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# Ability of a novel primer to enhance the polymerization of a self-cured resin composite

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ARTICLE INFO	A B S T R A C T
<i>Keywords:</i> Self-curing dental resins Polymerization Degree of conversion Temperature	<i>Objective:</i> This study evaluated the degree of conversion (DC), polymerization kinetics, and temperature of a new self-cure bulk-fill resin-based composite (Stela Automix, SDI). <i>Methods:</i> The study was divided into seven groups: (1) Stela Primer, (2) Stela Automix, (3) Stela Automix exposed to light for 20 s after 100 s, (4) Stela Primer with Stela Automix, (5) Stela Primer with Stela Automix and exposed to light for 20 s after 100 s, (6) Scotchbond Universal with Stela Automix, and (7) Scotchbond Universal with Stela Automix and exposed to light for 20 s after 100 s, (6) Scotchbond Universal with Stela Automix, and (7) Scotchbond Universal with Stela Automix and exposed to light for 20 s after 100 s. The real-time reaction rates and DC at the bottom of 2 mm thick specimens at ~32 °C were measured at 720 s after insertion using a spectrometer with an Attenuated Total Reflectance detector. The temperature of Stela, Stela exposed to light, Stela Primer with Stela, and Stela Primer with Stela exposed to light were measured by an infrared thermal camera in human molar teeth with Class I cavities. The temperature was recorded in real-time every 0.03 s for 720 s. The results were compared with ANOVA and Tukey's multiple comparison test ( $\alpha = 0.05$ ). <i>Results:</i> The highest degree of conversion (DC) at the 2 mm depth was achieved when Stela Primer was combined with Stela RBC exposed to light for 20 s. The Scotchbond Universal bonding system did not significantly increase the DC compared to Stela alone, except when exposed to light for 20 s, which slightly increased the final DC. Exposing Stela to light for 20 s did not improve the final DC of Stela as much as using the Stela Primer, but it did increase the temperature and prolonged the time taken to return to 37 °C. <i>Significance:</i> The Stela primer accelerates the degree of conversion of Stela. The reaction occurred rapidly and achieved a higher DC at the bottom of the specimens where the Stela was in contact with the Stela primer.

# 1. Introduction

Due to the global agreement to phase down the use of dental amalgam [1], there has been an increase in the use of resin-based composite (RBC) materials [2,3]. Most cavities are now filled with RBCs that must be photocured using a light-curing unit (LCU) [4], and manufacturers recommend light curing their RBCs in increments that are at most 2 mm thick for conventional RBCs and up to 5 mm thick for bulk-fill RBCs. Placing sculptable RBCs in increments may reduce the adverse effects of the shrinkage that occurs during polymerization [5,6],

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but this technique takes longer than when bulk-fill RBCs are used to fill the cavity [7,8]. To achieve this greater depth of cure [9], most bulk-fill RBCs are more translucent than conventional RBCs, allowing more light to reach the bottom of the RBC as the thickness increases [9,10].

Light-cured RBCs commonly use camphorquinone as the photoinitiator in combination with a tertiary amine as the co-initiator. When sufficient light at the correct wavelengths reaches the RBC, the photoinitiator and tertiary amine react together to produce free radicals that break the double bonds and initiate the polymerization process [11]. Tertiary amines help to improve the depth of cure of light-cured RBCs and to speed up the polymerization process [12], but the photoinitiator/tertiary amine combination has poor color stability [11,13]. When insufficient light reaches the RBC, the RBC is inadequately cured, leading to limited depth of cure [14], inferior mechanical properties [11, 15], lower bond strength, and potential fracture and failure of the restoration [16–19]. Additionally, when insufficient light is delivered, the monomers in light-cured RBCs, such as the BisGMA and TEGDMA, may not be consumed in the polymerization process. These remaining monomers are then released into the mouth and can cause allergic reactions [20–23]. Also, more biofilm may form in the regions where RBC is undercured [17,24], leading to more secondary caries.

It has been estimated that 5 to 10 % of healthcare budgets in industrialized countries are spent treating caries [25] and replacing failed restorations, which accounts for more than half (57 %) of the restorations placed by clinicians [26]. The two most common reasons for replacing failed RBC restorations are fractures of the RBC and secondary dental caries [3,27-29]. Failures due to recurrent caries occur most frequently at the gingival margin of the proximal box in Class II restorations [27,28]. Here, the RBC is furthest away from the light-emitting tip of the LCU [30] and the most challenging to reach with sufficient energy [31-33], especially considering that there is a significant reduction in irradiance from most LCUs as the distance from the light tip increases [30,34,35] and many studies have reported that the light output from many LCUs used in dental offices is inadequate [36-40]. Consequently the bonding agent and the RBC at the bottom of the proximal box are likely to be less well photocured than the resin at the occlusal surface and this will also reduce the bond strength to the tooth at the bottom of the proximal box [18,31,32,39,41,42].

Self-cured or dual-cured RBCs have an effectively infinite depth of cure and may be a better restorative material to use in deep cavities than light-cured RBCs [43-47]. Self-cured RBCs do not require exposure to light because they do not contain any photoinitiators in their composition and can be used if the dentist does not have a curing light. They also have a slower polymerization rate [43] and may have a higher degree of conversion if combined with newer monomers and primers [43,44]. Thus, the quality of the final restoration may be improved if a self-cured RBC is used [45]. A new resin-based bulk-fill restorative material, Stela (SDI, Bayswater, Victoria, Australia), was introduced recently. This self-cure RBC is available in automix syringes or capsules and is promoted as an "amalgam alternative" because it does not require an LCU, has an unlimited depth of cure, and has better aesthetics than amalgam. When the Stela RBC contacts the Stela primer, the manufacturer claims that polymerization starts at the tooth/primer-RBC interface and this accelerates the polymerization reaction of the Stela RBC. The manufacturer claims that this system uses a novel primer that is free from tertiary amine and includes glycerol-dimethacrylate (GDMA). Including this monomer may improve the polymerization, mechanical properties, and adhesion to dentin and reduce water uptake and solubility [48].

The polymerization kinetics and degree of conversion (DC) of RBCs can be measured using Fourier Transform Infrared (FT-IR) spectroscopy [49]. The final DC of light-cured RBCs never reaches 100 %, but usually ranges from 50–66 % [14,49]; the DC at the bottom is usually less than at the top of the RBC because the reaction depends on the initiators in the RBC receiving sufficient light at the correct wavelengths, irradiance, radiant exposure [11,14,15] and the temperature [50–52]. The initiation, propagation and termination rates determine the overall rate of

polymerization of unfilled resins. The rate constant for initiation  $(k_i)$  is independent of temperature. In contrast, the rate constants for propagation  $(k_p)$  and termination  $(k_t)$  increase with temperature according to the Arrhenius dependence [53]. Using typical activation energies for propagation and termination, Hiemenz and Lodge have estimated that the overall polymerization rate for unfilled dimethacrylate-based resins will increase by 1.90 % per °C [53]. For polymerization of dental resins, the reaction rate is further accelerated by the Trommsdorf effect that occurs in free radical polymerization reactions. As the polymer chains grow, the system becomes more entangled with large polymer molecules and the viscosity of the reaction mixture increases. The marked increase in viscosity during gelation decreases the mobility of the large polymer radicals, effectively reducing  $k_t$  for the termination step at a given temperature [53]. Thus, autoacceleration of the free radical polymerization reaction occurs. As the polymerization accelerates, the reaction can become exothermic, producing more heat, which further accelerates the reaction [50–53]. Since resin polymerization in the mouth does not occur at room temperature, but instead at 30 to 32 °C [54], it is expected that the rates and extents of polymerization will be different at mouth temperature compared to room temperature that is used in most studies [50-53].

One recent clinical trial compared 55 restorations made using Filtek One Bulk Fill (3 M) with 55 Stela restorations placed using Stela Automix and 55 restorations placed using Stela from capsules. The four dentists who placed the restorations reported that the Automix version of Stela had a longer and somewhat inconsistent polymerization time [55]. However, there is still little information available on the properties of Stela [45,55,56]. The authors could find no information on the DC, polymerization kinetics, or the thermal changes when Stela was used with or without the Stela primer. Thus, this study shall evaluate the DC and polymerization kinetics and temperature of Stela Automix self-cure bulk-fill RBC when used with and without its dedicated primer. The null hypotheses are:

(a) there will be no difference in the final DC of Stela self-cure RBC when the primer is used;.

(b) the DC will not be improved when using a different bonding agent in combination with the self-cure Stela RBC;.

(c) exposure to light from an LCU will not enhance the DC of Stela RBC and.

(d) there will be no difference in temperature when light curing the self-cure RBC.

# 2. Materials and methods

# 2.1. Degree of conversion and polymerization kinetics

One self-cured bulk-fill RBC was used: Stela Automix (SDI, Bayswater, Victoria, Australia). This RBC was analyzed alone or in combination with its dedicated Stela Primer (SDI, Bayswater, Victoria, Australia) or with Scotchbond Universal (3 M Oral Care, St. Paul, MN, USA). The compositions according to the manufacturers are reported in Table 1. The exact formula and the initiators used in the Stela Primer and the Stela RBC are protected by a patent [57]. The DC and polymerization kinetics were analyzed using a Bruker Vertex 70 Fourier Transform Infrared spectrometer (Bruker, Billerica, MA, US) with an Attenuated Total Reflectance (ATR) diamond crystal (Golden Gate, Specac, Orpington, Kent, UK) attachment. The ATR sensor temperature was set to 32 °C to better simulate a clinical scenario of the temperature of a cavity in a tooth [54].

The Instructions for Use (IFU) state that the self-cure setting time for Stela at 37  $^{\circ}$ C is 4 min from the start of the mix. Therefore, the DC was reported at 720 s (12 min). The rate of reaction after injecting the Stela directly into the mold was determined by calculating the first derivative of the conversion versus time. The time at which the reaction starts was estimated using the point of inflection (the maximum of the second derivative) of the conversion.

### Table 1

Composition according to the manufacturer of the materials used in the study. See patent US 2023/0320941 A1 [57] for more information about Stela.

Material (Abbreviation)	Manufacturer	LOT number	Composition
Stela Primer (SPrimer)	SDI Ltd, Bayswater, Victoria, Australia	1213099	10-MDP, dimethacrylates, methyl ethyl ketone (MEK), water, initiators, stabilizers
Stela Automix (SRBC)	SDI Ltd, Bayswater, Victoria, Australia	1212256	UDMA, GDMA, fumed silica, barium aluminoborosilicate glass, fluoro aminosilicate glass, ytterbium trifluoride (YbF <sub>3</sub> ), calcium aluminate, hydroperoxide-based initiators, stabilizers, piements
Scotchbond Universal (SB)	3 M Oral Care, St. Paul, MN, USA	10267886	2-hydroxyethyl methacrylate, bisphenol A diglycidyl ether dimethacrylate (BISGMA), 2- propenoic acid, 2-methyl-, reaction products with 1,10- decanediol and phosphorus oxide (P <sub>2</sub> O <sub>5</sub> ), ethanol, water, silane treated silica, copolymer of acrylic and itaconic acid, camphorquinone, dimethylaminobenzoate (-4), (dimethylamino) ethyl methacrylate, 2,6-di-tert- butyl-p-cresol

The DC and polymerization kinetics were analyzed for the Stela RBC in seven different combinations, with five repetitions in each group (n =5), as reported in Table 2. The primer/bonding agent and Stela RBC were injected directly into 4.0-mm internal diameter metal molds that were 2.0 mm thick. In the first group, only Stela primer was applied and airdried for 3 s onto the surface of the ATR crystal on the spectrometer. In the second group, no primer was used, and Stela RBC was applied directly onto the surface of the ATR crystal on the spectrometer. In the third group, no primer was used; Stela RBC was applied directly onto the surface of the ATR crystal on the spectrometer and at 100 s after insertion of the Stela, the Valo X (Ultradent, South Jordan, UT, USA) curing light was used for 20 s on Standard mode at 0 mm. The light output from the Valo X was measured using a 6-inch integrating sphere connected to a spectrometer (Flame USB; Ocean Insight, Orlando, FL 32817 USA). The total radiant power of the Valo X was 1230 mW, and the irradiance delivered to the RBC was 1017 mW/cm<sup>2</sup>. The emission spectrum is reported in Supplemental Figure 1. In the fourth group, Stela

Table	2
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Combinations of products examined.

Group	up Primer/Bonding Com Agent used		Light-Exposure
SPrimer	Stela primer	None	No light-exposure
SRBC	No primer or bonding agent	Stela	No light-exposure
SRBC+Light	No primer or	Stela	Yes, Valo X
	bonding agent		(Standard mode)
			20 s
SPrimer+SRBC	Stela Primer	Stela	No light-exposure
SPrimer+SRBC+Light	Stela Primer	Stela	Yes, Valo X
			(Standard mode)
			20 s
SB+SRBC	Scotchbond	Stela	No light-exposure
	Universal		
SB+SRBC+Light	Scotchbond	Stela	Yes, Valo X
	Universal		(Standard mode)
			20 s

primer was applied to the ATR crystal on the spectrometer for 5 s and then air-dried for 3 s. The Stela RBC was injected directly over the Stela primer. In the fifth group, Stela primer was applied to the ATR crystal on the spectrometer for 5 s and air-dried for 3 s. The Stela RBC was injected directly over this primer, and at 100 s after insertion of the Stela, the Valo X (Ultradent) curing light was used for 20 s on Standard mode at 0 mm. In the sixth group, Scotchbond Universal (3 M Oral Care, St Paul, MN, USA) was applied onto the surface of the ATR crystal on the spectrometer and air-dried for 5 s. The Stela RBC was then applied directly onto the surface of the uncured bonding agent on the ATR crystal. In the seventh group, the Scotchbond Universal was exposed to light through 2 mm of Stela at 100 s after insertion of the Stela using the Valo X (Ultradent) for 20 s on Standard mode and at 0 mm.

To measure the DC, data was collected in real-time using a doublesided forward-backward method on the FT-IR. During this time, the mirror completed a full forward and backward movement cycle, which took  $\sim$  79 ms. Thus, each spectrum used to calculate DC was the average of the spectra collected during each pair of forward/backward scans. This methodology provided approximately 13 spectra per second between 750 and 2000  $\text{cm}^{-1}$  at a resolution of 8  $\text{cm}^{-1}$ . The DC was calculated using standard methods [58] by analyzing changes in the height of the cured Stela's aliphatic (1635  $\text{cm}^{-1}$ ) peak. Since the aromatic peak (1609 cm<sup>-1</sup>) of the Stela did not change during photopolymerization, only changes in the height of the aliphatic peak were considered. Reference spectra of air and the uncured specimen were used. For SPrimer+SRBC and SPrimer+SRBC+Light groups, a different method of recording the background had to be used because the reaction started immediately and the beginning of the reaction could not be accurately measured. Therefore, for these analyses, the primer with the uncured composite was inserted, and the background scan was recorded. Then the Stela materials were removed from the ATR crystal and the ATR crystal was cleaned. Another coat of primer and uncured Stela were applied and the reaction was recorded following the same instructions as described previously.

An autocatalytic model was applied to understand the DC's behaviour and the rate of reaction [59]. This model relates the DC versus time to its first temporal derivative (the DC rate) according to the following differential equation:

$$\frac{\mathrm{d}}{\mathrm{d}t}(\mathrm{DC}(t)) = k \quad (\mathrm{DC}(t))^m \quad (\mathrm{DC}_{\mathrm{max}} - \mathrm{DC}(t))^n,$$

where *k* is a rate constant, and *m* and *n* are the characteristic autocatalytic and rate exponents. The quantity  $DC_{max}$  is the maximum DC reached at the end of the reaction. The noise in the DC rate was removed using a first-order Savitzky-Golay filter with an averaging window of 0.3 s [60]. The autocatalytic model was applied to the filtered data. The maximum value of the reaction rate was extracted from the fit, along with the time that this rate occurred. Previous studies have shown that this model can accurately describe the behaviour of the DC rate when highly time-resolved DC data is used [61,62].

# 2.2. Temperature analysis

To complement the DC results, the temperature increases of selected groups (Table 3) were examined in human molar teeth (Ethics Board Committee #2021–5703). Divergent 4 mm deep Class I cavities (8 mm

Table 3
Temperature recordings of the different combinations of Stela with and
without the Stela Primer.

Primer	Light Exposure		
No primer	No		
No primer	Yes: Valo X (Standard mode) 20 s		
Stela Primer	No		
Stela Primer	Yes: Valo X (Standard mode) 20 s		

wide and 5 mm long) were prepared in molar teeth so that at least 1 mm of dentin remained on the pulpal floor. The buccal wall was removed using a polishing machine (EcoMet® 30, Buehler, Lake Bluff, IL, USA) with 120 grit sandpaper to provide direct access to the cavities for the temperature analysis. The tooth was positioned over a warming plate (Cimarec, ThermoFisher Scientific, Waltham, MA, USA) to maintain a baseline temperature of 32 °C in front of a thermal camera (PI 640i, Optris Infrared Measurements, Berlin, Germany), as shown in Fig. 1. The same tooth was used for all temperature analyses for the groups that did not use the primer. To allow the removal of the composite after the analysis, a thin layer of hydrophilic gel (LOT 04322, KY, Semina Indústria e Comércio Ltda., Sao Paulo, SP, Brazil) was applied to the cavity walls before inserting the RBC. A piece of dental floss was incorporated into the RBC to help remove the RBC after the temperature had been measured. The Stela was inserted into the cavity while the software (Optris PIX Connect, Optris Infrared Measurements, Berlin, Germany) recorded the temperature for 720 s in real-time at a frame rate of 32 Hz (every 0.03 s). Two different temperature measurement sites were analyzed: at the pulpal floor and 1 mm below the pulpal floor. Six different teeth were used to analyze the groups that used the Stela Primer because it was impossible to remove the RBC from the cavity in these groups. The Stela Primer was applied into the cavity for 5 s, airdried for 3 s and then the Stela was inserted. The coloured temperature scale bar was set from 25 °C to 50 °C, but this did not limit the data collection to this range. For groups that were exposed to light after inserting Stela, the Valo X (Ultradent) was used for 20 s on Standard mode after 100 s, using the same exposure conditions as when the DC was recorded. The data was analyzed using the Optris PIX Connected software (Optris Infrared Measurements). Peak temperature (PT), temperature variation over baseline,  $(\Delta T)$  and the time to return from the maximum temperature to body temperature of 37 °C [63] was recorded.

### 2.3. Statistical analysis

DC and temperature data were analyzed using GraphPad Prism (GraphPad Software, Boston, MA, US). Data distribution was analyzed using the Shapiro-Wilk test. After passing the normality test, the results of the DC, polymerization kinetics, and temperature were compared using ANOVA and Tukey's multiple comparison test ( $\alpha = 0.05$ ).

### 3. Results

# 3.1. Degree of conversion and polymerization kinetics

As expected from the manufacturer's IFU [64], Stela Primer did not polymerize independently. However, upon contact with the Stela RBC,



Fig. 1. Molar tooth positioned in front of the thermal camera. Note the divergent cavity walls and 1 mm of dentin remained at the center of the pulpal floor.

the primer/RBC combination polymerized rapidly, and the DC started to change as soon as the Stela RBC touched the Stela Primer.

Table 4 shows the DC at 2 mm of all groups analyzed in the study at 720 s. The highest DC was achieved when the Stela Primer was used in combination with Stela RBC (72.4  $\pm$  3.5 % at 720 s). Light-exposure of the Stela RBC with (72.0  $\pm$  2.5 %) or without (57.8  $\pm$  1.8 %) the primer did not improve the DC of the Stela RBC. Tukey's test showed that the Scotchbond Universal bonding system did not improve or change the DC (58.9  $\pm$  1.9 %) at 720 s compared to the DC of Stela RBC used on its own (57.7  $\pm$  1.2 %). The DC did increase (62.0  $\pm$  2.1 %) when the Stela and the bonding agent were exposed to light for 20 s at 100 s, but it was not statistically different from the SRBC group, and the DC was still lower than when the Stela Primer was used.

Stela RBC self-cured independently, but not so fast or as well as when it was in contact with the Stela Primer (Table 4). Table 5 shows that the Stela Primer accelerated the polymerization rate of Stela RBC compared to the other groups, with the mean rate of reaction of 1.1  $\pm$  0.1 % per second for the SRBC group,  $3.8 \pm 0.6$  % per second for SPrimer+SRBC group, and 1.0  $\pm$  0.2 % per second for SB+SRBC group. It can be noticed that light exposure did not increase the rate of reaction of groups using the Stela RBC with or without the Primer, with SRBC+Light group having the same reaction rate as SRBC group (1.1  $\pm$  0.1 % per second), SPrimer+SRBC+Light group having a mean rate of reaction of 3.4  $\pm$  0.5 % per second, really close to SPrimer+SRBC group (3.8  $\pm$  0.6 % per second) and for SB+SRBC+Light group, that used another bonding agent, the mean rate of reaction was 1.0  $\pm$  0.2 % per second increasing to 3.7  $\pm$  1.1 % per second during the 20 s of light exposure at the 100 s point. The initiators in Stela RBC were sufficient to cure the Scotchbond Universal (DC=58.9%) at the interface between the Scotchbond and the Stela when no light was delivered (Fig. 2).

When comparing the time in which the groups reached the maximum rate, it can be noticed that when the Primer was used, the maximum rate was faster than the other groups ( $9.6 \pm 4.6$  s for the SPrimer+SRBC group and  $11.2 \pm 5.5$  s for the SPrimer+SRBC+Light group). When the light was turned on (at 100 s), the only group that had two different maximum rates was the SB+SRBC+Light group (Table 5, Fig. 2).

Fig. 3 shows the reaction rate of three DC experiment groups. In the SRBC group, the reaction took longer time to initiate. In the SPrimer+SRBC group, the reaction is started almost instantaneously. For the SB+SRBC+Light group, the arrow shows when the reaction accelerates because the light from the LCU is turned on. Therefore, the SB+SRBC+Light group has two different reaction rates. The first one is comparable with the reaction rate of the SRBC group (Table 5, Fig. 2), and the second one is a faster reaction rate because of the LCU, which is probably light-activating the Scotchbond bonding agent.

### 3.2. Temperature changes

The mean temperature change ( $\Delta$ T) and peak temperature of all four temperature groups at the pulpal floor and 1 mm below the pulpal floor were significantly different (Table 6).

When Stela RBC was exposed to light from the Valo X, the highest

# Table 4 Mean degree of conversion (DC) $\pm$ SD of Stela at 720 s at the bottom of the 2 mm thick specimens of Stela (five repetitions in each group).

Group	DC
Stela Primer	0 (no changes detected)
SRBC	57.7 (1.2) <sup>B</sup>
SRBC+Light	57.8 (1.8) <sup>B</sup>
SPrimer+SRBC	72.4 (3.5) <sup>A</sup>
SPrimer+SRBC+Light	72.0 (2.5) <sup>A</sup>
SB+SRBC	58.9 (1.9) <sup>B</sup>
SB+SRBC+Light	62.0 (2.1) <sup>B</sup>

Different superscripted letters represent the DC values that are statistically different (p < 0.05).

### Table 5

Mean ( $\pm$  S.D.) reaction rate (% per second) and the time of the maximum rate of reaction (s) of Stela at the 2 mm depth.

Experimental Group	Rate of Reaction (%/s) (SD)	Time at Maximum Rate (s)
SRBC SRBC+Light SPrimer+SRBC SPrimer+SRBC+Light SB+SRBC SB+SRBC+Light SB+SRBC+Light*	$\begin{array}{c} 1.1 \ (0.1)^{B} \\ 1.1 \ (0.1)^{B} \\ 3.8 \ (0.6)^{A} \\ 3.4 \ (0.5)^{A} \\ 1.0 \ (0.2)^{B} \\ 1.0 \ (0.2)^{B} \\ 3.7 \ (1.1)^{A} \end{array}$	71.8 $(14.4)^{C}$ 71.1 $(4.9)^{C}$ 9.6 $(4.6)^{A}$ 11.2 $(5.5)^{A}$ 42.9 $(7.6)^{B}$ 83.8 $(10.0)^{C}$ 103.0 $(1.7)^{D}$
SB+SRBC+Light SB+SRBC+Light*	$3.7 (1.1)^{A}$	83.8 (10.0) <sup>o</sup> 103.0 (1.7) <sup>D</sup> *LCU turned on at 100 s

Different superscripted letters represent reaction rates that are statistically different (p < 0.05).

\*Rate of reaction after light exposure

mean  $\pm$  standard deviation temperature increase (54.1  $\pm$  0.5 °C at 1 mm below the pulpal floor and 59.2  $\pm$  1.7 °C at the pulpal floor) and  $\Delta T$  (21.7  $\pm$  0.5 °C at 1 mm below the pulpal floor and 27.1  $\pm$  1.8 °C at the pulpal floor) occurred. When the Stela Primer was used with the Stela RBC without light exposure, the temperature rise was the lowest among all groups (41.2  $\pm$  0.9 °C 1 mm below the pulpal floor and 44.9  $\pm$  1.5 °C at the pulpal floor) and  $\Delta T$  (8.5  $\pm$  0.9 °C 1 mm below the pulpal floor) were the lowest among all groups.

When analyzing the time necessary for the composite to return to 37 °C, Table 6 shows that the Stela RBC exposed to light from the Valo X required the longest time to cool down (153 s at the point 1 mm below the pulpal floor and 143 s at the pulpal floor). Stela Primer with Stela RBC was the Group that needed the least amount of time to cool down (59 s at 1 mm below the pulpal floor and 80 s at the pulpal floor).

Fig. 3 illustrates a representative temperature graph of the four temperature groups. When the Valo X light was turned on at 100 s, the reaction had already started inside the Stela. Thus, there are two different temperature rates. At first, it is just the exothermic reaction of

the Stela RBC. This is then followed by a rise in temperature caused by heat from the LCU. Also, when the RBC was exposed to light, it took longer to return to the baseline temperature (Table 6). It is important to note that the peak temperatures are different when Stela is exposed to light than when Stela is used alone. When Stela is exposed to light, the maximum temperature is greater and remains higher than the groups that did not receive light.

Fig. 4 shows the beginning of the reaction when Stela Primer was used with Stela RBC compared with the beginning of the reaction when no primer was used. The Stela Primer caused the reaction to start from the bottom of the cavity where the Stela RBC was in contact with the Stela Primer. Fig. 4 shows the difference in the temperature reaction when Stela is used with or without the Primer. The video is avaliable as Supplementary Video S1.

Additional supplementary material related to this article can be found online at doi:10.1016/j.dental.2024.10.013.

# 4. Discussion

This study used a novel self-cured bulk-fill RBC in combination with two different bonding agents with and without exposure to additional light to analyze the DC and polymerization kinetics of the RBC. The Stela Primer and the Stela RBC use a polymerizable adhesive that does not include any photoinitiator to form an adhesive layer. The self-cure Stela uses a redox initiator system. The manufacturer states that the Stela Primer does not contain any tertiary amines and is intended to act as a catalyst that initiates the curing process. This study shows that the snapset fast curing claimed by the manufacturer is correct. When Stela was used with the Stela Primer, the rate of reaction (Table 5) and the final DC of Stela (Table 4) were significantly increased. Therefore, the first research hypothesis, that there would be no change in the final DC of Stela when it was used with Stela Primer was rejected. Instead, the Stela Primer self-polymerized together with the Stela RBC and enhanced the polymerization of Stela. When the Stela primer was used, the polymerization rate increased, initiating the conversion so rapidly at the junction



Fig. 2. Rate of reaction (%DC/s) of SRBC (A and D), SPrimer+SRBC (B and E) and SB+SRBC+Light (C and F) experimental groups. The arrow indicates when the light was turned on at 100 s for the SB+SRBC+Light group. The red line is the fit to the DC rate obtained from the autocatalytic equation. The grey line is the smoothed data.



Fig. 3. Temperature rise of four groups. Note the initial drop in temperature when the Stela was inserted. The arrow indicates when the composite was inserted.

### Table 6

Mean temperature change ( $\Delta T$ )  $\pm$  standard deviation (SD), peak temperature  $\pm$  SD (in °C) and mean time  $\pm$  SD (seconds) to return from the peak temperature to 37 °C for the different combinations of Stela.

Group	Mean peak temperature 1 mm below pulpal floor	Mean temperature change at 1 mm below pulpal floor (∆T)	Mean time to return to 37 °C at 1 mm below pulpal floor (seconds)	Mean peak temperature at the pulpal floor	Mean temperature change at the pulpal floor (ΔT)	Mean time to return to 37 °C at pulpal floor (seconds)
Stela RBC alone Stela RBC with light-exposure	44.0 (0.8) <sup>B</sup> 54.1 (0.5) <sup>A</sup>	12.3 (0.5) <sup>B</sup> 21.7 (0.5) <sup>A</sup>	91 (13.8) 153 (10.5)	48.1 (0.7) <sup>B</sup> 59.2 (1.7) <sup>A</sup>	16.5 (0.6) <sup>B</sup> 27.1 (1.8) <sup>A</sup>	98 (13.7) 143 (8.4)
Stela Primer with Stela RBC	41.2 (0.9) <sup>D</sup>	$8.5(0.9)^{\mathrm{D}}$	59 (8.7)	44.9 (1.5) <sup>D</sup>	12.6 (1.7) <sup>D</sup>	80 (1.7)
Stela Primer with Stela RBC with light- exposure	48.9 (3.0) <sup>C</sup>	16.3 (2.3) <sup>C</sup>	148 (8.6)	53.0 (2.7) <sup>C</sup>	20.4 (2.7) <sup>C</sup>	145 (11.0)

Different upper-case letters indicate statistically significant differences within the same column (p < 0.05).

between the Stela and the Stela Primer that the DC was already changing at the first scan, which occurred in less than 0.077 s (Fig. 2 – B and E). This produced the highest DC at the bottom of the RBC (72.4  $\pm$  3.5 % at 720 s).

This synergistic effect between a bonding agent and the Stela improvement did not occur when Scotchbond Universal (3 M Oral Care) was used. The use of the Scotchbond Universal without light exposure did not improve or change the DC (58.9  $\pm$  1.9 %) at 720 s compared to when the Stela was used without the Stela Primer (57.7  $\pm$  1.2 %) and the second hypothesis was accepted. The reaction rate increased when

the SB+SRBC+Light group was exposed to light (Fig. 2, Table 5). This increase was likely due to the heat from the LCU. When the Stela RBC was applied over the Scotchbond that had been air-dried at the interface, the materials combined to form a new mixture containing photoinitiators, which may have contributed to the increase in the reaction rate. Although there was an improvement in the reaction rate, the DC at 720 s was not significantly different from the SRBC and SRBC+Light groups (Table 4). This was possibly because the Scotchbond had been air-dried according to the IFU, and therefore, there was little Scotchbond present to mix with the Stela RBC. In the present study, light-curing the



Fig. 4. Thermal changes at the beginning of the reaction of Stela (A) compared to when Stela was used in combination with Stela Primer (B). Note that the thermal effects start in different locations. + marks the floor of the cavity and at the roof of the pulp chamber. The video is available as Supplementary Video S1.

Stela Primer did not result in a statistical increase in the DC when used with the Stela RBC (Table 4). This was expected because the manufacturer of the Stela Primer states that it does not contain any photo-initiators. This suggests that the exothermic reaction was already happening when the Valo X curing light turned on, and the heat from the LCU at 100 s had no influence on the reaction. In the present study, the  $RP_{max}$  of the Stela RBC when it was used combined with the primer was similar to the  $RP_{max}$  of the SB+SRBC+Light group that was exposed to light and used Scotchbond Universal (Table 5). This also suggests that the reaction starts as soon as the Stela primer touches the Stela composite, which is different from other dual- or self-cured materials that have slower reaction rates [43,65].

There is some evidence that dual- and self-cured materials generate lower stress than light-cured materials [66,67]. When the RBC has a slower polymerization rate, this material will usually generate lower stress because of the extended pre-gel phase and a slower initiation, which may, in turn, lead to less shrinkage stress [67] and a consequent reduction in gap formation. In a previous study, when Stela was applied in Class I cavities, it had better adaptation and no gaps at the resin-dentine interface compared to conventional resin composite, even after 12 months of storage in artificial saliva [56]. This was attributed to the low shrinkage effect that it may have had during its self-polymerization reaction [56]. The Stela Automix contains two types of fillers with high adhesion with the matrix [45] and mean sizes ranging from 2.8 to 4.0 µm [55]. The high cross-linking ability of the GDMA in Stela produces superior conversion and cross-linking capabilities of the resin matrix with these fillers. It should increase the mechanical properties when compared with dual-cure RBCs [45].

When light-curing RBCs, the photopolymerization reaction initiates from the surface of the RBC closest to the light source, typically at the top of the restoration. For self-cured RBCs, the polymerization reaction begins from the center of the restoration, where the temperature tends to be higher due to the greater mass of material. Then, it spreads outward toward the periphery of the RBC. In the present study, the Stela Primer initiated the reaction from the walls and bottom of the cavity. This effect was very evident in the thermal images (Fig. 4). The observation that the polymerization reaction starts at the tooth/primer/RBC interface and not at the center mass of the RBC may improve the bond strength to the tooth and reduce gap formation at the tooth/RBC interface.

The third hypothesis that light-curing would not improve the DC of the Stela composite was accepted. When another bonding agent (Scotchbond Universal) was used, the DC was not as high as the DC when using the Stela Primer with the Stela that was exposed to light, confirming the synergistic polymerization effect of the Stela Primer[56]. It is also interesting to note that the DC of the SPrimer+SRBC+Light group was similar to the SPrimer+SRBC group. SPrimer+SRBC+Light group was exposed to light, but exposing the combination of Stela Primer and Stela to light produced no improvement in DC at 720 s (Table 4). This was expected because neither the Stela Primer nor Stela RBC contains a photoinitiator. It is already well established that an increase in temperature will improve the polymerization of RBCs [50,52]. However, in the present study, when the Valo X was used for 20 s, there was no significant improvement in the DC that was measured in 2 mm deep metal molds (Table 4). The molds may have acted as a heat sink and reduced the temperature rise in DC specimens. The temperature in the teeth was consistently higher when the LCU was used for 20 s (Fig. 3), and the time required for the Stela to return to 37 °C was the longest, 153 s at the point 1 mm below the pulpal floor and 143 s at the pulpal floor (Table 6). Stela Primer with Stela RBC needed less time to cool down (59 s at 1 mm below the pulpal floor and 80 s at the pulpal floor) while producing the highest DC. For this reason, exposing Stela to light for 20 s is not recommended, instead use Stela with Stela Primer.

Adding additional light exposure resulted in a higher temperature increase in the tooth and no benefit regarding the final DC measured in the metal rings. When no light was used, just the temperature of the exothermic reaction was recorded from Stela RBC. When Valo X was used for 20 s, the heat from the LCU led to a greater increase in temperature (Fig. 3). Thus, the fourth hypothesis that there would be no difference in temperature when light curing the Stela RBC was rejected. The highest temperature change at the pulpal floor ( $\Delta T$ ) was observed when Stela RBC was exposed to light for 20 s, and no primer was used (Table 6 and Fig. 3) with  $\Delta T$  of 27.1 (  $\pm$  1.8)°C. The light-exposed groups took longer to return to baseline temperature (Table 6). That means that additional light exposure kept the temperature higher than the baseline for longer. The heat from the LCU was distributed around the structures surrounding the RBC and outward to the surrounding environment [68]. Due to its low thermal diffusivity ( $\approx 1.87 \times 10^{-1}$  $cm^2/s$ ) [69], dentin can store thermal energy and release it slowly [68, 70]. This characteristic is similar to high-filler RBCs, which also have low thermal diffusivity [71]. This phenomenon indicates that the heat will slowly be released through the restoration and surrounding tooth tissue. Therefore, this resulted in a sustained higher temperature, even after the LCU had shut off, which led to a longer time to return to 37 °C.

The primary purpose of this study was to examine the DC and polymerization kinetics of a novel self-cured bulk-fill RBC. The data was fitted using an autocatalytic model since a feedback mechanism plays a role in the polymerization process. The reaction rate increases rapidly over time because the product catalyzes the generation of more of itself. Taking the derivative of the DC resulted in a noisy DC rate signal, but this noise was removed using a numerical filter and the autocatalytic model was then applied to the smoothed data. The maximum value of the reaction rate was extracted from the fit, along with the time that this rate occurred. It was verified that the smoothing procedure did not bias the fitting to within reasonable deviation but it was necessary to achieve a robust fit of the data so that the maximum value of the reaction and the time that this rate occurred could be determined. This result supports using the autocatalytic model to follow the polymerization reaction of dental RBCs in real time. A recent clinical trial using Stela reported less postoperative sensitivity when a light-cured bulk-fill RBC (Filtek One 3 M) was used, both at the baseline measurement and after 48 h [55]. This is encouraging since all the groups had a peak temperature change greater than 5.5 °C. This temperature rise is concerning since a temperature increase in the pulp chamber above 5.5 °C may lead to irreversible damage to the pulp tissue [72]. In this study, only the temperature in the dentin at the pulpal floor was analyzed. However, further in vitro studies using teeth with a simulated fluid circulation through the pulp chamber or longer clinical trials are required to determine if this temperature rise may cause irreversible pulpal changes.

The FDI has recommended that "research is needed to improve overall material properties and, eventually, their clinical performance and cost-effectiveness" [3]. When used in combination with Stela Primer, the Stela RBC produced the highest DC and could be a suitable alternative for light-activated composites. However, in the present study, only the Stela Automix was used, and the tooth did not have any circulation of pulpal fluid. Additional studies are required to evaluate wear, polymerization shrinkage, shrinkage stress and bond strength because Stela has been reported to have a lower surface lustre and texture than one light-cured composite, Filtek One (3 M). In one clinical trial, the dentists reported that the Automix version of the Stela was easier to use due to its flowability. Still, it exhibited a longer and somewhat inconsistent polymerization time (4-8 min) compared to using Stela in the capsule, which had a shorter curing time, typically around 30-60 s from the start of the application of the Stela from the capsule [55]. This subjective wide range in handling Stela Automix was not observed in the present objective study, where the temperature and DC changes were measured in real time. A future study should confirm if there is a difference in the speed of the polymerization reaction between Stela in the capsule form and the Stela Automix.

### 5. Conclusion

Despite the limitations of the present in vitro study, it was concluded that:

1. Using the Stela Primer and the Stela Automix produced the highest DC due to a synergistic effect between these two products;.

2. The use of a different bonding agent did not improve the DC of Stela as much as using Stela Primer;.

3. Exposing Stela to light from the Valo X for 20 s did not improve its final DC as much as using the Stela Primer, but it did increase the temperature and prolong the time it took to return to 37  $^{\circ}$ C.

# 5.1. Relevance

The Stela Primer accelerates the degree of conversion of self-cure Stela Automix. The reaction occurred rapidly when Stela was in contact with the Stela Primer, and Stela achieved a higher DC at the bottom of the specimens. This may improve the bond to the tooth.

# **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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# Appendix A. Supporting information

Supplementary data associated with this article can be found in the

online version at doi:10.1016/j.dental.2024.10.013.

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