Fast, the GIC materials demonstrated a mean LCTE that was lower than 15 ppm $°K^{-1}$. The results of this study differ from those earlier reported for GIC restorative materials. Both Sidhu and others[10] and Yan and others[3] reported that GIC materials demonstrated shrinkage during thermal challenge, but these results did not involve maintaining moisture, and the resulting shrinkage was due to moisture loss. Yan and others[3] in the same study also maintained at least 65% humidity, which was reported to cause minimal dimensional change for GIC materials. The present study, which had humidity values closer to that of the oral environment, did demonstrate specimen dimensional change. Tooth structure LCTE has been reported to be approximately 17 ppm $°K^{-1}$ for enamel and 11 ppm $°K^{-1}$ for dentin.[8] With combined dentin and enamel, the noncarious molar occlusal surface has been reported to be approximately 16 ppm $°K^{-1}$ with the molar cervical area to be approximately 5 ppm $°K^{-1}$.[34] For restoration of cervical molar areas, it might be intuitive to reason that the conventional GIC materials would be indicated, but the reported longevity of RMGI materials in cervical restorations cannot be dismissed.[35-37]

Table 3: *Mean (SD) LCTE Results (ppm/°K)*[a]

GIC Materials	Heating LCTE	Cooling LCTE
Chemfil Rock	16.7 (2.0) D a	21.4 (2.7) ABCD a
Equia	12.7 (2.3) DE a	17.5 (3.8) ABCDE b
Equia Forte	5.6 (1.6) G a	7.6 (0.9) E b
Fuji Triage	9.0 (2.2) EFG a	13.7 (3.4) BCDE b
Fuji II LC	25.4 (3.5) BC a	30.0 (5.0) AB b
Ketac Fil	7.9 (2.4) EFG a	8.8 (3.4) E a
Ketac Molar Quick	6.9 (1.0) FG a	7.4 (1.4) E a
Ketac Nano	47.4 (8.5) A a	50.3 (10.7) A b
Ketac Silver	8.1 (1.8) EFG a	9.2 (2.6) DE a
Ketac Universal	8.5 (2.1) EFG a	11.1 (1.9) CDE b
Riva Protect Fast	20.6 (3.9) CD a	14.6 (3.9) ABC b
Riva LC	30.6 (6.4) B a	36.9 (7.2) A b
Riva LC HV	28.9 (5.6) B a	32.6 (6.7) AB b
Riva SC Fast	14.4 (4.0) D a	17.6 (5.1) ABCDE a
Riva SC HV	6.4 (1.9) FG a	8.4 (2.5) E b
Riva Silver	12.3 (3.1) DEF a	14.6 (3.9) BCDE a

[a] *n=12. Uppercase letters identify similar groups per column (Kruskal-Wallis/Dunn's; α=0.05); lowercase letters identify similar groups per row (paired Wilcoxon sign rank; p=0.05).*

This study is the first to report LCTE determination during a dedicated cooling challenge. The results found that the cooling challenge from 50°C to 15°C produced a higher LCTE trend as compared with the heating cycle. With the exception of Chemfil Rock, Ketac Fil, Ketac Molar Quick, Ketac Silver, Riva SC Fast, and Riva Silver, GIC and RMGI products demonstrated significantly more LCTE during the cooling cycle. The reasons for this hysteresis and its significance are presently not known, and further research is warranted to identify possible causes such as proscribed temperature rate, testing conditions, or artifact induced by hardware used. Nevertheless, although the results are interesting, the difference is of relatively little magnitude, and it is presently thought doubtful to be of clinical significance.

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CONCLUSION

Under the conditions of this study, the LCTE of conventional GIC materials during heating challenge ranged from approximately 5°C to 20°C ppm $°K^{-1}$, while the LCTE of the RMGI materials ranged from approximately 25°C to 47°C ppm $°K^{-1}$. With some exception, during a cooling challenge, the LCTE of all materials displayed a trend of higher LCTE, with the reason and significance unknown. However, the magnitude of the difference was relatively small and is presently thought to be of minor clinical significance.

Note

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors. Any opinions expressed in this work are of the authors only and do not represent the official opinion of the United States Air Force, Uniformed Services University of the Health Sciences, Department of Defense, or the United States Government.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the USAF Post Graduate Dental School, Keesler Air Force Base, MS.

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.

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