

Influence of temperature and primer application on setting time and degree of conversion of a dual-cure self-adhesive cement

Cristiane Maucoski^{a,*,} Juliana Anany Gonzales Guarneri^{a,b,} Paul Farrar^{c,} Richard Bengt Price^{a,*}

^a Department of Dental Clinical Sciences, Dalhousie University, Faculty of Dentistry, Halifax, Nova Scotia, Canada

^b Department of Restorative Dentistry, State University of Ponta Grossa, Ponta Grossa, Parana, Brazil

^c SDI Limited, Bayswater, Victoria, Australia

ARTICLE INFO

Keywords:

Dental cement
Resin cement
Temperature
Polymerization
Fourier transform infrared spectroscopy

ABSTRACT

Objectives: To examine the *in vitro* effect of different temperatures and use of a primer on the setting time (ST) and degree of conversion (DC) of a novel dual-cure self-adhesive cement (Set Maxx, SDI Limited).

Methods: Set Maxx was tested alone or with Stela Primer (SDI), both with light curing (LC) for 20 s with the Valo X (Ultradent), or it was allowed to self-cure (SC). The ST was determined according to ISO 4049 ($n = 5$). To measure DC, the cement was dispensed onto a temperature-controlled ATR sensor on an FT-IR spectrometer set at 23 °C, 32 °C, or 37 °C. The rates of polymerization and DC were measured for 1800 s ($n = 4$). The temperature increase was also measured at the bottom using a K-type thermocouple ($n = 3$). The results were compared using ANOVA and Tukey's multiple comparison test ($\alpha = 0.05$).

Results: The setting time was longer for the 23 °C group that used no primer ($149 \text{ s} \pm 2.6$). The highest DC was observed when using the primer at 37 °C in the SC ($75.1 \% \pm 2.2$) and LC ($76.5 \% \pm 1.8$) groups. Light exposure had a negligible effect on the final DC; however, it had the most significant impact on the maximum rate of change in the DC during polymerization. This was likely due to heat from the light, with the highest rate for the LC group at 37 °C ($7.7 \%/\text{s} \pm 0.4$) and a temperature increase of $41.7^\circ\text{C} \pm 3.2$.

Conclusions: Compared to using no Primer and at a temperature of 23 °C, using 37 °C and Stela Primer enhanced the DC of Set Maxx. Light exposure alone did not substantially increase the final DC, but it did increase the reaction rate and accelerate the polymerization.

Clinical Significance: Combining the Stela Primer with light exposure achieved the highest DC in Set Maxx. Laboratory studies conducted at room temperature should be reevaluated and repeated at intraoral temperatures.

1. Introduction

The longevity of indirect restorations is influenced by the luting cement used [1]. Resin cements have gained widespread clinical acceptance due to their ability to bond the restoration to the tooth [2] and their aesthetic qualities [3]. As a result, they are widely used for luting indirect restorations [3]. These resin cements can be exclusively light-cured, dual-cured, or chemically cured [2,4], with dual-cure cements being most commonly used today [3]. Dual-cured resin cements are recommended when the light from the light curing unit (LCU) may not adequately penetrate the restoration, such as for metal or thick ceramic indirect restorations, or due to the location in the mouth [2].

The setting mechanism of dual-cured resin cements is based on a redox reaction between the catalyst and base pastes. Typically, only one of these pastes contains the photoinitiator responsible for the light-curing component. The catalyst paste often contains benzoyl peroxide, which acts as the chemical initiator. The base paste usually includes a resin that can be photocured, along with a tertiary amine that participates in the redox reaction to trigger the self-curing mechanism [4].

Besides the polymerization reaction or the cement, the resin-based types of cement can also be divided by the luting procedure. Some resin cements require pre-treatment of the tooth using either an etch-and-rinse bonding system or they can use a self-etching adhesive system [4]. However, using an adhesive is complex, technique-sensitive

* Corresponding author: Faculty of Dentistry, Dalhousie University, 5981 University Ave, Halifax, Nova Scotia, B3H 3J5, Canada.

E-mail addresses: cris_maucoski@hotmail.com (C. Maucoski), julianaguarneri.jg@gmail.com (J.A.G. Guarneri), paul.farrar@sdi.com.au (P. Farrar), rbprice@dal.ca (R.B. Price).

<https://doi.org/10.1016/j.jdent.2025.105859>

Received 11 March 2025; Received in revised form 24 May 2025; Accepted 27 May 2025

Available online 28 May 2025

0300-5712/© 2025 The Author(s). Published by Elsevier Ltd. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

and requires additional steps. Therefore, self-adhesive resin cements have been developed to simplify the bonding and luting procedure [1,3]. These self-adhesive cements use acidic monomers that demineralize the enamel and dentin to produce micromechanical retention and some chemical attachment to the tooth [5,6]. These cements are claimed to be somewhat moisture tolerant and capable of providing excellent aesthetics, superior mechanical properties, better dimensional stability, and better micromechanical adhesion [1].

The polymerization of some resin cements can be accelerated when an adhesive is used [7]. Self-adhesive cements are designed for use in a one-step protocol, but some can also be combined with primers to enhance adhesion to the tooth. Some manufacturers, such as GC (Tokyo, Japan), recommend applying their primer (G-Cem One Adhesive Enhancing Primer) to the tooth before using their self-adhesive cement (G-Cem One) to optimize the bond strength to the tooth. This technology is designed to accelerate the polymerization process because the chemical cure accelerators in the adhesives should promote better polymerization of the G-Cem One cement, particularly in areas that are not exposed to light [8]. In a recent study, combining G-Cem One with the adhesive-enhancing primer significantly improved the interfacial bond strength of fiber posts to dentin [8]. This finding was supported by another study that showed that using a primer in combination with G-Cem One improved the microtensile bond strength between cement and dentin [9].

Dual-cured resin cements combine the advantages of both light and chemically activated systems, ensuring an optimal degree of conversion in the deeper areas of a restoration while also providing a controlled working time (WT) and a rapid setting time (ST). Most self-adhesive cements use a dual chemical and photochemical cure, where exposure to light provides an on-demand setting process [5]. This provides clinicians with an adequate working and setting time when cementing indirect restorations. The ST can be determined using rheological and temperature measurements [10], and it can be affected by various factors, including temperature, aging conditions, curing method, component ratios, exposure to oxygen, and when the product was manufactured [10,11].

The degree of conversion (DC) is a critical parameter for evaluating the physicochemical properties of resin-based materials, as it reflects the percentage of double carbon-carbon ($C = C$) bonds that have been converted into single carbon-carbon ($C - C$) bonds [12]. A common method for measuring the %DC is using Fourier Transform Infrared (FT-IR) spectroscopy, which measures the consumption of the $C = C$ bonds [12]. The more $C = C$ bonds consumed, the stronger the polymer network becomes [12], and resin with a low %DC is inadequately cured and can have a poor clinical performance. A study examining four self-adhesive resin cements reported that groups exposed to light curing achieved a significantly higher %DC than those not exposed to light [13], highlighting the importance of light activation to achieve optimal resin polymerization [13,14]. The temperature also influences the DC. Higher temperatures typically increase the polymerization process, resulting in a higher DC and faster polymerization rates [15]. Thus, the DC results recorded at room temperature compared to body temperature will likely be different [15–17].

Set Maxx (SDI Limited, Bayswater, VIC, Australia) is a novel dual-cure, self-adhesive resin cement designed for the cementation of a variety of indirect restorations, including metal, glass ceramics, zirconia, lithium disilicate, resin composites, and alumina. The Stela Primer (SDI Limited) is free from tertiary amines. It contains glycerol-dimethacrylate and has been recommended for use with Stela self-cure composite (SDI Limited). An investigation that used this primer with Stela [17] reported that the combination of Stela Primer and Stela composite resulted in the highest %DC. This was attributed to the synergistic effect between the two products. The manufacturer of Stela Primer also recommends using it with Set Maxx. They claim that polymerization is initiated by contact with the primer, thereby enhancing bond strength to the tooth. They also claim that there is no need for light-curing when using the primer.

However, currently, there is no data showing how this primer affects the setting time (ST), degree of conversion (DC), and polymerization kinetics of Set Maxx.

A recent study investigated whether there was a difference in the degree of conversion (DC) of six resin cements polymerized with or without an adhesive and found that there was no difference at 24 h [7]. However, the DC was measured at room temperature, which is different from the temperature in the mouth, the reaction kinetics were not determined, and output from the LCU was not described. The present study examined the *in vitro* use of the Stela primer at different temperatures on the setting time (ST) and degree of conversion (DC) of Set Maxx. The null research hypotheses are:

- (a) There will be no difference in the setting time when Stela Primer is applied before Set Maxx.
- (b) There will be no difference in the degree of conversion of Set Maxx when the Stela Primer is used.
- (c) Exposure to light will not enhance the degree of conversion of Set Maxx.
- (d) Different ambient temperatures will not affect the degree of conversion of Set Maxx.,

2. Materials and methods

2.1. Setting time measurements

One dual-curing, self-adhesive cement, Set Maxx (SDI Limited), was used. The cement was tested both alone and when used with Stela Primer (SDI Limited). The details and composition of both materials, as reported by the manufacturer, are presented in Table 1.

The setting time (ST) was determined according to ISO 4049 [18] for dual-cure class 3 luting materials at 37 °C (± 1 °C). In addition to using a body temperature of 37 °C, the study also used room temperature of 23 °C, and 32 °C to represent the temperature of a vital tooth in a clinical scenario [19]. The temperature was set using a digital block heater (Select BioProducts, UK), and a microtube was inserted into it. After the microtube reached the same temperature, the cement was inserted into the microtube using the Set Maxx automix tip (SDI Limited), according to the manufacturer’s instructions. This produced a specimen that was 4 mm high and 4 mm in diameter. A thermocouple with a conical tip was inserted 1 mm into the base of the specimen. The thermocouple was connected to a temperature recorder (AZ 8834 Thermometer Data Logger, AZ Instruments Corp., Taichung City, Taiwan), which recorded the real-time temperature every 1 s at a resolution of 0.1 °C. The time from the start of the mixing was measured until a plateau at maximum temperature was reached. To obtain the ST, the plateau was extended backward to meet an extension of the straight line of temperature increase. The data was analyzed (TRLog, AZ Instruments Corp, Taichung City, Taiwan) to obtain the ST. For the groups where the Stela Primer was used, the internal surfaces of the microtubes were sandblasted before applying the primer to improve its retention to the walls. The primer was used according to the manufacturer’s instructions and left to

Table 1
Composition according to the manufacturer of the materials used in the study.

Material	Shade	LOT number	Manufacturer	Composition
Set Maxx	A2	1243076	SDI Limited, Bayswater, Victoria, Australia	Organic components (39 % wt) (64 % vol), Urethane Dimethacrylate, Glycerol Dimethacrylate, 10-MDP
Stela Primer	–	1242568	SDI Limited, Bayswater, Victoria, Australia	10-MDP, dimethacrylates, methyl ethyl ketone (MEK), water, initiators, stabilizers

warm up on the digital block heater for 1 min before testing the ST. Five repetitions were made for each test group [18]. None of the ST groups were exposed to light.

2.2. Degree of conversion and polymerization kinetics

To analyze the degree of conversion (DC), the Set Maxx cement was either light cured (LC groups) for 20 s with Valo X (Serial Number 3F001009, Ultradent, South Jordan, UT, USA) or allowed to self-cure (SC groups). The light output from the Valo X was measured through a 6-mm diameter aperture into a 6-inch integrating sphere connected to a spectrometer (Flame USB, Ocean Optics, Orlando, FL, USA) that had been previously calibrated. The spectral radiant power from 350 to 550 nm was recorded using OceanView software (Ocean Optics, Orlando, FL, USA). Three measurements were made for each group to obtain the mean power (mW), irradiance (mW/cm²) that was then multiplied by the exposure time to calculate the radiant exposure (J/cm²).

The DC and polymerization kinetics were analyzed in real-time using a Bruker Vertex 70 Fourier Transform Infrared spectrometer (Bruker, Billerica, MA, US) with a temperature-controlled Attenuated Total Reflectance (ATR) diamond crystal (Golden Gate, Specac, Orpington, Kent, UK) attachment at a resolution of 8 cm⁻¹, from 1250 to 1800 cm⁻¹ with a background made from an average of 50 scans. The temperature of the ATR stage was set at 23 °C, 32 °C and 37 °C. The Set Maxx cement was inserted into a 1-mm deep, 6-mm diameter aluminum mold, and the DC was measured at the bottom of the specimen. In the groups where the Stela Primer was used, a thin layer of the primer was first applied to the ATR crystal, left for 5 s, and then air dried for 3 s, according to the manufacturer's instructions. For the LC groups, the LCU tip was positioned 0 mm away from and perpendicular to the center of the mold. The cement was light cured within a few seconds after it had been inserted into the mold.

The DC was calculated using standard methods from the ratio of the changes in the heights of the aliphatic peak (1635 cm⁻¹). Data was collected at a rate of 13 scans per second. The reaction rate after injecting the cement directly into the mold was determined by calculating the first derivative of the conversion *versus* time. The start time of the reaction was estimated by using the point of inflection (the maximum of the second derivative) of the conversion [17]. The DC was reported after 1800 s (30 min) for the six groups, and four repetitions were made in each group.

2.3. Temperature analysis

To record the temperature increase (ΔT - °C) as Set Maxx polymerized, the same 1 mm thick mold with a 6 mm diameter hole was placed on a white filter paper (Whatman Filter Paper Chart, Whatman Limited, England) on the top of a glass plate to provide a standard white background for the temperature measurements (Fig. 1). The glass plate was placed on top of a warming plate (Cimarec, Thermo Fisher Scientific, Waltham, USA) set to 23 °C, 32 °C and 37 °C. A K-type thermocouple (TP-01 Type K Braided Wire Probe, Reed Instruments, Taiwan) connected to a temperature acquisition software (T.C. Chart 1.03, Nomadics Inc., USA) was placed centrally at the bottom of the metal mold, and the temperature was monitored in real-time (every 0.5 s) until it stabilized. A thin layer of zinc oxide thermal paste (Thermal Grizzly, Thermal Grizzly Holding GmbH, Germany) was placed around the thermocouple to improve the temperature transfer from the Set Maxx cement to the thermocouple [20]. Once the baseline was reached (23 °C, 32 °C or 37 °C), the metal mold was filled with the cement. This caused the temperature to change. Once the temperature returned to the baseline value, the cement was exposed to the light from the Valo with the LCU tip at 0 mm. After the specimens had been exposed to light, the LCU was taken out of contact with the surface of the cement so that the light tip did not act as a thermal heat sink. For the groups that did not receive light from the Valo X (SC groups), the cement was allowed to self-cure until it

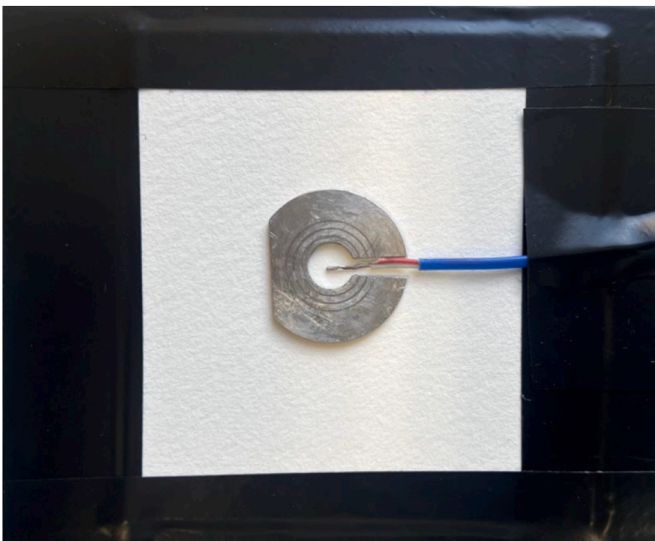


Fig. 1. The 1 mm thick aluminum mold with a 6 mm diameter hole placed on white filter paper on the top of a glass plate for the temperature measurements.

reached the maximum temperature (peak), and then it was allowed to return to the baseline temperature. The temperature increase (ΔT - °C) was calculated by subtracting the baseline temperature from the maximum temperature reached. The temperature was monitored in real time until it returned to the baseline temperature for all groups. Three temperature measurements were made for each group.

2.4. Statistical analysis

This study evaluated two independent variables—‘primer’ and ‘temperature’—for the ST analysis. For the DC and temperature analyses, three independent variables were considered: ‘light,’ ‘primer,’ and ‘temperature’. This resulted in six experimental groups for the ST analysis, and twelve for the DC/temperature analyses. The data distribution was analyzed using the Shapiro-Wilk test. After passing the normality test, the ST, DC, polymerization kinetics and temperature results were compared (StatView for Windows v. 5.0; SAS Institute, Cary, NC, USA) using ANOVA and Tukey’s multiple comparison test (α = 0.05).

3. Results

3.1. Setting time (ST)

Table 2 reports the Analysis of Variance (ANOVA) for the effect of temperature, the addition of Stela Primer, and their interaction on the ST (s). Both temperature ($p < 0.0001$) and primer had a significant impact ($p < 0.0001$) on the ST, with temperature being the most dominant factor (F-value = 65.168). The interaction between temperature and primer was also statistically significant ($p = 0.0164$), meaning the effect of primer on setting time depends on temperature. Fig. 2

Table 2
ANOVA for the effect of temperature, primer, and their interaction on ST of Set Maxx.

	DF	Sum of Squares	Mean Square	F-Value	P-Value
TEMPERATURE	2	28,204.467	14,102.233	1709.362	<0.0001
PRIMER	1	537.633	537.633	65.168	<0.0001
TEMPERATURE * PRIMER	2	80.867	40.433	4.901	0.0164
Residual	24	198	8.25		

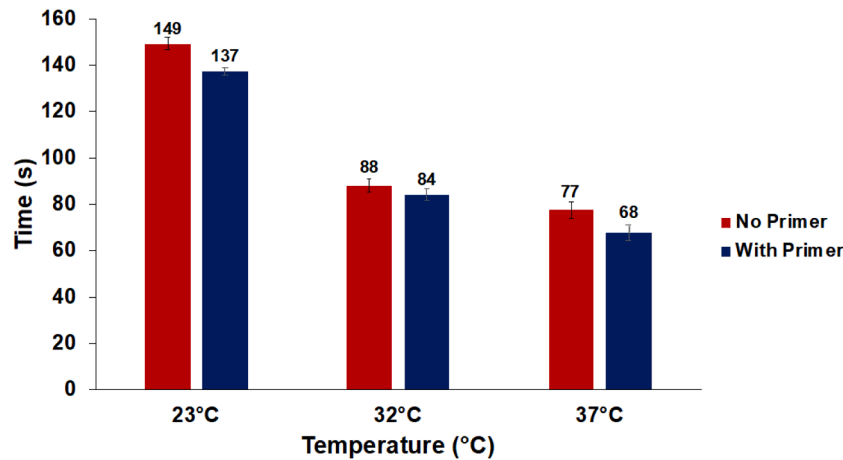


Fig. 2. Mean setting time(s) of Set Maxx used with or without using Stela Primer at 23 °C, 32 °C and 37 °C.

reports the ST for Set Maxx used with or without the Stela Primer. Overall, the ST was longer for the 23 °C group when no primer was used ($149 \text{ s} \pm 2.6$). The shortest ST was for the 37 °C groups when the Stela Primer was used ($68 \text{ s} \pm 3.3$). At 23 °C, the ST when using the primer was $137 \text{ s} \pm 1.5$. At 37 °C, when no primer was used, the setting time was $77 \text{ s} \pm 3.8$. At 32 °C, the mean ST was $88 \text{ s} \pm 2.9$ when no primer was used, and $84 \text{ s} \pm 2.5$ when the primer was used (Fig. 2).

3.2. Light output analysis

Fig. 3 shows the spectral radiant power from Valo X in the Standard mode. Valo X is a multi-peak broadband LED LCU that delivers three wavelength peaks: one in the violet region ($\lambda_1 = 395 \text{ nm}$), and two in the blue region ($\lambda_2 = 447 \text{ nm}$ and $\lambda_3 = 458 \text{ nm}$). The radiant power from the Valo X through the 6 mm aperture was $352 \pm 0.7 \text{ mW}$. The calculated irradiance delivered to the specimens in a 6 mm diameter mold was $1243 \pm 2.4 \text{ mW/cm}^2$, and the radiant exposure was $24.9 \pm 0.0 \text{ J/cm}^2$.

3.3. DC and polymerization kinetics

Table 3 reports the DC at the bottom of the 1 mm thick specimens after 1800 s (30 min) and the corresponding percentage increase in DC. Using higher temperatures always produced higher DC values. The highest DC values were observed when Stela Primer was used at 37 °C. In the self-curing mode, the DC reached $75.1 \% \pm 2.2$ and $76.5 \% \pm 1.8$ when light-cured. These results represented an increase of 35.8 % and 38.3 %, respectively, from the lowest DC that was recorded without primer or light exposure at 23 °C ($55.3 \% \pm 1.1$). The DC measurements at 32 °C demonstrated that primer application significantly increased conversion rates to $72.7 \% \pm 1.6$ (SC group) and $74.3 \% \pm 2.0$ (LC group), corresponding to 31.5 % and 34.4 % increase in DC,

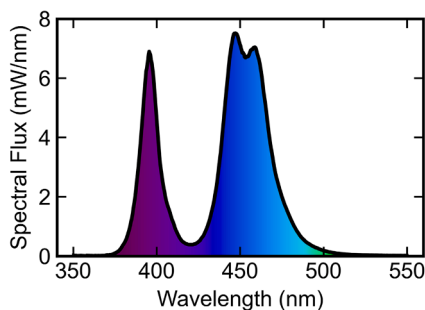


Fig. 3. Spectral radiant power (mW/nm) and emission spectra from the Valo X. Of note, this was not all the light emitted from the tip of the LCU, but it was the light received by the 6-mm diameter specimens.

Table 3

Mean degree of conversion (DC) $\pm$ SD and DC percentage increase (%) at 30 min of Set Maxx at the bottom of the 1 mm thick specimens.

Groups	23 °C		32 °C		37 °C	
	DC	% increase	DC	% increase	DC	% increase
SC without primer	55.3 (1.1)	–	60.3 (0.5)	9.0	62.9 (0.3)	13.7
SC with primer	64.8 (2.8)	17.2	72.7 (1.6)	31.5	75.1 (2.2)	35.8
LC without primer	56.3 (0.5)	1.8	61.6 (0.3)	11.4	64.1 (1.0)	15.9
LC with primer	72.5 (1.7)	31.1	74.3 (2.0)	34.4	76.5 (1.8)	38.3

respectively. Table 4 reports the ANOVA evaluating the effect of light, temperature, primer application, and their interaction on DC (%). Using the primer was the dominant factor affecting DC ($F = 813.72$, $p < 0.0001$). Temperature also played a substantial role ($F = 98.53$, $p < 0.0001$), and the light had a significant but small effect ($F = 29.35$, $p < 0.0001$). The interaction between light and primer was statistically significant ($F = 7.03$, $p = 0.0119$).

Table 3 shows that additional light exposure had a small positive effect on the %DC at 30 minutes; however, light exposure had a very significant effect on the reaction rate and it accelerated the polymerization process (Table 5). The highest rate of reaction was recorded for the LC group at 37 °C ($7.7 \%/\text{s} \pm 0.4$). The lowest reaction rate was observed in the SC group at 23 °C ($0.6 \%/\text{s} \pm 0.1$), regardless of whether the Stela Primer was used. This group took longer to reach the maximum reaction rate, with $99.9 \text{ s} \pm 14.4$ without primer and $80.9 \text{ s} \pm 5.9$ with

Table 4

ANOVA for the effect of light, primer, temperature and their interaction on the DC of Set Maxx.

	DF	Sum of Squares	Mean Square	F-Value	P-Value
LIGHT	1	68.217	68.217	29.346	<0.0001
PRIMER	1	1891.544	1891.544	813.723	<0.0001
TEMPERATURE	2	458.054	229.027	98.525	<0.0001
LIGHT * PRIMER	1	16.338	16.338	7.028	0.0119
LIGHT * TEMPERATURE	2	23.331	11.666	5.018	0.012
PRIMER * TEMPERATURE	2	0.582	0.291	0.125	0.8827
LIGHT * PRIMER * TEMPERATURE	2	27.421	13.71	5.898	0.0061
Residual	36	83.684	2.325		

Table 5

Mean (±SD) reaction rate (% per second) and time of the maximum rate of reaction (s) of Set Maxx.

Groups	Rate of reaction (%/s)			Time at maximum rate (s)		
	23 °C	32 °C	37 °C	23 °C	32 °C	37 °C
SC without primer	0.6 (0.1)	0.8 (0.0)	1.1 (0.0)	99.9 (14.4)	58.8 (0.8)	40.6 (1.4)
SC with primer	0.6 (0.1)	1.6 (0.1)	2.3 (0.4)	80.9 (5.9)	20.6 (1.7)	10.0 (2.8)
LC without primer	4.8 (0.4)	6.4 (0.3)	7.7 (0.4)	19.4 (2.2)	17.2 (1.6)	18.3 (2.6)
LC with primer	2.6 (0.8)	5.4 (0.8)	3.7 (0.5)	18.5 (2.7)	18.0 (0.3)	12.3 (4.6)

primer. Table 6 reports the ANOVA evaluating the effects of light, primer, temperature, and their interaction on the maximum rate of time. Light had the most substantial impact and the highest value (F-value = 582.22) on the reaction rate. Temperature was the second most influential factor ($F = 211.64$), significantly impacting how fast the setting reaction reached its maximum rate. The primer also had a significant effect ($F = 118.86$), and its interaction with the light was significant ($F = 92.49$).

3.4. Temperature analysis

Table 7 reports the temperature increase (ΔT - °C) at the bottom of the 1 mm thick specimens of Set Maxx. The SC group had only the Set Maxx exotherm, but the LC group had a combination of the exotherm reaction temperature from the Set Maxx plus the temperature rise caused by the LCU. The most significant temperature rises occurred when using the 20 s light exposure (LC). These increases ranged from $39.9\text{ }^{\circ}\text{C} \pm 3.2$ for 23 °C and up to $46.4\text{ }^{\circ}\text{C} \pm 2.4$ for the 37 °C group. The lowest temperature increase occurred for the SC group at 23 °C ($2.9\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$). At 32 °C, the group that had the highest temperature rise was when the cement was used in combination with primer and LCU, reaching $44.8\text{ }^{\circ}\text{C} \pm 2.2$. The lowest temperature rise, at 32 °C, was observed when Set Maxx was used in combination with a primer without light-activation, reaching $5.0\text{ }^{\circ}\text{C} \pm 0.8$. Table 8 reports the ANOVA of the effect of light, primer, temperature and their interaction on the temperature increase. Light was the dominant factor affecting temperature change ($F = 2063.63, p < 0.0001$). The impact of the primer on the temperature increase was not statistically significant ($p = 0.1024$).

4. Discussion

This study measured the *in vitro* setting time (ST), degree of conversion (DC), and polymerization kinetics of a dual-cure self-adhesive cement when it was used both with and without a primer (Stela Primer, SDI Limited) and at different temperatures (23 °C, 32 °C and 37 °C). The temperature and primer significantly affected the ST, with temperature

Table 6

ANOVA for the effect of light, primer, temperature and their interaction on the time of the maximum rate of DC change of Set Maxx.

	DF	Sum of Squares	Mean Square	F-Value	P-Value
LIGHT	1	14,255.81	14,255.81	582.219	<0.0001
PRIMER	1	2910.212	2910.212	118.855	<0.0001
TEMPERATURE	2	10,363.897	5181.948	211.635	<0.0001
LIGHT * PRIMER	1	2264.606	2264.606	92.488	<0.0001
LIGHT * TEMPERATURE	2	8414.301	4207.151	171.823	<0.0001
PRIMER * TEMPERATURE	2	207.175	103.587	4.231	0.0224
LIGHT * PRIMER * TEMPERATURE	2	219.732	109.866	4.487	0.0182
Residual	36	881.471	24.485		

Table 7

Mean (±SD) total temperature increase at the bottom of the specimens of Set Maxx.

Groups	Temperature increase (ΔT - °C)		
	23 °C	32 °C	37 °C
SC without primer	2.9 (0.5)	5.6 (1.2)	8.2 (0.9)
SC with primer	2.8 (0.2)	5.0 (0.8)	9.4 (0.4)
LC without primer	39.9 (3.2)	43.8 (1.1)	41.7 (3.2)
LC with primer	42.0 (6.1)	44.8 (2.2)	46.4 (2.4)

Table 8

ANOVA for the effect of light, primer, temperature and their interaction on the temperature increase of Set Maxx.

	DF	Sum of Squares	Mean Square	F-Value	P-Value
LIGHT	1	12,605.301	12,605.301	2063.632	<0.0001
PRIMER	1	17.612	17.612	2.883	0.1024
TEMPERATURE	2	126.013	63.007	10.315	0.0006
LIGHT * PRIMER	1	13.104	13.104	2.145	0.156
LIGHT * TEMPERATURE	2	23.074	11.537	1.889	0.1731
PRIMER * TEMPERATURE	2	11.68	5.84	0.956	0.3986
LIGHT * PRIMER * TEMPERATURE	2	1.42	0.71	0.116	0.8908
Residual	24	146.599	6.108		

being the most dominant factor (Table 2). Consequently, the first hypothesis was rejected. The study agrees with previous studies that reported the temperature significantly affected the setting reaction [10, 21]. According to the International Standards Organization (ISO) 4049, the minimum working time for dual-cure luting materials (Class 3 materials) at 37 °C is 90 s, with a maximum setting time of 10 min [18]. This study examined the ST in groups without light exposure following the ISO 4049 standard, and all the experimental groups were within the ISO 4049 guidelines for ST. Since the temperature at the pulp floor of a tooth cavity [19] is closer to 32 °C, the values obtained at 32 °C are considered to be more relevant. A rapid final set makes prosthesis repositioning more difficult and limits the clinician's working time. However, achieving a rapid final set once the working time has passed could be very beneficial clinically [6], allowing an improved workflow. Additionally, Fig. 2 demonstrate that light curing can shorten the setting time (ST) and Table 5 shows that delivering light accelerates the polymerization of the cement.

The data for DC was fitted using an autocatalytic model rather than an exponential model because a feedback mechanism played a role in the polymerization process [17]. The Set Maxx polymerization reaction was accelerated when the resin cement was in contact with the primer [8]. This reduced the ST (Fig. 2) and caused a significant improvement in the degree of monomer conversion (Tables 3 and 4). When Stela Primer was used with Set Maxx, the DC increased significantly ; therefore, the second hypothesis was rejected. A synergetic effect between the Stela Primer and the Stela self-cure composite, also made by SDI, has been previously reported [17], where it was found that using the primer was the most important factor for improving the DC.

This study showed that applying Stela Primer before Set Maxx increased the percentage of DC (Table 3). This may enhance the strength of the interfacial bond of endodontic posts [8] and improve bonding to the tooth. The functional monomer10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), contained in both Stela Primer and in Set Maxx, is known to enhance the adhesion to tooth structure [3,8], and it has also been shown to improve the bonding to zirconia ceramics [22]. This is due to the formation of a strong, aqueous, insoluble salt complex between calcium and the relatively hydrophobic MDP [5]. Additionally, using a primer containing 10-MDP should increase the penetration of the

self-adhesive resin cement into the dentin, compared to when no primer is used [9].

An unexpected finding was that the polymerization rate decreased in the light-cured (LC) groups when the primer was applied compared to when it was omitted (Table 5). This occurred despite the primer plus LC groups reaching a higher overall degree of conversion (%DC) and producing a greater temperature increase (Tables 3 and 7). This difference in the reaction kinetics between the light-cured and the self-cured groups that used the Stela Primer was likely only observed because of the rapid data collection rate on the FT-IR equipment. In this study, Stela Primer was applied directly to the ATR crystal of the spectrometer for 5 s, then air-dried for 3 s. The Set Maxx resin was then placed directly over the Stela Primer. However, light does not polymerize Stela Primer. Instead, it must be in contact with an overlying RBC before the primer starts to polymerize [17]. For a wavenumber of 1600 cm^{-1} in ATR-FTIR spectroscopy, the depth of penetration of the “evanescent wave” is only about 1 to $2\text{ }\mu\text{m}$ [23] and thus the DC being measured is the thin layer of Stela plus the Set Maxx over the crystal. When the overlying Set Maxx is allowed to ‘slowly’ self-cure, the thin layer of Stela Primer polymerizes as expected, but when light-cured, the Set Maxx cement polymerizes at a much faster rate, and the Stela Primer that is in contact with the ATR crystal takes longer to polymerize compared to the overlying Set Maxx.

The third hypothesis was partially accepted because the addition of light produced a small but significant improvement in the DC of Set Maxx. The DC of Set Maxx exposed to light at $23\text{ }^{\circ}\text{C}$ increased from 55.3 % to 56.3 %, at $32\text{ }^{\circ}\text{C}$ the DC increased from 60.3 % to 61.6 %, and at $37\text{ }^{\circ}\text{C}$ the DC increased from 62.9 % to 64.1 % (Table 3). When Set Maxx was used with Stela Primer and exposed to light at $23\text{ }^{\circ}\text{C}$ there was a larger increase in from 64.8 % to 72.5 %, but at $32\text{ }^{\circ}\text{C}$ the DC increased from 72.7 % to 74.3 %, and at $37\text{ }^{\circ}\text{C}$ the DC only increased from 75.1 % to 76.5 %. This agrees with a previous study, which showed that the DC of some dual-cure resin cements was not dependent on light curing [7]. The observation that Set Maxx will cure in the absence of light (Table 3) is beneficial [24], particularly in regions with limited light penetration, such as in metal or thick ceramic indirect restorations and endodontic post-cementation where the self-curing ability of Set Maxx is essential. The additional light did significantly accelerate polymerization and increase the reaction rate of Set Maxx (Table 5). This finding agrees with another study that reported better conversion after light curing the self-adhesive resin cement [13]. However, the DC (Table 3) and polymerization rates (Table 5) were still not as high as when Set Maxx was light cured. Thus, light curing increases the DC, but its effect is much stronger when Stela Primer is used (Table 3). Therefore, when using Set Maxx, clinicians should use both light and the Stela Primer to maximize the polymer conversion.

The three different temperatures affected the DC of Set Maxx (Table 3), leading to the rejection of the fourth hypothesis. Increasing the temperature during polymerization enhances the mobility of the free radicals and monomers, resulting in higher polymerization rates and increased DC values [21]. The baseline temperatures used in the study were representative of clinical scenarios, with $32\text{ }^{\circ}\text{C}$ simulating the temperature of a vital tooth [19] and $37\text{ }^{\circ}\text{C}$ corresponding to the temperature used in the ISO 4049 standard. The temperature increase during the polymerization of a dual-cure cement is influenced by the light from the LCU and the resin’s exothermic reaction [25]. Although the values obtained for the total temperature increase within the cement (Table 7) were higher than what is considered safe to the pulp [26], they may not reflect the temperature increase that can occur inside the pulp because factors such as pulp flow [27] and the thermal properties of enamel and dentin [28] were not considered in this study. While some temperature increases can be attributed to the exothermic reaction from the resin cement (SC group - Table 7), the radiant energy from the LCU seemed to have a greater effect on the total temperature increase [29]. For the SC groups, the higher the temperature ($37\text{ }^{\circ}\text{C}$), the greater the exothermic reaction. Note that this was only a $14\text{ }^{\circ}\text{C}$ change in temperature from 23 to $37\text{ }^{\circ}\text{C}$.

In this study, the LCU was the primary contributor to the temperature rise, suggesting that different light sources can lead to very different temperature changes since the choice of LCU, the exposure time, and the exothermic contribution from the composite can all affect the temperature increase [20,30]. Previous studies have shown that when RBCs are polymerized at body temperature, the DC is higher than when the RBCs are polymerized at room temperature [15,16]. This occurs because higher temperatures increase the mobility of molecules during chemical reactions, thus increasing the DC [13]. Higher temperatures allow unreacted monomers to diffuse more efficiently and participate in the cross-linking reaction before vitrification occurs. These combined effects promote an increase in the extent of monomer conversion. Furthermore, unlike light-cured resin composites, dual-cured resin cements contain both photoinitiators and self-curing components. The degradation of these components into radicals is accelerated as the temperature increases, further promoting polymerization [21]. Therefore, this research supports that DC studies should be conducted at clinically relevant temperatures because results obtained at room temperatures may not accurately represent clinical scenarios [15,16].

This *in vitro* study has several limitations that should be addressed in future research. First, only 1 mm-thick samples were used. This is likely thicker than the typical cement thickness in most, but not all, clinical situations. The DC was only analyzed at the bottom of the specimens of Set Maxx. Future studies should evaluate the DC and bonding strength of various cement thicknesses. Additionally, this study focused on the effect of a dedicated primer on a matched resin cement system from one manufacturer. While this restriction limits the generalizability of the findings to other cements, it reflects clinical practice, where manufacturers recommend matched systems for optimal performance and dentists should not go ‘off label’ and deviate from the manufacturer’s recommended instructions for use. Further research is needed to assess the performance of other resin cement systems that may also have dedicated primers. The DC was measured for only 30 min, and longer observation periods are required to understand the effects of primer use over time [7]. Ultimately, clinical studies are necessary to evaluate the long-term impact of using Set Maxx.

5. Conclusions

Within the limitations of this *in vitro* study, it was concluded that:

- (1) Using $37\text{ }^{\circ}\text{C}$ and the use of Stela Primer reduced the setting time of Set Maxx,
- (2) Stela Primer increased the degree of conversion of Set Maxx at 30 min, suggesting a synergistic effect.
- (3) Light exposure from the LCU did not have a strong effect on the final degree of conversion of Set Maxx; however, it increased the temperature, accelerated the polymerization and increased the reaction rate of Set Maxx. Combining the use of the Stela Primer and the light is recommended to achieve the highest degree of conversion.
- (4) *In vitro* studies should be carried out at a temperature that simulates the temperature in the tooth cavity.

CRedit authorship contribution statement

Cristiane Maucoski: Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Juliana Anany Gonzales Guarneri:** Writing – review & editing, Methodology, Investigation, Funding acquisition. **Paul Farrar:** Resources, Methodology, Conceptualization. **Richard Bengt Price:** Funding acquisition, Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Paul Farrar is the R&D Manager at SDI Limited, however, due to this conflict of interest, he was not involved in testing or analysis of the resulting data. The other authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by MITACS grants (#IT40995 and #IT42399) and partially supported by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brasil (CAPES) – Finance Code 001. We also acknowledge the help that Alex Gareau, Brett MacNeil, and Karolinne Martins de Sousa provided on this project.

References

- [1] I. Radovic, F. Monticelli, C. Goracci, Z.R. Vulicevic, M. Ferrari, Self-adhesive resin cements: a literature review, *J. Adhes. Dent.* 10 (4) (2008) 251–258.
- [2] A.P. Manso, N.R. Silva, E.A. Bonfante, T.A. Pegoraro, R.A. Dias, R.M. Carvalho, Cements and adhesives for all-ceramic restorations, *Dent. Clin. North Am.* 55 (2) (2011) 311–332, ix.
- [3] T. Maravić, C. Mazzitelli, E. Mancuso, F. Del Bianco, U. Josić, M. Cadenaro, L. Breschi, A. Mazzoni, Resin composite cements: current status and a novel classification proposal, *J. Esthet. Restor. Dent.* 35 (7) (2023) 1085–1097.
- [4] A. Alshabib, K. AlDosary, H. Algamaiah, A comprehensive review of resin luting agents: bonding mechanisms and polymerisation reactions, *Saudi. Dent. J.* 36 (2) (2024) 234–239.
- [5] J.L. Ferracane, J.W. Stansbury, F.J. Burke, Self-adhesive resin cements - chemistry, properties and clinical considerations, *J. Oral Rehabil.* 38 (4) (2011) 295–314.
- [6] L. Ling, Y. Ma, Y. Chen, R. Malyala, Physical, mechanical, and adhesive properties of novel self-adhesive resin cement, *Int. J. Dent.* 2022 (2022) 4475394.
- [7] S.Y. Lee, Y. Shimada, A. Sadr, T. Tabata, T. Sato, J.E. Byun, S.H. Han, Polymerization of resin cements by self-curing with or without adhesive treatment, *Dent. Mater. J.* 44 (2) (2025) 129–139.
- [8] R.S. Jamel, E. Farhan Alkhalidi, A. Edrees Dawood, The effect of touch-cure polymerization on the push-out bond strength of fiber posts, *Saudi. Dent. J.* 36 (4) (2024) 627–632.
- [9] S.A. Son, B.N. Kim, J.H. Kim, D.G. Seo, J.K. Park, Influence of Dentin surface roughness, drying time, and primer application on self-adhesive composite-cement bond strength, *J. Adhes. Dent.* 24 (2022) 137–146.
- [10] T.A. Pegoraro, R. Fulgêncio, L.E. Butignon, A.P. Manso, R.M. Carvalho, Effects of temperature and aging on working/setting time of dual-cured resin cements, *Oper. Dent.* 40 (6) (2015) E222–E229.
- [11] X. Zhang, Q. Zhang, X. Meng, Y. Ye, D. Feng, J. Xue, H. Wang, H. Huang, M. Wang, J. Wang, Rheological and mechanical properties of resin-based materials applied in dental restorations, *Polymers* 13 (17) (2021).
- [12] Ö. Hatipoğlu, M. Par, F. Pertek Hatipoğlu, Comparison of degree of conversion performance of bulk-fill resin composites: a systematic review and network meta-analysis of *in vitro* studies, *J. Dent.* 149 (2024) 105289.
- [13] A.D. Vrochari, G. Eliades, E. Hellwig, K.T. Wrbas, Curing efficiency of four self-etching, self-adhesive resin cements, *Dent. Mater.* 25 (9) (2009) 1104–1108.
- [14] M. Aldhafyan, N. Silikas, D.C. Watts, Influence of curing modes on conversion and shrinkage of dual-cure resin-cements, *Dent. Mater.* 38 (1) (2022) 194–203.
- [15] R.B. Price, J.M. Whalen, T.B. Price, C.M. Felix, J. Fahey, The effect of specimen temperature on the polymerization of a resin-composite, *Dent. Mater.* 27 (10) (2011) 983–989.
- [16] M.M. AlShaafl, Effects of different temperatures and storage time on the degree of conversion and microhardness of resin-based composites, *J. Contemp. Dent. Pr.* 17 (3) (2016) 217–223.
- [17] J.A.G. Guarneri, C. Maucoski, S. Ghaffari, B.D. MacNeil, R.B. Price, C.A.G. Arrais, Ability of a novel primer to enhance the polymerization of a self-cured resin composite, *Dent. Mater.* 41 (1) (2025) 42–50.
- [18] Dentistry Polymer-based filling, Restorative Materials, 4049, International Standards Office, Geneva, Switzerland, 2019.
- [19] D.C. Zarpellon, P. Runnacles, C. Maucoski, D.J. Gross, U. Coelho, F.A. Rueggeberg, C. Arrais, *In vivo* pulp temperature changes during class V cavity preparation and resin composite restoration in premolars, *Oper. Dent.* 46 (4) (2021) 374–384.
- [20] C. Maucoski, R.B. Price, C.A.G. Arrais, Temperature changes and hardness of resin-based composites light-cured with laser diode or light-emitting diode curing lights, *Odontology* 111 (2) (2023) 387–400.
- [21] M. Oliveira, P.F. Cesar, M. Giannini, F.A. Rueggeberg, J. Rodrigues, C.A. Arrais, Effect of temperature on the degree of conversion and working time of dual-cured resin cements exposed to different curing conditions, *Oper. Dent.* 37 (4) (2012) 370–379.
- [22] R.Q. Ramos, B. Mercelis, M.H. Ahmed, M. Peumans, G.C. Lopes, B. Van Meerbeek, Bonding of composite cements containing 10-MDP to Zirconia ceramics without dedicated ceramic primer, *J. Adhes. Dent.* 26 (2024) 135–145.
- [23] M. Milosevic. Internal reflection and ATR spectroscopy, John Wiley & Sons, Hoboken, N.J., 2012, ISBN 978-1-118-30974-2.
- [24] E. Faria, C.S. Pfeifer, Development of dual-cured resin cements with long working time, high conversion in absence of light and reduced polymerization stress, *Dent. Mater.* 36 (10) (2020) e293–e301.
- [25] A. Balestrino, C. Verissimo, D. Tantbirojn, F. Garcia-Godoy, C.J. Soares, A. Versluis, Heat generated during light-curing of restorative composites: effect of curing light, exotherm, and experiment substrate, *Am. J. Dent.* 29 (4) (2016) 234–2240.
- [26] L. Zach, G. Cohen, Pulp response to externally applied heat, oral surg oral med oral, *Pathol.* 19 (4) (1965) 515–530.
- [27] S. Braga, L. Oliveira, M. Ribeiro, A. Vilela, G.R. da Silva, R.B. Price, C.J. Soares, Effect of simulated pulpal microcirculation on temperature when light curing bulk fill composites, *Oper. Dent.* 44 (3) (2019) 289–301.
- [28] Y.C. Chiang, B.S. Lee, Y.L. Wang, Y.A. Cheng, Y.L. Chen, J.S. Shiau, D.M. Wang, C. P. Lin, Microstructural changes of enamel, dentin-enamel junction, and dentin induced by irradiating outer enamel surfaces with CO₂ laser, *Lasers. Med. Sci.* 23 (1) (2008) 41–48.
- [29] B.W. Nilsen, M. Mouhat, T. Haukland, U.T. Ortengren, J.B. Mercer, Heat development in the pulp chamber during curing process of resin-based composite using multi-wave LED light curing unit, *Clin. Cosmet. Investig. Dent.* 12 (2020) 271–280.
- [30] C. Maucoski, R.B. Price, B. Sullivan, J.A.G. Guarneri, B. Gusso, C.A.G. Arrais, *In-vitro* pulpal temperature increases when photo-curing bulk-fill resin-based composites using laser or light-emitting diode light curing units, *J. Esthet. Restor. Dent.* 35 (4) (2023) 705–716.