



# Dual-curing resin cement with colour indicator for adhesively cemented restorations to dental tissues: Change of colour by curing and some physical properties

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## ABSTRACT

The study was aimed to investigate a color indicator containing dual curing resin composite luting cement and to plot the color change to the time of solidification of the cement. In addition some physical properties were studied. Specimens were made of a dual-cure resin cement (Maxcem Elite™ Chroma, Kerr, Orange, CA, USA) and polymerized by autopolymerization only, or with light initiated polymerization. A spectrophotometer was used to quantify the color change of the cement as plotted with the curing time. The efficacy of the curing process was studied by measuring water sorption and the ultimate flexural properties of the cement. The results showed that the flexural strength of cement after autopolymerization was 27.3 MPa and after light initiated polymerization 48.1 MPa. Young's modulus of bending was 2089.3 MPa and 3781.5 MPa respectively for the same cement samples. Water sorption after two weeks for the autopolymerization cement samples was –1.12 wt% and for the light initiated polymerization samples 0.56 wt%. Non-parametric Spearman's correlation was measured for autopolymerized cement samples between variables for color and solidification load (N), which showed a strong correlation between curing process and color change ( $p < 0.05$ ). There was a correlation between the color change and degree of monomer conversion of the dual curing resin composite luting cement which contained a color indicator system for polymerization reaction. The study also suggested that autopolymerization only resulted in suboptimal polymerization of the cement. By additional light curing considerably higher flexural properties were obtained.

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## 1. Introduction

An effective adhesion of resin cements to dental tissues is foremost for the success of adhesively cemented restorations. However, the clinical success of these kinds of restorations is complex and exceeds a good adhesion between luting agent, restoration and abutment (Anusavice, 2012; Pang et al., 2015; Nicolaisen

et al., 2016). A number of factors add to the clinical performance of indirect restorations, including the type of resin cement, the activation mode of the polymerization, the photo polymerization time and the thickness of the luting cement (Hardy et al., 2018; Novais et al., 2017; Ozcan and Volpato, 2016). Resin cements that are light-cured have been commonly used for the cementation of indirect ceramic and resin composite restorations due to their better time control during the cementation process. However, since the type of material and the thickness of the restoration can hamper the degree of monomer conversion by the polymerization reaction of the resin cement, a resin cement that does not depend entirely on light activation might be desired (Ilie and Hickel, 2008; Lise et al., 2018; Meng et al., 2008; Passos et al., 2013).

Resin cements can be light activated or chemical activated or dual activated (combinations of light and chemical) (Ferracane, 2011; Blatz et al., 2003), because of the lack of control over the

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working time in chemically cured resin cements, clinicians prefer either light activated or dual activated resins (Hofmann et al., 2001; de Lima et al., 2016; Lee et al., 2008; Cho et al., 2015). Dual-cured resin cements provides the advantages of both light and chemically activated resins, reach adequate degree of polymerization and provide an extended working time due to the easily controlled light-curing mechanism. As a result, a fast initial setting of the resin cement contributes to a stabilization of the restoration (Yoshida and Atsuta, 2006; Faria-E-Silva and Pfeifer, 2017). A resin cement should be able to achieve a high degree of monomer conversion, which guarantees a more effective sealing ability, lower solubility of the cement, higher bond strength and superior color stability of the cement (Archegas et al., 2011; Lohbauer et al., 2010; Kumbuloglu et al., 2004). Dual-cured cements along with the benefit of favoring a better polymerization in deep areas of teeth, show elevated mechanical properties such as flexural strength and hardness when compared to those where other activation mechanisms are involved (Braga et al., 2002). Interferences in the polymerization process can compromise the mechanical properties and clinical performance of the restorative material (Mounajjed et al., 2017).

Removal of excess cement associated with indirect restorations is a crucial working step, and if done improperly excesses can be a risk factor of gingivitis, periodontitis, as well as perimplantitis in the case of implant supported restorations (Pesce et al., 2015; Anami et al., 2012; Galvan et al., 2015). The resin cements may also adhere to the oral surface of restorations, which can be an inconvenience for the cleanup process and also add time to the cementation process. The removal of excess cement is important along with the determination of the correct moment of removal. The interruption of the light curing at an inappropriate time for removal of cement from non-desirable places has been investigated. Some affirm that this practice may result in a low cross-link density of the luting cement, translated into resin luting cement with higher susceptibility to wear (Asmussen and Peutzfeldt, 2003; Soh and Yap, 2004). Determining the appropriate time for cement removal can be difficult although material manufacturers offer time estimates. Some manufacturers offer a color cleanup indicator to identify the optimal time to remove excess cement.

To the authors knowledge there are no reports on visual indicators that state the precise time for excess cement removal in conjunction with the level of the degree of cure of resin composite luting cements. Therefore, the aim of this study was to investigate a dual-curing cement that provides a color cleanup indicator with relation of the color change to the degree of monomer conversion and to some mechanical properties of the cement.

## 2. Materials and methods

A self-etch, self-adhesive dual-cure resin cement was investigated (Maxcem Elite™ Chroma, Kerr, Orange, CA, USA). Clear color shade from the manufacturer's standard selection was used. A single operator prepared the specimens and performed the measurements. The cement was handled under ambient laboratory conditions ( $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ) using the normal lightning of the laboratory (630 lx at the working level). The cement was dispensed according to the manufacturer's instructions and inserted into a round shaped acrylic mold (1 mm thick and 4 mm in diameter) placed on a mylar strip.

The curing process of the autopolymerizing cement samples ( $n = 6$ ) was visually inspected with 1 min intervals for 10 min and photographed. Color change of the cement during autopolymerization was further quantified by measuring spectrum of the reflected light using a spectrophotometer (CM-700d, Konica

Minolta, Osaka, Japan). The color spectrum (CIELAB color space) was measured with 30 s intervals up to 10 min. Number of samples used for spectrophotometer measurement was 6 ( $=n$ ).

The curing process (solidification) during autopolymerization of the cement was studied with a custom arrangement on a single column materials testing machine (Lloyd Instruments LRX, Ametek Inc, Largo, FL, USA) (Fig. 1). The cement was dosed into a 0.5 ml syringe which was cut 1 cm long and attached to a measuring table with two-sided adhesive tape. Next, a solid metal bar (diameter 1.2 mm) was pressed into the curing cement at constant speed of 0.3 mm/min ( $n = 6$ ). The force was limited to 50 N to prevent damage to the testing equipment and the results in Newton were recorded as force overtime which reflected autopolymerization of the cement. Force in Newton was used as an indicating unit for solidification, and further for plotting the state of autopolymerization to the color change.

Additional specimens were made for measuring the flexural properties of the cement with a three-point bending test. The cement was dispensed into a  $2\text{ mm} \times 2\text{ mm} \times 25\text{ mm}$  mold, with a microscope glass on top. The samples were divided into two groups. The first group ( $n = 8$ ) was light cured for 20 s at a distance of 1 mm at three overlapping positions of the specimen using a hand held light curing unit with reported light intensity of  $1200\text{ mW/cm}^2$  by manufacturer (Elipar™ S10, 3 M Espe, St. Paul, MN, USA). The second group ( $n = 8$ ) was let to autopolymerize for up to three minutes before taking the glass off. The specimens were then extracted from the molds, any excess material was removed, and were subsequently wrapped in aluminum foil and stored in a desiccator at  $22\text{ }^{\circ}\text{C}$  for three days. Next, the samples

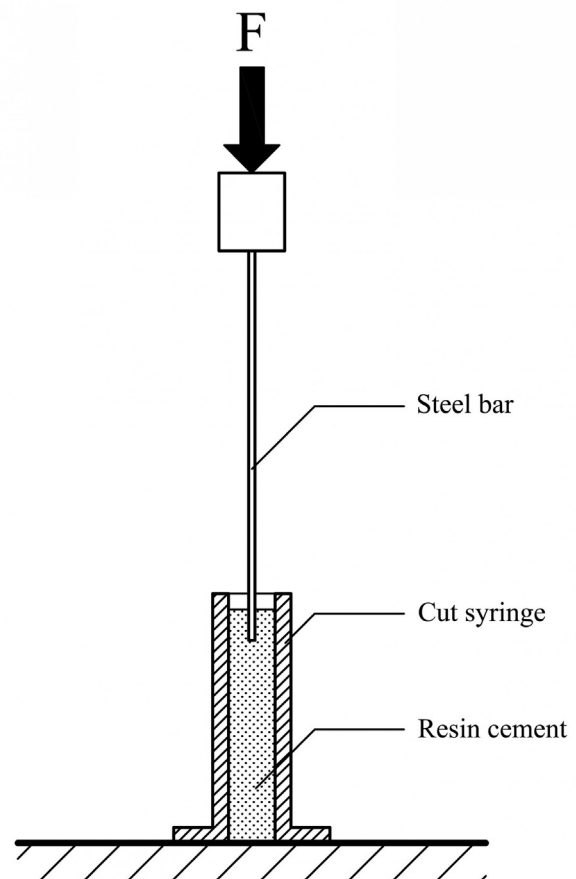


Fig. 1. Custom test arrangement for measuring solidification of the cement during autopolymerization. Force in N was used as the indicating unit.

were unwrapped and placed in water and stored at 37 °C. Water sorption was measured by gently drying the specimens and placing them on a scale. The specimens were measured on days 1, 2, 4, 7 and 14 after placing them in water and the weight change (water sorption) was measured.

After the last weighing of the specimens was carried out, a three-point bending test was performed using a dual column universal testing machine (Lloyd Instruments LR30K, Ametek Inc, Largo, FL, USA). The span was set to 20 mm and a 2.5 kN load cell was used. The results were recorded as maximum bending stress (MPa) over extension from preload (mm). In addition Young's modulus of bending was recorded for each sample using nano-indenter (Bruker NH-2, 1041, Tucson, AZ, USA) equipped with three sided Berkovich diamond indenter as described by Alaqeel et al (Alaqeel et al., 2019). During the testing, the sample was placed on the sample holder and held with glue tape and the tip was pressed onto the surface with preset loading and unloading values of 0.01 and 0.02 mN/s respectively,

The statistical analyses were performed using SPSS software (SPSS 21.0, IBM SPSS Statistics, Armonk, NY, USA) to determine non-parametric Spearman's correlation between variables for color change and unit of solidification. In addition mean values of flexural strength and water sorption at the time point of 14 days were compared using an independent samples T-test.

### 3. Results

The values obtained for color variation, curing speed (time for solidification), water uptake and flexural properties are represented in Figs. 2–6 accordingly. Results show the correlation

between curing process and color change during the autopolymerization. Also, the final flexural strength results underline the importance of additional light curing to reach maximal material strength.

In Fig. 2 the photographed change of color from pink to final shade can be seen to happen mostly during time period of 2–4 min after mixing. Quantification of the color change was further measured by spectrophotometry which were in line with the visual findings by placing the time of most color change between 1 min 30 s and 4 min after mixing. Results were measured in CIELAB color space, which uses one value (L) for luminance and two (“a” and “b”) for color. “L” is a value for lightness from black to white (0–100, respectively), “a” is a value for color from green to red (–128–127, respectively) and “b” is a value for color from blue to yellow (–128–127, respectively). The results are presented in Fig. 3.

In Fig. 4 the solidification of the cement (n = 6) starts at around 160 s mark after mixing (2 min 40 s) and thus seems to align well with the color change. Solidification reaches steady rise from 230 s (3 min 50 s) mark onward up to the testing limit (force of 50 N).

Weight change in Fig. 5 was measured to be up to 0.56 wt% (SD = 0.50, n = 8) for light initiated polymerization samples after 14 days and –1.12 wt% (SD = 0.75, n = 8) for the autopolymerized samples. Negative values seem to suggest some material was dissolving during the test.

Finally the same samples used in water sorption were tested by the three-point bending. Results are shown in Table 1 and representative load-deflection curves in Fig. 6. Results show that light initiated polymerization samples reached higher flexural strength in comparison with the autopolymerized samples.

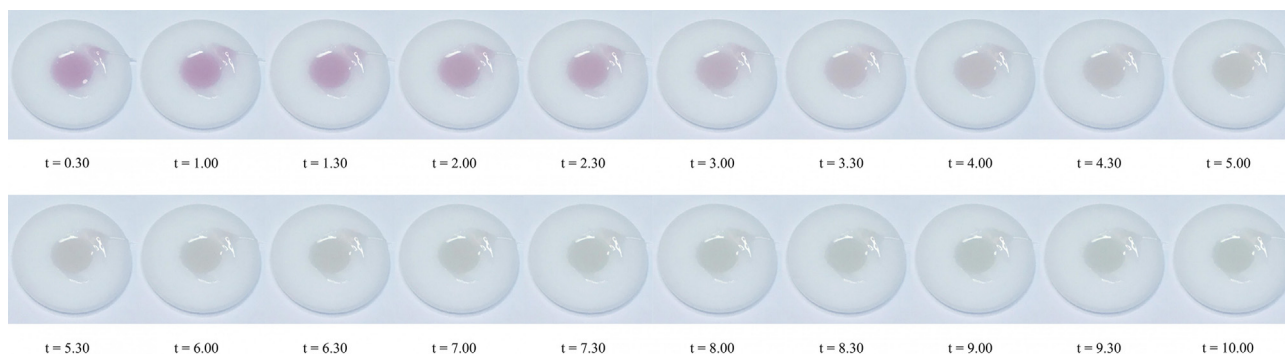


Fig. 2. Photographs of the color change of the cement during autopolymerization (“t” refers to time in minutes after the start of the autopolymerization).

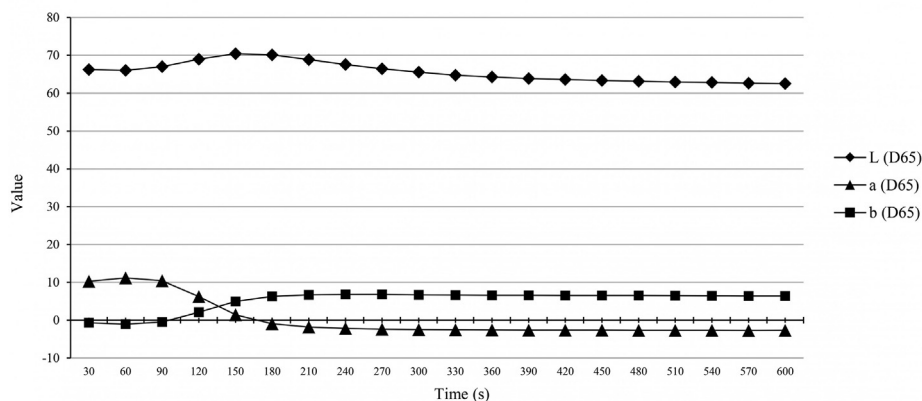


Fig. 3. Spectrometry values of the color change of the cement during autopolymerization. Results in CIELAB color space where “L” is the value for lightness (0–100), “a” is the value for color from green to red (–128–127) and “b” is the value for color from blue to yellow (–128–127). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

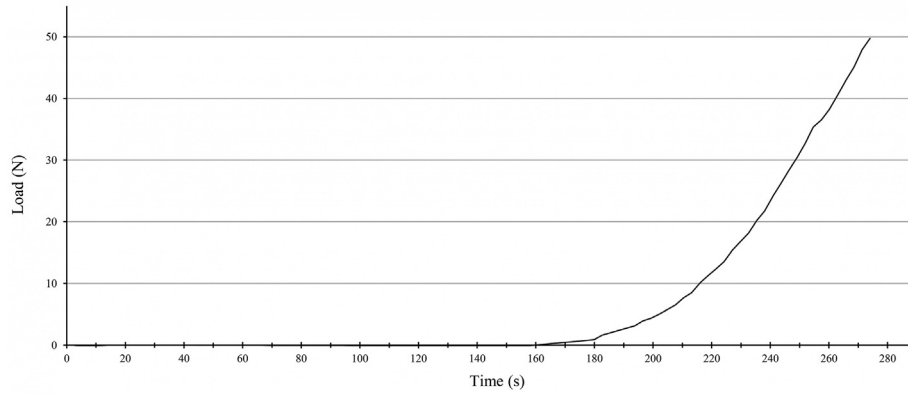


Fig. 4. Solidification profile of the cement during autopolymerizing.

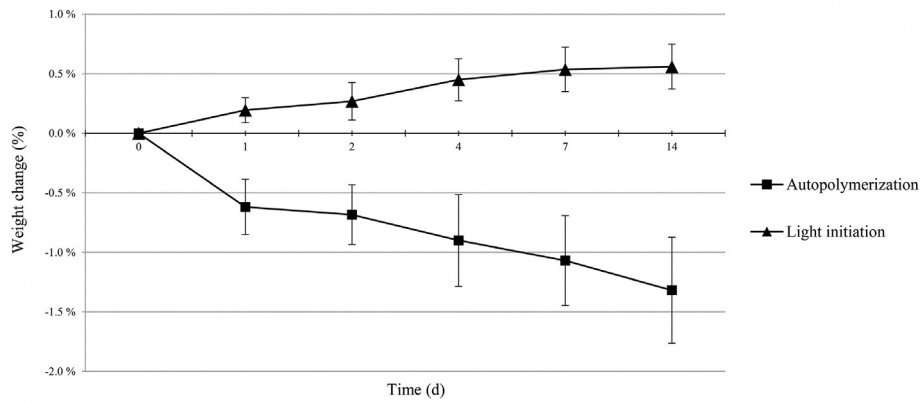


Fig. 5. Weight change in water of samples after the light initiated and autopolymerized cement samples after 1/2/4/7/14 days.

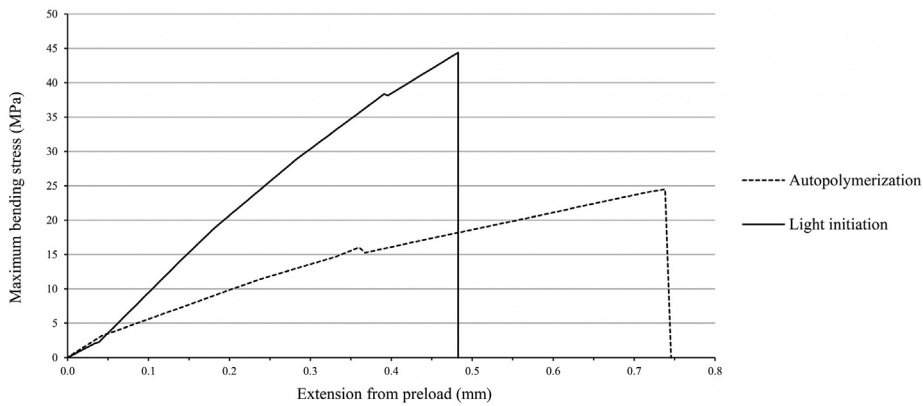


Fig. 6. Representative load-deflection curves for test specimens which were polymerized by light initiation and autopolymerization.

**Table 1**  
Results from three-point bending test for autopolymerized and light initiation polymerized cement samples.

	Flexural strength (MPa)		Young's modulus of bending (MPa)	
	Autopolymerization	Light polymerization	Autopolymerization	Light polymerization
Mean	27.34	48.06	2089.31	3781.51
S.D	5.06	7.52	586.62	425.31

Non-parametric Spearman's correlation was measured between three variables – two for color ("a" and "b" values) and one for curing (load value). For analysis time window of the color change between 01:00 and 04:30 (minutes) was chosen. Curing measurement data was further formatted to fit the spectrometry measurement (30 s intervals) by taking the mean value of 30 s interval (for the 1 min value the mean load from 30 s to 1 min was used).

The results show strong correlation between curing and color change with the correlation between the variable A and load varying from  $-1.000$  to  $-0.833$ , and between the variable B and load varying from  $0.833$  to  $1.000$ . The results were statistically significant ( $p < 0.05$ ).

Additionally, means of the final flexural strength and water sorption between autopolymerized and light initiation polymerized groups ( $n = 8$ ) were compared using an independent samples T-test. Analysis showed statistically significant difference between the groups ( $p < 0.05$ ) in both categories.

#### 4. Discussion

The aim of this study was to investigate the color change of a self-adhesive, dual-cured cement that provides a color cleanup indicator (Maxcem Elite™ Chroma, Kerr, Orange, CA, USA) and to evaluate its mechanical properties. A correlation between the color variation in the cement and its mechanical properties was found. The color fading indicated the optimal window of time to clean up excess cement.

The material investigated is available in a dual syringe, dosed through automix mixing tips and is of pink color when applied. The color then gradually changes into the final color shade, which occurs during the first minutes after mixing, when the cement is let to autopolymerize. After the pink color has faded any excess material is safe to be removed, according to manufacturer. The final bond strength to tooth substance and restoration is then achieved by light curing. The manufacturer also states that it is possible to use tack curing method to speed up the excess removal. When tack curing, cement is light cured for 1–2 s and then excess material is removed. The final curing is done according to the manufacturer's specifications. Tack curing method was not used in this study as the color change is near instant with light curing. As the initial visual inspection, digital photography and further testing of the autopolymerization process showed, the fading of most of the original pink color took place during the first 2–3 min, by which time the cement had started to cure and was already transformed from a gel state into a stiffer form.

Although the exact substances or mechanics causing the color change of the studied cement are not known, this study was undertaken to get more in-depth data on the nature of the color change and its relation to curing. In the field of polymer sciences, it has been shown, that the use of commercially available dyes and certain other reagents can result in a color change during polymerization (Hanna et al., 2001; Masere and Pojman, 1998). For example, the use of lead dithizone or carbazono in poly(methylmethacrylate) (PMMA) results in a color change from red to yellow during the polymerization (Hanna et al., 2001). This was explained by the previous study conducted by Hanna et al., which was mainly because of formation of lead dithizone complex. When lead dithizone is present, the PMMA polymerization proceeds by a free radical mechanism and the color change is attributed to formation of azo group by lead dithizone (affected by free radicals) (Hanna et al., 2001). When the polymerization proceeds and all of lead dithizone has reacted, the final color is achieved. The use of bromophenol blue with trimethylene glycol dimethacrylate (TGDMA) has also been recorded (Masere and Pojman, 1998). Masere and Pojman presented an alternative method to monitor the progress of

polymerization using dyes to scavenge free radicals. In this case the polymerization also proceeds by a free radical mechanism and the dye loses its color when reacting with the free radicals. By adjusting the amount of dye in the final product it could be possible to time the color change with a certain curing level. While bromophenol blue interacts via free radicals, it is worth mentioning that it also acts as a pH indicator, thus changing its color via a different reaction. As some resin cements exhibit a pH change when curing (Saskalauskaitė et al., 2008) it might also be possible to use such common pH indicator as pentamethoxy red to determine the degree of cure.

The curing process, i.e. the solidification of the autopolymerizing cement was studied in a custom testing arrangement. The used method was chosen by reason of that spectrometry was not an option due to the self-etching properties of the cement, and thus a potential damage to the infrared sensor would be involved. The testing arrangement provided results of the time needed for solidification of the cement and showed a slowly speeding up curing from around 160 s after mixing. The curing speed then started to accelerate and reached a steady rise after 230 s. The curing speed and timing was in relation to the color change happening around 2–3 min mark as shown in the spectrometry and visual examination.

The weight change (water uptake) of the light cured samples was measured to be 0.56 wt% after 14 days but the autopolymerized samples resulted in a negative value of  $-1.12$  wt% during the same period of time. According to Rashin et al., this can be explained by the property of solubility and absorption seen in most of the resin cements, and this property also depends on resin cement composition and surrounding environment (Rashin et al., 2016). Resins with lower filler content shows higher water sorption and resins with higher matrix are more vulnerable to hydrolytic degradation. This would suggest that there are compounds leaching from the non-light cured samples. It is likely that major releasing compounds are residual monomers. More detailed analysis of the leaching compounds could have been performed by high performance or gas chromatography and there are interests by the authors for studying this aspect in the future studies. The three-point bending test showed a clear difference in flexural strength in favor of the light cured samples. Autopolymerized samples did show higher extension from preload before breaking but with considerably lower flexural strength. Findings are in line with the fact that composite cements such as the one under study here achieve higher degree of conversion, even if light cured through ceramic material (Saskalauskaitė et al., 2008; Luhrs et al., 2014). Thus, it is suggested that light curing is required for optimizing curing of the cements and clinical outcome of indirect restorations cemented with the studied luting cement.

#### 5. Conclusion

The results of this study showed that there was a correlation between the color change and state of curing of the dual curing resin composite luting cement which contained a color indicator system for polymerization reaction. The study also suggested that autopolymerization only was not adequate for the optimal polymerization of the cement. By light curing considerably higher flexural strength was obtained.

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## Declaration of Competing Interest

The author declares no conflicts of interest.

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