Effect of Simulated Tooth Temperature on the Degree of Conversion of Self-adhesive Resin Cements Exposed to Different Curing Conditions

CAG Arrais • CL Chagas • A Munhoz
M Oliveira • AF Reis • JA Rodrigues

Clinical Relevance

Self-adhesive resin cements present a higher degree of conversion and faster polymerization kinetics when exposed to tooth temperature rather than to room temperature. Clinicians may expect differences in product setting time and some physical properties compared with what manufacturers report.

*Cesar AG Arrais, DDS, MS, PhD, Ponta Grossa State University, Department of Operative Dentistry, Ponta Grossa, Brazil

Carolina Levatti Chagas, DDS, MS, University of Guarulhos, Department of Operative Dentistry, Guarulhos, Brazil

Aurea Munhoz, BMSc, University of Guarulhos, Department of Operative Dentistry, Guarulhos, Brazil

Michele Oliveira, MS, DDS, University of Guarulhos, Department of Operative Dentistry, Guarulhos, Brazil

Andre F Reis, DDS, MS, PhD, University of Guarulhos, Department of Operative Dentistry, Guarulhos, Brazil

José Augusto Rodrigues, DDS, MS, PhD, University of Guarulhos, Department of Restorative Dentistry, Guarulhos, Brazil

*Corresponding author: Av. Carlos Cavalcanti, 4748, Uvaranas, Ponta Grossa, Parana 08030-900, Brazil; r-mail: cesararrais@yahoo.com.br

SUMMARY

Objectives: This study evaluated the degree of conversion (DC) of two commercial, self-adhesive resin cements (SARCs) using Fourier transform infrared analysis (FTIR) polymerized at simulated prepared tooth surface temperatures and under different curing conditions.

Materials and Methods: RelyX U100 (U100, 3M ESPE) and Maxcem Elite (MX, Kerr Corporation) were mixed at 25°C and applied to the surface of a horizontal attenuated total reflectance (ATR) unit, which was near room temperature (RT, control) (25°C) or heated to simulate prepared tooth surface temperatures (28°C and 32°C) and then attached to an infrared spectrometer. The products were polymerized using one of three conditions: direct light exposure through a glass slide (DLE), exposure through a 1.5-mm thick ceramic disc overlay

(CO) (A2 shade, IPS e.max, Ivoclar Vivadent), or self-curing (SC). FTIR spectra were recorded for 12 minutes (1 spectrum/s, 16 scans/spectrum, resolution 4 cm^{-1}) immediately after application to the ATR. The DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios before and 12 minutes after curing, as well as during each 1-second interval. DC data (n=7) were analyzed by two-way analysis of variance and Tukey's post-hoc test (*p*=0.05).

Results: Both simulated tooth temperatures significantly increased DC in all groups of MX and in the CO and SC groups of U100 compared with the RT control. For MX, the self-cure groups exposed to tooth temperatures showed DC values similar to those of the CO groups. For U100, the CO groups showed higher DC values than SC groups regardless of temperature. Time-based conversion profiles ranged according to product, temperature, and curing mode.

Conclusions: Causing SARCs to polymerize at simulated tooth temperatures increases DC of SARCs compared with room-temperature curing values, mainly in the SC mode.

INTRODUCTION

The success of an indirect restoration relies on bonding materials to ensure an optimum link between the prepared tooth and the indirect restorative material. Within this context, dual-cured resin cements have been the choice of most clinicians to cement esthetic indirect restorations. To reduce technique sensitivity due to multistep bonding procedures involved in cementation techniques, manufacturers have developed the so-called self-adhesive resin cements (SARCs). These materials were introduced in the market as a new subgroup of resin cements that require no pretreatment of tooth substrate before its application.[1,2]

Similar to conventional dual-cured resin cements, SARCs depend on optimal polymerization to exhibit clinically acceptable mechanical properties and bond strength.[3-5] Because these products use similar mechanisms to initiate polymerization compared with conventional resin cements, their polymerization mode is crucial to ensure an optimal degree of conversion.[6] In this regard, the self-curing mode of such products is generally less effective than the light-activated mode,[6] and little information is available in the literature regarding the effects of light attenuation due to the presence of an indirect

restoration interposed between the light-curing unit tip and the SARC layer.[7] This issue raises some concern, however, because the presence of a 1.5-mm-thick indirect restoration can cause a severe reduction of 80% to 90% in light intensity.[8,9]

Most comparisons between curing conditions and monomer conversion of SARCs are based on *in vitro* tests performed at room temperature (approximately 25°C).[6,10-12] Conversely, SARCs are applied directly to a prepared tooth, where temperature ranges from 27.8°C to 32.0°C, depending on the tooth,[13-15] and not at 37°C, as many people assume. For instance, if SARCs are used to cement indirect restorations in anterior teeth, these products will be in contact with tooth surfaces at approximately 28.0°C, while the tooth surfaces of premolars and molars will warm SARCs to approximately 32°C.[15] Previous studies have demonstrated that increased temperatures of 50°C or 60°C result in higher monomer conversion in resin cements and resin composites, regardless of the curing condition.[16,17] Therefore, it would be reasonable to expect some effect of tooth temperature in the monomer conversion of SARCs. In this regard, a previous study demonstrated that polymerization of SARCs at 37°C develops more shrinkage stress than it does at 23°C.[18]

Only a few studies have evaluated the effects of tooth temperature on SARCs, and all of them focused on bond-strength values, electron microscopy analysis, and shrinkage stress.[18-20] Although the authors attributed high strength values to elevated degree of conversion,[18-20] there is no study available in the literature directly evaluating the degree of conversion of SARCs with increased temperature.

The purpose of this study was to evaluate the effects of tooth temperatures on the degree of conversion of dual-cured SARCs exposed to different curing conditions: direct light exposure (DLE), light exposure attenuated by an indirect ceramic restoration, and activation by self-curing components with no light exposure involved. Two research hypotheses were addressed. The first hypothesis anticipated that when a ceramic restoration attenuates or totally blocks the curing light, the degree of conversion of self-adhesive cements increases as the temperature during polymerization approaches that of the clinically prepared tooth. The second research hypothesis was that, despite temperature during polymerization, the degree of conversion value when directly light-curing the SARC will not be significantly different from that attained when exposing the specimen through a porcelain overlay or when the specimen is allowed to totally self-cure without light.

Table 1:	*Brand, Composition, and Batch Number of SARCs Tested*	
Product (Manufacturer)	Composition (Batch Number)	
Rely X U100 (3M ESPE, St Paul, MN, USA)	Base: glass fiber, methacrylated phosphoric acid esters, dimethacrylates, silanated sílica, sodium persulfate	
	Catalyst: glass fiber, dimethacrylates, silanated sílica, p-toluene sodium sulfate, calcium hydroxide (367405)	
Maxcem Elite (Kerr Corporation, Orange, CT, USA)	Resin: multifunctional DMAs, GPDM, proprietary Redox initiators and photoinitiators.	
	Filler: barium, fluoroaluminosilicate, fumed silica (66 wt%) (423636)	
Abbreviations: GPDM, glycerophosphate dimethacrylate; DMAs, dimethacrylates.		

MATERIALS AND METHODS

Specimen Preparation

Two commercially available, dual-cure SARCs were tested: Rely X U100 (U100, 3M ESPE, St Paul, MN, USA) and Maxcem Elite (MX, Kerr Corp, Danbury, CT, USA). The products were selected because of their differences in composition (Table 1); only one uses calcium hydroxide to accelerate pH increase. As the product pH may have detrimental effects on the polymerization features of a resin-based material, it was expected that tooth temperature might have a different effect on the monomer conversion of a product with another pattern of pH increase caused by calcium hydroxide. A ceramic disc overlay (CO) (2-cm diameter, 1.5-mm thick; IPS e.max, A2 shade, Ivoclar Vivadent, Schaan, Liechtenstein) was selected to model an indirect restorative material through which irradiating light would pass.

Base and catalyst pastes of each product were equally dispensed on a glass plate and were hand-mixed using a metal spatula. The mixture was then applied to the horizontal diamond element of an attenuated total reflectance (ATR) unit attachment (Golden Gate, Specac, Woodstock, GA, USA) in the optical bench of a Fourier transform infrared spectrometer (Tensor Series, Bruker Optik GmbH, Ettlingen, Germany). For groups simulating tooth temperature, the diamond surface temperature was elevated to 28°C or 32°C using a custom-made heating device, and the surface temperature was constantly monitored using a K-type thermocouple (SmartMether, Novus, Porto Alegre, Brazil) during resin cement polymerization. Before resin placement, adhesive tape (3M ESPE) was placed around the diamond surface to act as a spacer, ensuring standard thickness for all specimens (100-120 µm).

The deposited cement was covered with a Mylar strip and polymerized using one of three curing modes. Specimens were exposed directly to light activation without any overlying restorative material (DLE or control group) for 20 seconds for MX and 40 seconds for U100 (manufacturers' instructions) to a blue light-emitting diode light-curing source emitting 1500 mW/cm^2 (Radii Plus, SDI Limited, Victoria, Australia). Seven replications were made for each test condition (n=7) based on previous studies using the same methodology.[6,21-23] To ensure delivery of consistent levels of irradiance during testing, the output of the light was continuously checked during testing, using a handheld dental curing radiometer (Cure Rite, Dentsply Caulk, USA). The distal end of the light-emitting guide was placed directly against a 1-mm-thick glass slide, positioned directly over top of the Mylar-covered resin specimen. When exposing cements to light by overlying a ceramic disc, the disc was placed directly between the Mylar sheet and the light-emitting guide end (Figure 1). In addition, specimens were allowed to self-cure without light exposure.

Degree of Conversion

Infrared (IR) spectra were collected between 1680 and 1500 cm^{-1} at a rate of one spectrum per second (16 scans per spectrum) at 4 cm^{-1} resolution. Data were counted from the moment the IR scan demonstrated that the resin was stabilized on the ATR
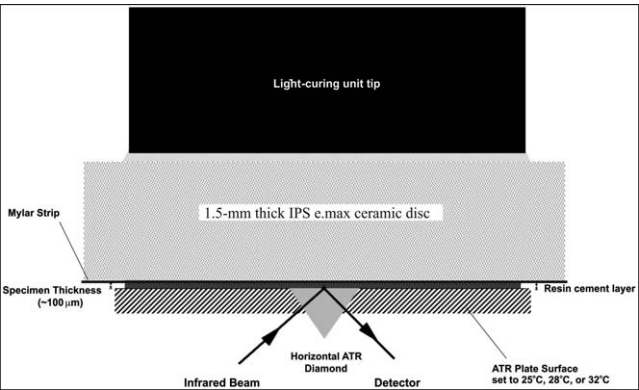

Figure 1. *Illustrative diagram demonstrating the interaction between the infrared beam and the specimen, as well as the position of the 1.5-mm thick ceramic disc and the light-curing unit tip.*

Table 2:	*Mean (SD) Direct Conversion Values of MX at Room (25°C) and Tooth Temperatures (28°C and 32°C)*[a]		
Temperature at Polymerization	Direct Light Exposure	Indirect Ceramic Disc Overlay	Self-curing
25°C	58.1 (2.7) Ba	52.4 (1.3) Cb	47.6 (1.8) Bc
28°C	66.2 (2.7) Aa	59.5 (2.4) Bb	61.8 (3.2) Aab
32°C	70.9 (2.0) Aa	66.2 (3.3) Aab	62.1 (1.4) Ab
[a] Mean *values with similar letters (upper case within column; lower case within row) are not significantly different* (p>0.05).			

surface and any overlying objects had been placed. Spectra were recorded continuously during each 1-second interval for 12 minutes to generate kinetic spectra of monomer conversion. Monomer conversion was calculated using standard methods that evaluated changes in the ratios of aliphatic-to-aromatic C=C IR absorption peaks (1636 cm^{-1}/1608 cm^{-1}) in the uncured and cured states,[24,25] according to the following equation:

$$DC = 1 - \frac{(C = C_{aliphatic}/C = C_{aromatic})\,polymer*100}{(C = C_{aliphatic}/C = C_{aromatic})\,monomer}$$

Where DC is the degree of conversion, $C=C_{aliphatic}$ corresponds to the IR absorbance of aliphatic double carbon bonds, and $C=C_{aromatic}$ is the IR absorbance of aromatic carbon bonds.

Before determining conversion, calibration graphs were made relating the absorbance ratios of known molar concentrations of aliphatic and aromatic C=C to their respective absorbance height ratios as previously established.[25] Conversion values among all curing modes were compared statistically within each product at 12 minutes from the time the resin cement was stabilized on the ATR surface. All polymerized specimens were carefully removed from the ATR plate and measured for thickness to the nearest 0.01 mm using a digital micrometer (Series 406; Mitutoyo America Corp, Aurora, IL, USA) to ensure similar thickness among all specimens.

Statistical Analyses

DC data were evaluated within each dual-cure resin product using a two-way analysis of variance (ANOVA) (factors: temperature [three levels] and curing condition [three levels]) followed by Tukey's post-hoc test. Direct comparison of conversion values between products was not made, because such comparisons can only be validly made when resins have the same chemical formulation. All testing was performed at a preset $\alpha=0.05$ using personal statistical software (SAS 8.0 for Windows; SAS Institute Inc, Cary, NC, USA). Post-hoc power analysis was performed for the statistical analysis of degree of conversion data using additional software (Statistics 19, SPSS Inc, IBM Company, Armonk, NY, USA).

RESULTS

Degree of Conversion

The study was adequately powered for both factors, temperature and curing mode (over 95%; $\alpha=0.05$). The two-way ANOVAs indicated that the interaction between curing mode and temperature was a significant factor in affecting DC for both MX ($p=0.0098$) and U100 ($p<0.00001$).

Tables 2 and 3 present DC of dual-cure SARCs exposed using the three curing conditions at three temperatures. Temperature increases from 25°C to 28°C resulted in a significantly higher DC in all groups of MX (Table 2), while a significant increase in DC of U100 (Table 3) was only observed in groups exposed to the CO condition and in the self-curing groups ($p<0.0001$). No significant increase in DC was observed in the DLE group when the temperature increased from 25°C to 28°C. U100 exposed at 32°C showed higher DC values than when it was exposed to lower temperatures ($p<0.00001$).

For MX, the effect of different curing conditions on DC was temperature-dependent. When exposed to tooth temperatures, the self-curing mode provided similar DC values to those when curing was through the CO. The self-polymerizing mode at 25°C led to

Table 3:	*Mean (SD) Direct Conversion Values of U100 Polymerized at Room (25°C) and Tooth Temperatures (28°C and 32°C)*[a]		
Temperature During Polymerization	Direct Light Exposure	Indirect Ceramic Disc Overlay	Self-curing
25°C	49.9 (1.0) Ba	45.1 (2.6) Cb	25.2 (3.7) Cc
28°C	50.7 (2.3) Ba	46.8 (1.0) Bb	29.5 (1.8) Bc
32°C	57.4 (0.9) Aa	47.9 (1.5) Ab	34.1 (1.6) Ac
[a] Mean values with similar letters (upper case within column; lower case within row) are not significantly different (p>0.05).			

lower DC values than did the attenuated light-activated mode (p=0.0427). On the other hand, the effects of curing conditions on DC values were not influenced by temperature when U100 was evaluated. The DLE groups demonstrated higher DC values than groups exposed to light attenuated by the ceramic overlay (p<0.0001). The self-cure mode led to the lowest DC values in all temperatures for this material.

Figures 2 and 3 show the polymerization kinetic of representative specimens, representing the pattern observed in most specimens regarding the effects of tooth temperature on the time-based conversion profiles of U100 and MX, respectively. All Figures included the comparison of these values to the 12-minute degree of conversion value from the DLE group at 25°C (dashed line) as a control. At 28°C and 32°C (Figure 2), time-based conversion changes in the CO condition of U100 exhibited polymerization kinetics similar to that obtained in the room-temperature DLE groups at 12 minutes. The polymerization kinetics of the CO groups exposed to tooth temperatures showed higher slopes during the initial stages of polymerization, indicating higher rates of conversion, until the light curing unit shut off, in comparison to the polymerization kinetics observed at room temperature, where the slopes of these time-based conversion values were much lower. Then, a slow, continual increase was noted in all CO groups. The slowest initial curing rate was noted for the self-cure only mode at room temperature, resulting in the lowest DC values. The effects of temperature were more evident in the self-cure groups, as the conversion rate at tooth temperatures was apparently much faster than that at room temperature. Less time was required for the self-curing mode of Rely X U100 to begin polymerization when it was exposed to tooth temperatures.

Overall, the time-based conversion profile of MX appeared to be more influenced by tooth temperatures than U100 was. Interestingly, the basic profile of time-based conversion for the CO groups was similar to that of the self-curing only group, indicating that the overall polymerization reaction, when curing through the ceramic disc, was related to the self-cure reaction. At room temperature, the CO group exhibited higher slope at the beginning of polymerization in comparison to the CO group at 28°C, indicating higher initial curing rates at room temperature. The time-based conversion profile of the self-curing mode of MX apparently provided faster curing rates than did U100, regardless of temperature. The polymerization of MX at simulated tooth temperatures
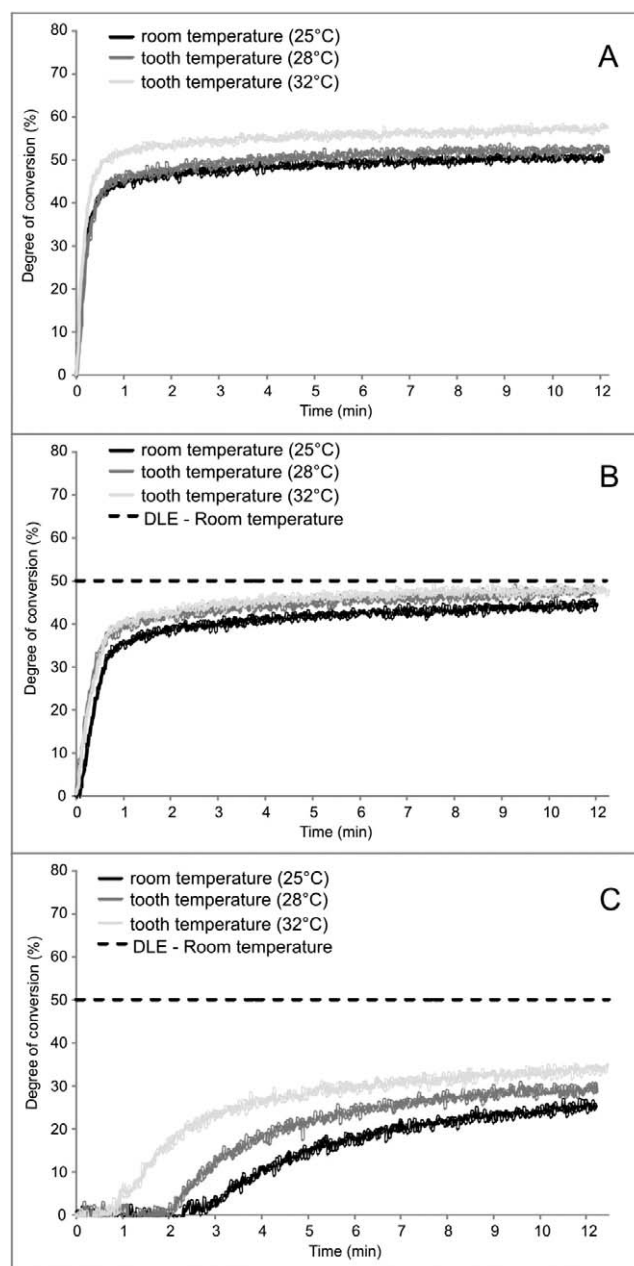

Figure 2. *Polymerization kinetics of U100 at 25°, 28°, and 32°C exposed to direct light exposure (DLE) (A), light exposure through ceramic disc overlay (CO) (B), and self-curing (SC) (C). The dashed line represents the degree of conversion after direct light exposure at 25°C.*

promoted faster curing rates than polymerization occurring at room temperature in groups relying solely on self-curing, which led to 12-minute DC values as high as those at room temperature.

DISCUSSION

The results validated the first research hypothesis, which anticipated that tooth temperature increases
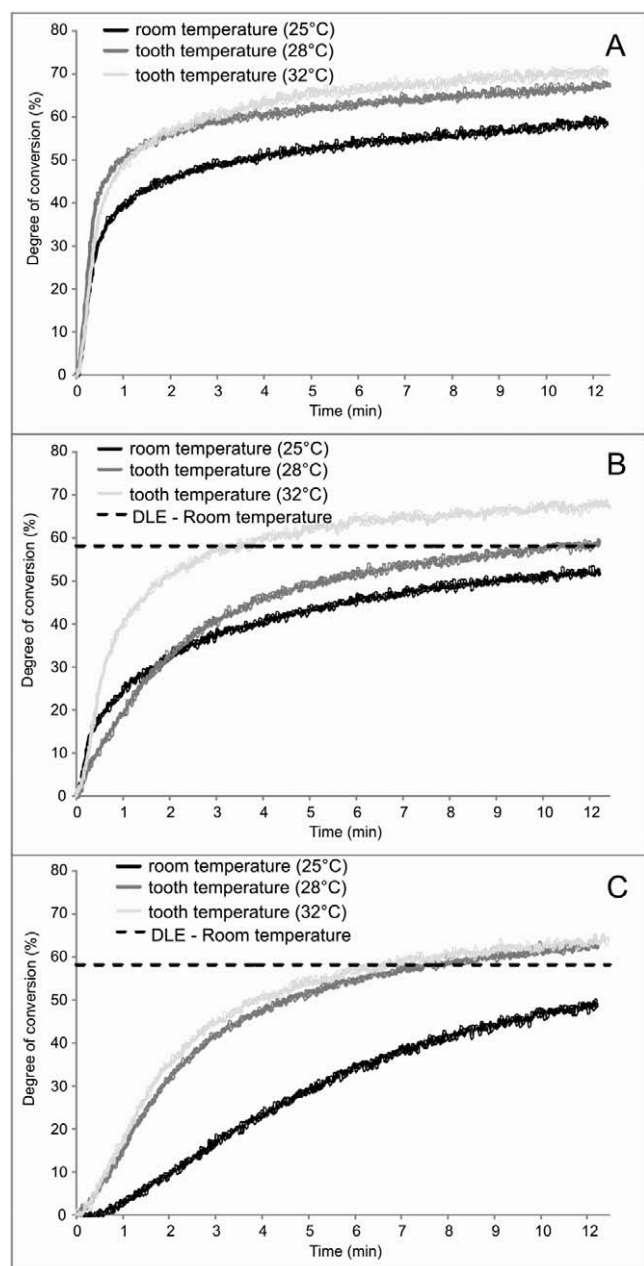
Figure 3. *Polymerization kinetics of MX at 25°, 28°, and 32°C exposed to 3 curing conditions: direct light exposure (DLE) (A), light exposure through ceramic disc overlay (CO) (B), and self-curing (SC) (C). The dashed line represents the degree of conversion after direct light exposure at 25°C.*

DC of SARCs using simulated clinical conditions. Because the products tested are relatively new and involve acid-base reactions and radical polymerization, it is difficult to anticipate the effects of such increases in DC on cement mechanical properties and bonding to dentin. It has been shown that high temperature can catalyze the acid-base reaction between acid groups and calcium or aluminum from

fillers to increase pH.[19] As a consequence, the pH would rise more quickly at a high temperature than at room temperature. Therefore, polymerization would proceed without the well-known detrimental effect from low pH[21,26] but could, for this reason, compromise bonding between such products and dentin. However, it should be pointed out that studies evaluating the effects of temperature on SARCs have focused on a temperature of 60°C,[19] which is considerably higher than that of the tooth. Thus, it is expected that tooth temperatures may increase the initial pH of these products more quickly than when these products set at room temperature. On the other hand, it is reasonable to expect that such effects caused by the cement exposure to tooth temperature are less pronounced than the effects on pH increase observed when such products are exposed to high temperature.

The self-curing mode of SARCs at room temperature was less effective in elevating DC than were the light-activating modes, corroborating previous findings.[6,10,27] This curing mode was also more influenced by temperature ranges than light-activated modes. More specifically, the DC of MX in self-curing mode increased approximately 14.2% from 25°C to 28°C, while the light-activated groups showed an approximately 7.1% to 8.1% increase within the same temperature range. For U100, self-curing mode groups increased 4.3% from 25°C to 28°C, while light-activated modes increase approximately 1.8%. This finding demonstrates that the effectiveness of self-curing components in SARCs exposed to tooth temperature is not as poor as that observed at room temperature. However, it should be noted that Rely X U100 still led to low DC values, even at 32°C. In addition, these results help explain those observed by Schmid-Schwap and others,[28] who evaluated the cytotoxicity of conventional and self-adhesive resin cements. In that study, U100 was one of the most cytotoxic cements when its effect was evaluated 10 minutes after it was allowed to self-cure.

The current results demonstrated that, regardless of the temperature, both curing conditions that simulated the cementation of an overlay or the complete absence of curing light led to lower DC values than those obtained from the control group, in which the SARCs were directly exposed to curing light. Such differences were observed when all experimental groups of U100 were compared with the DLE groups. For MX, only two curing conditions led to no significant differences in DC values, in comparison to the values from DLE groups: light activation through ceramic overlay at 32°C and self-

cure at 28°C. Thus, the data invalidated the second research hypothesis for MX and U100, which anticipated that DC values will not be significantly different from those obtained when exposing the specimen through a porcelain overlay or when allowed to totally self-cure without light. It should be mentioned that most statistical comparisons were made within each temperature. A similar increase in DC values was also observed in DLE groups, the differences between control group and experimental groups within each temperature remained constant. Despite such differences, when SARCs were exposed to attenuated light (CO groups) or allowed to self-cure at simulated tooth temperature, their DC values were apparently as high as those of the control (DLE) group at room temperature.

Although the comparisons between time-based conversion profiles (Figures 2 and 3) were not based on statistical methods but only on visual analyses, the effects of tooth temperature on polymerization kinetics were apparently more pronounced when the SARCs were allowed to self-cure. Besides, and still based on the conversion profiles, the increase in temperature also reduced the delay in the polymerization start. Based on this evidence, two assumptions can be made: (1) despite the lack of information from the manufacturers, MX presents higher amounts of self-curing components than U100; and (2) the differences in DC and conversion profiles caused by the increased temperature were apparently related to the amount of self-curing component in each resin cement. The degradation rate of self-curing components into radicals increases with increased temperature,[29] so radicals are created more rapidly when heated. For MX at 25°C (Figure 3A), the importance of self-curing components to compensate for lower light levels reaching the resin cement was evident, as a significant increase in conversion values with time was noted after light exposure. On the other hand, the light-activating components in U100 that are responsible for compensating for light attenuation were seen to work more effectively than in MX. For instance, at 25°C, the conversion-based profile using a ceramic disc placed over U100 produced nearly the same result as when the SARC was exposed to unattenuated light.

Time-based conversion profiles also demonstrated that the increase in DC values in all curing conditions is a consequence of increased polymerization rate (Figures 2 and 3). In this type of resin cement, a high polymerization rate can have detrimental effects on bonding to dentin, because higher shrinkage stress is developed as a consequence.[18]

Also, high polymerization rates enhance cross-link density and network quality,[30] resulting in a reduction of resin matrix permeability for fluoride ion release from the SARC.[31,32] As a consequence, SARCs polymerized at tooth temperature may release fewer fluoride ions than they do at room temperature, so the clinical benefits of fluoride release may be compromised as well. Conversely, the consequent high DC may improve mechanical properties.[3,30] Based on such assumptions, it is reasonable to assume that bonding and longevity of indirect restorations cemented with SARCs may be influenced by tooth temperature. However, only further studies evaluating short- and long-term mechanical properties and bond strength of such products to dentin and enamel might confirm how positive or negative such an influence would be.

Curiously, when light-activation of MX was performed through a CO, the conversion profiles at room temperature showed an apparently higher slope in the curve at the initial conversion than did specimens heated to 28°C. This finding indicates that the resin cement exhibited a higher curing rate at room temperature than at 28°C in this curing condition. It is important to point out that the moment of initial polymerization corresponds to the moment when the resin cement is being exposed to light from the curing unit. Therefore, based on the monomer conversion versus time plot, it seems that light activation was more effective at room temperature than at 28°C. Because the same energy density was delivered to the resin cement in both conditions, such a difference in the time-based conversion profile at the beginning of the polymerization may be related to the effects of tooth temperature on the self-curing components. As discussed earlier, because the degradation rate of such components into radicals increases greatly with increased temperature, the radicals are created more rapidly when heated. Conversely, it seems that such a premature activation of self-curing components might have interfered with components used in the light activation process.

The resin cement polymerization was evaluated for 12 minutes, which corresponds approximately to the time spent by clinicians to cement an indirect restoration to a prepared tooth. Therefore, the exposure of SARCs to tooth temperatures will allow the cement to be better polymerized at the moment of occlusal adjustments and first occlusal stress. However, it must be emphasized that further polymerization, along with an additional acid–base setting reaction, may proceed during the following

24 hours.[2,33] For this reason, no assumptions can be made regarding long-term bonding and resin cement mechanical properties, so only evaluation of SARCs after all curing reactions are completed can provide evidence of how tooth temperature affects their final mechanical properties and tooth-bonding capabilities.

CONCLUSIONS

Within the limitations imposed by the current study, the following conclusions may be made:

1. SARCs allowed to polymerize at temperatures simulating that of a prepared tooth (28°C or 32°C) showed increased monomer conversion values compared with the same products polymerized at room temperature (25°C).
2. At temperatures similar to those of the prepared tooth surface, SARCs exposed to attenuated light through the thickness of an indirect ceramic restoration exhibited degree of conversion values as high as those observed when resin cements were exposed to direct light at room temperature.
3. Despite the significant increase in DC promoted by polymerization at simulated prepared tooth temperatures for self-cured SARCs, the effectiveness of self-curing components at higher temperatures, in comparison to dual-curing polymerization at room temperature, was product-dependent.

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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