Effect of solution temperature on the mechanical properties of dual-cure resin cements

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PURPOSE. This study was to evaluate the effect of the solution temperature on the mechanical properties of dual-cure resin cements. **MATERIALS AND METHODS.** For the study, five dual-cure resin cements were chosen and light cured. To evaluate the effect of temperature on the specimens, the light-cured specimens were immersed in deionized water at three different temperatures (4, 37 and 60℃) for 7 days. The control specimens were aged in a 37℃ dry and dark chamber for 24 hours. The mechanical properties of the light-cured specimens were evaluated using the Vickers hardness test, three-point bending test, and compression test, respectively. Both flexural and compressive properties were evaluated using a universal testing machine. The data were analyzed using a two way ANOVA with Tukey test to perform multiple comparisons (α=0.05). **RESULTS.** After immersion, the specimens showed significantly different microhardness, flexural, and compressive properties compared to the control case regardless of solution temperatures. Depending on the resin brand, the microhardness difference between the top and bottom surfaces ranged approximately 3.3-12.2%. Among the specimens, BisCem and Calibra showed the highest and lowest decrease of flexural strength, respectively. Also, Calibra and Multilink Automix showed the highest and lowest decrease of compressive strength, respectively compared to the control case. **CONCLUSION.** The examined dual-cure resin cements had compatible flexural and compressive properties with most methacrylate-based composite resins and the underlying dentin regardless of solution temperature. However, the effect of the solution temperature on the mechanical properties was not consistent and depended more on the resin brand. ***[J Adv Prosthodont 2013;5:133-9]***

KEY WORDS: Solutiontemperature; Microhardness; Flexural properties; Compressive properties; Dual-cure resin cements

INTRODUCTION

Methacrylate-based resin cements are popular for attaching indirect restoratives, appliances, and fiber posts to the prepared teeth due to their less technique sensitivity, good esthetics, high bond strength, and a potential for fluoride release.[1-4] As luting agents for indirect restorations, resin cements are needed to keep the restoration in place and prevent dislodgement by forming a hard mass with sufficient strength to resist functional forces. In the oral cavity, however, shear forces that interact with the restoration inevitably induce dislodgement and retention loss. In addition, a low flexural strength, high shrinkage, water uptake, and expansion of the restorative materials also provoke debonding.[5-7]

Among the resin cements, dual-cure resin cements have been adopted widely because of their dual cure nature by light and chemical agents. Since restorative materials with opacity or dark shades are common, insufficient light transmission to the underlying cement and insufficient curing

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(degree of conversion) can occur. So any additional curing will be beneficial to compensate insufficient curing of resin cement because the curing conditions can be affected by the external light (light intensity, distance between the light-guide and resin cement, light-curing unit, etc.) and restoration thickness.[8-11] In a dual-cure system, light-curing process is initiated by the light-activated photoinitiators and terminates the process mostly after the end of light irradiation. On the other hand, the self-curing process initiated by a chemical activator (benzoyl peroxide) lasts longer, even after the termination of light irradiation. Therefore, in a situation of insufficient light transmission, the free radicals formed by the benzoyl peroxide/amine system compensate for the lack of monomer to polymer conversion.[12] The dual–cure resin cements give higher hardness or better bond strength than those of the chemically-activated resin cements.[13,14] The overall properties of resin cements using the dual-cure system seem to be superior when compared to the single-cure system.

Although the temperature in the oral cavity remains constant in most times, it can vary from 0 to 60-70℃ due to the foods served and smoking.[15,16] Such a variation in temperature may chill or heat the restorative materials and teeth and can change their mechanical properties. The short-term thermal effect on the resin cements can be understood indirectly by examining the effect of pre-heating on the resin cement. According to the limited studies, the results of pre-heating were inconsistent and product-dependent.[17,18] Thus long-term thermal effect on the dual-cure resin cements in conjunction with their mechanical properties was not widely studied. The present study investigated the effect of temperature on the light-cured resin cements that were immersed in water. The microhardness, flexural and compressive properties were evaluated. The hypothesis was that the mechanical properties of dual-cure resin cements consistently depend on the temperature of immersion solution.

MATERIALS AND METHODS

For the study, five dual-cure resin cements (MaxCem Elite (MC), BisCem (BC), Calibra (CA), RelyX ARC (RX), and Multilink Automix (MA)) were selected, and their compositions are summarized in Table 1. For light curing, a quartz-tungsten-halogen (QTH) light-curing unit (Hilux 601, First Medica, Greensboro, NC, USA) of 900 mW/cm^2 was used. To evaluate the temperature effect on the specimens, the light-cured specimens were immersed in deionized water of three different temperatures (4, 37 and 60℃). The control specimens were aged only for 24 hours in a 37℃ dry and dark chamber.

To measure the surface microhardness of the specimens, a metal mold (4 × 2 × 3 mm) was filled with the resin cement and light cured for 40 seconds. The cured specimens was removed from the mold and aged for 24 hours in a 37℃ dry and dark chamber. The specimens were then immersed in deionized water (4, 37 and 60℃) for 7 days. At this time, each specimen (n=10 for each temperature condition) was placed in a 1.5 mL tube, and the solution in the tube was replaced daily. After 7 days, specimens were removed from the tube and the solution on the surfaces was blotted away. The microhardness of the light cured top side (z=0) and bottom side (z=3 mm) surfaces was measured using a Vickers hardness tester (MVK-H1, Akashi, Tokyo, Japan) by making a microindentation (n=12 for each test condition) under a 200 gf load and 10 seconds dwell time.

Table 1. Composition of the tested resin cements

Product (Code)	Composition	Filler content* wt/vol%	Manufacturer
MaxCem Elite (MC)	Base: UDMA, fluoroaluminosilicate glass Catalyst: Bis-GMA, TEGDMA, GPDM, bariumaluminosilicateglas	66/46	Kerr, Orange, CA, USA
BisCem (BC)	Base: Bis-GMA, TEGDMA, glass filler Catalyst: Bis HEMA phosphate, glass filler	> 60	Bisco Inc., Schaumburg, IL, USA
Calibra (CA)	Base: Bis-GMA, TEGDMA, barium-boron-fluoroaluminosilicate glass, TiO_2 Catalyst: Bis-GMA, TEGDMA, barium-boron-fluoroaluminosilicate glass, TiO_2, benzoyl peroxide	> 60	Dentsply/DeTrey, Konstanz, Germany
RelyX ARC (RX)	Bis-GMA, TEGDMA, silanated ceramic /silica	60-70	3M ESPE, St. Paul, MN, USA
Multilink Automix (MA)	Bis-GMA, TEGDMA, HEMA, benzoyl peroxide, barium glass, ytterbium trifluoride	68.5/40	IvoclarVivadent, Schaan, Liechtenstein

* Information provided by the manufacturers.
Bis-GMA: bisphenol A diglycidyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethyleneglycoldimethacrylate, GPDM: glycerophosphatedimethacrylate, HEMA: 2-hydroxyethyl methacrylate.

A three-point bending test was performed to determine the flexural properties [flexural strength (FS) and modulus (FM)]. To make the specimens, a metal mold (25 × 2 × 2 mm) was filled with the resin cement according to the ISO 4049.[19] After filling the mold, both top and bottom surfaces were covered with slide glasses to make a flat surface. The specimen was irradiated for 40 seconds using a light-curing unit. Since the specimen was much wider (25 mm) than the tip size (8 mm), five light exposures were performed on each side by overlapping the curing light. After light curing, one group of specimens was removed from the mold and aged for 24 hours in a 37℃ dry and dark chamber (control), the other groups of specimens were immersed in the three test solutions (4, 37 and 60℃) for 7 days. After aging or immersing in the test solutions, the specimens (n=10 for each test condition) were loaded to a universal testing machine (Instron 3345, Grove City, PA, USA) at a crosshead speed of 1 mm/min. The FS (σ_f in MPa) was obtained using the following formula

$\sigma_f = 3DP / (2WH^2)$

where D is the distance between the supports (20 mm), P is the maximum failure load (N), W is the width (2 mm), and H is the height (2 mm) of the tested specimen. The FM (E in GPa) was obtained using the following formula

$E = (P / D) \cdot (D^3 / (4WH^3))$

where P/D is the slope in the linear portion of the load-displacement curve.

To measure the compressive properties [compressive strength (CS) and modulus (CM)], a metal mold (3 mm in diameter, 6 mm in height) was filled with the resin cement. The metal mold was made of two identical hollow hemicylinders by combining together. After filling the mold, both the top and bottom surfaces were covered with slide glasses to make the surface flat and then irradiated for 5 seconds (since light does not reach to the bottom surface, light curing would be better through the lateral surface after exposure). Subsequently, one part of the metal mold was removed by sliding. The exposed surface was light cured for 40 seconds. The opposite side was light cured for 40 seconds again after removing the other part. After light curing, one group of specimens was removed from the mold and aged for 24 hours in a 37℃ dry and dark chamber (control). The other group of specimens was immersed in the testing solutions (4, 37 and 60℃) for 7 days. After aging or immersing in the test solutions, compression tests were performed using a universal test machine at a crosshead speed of 1 mm/min. The CS (σ_c in MPa) was obtained using the following formula

$\sigma_c = P / A$

where P is the maximum failure load (N) and A is the cross-sectional area of the specimen. The CM of the specimens is the slope of the linear portion of the load-displacement curve.

The data acquired from the microhardness, three-point bending test, and compression test was analyzed by two way ANOVA and post-hoc Tukey tests at the 0.05 level of significance.

RESULTS

Table 2 shows the microhardness of the top and bottom surfaces before and after immersion in the solutions of different temperatures. Before immersion, MA and MC showed the highest (50.9/47.6 HV) and lowest (33.1/30.1 HV) microhardness, respectively, among the specimens. After immersion for 7 days, the specimens showed a significant decrease in microhardness compared to the control case regardless of the solution temperatures (P<.001). On the top surface, CA and BC showed the lowest (4.8-15.9%) and highest (42.4-54.6%) microhardness difference between

Table 2. Microhardness of the specimens immersed in the solutions of different temperatures

		Resin cement					
		MC[A]	BC[B]	CA[C]	RX[D]	MA[E]	*P* value
Top surface	Control[1]	33.1 ± 2.6	46.9 ± 2.8	42.1 ± 2.5	48.1 ± 3.2	50.9 ± 3.3	α<.001
	4℃[2]	20.9 ± 1.7	21.3 ± 1.2	40.1 ± 3.2	41.4 ± 1.2	44.5 ± 0.4	β<.001
	37℃[3]	22.5 ± 1.5	25.4 ± 0.7	35.4 ± 2.8	39.3 ± 2.1	38.1 ± 0.7	α*β<.001
	60℃[3]	21.1 ± 1.6	27.0 ± 1.1	36.2 ± 2.3	35.6 ± 3.2	37.9 ± 0.5	
		MC[A]	BC[B]	CA[C]	RX[D]	MA[E]	
Bottom surface	Control[1]	30.1 ± 3.9	41.2 ± 2.3	37.2 ± 5.9	46.5 ± 2.3	47.6 ± 2.3	α<.001
	4℃[2]	20.2 ± 1.4	20.2 ± 1.3	37.6 ± 2.3	38.8 ± 1.8	42.6 ± 1.1	β<.001
	37℃[2]	22.3 ± 1.8	24.5 ± 1.1	34.1 ± 2.9	36.4 ± 1.1	36.2 ± 1.1	α*β<.001
	60℃[2]	20.6 ± 0.9	25.7 ± 0.8	34.2 ± 1.8	34.3 ± 2.1	35.6 ± 1.4	

* Statistically significant difference among the resin cements is shown by superscript letters[A, B, ...], temperature conditions by superscript numbers[1, 2, 3]. Same letters or numbers are not significantly different (*P*>.05).

* On *P*-values, the letters α and β denote temperature and resin cement, respectively.

MC: MaxCem Elite, BC: BisCem, CA: Calibra, RX: RelyX ARC, MA: Multilink Automix.

the control and immersed specimens, respectively. RX and MA showed a consistent microhardness decrease as the solution temperature increased. On the bottom surface, for the control case, the microhardness decreased approximately 3.3-12.2% compared to that of the top surface. The change of microhardness for solutions of different temperatures was not consistent both on the top and bottom surfaces.

Flexural properties (FS and FM) of the resin cements immersed in the solutions of different temperatures are shown in Table 3. The highest and lowest FS, before (control) and after immersion, were achieved by CA (116.8-147.1 MPa) and BC (51.9-85.5 MPa), respectively. After immersion for 7 days, in most cases, FS decreased with increasing solution temperature. Among the specimens, BC and CA showed the highest (39.3%) and lowest (20.6%) FS decrease, respectively. Before immersion, MA and RX showed the highest (12.95 GPa) and lowest (10.72 GPa) FM, respectively. After immersion, most specimens (except CA) showed a significant decrease in modulus with increasing solution temperature (P<.001). Among the specimens, CA and BC showed the highest (10.82-11.49 GPa) and lowest (6.62-6.81 GPa) FM, respectively.

Table 4 shows the compressive properties (CS and CM) of the resin cements immersed in the solutions of different temperatures. Before immersion, RX and BC showed the highest (299.3 MPa) and lowest (211.7 MPa) CS, respectively. After immersion for 7 days, CS of the specimens decreased significantly (P<.001). Among the specimens, CA and MA showed the highest (22.6%) and lowest (2.2%) CS

Table 3. Three-point flexural properties of resin cements immersed in solutions of different temperatures

		Resin cement					
		MC[A]	BC[B]	CA[C]	RX[D]	MA[CD]	*P* value
Flexural strength	Control[1]	117.48 ± 3.25	85.49 ± 9.23	147.13 ± 12.69	141.46 ± 11.24	132.95 ± 18.36	α<.001
(MPa)	4℃[2]	82.18 ± 12.82	55.79 ± 8.15	135.27 ± 9.37	113.82 ± 132.68	127.57 ± 12.84	β<.001
	37℃[3]	81.47 ± 18.52	54.56 ± 10.27	116.84 ± 11.05	106.07 ± 12.56	124.12 ± 10.69	α*β=.048
	60℃[3]	81.29 ± 7.47	51.88 ± 6.51	118.59 ± 11.37	94.38 ± 12.31	99.02 ± 17.36	
		MC[A]	BC[B]	CA[C]	RX[A]	MA[C]	
Flexural modulus	Control[1]	12.49 ± 1.43	11.28 ± 0.94	11.34 ± 1.19	10.72 ± 1.23	12.95 ± 0.92	α<.001
(GPa)	4℃[2,3]	6.82 ± 0.96	6.69 ± 0.84	10.82 ± 0.73	8.95 ± 0.91	11.37 ± 1.06	β<.001
	37℃[3]	8.38 ± 1.07	6.62 ± 0.78	11.49 ± 0.74	8.83 ± 0.66	10.81 ± 0.74	α*β<.001
	60℃[3]	8.32 ± 0.85	6.81 ± 0.86	11.05 ± 0.58	8.62 ± 0.52	9.96 ± 0.67	

* Statistically significant difference on resin cements is shown by superscript letters[A, B,...], temperature on by superscript numbers[1, 2, 3]. Same letters or numbers are not significantly different (P>.05).

* On P-values, the letters α and β denote temperature and resin cement, respectively.

MC: MaxCem Elite, BC: BisCem, CA: Calibra, RX: RelyX ARC, MA: Multilink Automix.

Table 4. Compressive properties of resin cements immersed in solutions of different temperatures

		Resin cement					
		MC[A]	BC[B]	CA[A]	RX[C]	MA[C]	*P* value
Compression	Control[1]	248.97 ± 13.12	211.67 ± 14.35	275.38 ± 12.76	299.25 ± 13.73	278.61 ± 11.57	α<.001
Strength	4℃[2]	245.83 ± 15.34	197.79 ± 14.37	220.69 ± 14.23	288.93 ± 7.95	273.35 ± 11.38	β<.001
(MPa)	37℃[2,3]	248.89 ± 6.68	189.68 ± 11.38	223.49 ± 8.94	279.49 ± 11.67	275.21 ± 14.76	α*β<.001
	60℃[3]	237.98 ± 8.68	178.17 ± 9.49	213.15 ± 10.68	246.38 ± 13.58	272.54 ± 13.16	
		MC[A]	BC[A]	CA[B]	RX[A]	MA[B]	
Modulus	Control[1]	4.21 ± 0.57	4.19 ± 0.37	3.92 ± 0.47	3.62 ± 0.55	3.85 ± 0.68	α<.001
(GPa)	4℃[2]	2.87 ± 0.57	2.84 ± 0.59	3.39 ± 0.59	3.60 ± 0.62	3.74 ± 0.38	β<.001
	37℃[3]	2.92 ± 0.61	2.25 ± 0.63	3.64 ± 0.61	2.93 ± 0.47	3.64 ± 0.58	α*β<.001
	60℃[3]	3.01 ± 0.49	2.16 ± 0.51	3.48 ± 0.63	2.73 ± 0.57	3.39 ± 0.57	

* Statistically significant difference on resin cements is shown by superscript letters[A, B,...], temperature on by superscript numbers[1, 2, 3]. Same letters or numbers are not significantly different (P>.05).

* On P-values, the letters α and β denote temperature and resin cement, respectively.

MC: MaxCem Elite, BC: BisCem, CA: Calibra, RX: RelyX ARC, MA: Multilink Automix.

decrease, respectively. For CM, before immersion, BC and RX showed the highest (4.19 GPa) and lowest (3.62 GPa) modulus, respectively. However, after immersion, specimens showed a significant decrease in modulus ($P<.001$). Among the specimens, BC showed the lowest modulus (2.16 GPa) when immersed in the solution of 60℃.

DISCUSSION

The present study evaluated the mechanical properties of dual-cure resin cements under different solution temperatures, which can minimally reflect the oral situation. The choice of solution temperature was arbitrary because the range of temperatures in the oral cavity is complicated and dynamic. In most situations, a constant temperature is maintained if there is no eating or drinking. However, the temperature can vary from 0 to 60-70℃ once cold or hot food is served.[15,16] The range in this case can change depending on the dietary habits of each individual. In that sense, the choice of three different temperatures (4, 37 and 60℃) appears to be minimally appropriate for mimicking the oral situation.

The microhardness test is a useful indirect assessment for evaluating the degree of cure of methacrylate-based specimens. The microhardness of the specimens is affected by many other factors which constitute the specimens. Among the factors, the filler content, monomers types and ratios were found to be the determinant factors.[20,21] Regarding the filler content, in the present study, manufacturers released only the weight% data. According to the data, the resin cements have much lower filler content than many other composite resins. Generally, in light-curing composite resins, the microhardness is positively correlated with the filler content. Therefore, in many cases, specimens with high filler content have high microhardness. The degree of cure is basically the degree of monomer conversion to the polymer networks. Most methacrylate-based dental restorative materials contain monomers, such as Bis-GMA, Bis-EMA, UDMA, and TEGDMA, with many different combinations and ratios. This means that the degree of cure (conversion) changes inconsistently depending on the commixtures and their combination ratios.[22,23] In the present study, the filler content of the specimens showed a low correlation with the measured microhardness ($R<0.4$) regardless of the solution temperatures. Such a low correlation may be due to the low filler content of the tested specimens. Within the resin matrix, the distributed fillers are the source of light dissipation because fillers induce light scattering and absorption, so low filler content may result in less light loss within the specimen. Actually, resin cements are not used for the restorations in which a direct mastication load is impressed, so the situation of low filler content would be tolerable both by itself and due to the dual-cure nature. The process of dual-cure is the curing of resin cements both by the external light and chemical agent. The dual-cure polymerization is beneficial in which light transmission is not sufficient. In this process, light-induced polymerization occurs first and then chemical agent-induced polymerization occurs later. The latter process compensates incomplete polymerization due to insufficient light transmission at the lower part of the specimen. On the control case (before immersion), minor microhardness difference (3.3-12.2% depend on resin brand) between the top and bottom surfaces would be due to the additional curing by chemical agent. Such low microhardness difference is not common in the light-curing composite resins of 3-mm thick.

After immersion in the test solutions for 7 days, the specimens showed a significant decrease in microhardness both on the top and bottom surfaces. Such decrease in microhardness has also been observed in composite resins due to softening of the surface.[24] Once the specimens are immersed in the test solutions, water uptake and subsequent attacks at the interface between the inorganic fillers and resin matrix will occur. Weakening of the bond between the filler and matrix and leaching of the surface by water make the specimen soft, which leads to a decrease in hardness. The solution temperature also significantly affects the microhardness. However, in the present study, the results were not consistent. In the case of RX and MA, the microhardness decreased gradually with increasing solution temperature. On the other hand, in the case of BC, the microhardness increased gradually with increasing solution temperature even though the microhardness had significantly decreased after immersion at 4℃ solution. In general, temperature increase enhances the activation of free radicals. Moreover, any additional curing by the external heat after the termination of light curing will depend on the residual monomers. In dual-cure resin cements, the residual monomers will be polymerized further by the subsequent chemical curing, so the remaining monomers will be reduced further with time (even though the situations can be different in each specimen). In the present study, hot water may enhance further polymerization, but, it may also enhance the softening and leaching of specimen through the specimen expansion and dissolution, so subsequent weakening is possible. On the other hand, if chemical curing after light curing is not complete and any monomers remain, external heat from the immersion solution may lead to some further polymerization, resulting in a subsequent increase in microhardness.

FS and FM, the flexural properties, are determined using a three-point bending test. These flexural properties are related to the resistance to the mastication stress without fracture or permanent deformation. Generally, FS depends on the internal defects or voids that were generated during manufacturing process.[25-27] In the present study, FS and FM showed a significant decrease after immersion in the test solution, but the decrease was not consistent in the specimens. The FS of the resin cements tested ranged approximately 52-147 MPa depending on the conditions (control or immersion). These values meet the minimum FS requirement for dual-cure luting materials (50 MPa) set by ISO 4049.[19] As to the many methacrylate-based composite

resins from more than 70 proprietary products (approximately 63-161 MPa), the tested resin cements showed a similar range of FS values.[28] After immersion for 7 days, the initial (control) FM (10.72-12.95 GPa) decreased to 6.62-11.49 GPa depending on the solution temperature and brand. In the oral cavity, the preferred FM of the resin cements should be near the value of dentin and the restorative materials in order to have a similar deformation nature against the external load. According to the studies, the modulus of methacrylate-based restoratives and dentin was approximately 3-12 GPa and 17-25 GPa, respectively.[28-30] A similar FM of the tested resin cements as to the many other restoratives is desirable for more durable cementation. Otherwise, any resin cement may cause an adverse effect on the dentin-restorative structure due to the improper resistance at the interface.

A compression test is an important in vitro test that evaluates the material's sustained resistance against a heavy load during mastication. After immersion, the compressive properties (CS and CM) significantly decreased, but the correlation with solution temperature was not consistent. MC and MA showed minor CS change (2.2-4.4% decrease after immersion compared to that of the control case). On the other hand, the rest resin cements showed much greater CS change (15.8-22.6%) than MC and MA. Generally, as the solution temperature increases, the immersed specimens expand and it results in the increase of water uptake through the filler-matrix interface. Also, dissolution of fluoride from the contained fluoride-containing fillers will occur. As a result, weakening of the bond between the filler and matrix and the structure itself can lead to a decrease in CS. However, if there is any additional curing by the hot solution, such curing may compensate the weakening of the specimens by the absorbed water. The similar CS pattern in MC and MA may be due to a combination of these two effects. In the present study, CS after immersion for 7 days ranged approximately 178-299 MPa. This range matches with most methacrylate-based composite resins (approximately 100-290 MPa based on the study with more than 70 proprietary products).[28] Specimens showed much lower CM (2.2-4.2 GPa) than that of dentin (11.0-18.5 GPa) whether they were immersed or not.[31,32] As a luting material, since resin cement forms a thin layer at the interface, such low CM may not make any serious mechanical problem after bonding. According to the evaluation, immersion in solution made the specimens to have significantly lower microhardness, flexural and compressive properties; yet, the values inconsistently changed for varying solution temperature. The hypothesis has to be rejected.

CONCLUSION

The mechanical properties of the dual-cure resin cements were tested for different solution temperatures. The microhardness, flexural properties (FS and FM), and compressive properties (CS and CM) decreased significantly after immersion in the deionized water regardless of change in temperature. However, the effect of the solution temperature on the mechanical properties was not consistent and was rather brand-dependent. The flexural and compressive properties of the dual-cure resin cements were similar to those of the most methacrylate-based composite resins and dentin.

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