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Effect of Tack Cure on Polymerization Shrinkage of Resin-based Luting Cements

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Clinical Relevance

Self-cure after tack cure could result in a lower polymerization shrinkage in some resin-based luting cements, which is closely related to lower degree of cure.

SUMMARY

Objectives: To evaluate the effect of tack cure on polymerization shrinkage (PS) of resin-based luting cements.

Methods and Materials: One composite resin cement, Duo-Link (Duolink); two self-adhesive resin cements, RelyX U200 (U200) and G-CEM LinkAce (GCem); and one resin-modified glass ionomer cement, RelyX Luting Plus (Luting+), were used for measuring PS in light-cure (LC group), self-cure (SC group), and two tack-cure modes that were light cured (TC-LC group) or self-cured (TC-SC group) after tack cure. PS was measured by a modified bonded disc method for 1600 seconds and analyzed with two-way analysis of variance and Tukey honestly significant difference test. To investigate the effect of tack cure on light cure or self-cure, data were analyzed with an independent-samples *t*-test with tack cure as a variable. The significance level was 5%.

Results: Regarding cure mode, Duolink showed a significantly lower PS in the TC-SC group compared with the other groups. Luting+ showed a significantly lower PS in the TC-SC group than in the SC group. U200 showed a significantly lower PS in the self-cure groups compared with that in the light-cure groups. The PS of GCem was not affected by cure mode.

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Regarding cements, Luting+ showed the highest PS, followed by GCem, Duolink, and U200 ($p<0.05$). Self-cure of Duolink and Luting+ was negatively affected by tack cure, while light cure was not affected. U200 and GCem were not affected by tack cure either in the self-cure or light-cure groups.

Conclusion: For the tested cements, tack cure decreased the PS of Duolink and Luting+ when they were self-cured after tack cure. When the cements were light cured after tack cure, PS was not affected by tack cure in any cement.

INTRODUCTION

The tack cure procedure is generally used to remove excess cement from the margins of indirect restorations when using resin-based luting cements with the tack-cure option.[1] Briefly, a light-cure tip is placed over the margins and light cured for a short time to polymerize cements to a semi-gel state, which makes the removal of excess cement easier while fixing the restoration in place. Then, the cements can be light cured or self-cured for final cure according to the manufacturer's instructions.[2,3]

As dual-cure cements incorporate both light-cure and self-cure components, the polymerization initiates independently but overlaps with the other cure mode during the cure reaction, as both cure modes initiate free-radical formation and monomer conversion.[4] The exact mechanism for the interaction between light cure and self-cure is not yet known. However, studies have shown that both cure modes could not guarantee a uniform maximum cure.[2,5,6] The self-cure reaction is generally slower and shows less effective monomer conversion than when light cure is used as a supplement,[4,7] as there is a limit to the amount of self-cure promoters that can be added to dual-cure cements in order not to impair their working time. Therefore, the self-cure reaction is generally restricted to some extent.[7]

One issue encountered with the tack cure procedure is that immediate light cure of some dual-cure cements appears to interfere with the following self-cure reaction and restricts the cement from achieving its maximum mechanical properties.[7-9] Immediate light cure induces rapid formation of polymer chain cross-linking, which results in entrapment of initiators and unreacted monomers because of increased steric hindrance. Thus, segmental mobility of methacrylate groups is decreased along with the reactivity of the self-cure reaction.[10,11] In addition, studies showed that immediate low-dose light cure, which is more closely related to tack cure, could lower the degree of cure (DC) of dual-cure cements.[7,9]

Another issue is encountered when the cement is light cured after tack cure to reduce chair time. This is because the self-cure reaction is in progress during excess cement removal. The resulting interruption of the light-cure procedure could also result in the formation of relatively linear polymer structures with lower cross-link density, which makes polymers more susceptible to softening, as shown in pulse-delay cure of light-cure composite resins.[12,13]

Therefore, the incorporation of a tack-cure procedure offers a complex cure scenario consisting of light cure and self-cure in dual-cure cements. Previous studies showed that delaying or omitting light cure might modify the polymeric structure[14] and the extent of polymerization of dual-cure cements.[9] The moment of light cure, light intensity, duration of interruption, and frequency of interruption determine the way structure networks will be formed,[15,16] thus determining the structural integrity of the materials.[9] However, there are only limited studies on the evaluation of clinically relevant tack-cure scenarios for polymerization of dual-cure cements.[2,3,17] Therefore, the aim of this study was to evaluate the effect of tack cure on the polymerization shrinkage (PS) of resin-based luting cements. The null hypothesis was that tack cure would not affect the PS of resin-based luting cements.

METHODS AND MATERIALS

One conventional composite resin cement, Duo-Link (Duolink); two self-adhesive resin cements (SARCs), RelyX U200 (U200) and G-CEM LinkAce (GCem); and one resin-modified glass ionomer cement (RMGIC), RelyX Luting Plus (Luting+) were used for measuring the PS in light-cure mode (LC group), self-cure mode (SC group), and two tack-cure modes that were light cured (TC-LC group) or self-cured (TC-SC group) after tack cure (Table 1). Duolink and GCem were of the automix type, whereas U200 and Luting+ were of the clicker type. For the clicker type, the cement was dispensed on a mixing pad and hand mixed with a plastic spatula. Mixed cement was delivered into the C-R syringe (Needle Tip, Seil Global, Busan, Korea) and plugged and loaded into a gun. A volume of 14 mm^3 of each cement was measured with a flat 3-mm thick stainless-steel mold with a hole of 2.44 mm diameter, which was placed on a glass slide. After filling the mold to the upper limit, it was carefully lifted from the glass slide,

Table 1: *Materials Used in This Study and Tack-Cure Time for Each Material*

Group	Cement Type	Product (Lot No.)	Composition	Tack Cure Time, s
Duolink	Composite resin cement	Duo-Link Trans, Bisco, Schaumberg, IL, USA (1600006736)	Base: Bis-GMA, TEGDMA, glass filler Catalyst: Bis-GMA, TEGDMA, glass filler Filler load: 38 vol%, 61 wt%	2
U200	Self-adhesive resin cement	RelyX U200 TR, 3M, Neuss, Germany (630798)	Base paste: fiberglass, phosphoric acid esters methacrylate, TEGDMA, silica treated silane, sodium persulfate Catalyst paste: fiberglass, substitute dimethacrylate, silane-treated silica, p-toluenesulfonate sodium, and calcium hydroxide 70 wt%, 50 vol%	1
GCem	Self-adhesive resin cement	G-CEM LinkAce A2, GC, Tokyo, Japan (1610111)	Paste A: Fluoro-alumino-silicate glass, UDMA, dimethacrylate, silicon dioxide, initiator, inhibitor Paste B: Silicon dioxide, UDMA, dimethacrylate, initiator, inhibitor 55.3 wt%	1
Luting+	Resin-modified glass ionomer cement	RelyX Luting Plus, 3M, St Paul, MN, USA (N820543)	Paste A: Silane-treated filler, water, 2-hydroxyethyl methacrylate, silane-treated silica, 4-(dimethylamino)-benzene-ethanol, titanium dioxide Paste B: Silane-treated ceramic, copolymer of acrylic and itaconic acids, 2-hydroxyethyl methacrylate, water, glycerol 1,3 dimethacrylate 1-5%, potassium persulfate, potassium diphosphate, glyceryl methacrylate, 2,6 di-tert-butyl-P-cresol (BHT)	5

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

leaving the cement on the glass slide due to its flow. When there was a cement remnant inside the mold, a 0.5-mm-thick metal wire was used to remove and incorporate the remnant to the specimen. Then, the cement was pressed between the glass slide and a flexible cover glass (Marienfeld, Lauda-Koenigshofen, Germany) with the help of a 0.5-mm-diameter metal orthodontic wire, resulting in a specimen size of 0.5 mm in thickness and 6 mm in diameter (n=7). The glass slide was positioned on the metal shelf of a PS-measuring device (u-BioMechanics, IB Systems, Seoul, Korea), so that the specimen was located in the center of a blue light-emitting diode (LED) light (9 mm in diameter, 500 mW/cm^2 total irradiance) mounted in the middle of the metal shelf. The distance from the LED light to the specimen was 3 mm (glass slide of 1 mm + distance from the LED light source to the upper surface of 2 mm). Then, the linear variable differential transformer (LVDT) probe was set to touch the cover glass and adjusted to zero. On curing of the cement, the LVDT probe measured the real-time axial PS for 1600 seconds at a rate of 10 data points/s.[18] Baseline data were obtained for 20 seconds before tack cure or light cure to check the consistency of data collection before PS measurement. Also, 20 seconds could correspond to the time for a clinician to apply the cement to inlays. In the LC group, the specimen was light cured for 20 seconds. In the tack-cure groups, the specimen was tack cured for either one second, two seconds, or five seconds, respectively, according to the manufacturer's instructions. The cement was then light cured for 20 seconds in the TC-LC group after a two-minute delay, which simulated the time to remove the excess cement in the clinical situation. In the TC-SC group, the cement was left to self-cure after tack cure. In the SC group, the specimen was left to self-cure without tack cure or light cure.

Data acquired at 1600 seconds were analyzed with two-way analysis of variance (ANOVA; SPSS 23, SPSS, Chicago, IL, USA) to examine two factors, cure mode and cement, followed by post hoc comparisons with Tukey honestly significant difference (HSD) test. To investigate the effect of tack cure on light cure or self-cure, data were analyzed with an independent-samples *t*-test with tack cure as the variable. In addition, the PS data of each cement were analyzed with one-way ANOVA to examine either the cure mode factor or the time factor followed by Tukey HSD. The significance level was 5%.

RESULTS

Two-way ANOVA showed significant effects for cure mode factor, cement factor, and their interaction ($p<0.001$; Tables 2 and 3) on PS data at 1600 seconds. Independent-samples *t*-test showed that tack cure had a negative effect on self-cure in Duolink and Luting+ ($p<0.05$; Table 4). One-way ANOVA showed significant effects for cure mode factor and time factor on the PS data of each cement ($p<0.05$; Tables 5-8).

Table 2: *Summary of Two-Way ANOVA for Main Factors (Cement and Cure Mode) and Their Interactions for Polymerization Shrinkage of Resin-Based Luting Cements*

Source of Variation	Sum of Squares	*df*	Mean Square	*F*	Significance
Cement	23.328	3	7.776	402.147	<0.001
Cure mode	2.702	3	0.901	46.586	<0.001
Cement × cure mode	4.376	9	0.486	25.149	<0.001
Error	1.856	96	0.019		
Total	1961.349	112			

Regarding cure mode, PS of the LC or TC-LC group was higher than that of the SC group, which was higher than that of the TC-SC group ($p<0.05$). For Duolink, PS in the SC group was significantly lower than that in the other groups at 200 seconds. However, at 1600 seconds, PS was significantly lower in the TC-SC group, while there was no significant difference in PS among the LC, TC-LC, and SC groups (Figure 1a). Accounting for this, the PS rate in the SC group was higher between 200 seconds and 400 seconds than between 0 and 200 seconds only for Duolink (Figure 2a). For U200, PS was the highest in the LC group, followed by that in the TC-LC, TC-SC, and SC groups at 200 seconds ($p<0.05$). However, at 1000 seconds and thereafter, U200 showed a significantly higher PS in the LC or TC-LC groups compared with that in the SC or TC-SC groups, without significant difference between light-cure groups or between self-cure groups (Figure 1b). The PS or PS rate of GCem was not significantly affected by tack cure or cure mode (Figures 1c and 2c). For Luting+, PS was the highest in the TC-LC group, followed by that in the LC, TC-SC, and SC groups at 200 seconds ($p<0.05$). At 800 seconds and thereafter, however, PS was higher in the SC group than that in the TC-SC group ($p<0.05$). The SC, TC-LC, and LC groups showed no significant difference in PS (Figure 1d). Regarding cements, Luting+ showed the highest PS, followed by GCem, Duolink, and U200, respectively, with significant differences (Table 3).

DISCUSSION

The PS of resin-based luting cements is frequently identified as an important cause of the failure of indirect restorations.[19] Also, as a strong positive correlation has been observed between PS and DC,[20,21] PS is considered a feasible alternative to the conventional infrared method of measuring DC.[22] Therefore, the extent of PS in this study could be used to determine DC indirectly within the same material. In other words, high or low PS could be regarded as high or low DC within a resin-based luting cement.

As the independent-samples *t*-test showed that tack cure had a negative effect on self-cure in Duolink and Luting+, the null hypothesis was partially rejected. The short tack-cure procedure with a low radiant exposure increased the PS abruptly at the beginning of polymerization in all cements. It could be speculated that tack cure induced polymer growth centers, although such induction was relatively less than that induced by a final light cure.[12] Accordingly, the increased viscosity of cements entrapped activated free radicals and unreacted monomers as well as self-polymerization promoters needed for self-cure, which is known to usually have a lower polymerization rate than light cure.[2,23] Therefore, the PS of Duolink and Luting+ was lower in the TC-SC groups compared with that in the SC groups. However, GCem and U200 did not show any significant difference in PS between the TC-SC and SC groups.

Table 3: *Mean Polymerization Shrinkage (%±SD) of Resin-Based Luting Cements When Light Cured or Self-Cured With or Without Tack Cure at 1600 Seconds*[a]

	LC	TC-LC	TC-SC	SC	Pooled Average
Duolink	4.29 ± 0.11 Ab	4.23 ± 0.20 Ab	3.89 ± 0.05 Bc	4.14 ± 0.11 Ac	4.14 ± 0.20 c
U200	3.95 ± 0.07 Ac	3.89 ± 0.08 Ac	3.04 ± 0.10 Bd	2.96 ± 0.15 Bd	3.46 ± 0.48 d
GCem	4.28 ± 0.08 Ab	4.23 ± 0.15 Ab	4.19 ± 0.15 Ab	4.35 ± 0.13 Ab	4.26 ± 0.14 b
Luting+	4.72 ± 0.20 ABa	4.76 ± 0.14 ABa	4.57 ± 0.22 Ba	4.90 ± 0.15 Aa	4.74 ± 0.21 a
Pooled average	4.31 ± 0.30 A	4.28 ± 0.35 A	3.92 ± 0.59 C	4.09 ± 0.73 B	

Abbreviations: Duolink, Duo-Link; GCem, G-CEM LinkAce; LC, light cure; Luting+, RelyX Luting Plus; SC, self-cure; TC, tack cure; U200, RelyX U200.

[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD* ($p<0.05$).

Table 4: *Mean Polymerization Shrinkage (%±SD) of Resin-Based Luting Cements When Light Cured or Self-Cured at 1600 Seconds With Tack Cure as the Variable*[a]

Cement	Cure Mode	With Tack Cure	Without Tack Cure
Duolink	Light cure	4.23 ± 0.20 A	4.29 ± 0.11 A
	Self-cure	3.89 ± 0.05 B	4.14 ± 0.11 A
U200	Light cure	3.89 ± 0.08 A	3.95 ± 0.07 A
	Self-cure	3.04 ± 0.10 A	2.96 ± 0.15 A
GCem	Light cure	4.23 ± 0.15 A	4.28 ± 0.08 A
	Self-cure	4.19 ± 0.15 A	4.35 ± 0.13 A
Luting+	Light cure	4.76 ± 0.14 A	4.72 ± 0.20 A
	Self-cure	4.57 ± 0.22 B	4.90 ± 0.15 A

Abbreviations: Duolink, Duo-Link; GCem, G-CEM LinkAce; RelyX Luting Plus; U200, RelyX U200; Luting+.

[a] *Mean values followed by different uppercase letters (row) are significantly different by independent-samples* t*-test (*$p<0.05$*) within a cure mode.*

Differences in compositions of cements, including the initiator and catalyst system, might have caused the difference in the PS of these dual-cure cements.[10,17]

On the other hand, tack cure had no significant effect on light cure in all resin-based luting cements. This result was consistent with a previous study showing that light cure after tack cure did not negatively affect the microhardness of dual-cure cements.[17] In addition, a study on dual-cure cements reported that there was no significant difference in DC between the immediate light-cure group and two-minute delayed light-cure group, which had the same delay time as in the current study, except for the tack-cure procedure.[7] Therefore, it could be speculated that radiant exposure of final light cure in the TC-LC group might have induced enough growth centers to result in a PS similar to the LC group[12] despite the tack cure procedure and a two-minute delay time.

Duolink, a conventional composite resin cement, showed higher PS in the TC-SC group than in the SC group at the beginning of polymerization because a short light-cure reaction due to tack cure was added to the self-cure reaction. However, the PS rate in the SC mode increased thereafter, resulting in higher PS in the SC group than in the TC-SC group. Thus, it could be speculated that tack cure induced premature polymer growth centers before the self-cure PS rate was dominant and resulted in the interference of the self-cure reaction. In addition, the time to the plateau of PS decreased in the TC-SC group. This could also be a sign that the tack cure interfered with the self-cure reaction, resulting in premature termination of polymerization. For the light-cure groups, Figure 1a shows that the PS curves of LC and TC-LC groups were lined up exactly if the PS curve of the LC group was shifted 122 seconds (two seconds for tack cure and two minutes for delay time) on the x-axis. PS after 20-second light cure was 3.32% ± 0.12% at 40 seconds in the LC group and 3.35% ± 0.14% at 162 seconds in the TC-LC group. The final comparable PS values for both groups were 4.28% ± 0.11% at 1478 seconds in the LC group and 4.23% ± 0.20% at 1600 seconds in the TC-LC group. Data at those time points showed no significant difference between the LC and TC-LC groups ($p>0.05$, independent-samples t-test). Therefore, the tack cure and the self-cure reaction during the two-minute delay time seemed to have no significant effect on the light-cure reaction. Similar to this study, Pereira and others[9] reported that there was no significant difference in the extent of polymerization among light-cure, self-cure, and five-minute delayed light-cure modes with Duolink. The extent of polymerization in the five-minute delayed light-cure mode could be explained with the result of this study. As the PS rate of the SC group was the highest between 200 seconds and 400 seconds, the five-min delayed light cure might not have interfered with the self-cure reaction because the cement was light cured when the self-cure PS rate was high.

U200, an SARC, showed significantly higher PS in the light-cure groups than in the self-cure groups,[2,6,24] regardless of tack cure. In addition, the PS of U200 was the lowest among the dual-cure cements tested. Previous studies reported that water produced during neutralization of acidic monomers in SARCs could interfere with the polymerization reaction.[25] In addition, free radicals formed by acidic monomers are less reactive than free radicals formed by unmodified dimethacrylate monomers. This might be a reason for the lower PS of this cement especially in the self-cure mode when the PS rate is low.[2,24,26] Regarding the self-cure groups, although tack cure increased PS initially, the difference in PS between the TC-SC and SC groups was not significant after 1000 seconds. As the self-cure PS rate was the highest when the cement was tack cured, namely, between 0 and 200 seconds, it seemed that tack cure did not affect the self-cure reaction (Figure 2b). In addition, tack cure extended the time to reach the plateau of PS from 1200 seconds in the SC group to 1400 seconds in TC-SC group. Since the self-cure reaction had low PS and PS rate, it could be assumed that the light-cure reaction by the tack-cure procedure was added to the self-cure reaction to extend the time for polymerization. However, radiant exposure of the tack-cure procedure was not high

Table 5: *Polymerization Shrinkage (%±SD) of Duolink for 1600 Seconds at 200-Seconds Intervals*[a]

Group	**Time, s**					
	200	**400**	**600**	**800**	**1000**	**1200**
LC	3.89 ± 0.12 Ca	4.04 ± 0.12 BCa	4.13 ± 0.11 ABa	4.19 ± 0.11 ABa	4.23 ± 0.12 ABa	4.26 ± 0.12 Aa
TC-LC	3.63 ± 0.16 Ca	3.91 ± 0.16 BCa	4.01 ± 0.17 ABa	4.08 ± 0.17 ABa	4.13 ± 0.18 ABa	4.17 ± 0.19 ABab
TC-SC	2.41 ± 0.36 Eb	2.98 ± 0.25 Db	3.34 ± 0.16 Cb	3.54 ± 0.11 BCc	3.68 ± 0.08 ABc	3.77 ± 0.07 ABc
SC	0.97 ± 0.16 Fc	2.89 ± 0.11 Eb	3.51 ± 0.10 Db	3.78 ± 0.10 Cb	3.92 ± 0.10 BCb	4.02 ± 0.10 ABb

Abbreviations: Duolink, Duo-Link; LC, light cure; SC, self-cure; TC, tack cure.

[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

Table 6: *Polymerization Shrinkage (%±SD) of U200 for 1600 Seconds at 200-Second Intervals*[a]

Group	**Time, s**					
	200	**400**	**600**	**800**	**1000**	**1200**
LC	3.48 ± 0.07 Fa	3.66 ± 0.07 Da	3.75 ± 0.06 CDa	3.81 ± 0.07 BCa	3.86 ± 0.07 ABCa	3.90 ± 0.07 ABa
TC-LC	3.17 ± 0.08 Db	3.55 ± 0.07 Ca	3.69 ± 0.08 Ba	3.77 ± 0.07 ABa	3.82 ± 0.08 Aa	3.85 ± 0.08 Aa
TC-SC	1.55 ± 0.07 Gc	2.03 ± 0.08 Fb	2.33 ± 0.09 Eb	2.55 ± 0.09 Db	2.71 ± 0.10 Cb	2.85 ± 0.10 BCb
SC	0.90 ± 0.18 Fd	1.67 ± 0.16 Ec	2.10 ± 0.15 Dc	2.38 ± 0.15 Cc	2.58 ± 0.15 BCb	2.74 ± 0.15 ABb

Abbreviations: LC, light cure; SC, self-cure; TC, tack cure; U200, RelyX U200.

[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

Table 7: *Polymerization Shrinkage (%±SD) of GCem for 1600 Seconds at 200-Second Intervals*[a]

Group	**Time, s**					
	200	**400**	**600**	**800**	**1000**	**1200**
LC	3.28 ± 0.04 Fa	3.64 ± 0.05 Ea	3.84 ± 0.07 Da	3.98 ± 0.08 Ca	4.09 ± 0.08 BCa	4.17 ± 0.08 ABa
TC-LC	3.26 ± 0.13 Ea	3.66 ± 0.14 Da	3.85 ± 0.15 CDa	3.98 ± 0.15 BCa	4.06 ± 0.15 ABCa	4.13 ± 0.15 ABa
TC-SC	2.96 ± 0.15 Eb	3.47 ± 0.15 Da	3.73 ± 0.15 Ca	3.89 ± 0.14 BCa	4.01 ± 0.14 ABa	4.08 ± 0.15 ABa
SC	2.93 ± 0.20 Eb	3.55 ± 0.16 Da	3.83 ± 0.14 Ca	4.01 ± 0.14 BCa	4.13 ± 0.13 ABa	4.22 ± 0.13 ABa

Abbreviations: GCem, G-CEM LinkAce; LC, light cure; SC, self-cure; TC, tack cure.

[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

Table 8: *Polymerization Shrinkage (%±SD) of Luting+ for 1600 Seconds at 200-Second Intervals*[a]

Group	**Time, s**					
	200	**400**	**600**	**800**	**1000**	**1200**
LC	3.89 ± 0.19 Cb	4.36 ± 0.18 Ba	4.52 ± 0.20 ABab	4.60 ± 0.21 ABab	4.64 ± 0.20 ABab	4.66 ± 0.20 ABab
TC-LC	4.36 ± 0.13 Ba	4.57 ± 0.14 ABa	4.64 ± 0.15 Aa	4.68 ± 0.15 Aab	4.71 ± 0.15 Aab	4.73 ± 0.15 Aab
TC-SC	3.03 ± 0.09 Cc	4.02 ± 0.16 Bb	4.30 ± 0.18 ABb	4.43 ± 0.20 Ab	4.49 ± 0.20 Ab	4.52 ± 0.21 Ab
SC	2.52 ± 0.30 Dd	4.07 ± 0.15 Cb	4.52 ± 0.12 Bab	4.69 ± 0.13 ABa	4.78 ± 0.14 ABa	4.83 ± 0.14 Aa

Abbreviations: LC, light cure; Luting+, RelyX Luting Plus; SC, self-cure; TC, tack cure.

[a] *Mean values followed by different uppercase letters (row) or lowercase letters (column) are significantly different by Tukey HSD ($p<0.05$).*

enough to affect the final PS at 1600 seconds.[27] Regarding the light-cure groups, the relationship between the LC and TC-LC groups seemed to be just like that of Duolink: exactly the same PS curves with a 121-second (one second for tack cure and two minutes for delay time) time difference (Figure 1b). After 20 seconds of light cure, the PS was 2.66% ± 0.10% in the LC group at 40 seconds and 2.74% ± 0.08% in the TC-LC group at 161 seconds. The final comparable PS values for both groups were 3.94% ±

Table 5: *Extended.*

Group	Time, s	
	1400	1600
LC	4.28 ± 0.11 Aa	4.29 ± 0.11 Aa
TC-LC	4.20 ± 0.20 ABab	4.23 ± 0.20 Aa
TC-SC	3.83 ± 0.05 ABc	3.89 ± 0.05 Ab
SC	4.08 ± 0.11 ABb	4.14 ± 0.11 Aa

Table 6: *Extended.*

Group	Time, s	
	1400	1600
LC	3.93 ± 0.07 Aa	3.95 ± 0.07 Aa
TC-LC	3.87 ± 0.08 Aa	3.89 ± 0.08 Aa
TC-SC	2.95 ± 0.11 ABb	3.04 ± 0.10 Ab
SC	2.86 ± 0.15 Ab	2.96 ± 0.15 Ab

Table 7: *Extended.*

Group	Time, s	
	1400	1600
LC	4.23 ± 0.08 Aa	4.28 ± 0.08 Aa
TC-LC	4.18 ± 0.15 ABa	4.23 ± 0.15 Aa
TC-SC	4.14 ± 0.15 Aa	4.19 ± 0.15 Aa
SC	4.30 ± 0.12 Aa	4.35 ± 0.13 Aa

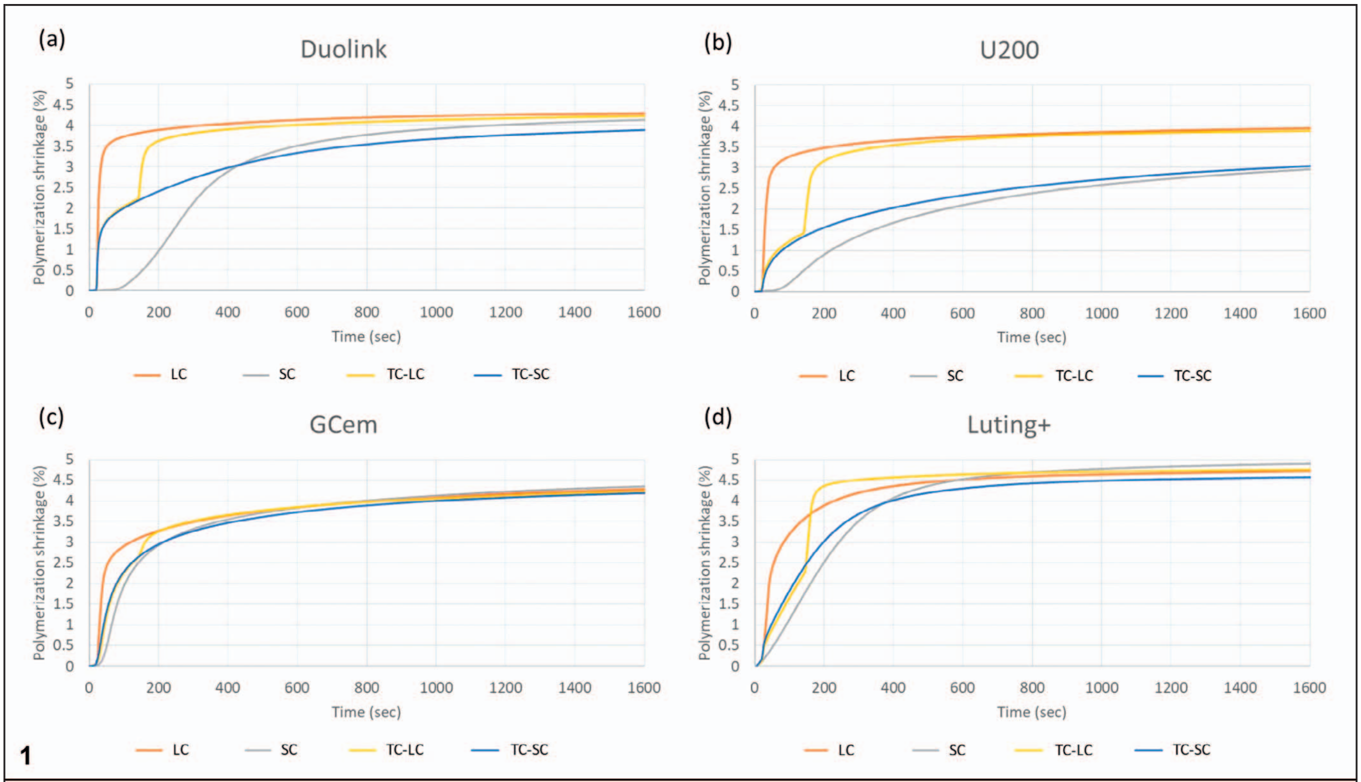
Table 8: *Extended.*

Group	Time, s	
	1400	1600
LC	4.70 ± 0.20 Aab	4.72 ± 0.20 Aab
TC-LC	4.75 ± 0.15 Aab	4.76 ± 0.14 Aab
TC-SC	4.55 ± 0.22 Ab	4.57 ± 0.22 Ab
SC	4.87 ± 0.15 Aa	4.90 ± 0.15 Aa

0.07% in the LC group at 1479 seconds and 3.89% ± 0.08% in the TC-LC group at 1600 seconds. At those time points, data showed no significant difference in PS between the LC and TC-LC groups ($p > 0.05$, independent-samples t-test). Thus, it could be stated that the tack cure and the self-cure reaction during the two-minute delay time had no significant effect on the final light-cure reaction of this cement. The time to reach the plateau of PS was reduced to 800 seconds in the TC-LC group from 1000 seconds in the LC group. The increase in total light-cure time due to additional light exposure of the tack-cure procedure might be one reason for such a result.[3]

GCem, another SARC, showed no significant difference in PS regardless of tack cure or cure mode. Figures 1c and 2c showed that PS and PS rate in the SC group were as high as those in the LC group, unlike U200. As the self-cure PS rate was the highest when the cement was tack cured, namely, between 0 and 200 seconds, it seemed that tack cure did not affect the self-cure process. Therefore, it could be assumed that the self-cure reaction was not interfered by tack cure because of the high initial PS rate of the SC group. The relationship between the LC and TC-LC groups was different from that of Duolink or U200. High self-cure PS rate at the time of tack cure and during the two-minute delay time seemed to be one reason for the difference. Nevertheless, there was no significant difference in PS between the LC and TC-LC groups. The time to reach the plateau of PS was decreased in the TC-LC group compared with the LC group, probably because of the additional light exposure of the tack-cure procedure. One study reported that the effect of light cure was compensated by self-cure within five minutes, showing the high self-cure capacity of this cement.[28] The manufacturer has claimed that they incorporated a metal ion accelerator to increase the free radical in the self-cure reaction and called it the "high-efficiency triple initiator self-cure technology."[29]

Duolink showed a PS difference between the TC-SC and SC groups, while GCem or U200 showed no significant PS difference between these groups. The PS rates of Duolink between 0 and 200 seconds and between 200 seconds and 400 seconds in the TC-SC group were 2.41% and 0.57%, respectively, compared with PS rates in the SC group, which were 0.97% and 1.92%, respectively. Thus, tack cure increased the PS rate between 0 and 200 seconds but decreased the PS rate between 200 and 400 seconds. This reduction in self-cure PS rate due to tack cure contributed to lower PS in the TC-SC group. On the other hand, PS rates of U200 between 0 and 200 seconds and between 200 and 400 seconds in the TC-SC group were 1.55% and 0.48%, respectively, compared with PS rates in the SC group, which were 0.90% and 0.77%, respectively. Although tack cure increased the PS rate between 0 and 200 seconds and decreased the PS rate between 200 and 400 seconds, the PS rate at those time periods



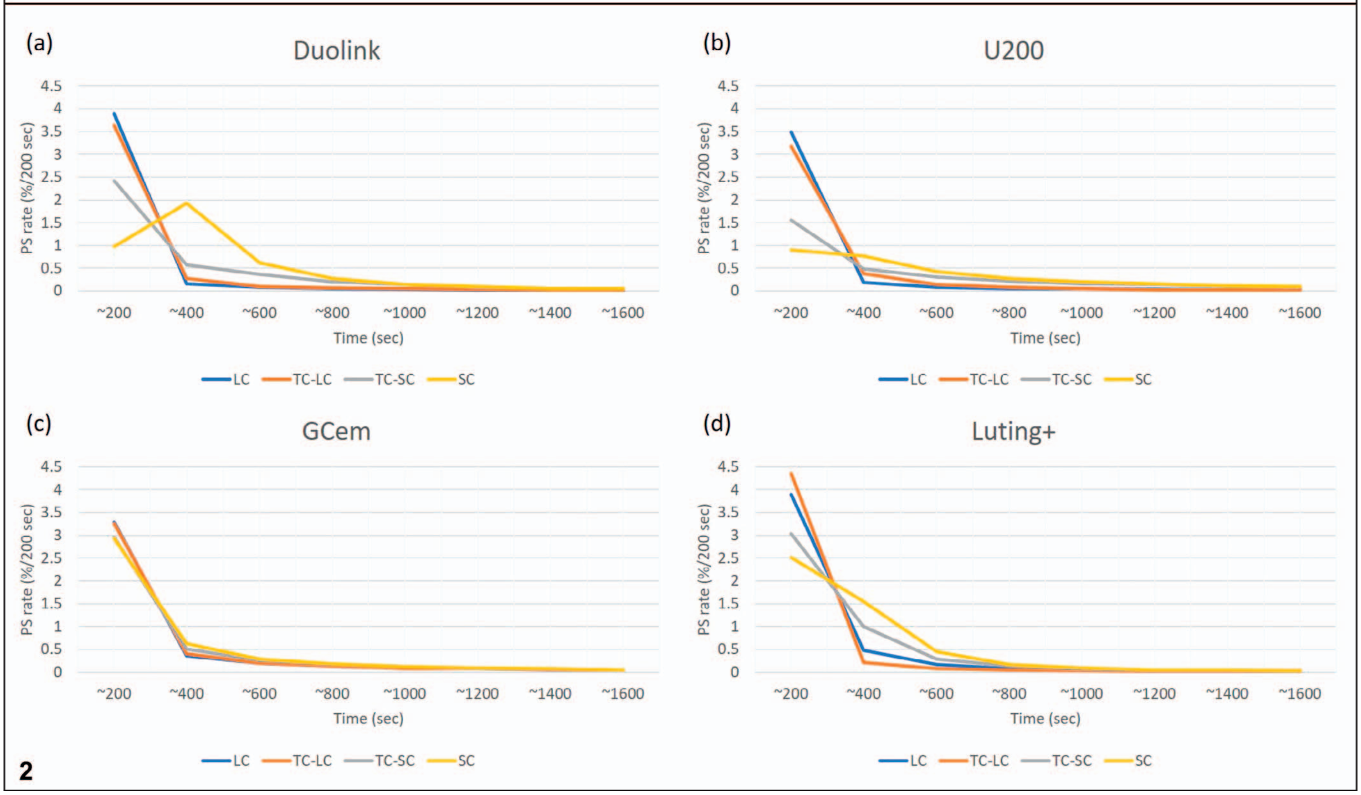


Figure 1. *Polymerization shrinkage of resin-based luting cements according to cure mode. (a): Duo-Link. (b): RelyX U200. (c): G-CEM LinkAce. (d): RelyX Luting Plus. LC, light cure; SC, self-cure; TC, tack cure.*

Figure 2. *Polymerization shrinkage rate (%/200 seconds) of resin-based luting cements. (a): Duo-Link. (b): RelyX U200. (c): G-CEM LinkAce. (d): RelyX Luting Plus. LC, light cure; SC, self-cure; TC, tack cure.*

was lower than those of Duolink, resulting in no significant difference between those groups and lower final PS for both self-cure groups. The PS rates of GCem between 0 and 200 seconds and between 200 and 400 seconds in the TC-SC group were 2.96% and 0.51%, respectively, similar to PS rates in the SC group, which were 2.93% and 0.62%, respectively. Therefore, it seemed that tack cure did not affect the self-cure reaction, as both cure modes exhibited a high PS rate and high final PS. Thus, the extent of decreased self-cure PS rate due to tack cure seemed to be one reason for the decreased PS of the TC-SC groups for the composite resin cements. In addition, if the dual-cure cements were tack cured when the self-cure PS rate was high, as in case of GCem and U200, the tack cure did not interfere with the self-cure reaction. However, if the dual-cure cements were tack cured before the self-cure PS rate reached its maximum, like in case of Duolink, the premature polymerization due to tack cure seemed to interfere with the self-cure reaction. Further study is needed to verify the relation between the timing of tack cure and the self-cure PS rate in detail.

Luting+, an RMGIC, showed higher PS than composite resin cements, similar to other studies.[30,31] The main setting of an RMGIC is achieved by an acid-base reaction, which has been reported to have higher setting shrinkage than composite resin.[31] In addition, HEMA contained in the RMGIC is a hydrophilic resin that also exhibits high PS.[32] Therefore, PS should be referred to as setting shrinkage in RMGIC. However, the term *polymerization shrinkage* was used throughout the text as in previous studies.[31,33] This is probably because PS is part of the setting shrinkage of RMGIC, making it difficult to distinguish between PS and shrinkage from an acid-base reaction. The highest PS was observed in the SC group, and there was a significant difference in PS between the SC and TC-SC groups. However, there was no significant difference in PS among the LC, TC-LC, and SC groups, although the manufacturer has claimed that this cement is basically a self-cure material with a five-second tack-cure option. In the self-cure groups, the PS rates of Luting+ between 0 and 200 seconds and between 200 and 400 seconds in the TC-SC group were 3.03% and 0.99%, respectively, compared with the PS rates in the SC group, which were 2.52% and 1.55%, respectively. Thus, tack cure seemed to interfere with the self-cure reaction of resin components as well as the acid-base reaction of glass ionomer components between 200 and 400 seconds, since the light-cure reaction and acid-base reaction could compete with each other and inhibit each other during the early RMGIC setting.[34] To support this, the acid-base reaction of an RMGIC in the SC mode was reported to show an exotherm peak at 4.2 minutes after mixing,[34] which is in the range of 200 seconds and 400 seconds. The time to reach the plateau of PS was reduced in the TC-SC group compared with that in the SC group, resulting in premature termination of polymerization. For the light-cure groups, tack cure did not affect the final PS, although the PS curves were different from those of resin cements. At 200 seconds, the PS in the TC-LC group was significantly higher than that in the LC group only for Luting+ (Figure 1d). One reason could be that the total light-cure time in the TC-LC group was 25 seconds, which was the longest light-cure time among the test groups. The longer tack-cure time might have resulted in a more crosslinked polymer network with higher exothermic heat, which accelerated the acid-base reaction and self-cure reaction to even earlier times. Nevertheless, it seemed that the light-cure time of 20 seconds in the LC group was enough to light cure the RMGIC, as the final PS showed no significant difference between the LC and TC-LC groups. As PS was not significantly different among the LC, TC-LC, and SC groups, Luting+ could be referred to as a dual-cure cement in terms of PS.

CONCLUSION

For the tested cements, tack cure decreased the PS of Duolink and Luting+ when they were self-cured after tack cure. However, light cure after tack cure showed no significant difference in PS compared with light cure alone. Duolink showed lower PS in the TC-SC group compared with the other cure groups. Luting+ showed lower PS in the TC-SC group than in the SC group. On the other hand, U200 showed lower PS in the self-cure groups compared with the light-cure groups regardless of tack cure. GCem was not affected by tack cure or cure mode.

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Conflict of Interest

The authors of this article 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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