AN ALTERNATIVE SPECIMEN PREPARATION TECHNIQUE FOR 3-POINT BENDING TESTS ON DUAL-CURED DENTAL RESIN CEMENTS

TÉCNICA ALTERNATIVA DE CONFECÇÃO DE ESPÉCIMES PARA TESTES DE FLEXÃO DE 3 PONTOS EM CIMENTOS DE RESINA DENTÁRIA DE DUPLA ATIVAÇÃO

Ana Carla Assunção OLIVEIRA[1]; Christiane Marques Tavares BARRETO[2]; Luiz Alves OLIVEIRA-NETO[3], Rafael Ratto MORAES[4]; André Luis FARIA-E-SILVA[5]

1. Graduate student, Graduate Program in Dentistry, Federal University of Sergipe, Brazil; 2. Graduate student, Graudate Programa in Health Sciences, Federal University of Sergipe, Brazil; 3. Posdoctoral researcher, Graduate Program in Dentistry, Federal University of Sergipe, Brazil; 4. Professor at Department of Restorative Dentistry, Dental School, Federal University of Pelotas, Brazil; 5. Professor at Department of Dentistry, Federal University of Sergipe, Brazil.

ABSTRACT: The proper mechanical properties of resin cements are essential to the longevity of indirect restoration, whereas the 3-point bending test is recommended for measuring the flexural strength. The ISO 4049 specification requires light-curing of specimens in three consecutive points; however, this approach cannot be used for dual-cured resin cements. The aim of this study was to investigate the effect of two different specimen preparation techniques on the flexural strength and elastic modulus of experimental dual-cured resin cements immediately or 5 after minutes light curing. Experimental dual-cured resin cements were formulated, and the specimens of these cements were confectioned with the dimension of ISO 4049 specification. Light-activation was performed at one or three points immediately or 5 minutes after the insertion of cement into the matrix (n=7), resulting in four experimental conditions (2 methods × 2 moments of light-activation). The three-point bending test was performed and the values of the flexural strength and elastic modulus were recorded. Data were individually analyzed using 2-way ANOVA followed by the Tukey's post hoc test ($P<0.05$). Regardless of the points of light-activation, the specimens that were light-cured at 3 points showed the highest values of flexural strength. Only for light-activation at 1 point did the time before the light-activation affect the elastic modulus, whereas delayed light-activation had the highest values. In conclusion, the number of light-curing points on specimen preparation for the 3-point bending test seems to affect the mechanical properties of dual-cured resin cements.

KEYWORDS: Compressive strength. Polymerization. Resin cements.

INTRODUCTION

Dual-cured resin cements are commonly used to lute indirect restorations and intra-radicular dental posts with the aim of combining the advantages of chemically and light-cured polymer-based materials (FARIA-E-SILVA, et al. 2007). The rationale is to have a material that combines extended working time with capacity for reaching proper polymerization in either the presence or absence of light. Proper polymerization is related to improved mechanical properties and may ultimately impact the longevity of the restorative procedures (ILIE; SIMO, 2012). One of the more commonly used laboratory tests for evaluating the mechanical properties of dental resin cements is the 3-point bending test, which is a useful test for determining the flexural strength and flexural modulus (elastic modulus) of the materials (CHUNG, et al. 2004; DUYMUS, et al. 2013; GONÇALVES, et al. 2013). These mechanical properties are largely used for characterizing dental materials.

The International Organization for Standardization (ISO) specification 4049 establishes the standards for performing the 3-point bending test for polymer-based dental materials (ISO, 2009). The test consists of the application of a compressive load until there is failure on the center of a bar-shaped specimen that is 25 mm in length, 2 mm in height and 2 mm in width. The bar specimen is supported by two rods, 2 mm in diameter and mounted parallel with a 20 mm span between the supports; meanwhile, the load is applied through a rod that is 2 mm in diameter. This standardization establishes that the specimen fabrication should be performed with the light-cured resin materials inserted into a mold and then followed by light-activation in three consecutive, overlapping areas in both sides of the specimen. This approach is necessary because the length of the specimens is larger than the diameter of the light-curing unit guide. The center of the specimen receives the first light exposure, which is followed by two complementary exposures at equidistant points from the center.

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Although the ISO 4049 specification is largely used for testing composites that are light-cured only, the technique recommended for specimen preparation hinders the evaluation of the possible effects of light-activation moment on mechanical properties of dual-cured resin-based materials (MORAES, et al. 2009; FARIA-E-SILVA, et al. 2011; KHOROUSHI, et al. 2012; FARIA-E-SILVA, et al. 2012; SOUZA, et al. 2013; FARIA-E-SILVA, et al. 2014)

By the time dual-cured materials are inserted into the mold, the polymerization reaction has already started via chemical activation. Therefore, each light-activation is performed over areas of the polymer, presenting with differences in the degrees of C=C conversion impairing the standardization of time for light-activation (FARIA-E-SILVA, et al. 2012; KHOROUSHI, et al. 2012). Using single light-activation in the center of the specimen with the tip of the light-curing unit, away from the composite, is an alternative. However, to provide light exposure covering the entire bar specimen, a reduction in light irradiance in areas away from the center is expected. Another alternative is to perform light-activation at three equidistant points simultaneously using three light-curing units. To the authors' knowledge, this simple approach has not yet been reported.

The aim of this study was to evaluate the effect of light-curing methods using exposure either at a single area, at the center of the bar, or three simultaneous light exposures, covering the entire bar on the flexural strength and elastic modulus of dual-cured resin cements. We tested the hypothesis that the use of the method of three simultaneous light exposures affects the flexural strength and flexural modulus data.

MATERIAL AND METHODS

Study design

This investigation was conducted using a 2 × 2 factorial study design to evaluate the 'light-curing method' in two levels (single exposure at the center or three simultaneous exposures along the bar) and 'time before light-activation' of an experimental dental resin cement in two levels (immediately or 5 min after inserting the cement into the mold). The response variables evaluated were the flexural strength (σ_f) and flexural modulus (E_f) obtained through a 3-point blending test.

Formulation of the experimental dual-cured resin cement

A model dual-cured resin luting agent was formulated using the monomers 2,2 – bis[4–(2-hydroxy-3-methacryloxyprop-1-oxy)pheny1]propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) at a 3:1 mass ratio. The monomers were obtained from Esstech Inc. (Essington, PA, USA). Silanated barium borosilicate glass fillers, 2 µm in average diameter (Esstech Inc.), were added at 65 mass%. The cement was consisted of two pastes, one labelled base paste and another labelled catalyst paste. Camphorquinone (0.8 mass%) and diethanol-*p*-toluidine (3 mass%), both from Esstech Inc., were added to the base paste as the photoinitiator and co-initiator. Benzoyl peroxide (Vetec, Rio de Janeiro, RJ, Brazil) was added to the catalyst paste at a 3 mass% as self-activated initiation system. Butylated hydroxytoluene (0.2 mass%) was added to both pastes as a radical scavenger.

Specimen preparation for the 3-point bending test

Specimens were prepared according to the bar-shaped dimensions specified by the ISO 4049 standard.[6] Equal volumes of base and catalyst pastes were mixed for 15 s and inserted into a metallic split mold with 25 mm in length, 2 mm in width and 2 mm in height. The material was covered by an acetate strip and light-activation was performed using identical light-emitting-diode (LED) light-curing units (Radii-Cal; SDI, Bayswater, Victoria, Australia) with 1200 mW/cm^2 irradiance each.

For the light-activation using a single light exposure area, the tip of the light-curing unit was fixed 1 cm away from the mold and positioned at the center of the specimen for the polymerizing light to reach the entire bar. Light-activation was performed for 180 s. For light-activation using three simultaneous light exposures, three light-curing units that were used at the same time were also positioned 1 cm away from the specimen, but they were distributed at equidistant points from the center of the bar (Figure 1). Light-activation was performed for 60 s to generate the same radiant exposure of the method using a single light exposure area. For both light-curing methods, light-activation was performed immediately or 5 min after inserting the resin cement into the mold. The light-activation procedures were performed at both the top and bottom sides of each specimen; therefore, the total radiant exposure for each specimen was 43.2 J/cm^2. The cured specimens were wet-polished with #1200-grit SiC papers and stored in distilled water at 37±1°C for 24 h in the dark. Specimens presenting any void or otherwise defect under visual analysis were replaced.

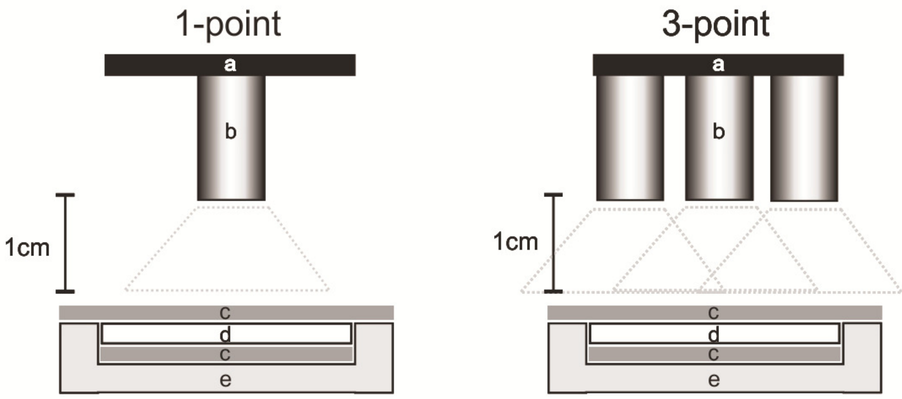

Figure 1. Schematic illustration of the light-activation procedures during specimen preparation. a – Hold to standardize the tip position of light-curing unit; b – Tip of light-curing unit; c – Acetate strips; d – Resin cement; and e – Mold.

Three-point bending test

The dimensions of the bars were checked with a digital caliper accurate to 0.01 mm (Mitutoyo Corporation, Tokyo, Japan). The specimens were positioned in a 3-point bending device coupled to a mechanical testing system (Instron 3367, Instron Corp., Canton, MA, USA). The distance between supports was 20 mm and the load was applied to the center of specimen. The diameter of both supports and of the loading rod was 2 mm (SOUZA, et al. 2013). The tests were performed at a crosshead speed of 0.5 mm/min until failure and was monitored by the testing machine software (Bluehill 2, Instron Corp.). To calculate σ_f (MPa), the following equation was used:

$$\sigma_f = \frac{3Fl}{2bh^2} \quad \text{(Eq. 1)}$$

where F is the maximum load (N) exerted on the specimen, l is the distance (mm) between the supports, and b is the width (mm) and h the height (mm) at the center of the specimen. The E_f was calculated using the following equation:

$$E = \frac{F_1 l^3}{4bdh^3} \quad \text{(Eq. 2)}$$

where F_1 is the load (N) exerted on the specimen and d is the deflection corresponding to the load F_1. Data for the σ_f and E_f showed normality (Kolmogorov-Sminorv, P > 0.05) and equal variance (Levene's test, P > 0.05) were individually submitted to 2-way analysis of variance. All pairwise, multiple comparison procedures were performed using the Tukey's method (α = 0.05). Data analysis was performed using the SigmaStat v.3.5 statistical software package (Systat Software Inc., Chicago, IL, USA).

RESULTS

The results for σ_f are shown in Table 1. The statistical analysis revealed a significant effect only for the factor 'light-curing method' (P < 0.001), while the factor ''time before light-activation' (P = 0.646) and interaction between the factors (P = 0.483) were not significant. The results were expressed as pooled averages for both times. The σ_f was significantly higher for the light-curing method using three simultaneous exposures.

Table 1. Means (SD) of flexural strength in MPa (n=7).

Light-curing method	Time before light-activation		Pooled averages
	Immediate	Delayed	
Single exposure	65.0 (11.3)	63.3 (12.6)	64.2 (11.2) B
Three simultaneous exposures	108.4 (25.8)	116.3 (15.6)	112.3 (20.9) A

For pooled averages, distinct letters indicate significant differences ($P \leq 0.05$).

Results for the E_f are shown in Table 2. The statistical analysis showed a significant effect for the factor 'light-curing method' (P = 0.038) and for the interaction between factors (P = 0.013). The factor ''time before light-activation' (P = 0.94) was not significant. Differences between light-curing methods were observed only for the delayed light-activation procedure, whereas the method that used three simultaneous exposures had the highest values. Irrespective of the light-curing method, no significant difference was observed between the times before light-activation.

Table 2. Means (SD) of the flexural modulus in GPa (n=7)

Light-curing method	Time before light-activation	
	Immediate	Delayed
Single exposure	7.6 (0.3) A.a	7.0 (0.4) A.b
Three simultaneous exposures	7.6 (0.8) A.a	8.4 (1.2) A.a

Distinct letters (uppercase for line, lowercase for column) indicate significant differences ($P \leq 0.05$).

DISCUSSION

The findings of this study demonstrated that the number of light-curing points significantly affected the σ_f of the dual-cured resin cement, irrespective of the time before light-activation. Light-curing the samples in 3 points resulted in almost 2-fold higher σ_f compared to light-curing in a single point. For the measured values of E_f, the number of light-curing points only affected the values 5 minutes after light-curing was performed, whereas 3-point light-curing increased the elastic modulus. As a result, the hypothesis of study was accepted.

Despite the presence of a polymerization reaction activated by a chemical reaction, several studies have demonstrated that dual-cured resin cements require light curing to improve their polymerization potential and mechanical properties (FARIA-E-SILVA, et al. 2007; FARIA-E-SILVA, et al. 2012; CALGARO, et al. 2013; KIM, et al. 2013; MAGALHÃES, et al. 2014). In this study, we used a light-curing device tip with an approximately 8-mm diameter, whereas the ISO 4049 specification recommends samples with a 25-mm length. Considering that the light beams emitted by device tip are divergent, the diameter of light over the samples tends to increase with longer distances from the tip (FELIX; PRICE, 2003). However, increasing the distance between the tip and sample also reduces the energy density (PRICE, et al. 2011). Attempting to polymerize the sample with single-point light-curing, the tip of the light-curing device was positioned 10 mm from the sample in the present study. A reduction of approximately 75% in the energy density when the tip is positioned at 10 mm from the sample has been reported (PRICE, et al. 2011). This significant reduction in the energy density reduces the polymerization of resin cement and its mechanical properties, explaining the lowest values observed when only one point was used to light-cure the samples (HALVORSON, et al. 2002; BAEK, et al. 2008; GRITSCH, et al. 2008). The use of the other two additional points for light-curing increases the energy available to polymerize the resin cement. Additionally, 3-point light curing reduces the inhomogeneous irradiance profile of irradiance provided by the light-curing device, resulting in more proper polymerization of cement (PRICE, et al. 2010; PRICE, et al. 2011; MICHAUD, et al. 2014). However, it is important to emphasize that the values of the σf (approximately 65 MPa) reached by samples that were light-cured in a single point was superior to the minimum values required by ISO standardization (50 MPa) (ISO, 2009).

Interestingly, the number of light-curing points only affected the E_f of resin cement for the delayed light-activation. Delayed light-activation has been advocated to slow the polymerization reaction and reduce the polymerization stress of dual-cured resin cements (STAVRIDAKIS, et al. 2005; FARIA-E-SILVA, et al. 2011) The slower chemical polymerization in the first minutes allows for an increase in the duration of the pre-gel polymer stage, resulting in increased flow of the cement and reduced polymerization stress (FARIA-E-SILVA, et al. 2011; FENG; SUH, 2006a; FENG; SUH, 2006b)

Therefore, delaying the light-activation of dual-cured cements allows for relief of this stress, whereas the light-activation is performed when there is a significant conversion of materials (FARIA-E-SILVA, et al. 2011). It has been demonstrated that the slowest polymerization reaction may results in polymers with reduced elastic modulus (SOH; YAP, 2004; YAP, et al. 2004; FENG; SUH, 2006b). However, no significant differences were observed in the times before light-activation, irrespective the number of light-curing points.

An important observation of the outcomes in the present study was that the number of light-curing points affected the E_f for only the delayed light-activation mode. A reasonable explanation for these findings can be related to the molecular mobility of reactional media in the moment of light-activation. For immediate light-curing, only a small number of resin monomers react in the moment of light incidence and the high mobility of reactional media allows for achieving additional polymerization even for a low energy density (RUEGGEBERG; CAUGHMAN, 1993). By contrast, higher conversion is expected 5 minutes after mixing resin cement with reduced mobility reaction media. Therefore, a higher energy density can be required to promote a significant improvement in the reactive sites. A higher number of polymerization reactive sites has been related to increased elastic modulus (FENG; SUH, 2006b; SOH; YAP, 2004; YAP, et al. 2004).

In the present study, the sample preparation method for the 3-point bending test significantly affected the mechanical properties of dual-cured resin cements. The alternative method suggested in this study is simultaneous light-curing of the sample in three different points, resulting in increased σ_f. This method also increases the E_f of cements that are light-cured after 5 minutes of mixing. According to ISO 4049 specification, this method uses 3 points of light-curing, allowing for a more homogeneous polymerization. In the present study, the method using consecutive light-activations (similar to ISO 4049) was not evaluated once that this does not allow to evaluate the effect of the moment of light-activation, which is one factor evaluated. The recommendation of ISO 4049 can be suitable to dual-cured materials when the moment of light-activation is not factor of study. However, differences between the polymer obtained using ISO recommendation and the alternative technique proposed in this study require further evaluation.

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RESUMO: Cimentos resinosos com propriedades mecânicas adequadas são essenciais para a longevidade de restaurações indiretas, sendo que o ensaio de flexão de 3 pontos é recomendado para mensurar a resistência flexural. A especificação ISO 4049 requer a fotoativação das amostras em três pontos consecutivos, entretanto, esta abordagem não pode ser usada para cimentos resinosos duais. O objetivo deste estudo foi investigar o efeito e duas diferentes técnicas de preparo de amostras na resistência flexural e módulo de elasticidade de cimentos resinosos duais experimentais fotoativados imediatamente ou após 5 minutos. Cimentos resinosos duais experimentais foram formulados, e amostras destes cimentos foram confeccionadas com as dimensões da especificação ISO 4049. A fotoativação foi realizada em um ou três pontos imediatamente ou após 5 minutos da inserção do cimento na matriz (n=7), resultando em quatro condições experimentais (2 métodos × 2 tempos antes da fotoativação). O teste de flexão de três pontos foi realizado e os valores de resistência flexural e módulo de elasticidade mensurados. Os dados foram individualmente analisados por ANOVA de dois fatores seguido pelo teste de Tukey (P<0,05). Em relação aos pontos de fotoativação, as amostras que foram fotoativadas em três pontos mostraram os maiores valores de resistência flexural. Apenas para a fotoativação em um ponto, o tempo antes da fotoativação afetou o módulo de elasticidade, sendo que a fotoativação tardia apresentou maiores valores. Em conclusão, o número de pontos de fotoativação no preparo das amostras para teste de flexão de 3 pontos parece afetar as propriedades mecânicas dos cimentos resinosos.

PALAVRAS-CHAVE: Resistência à compressão. Polimerização. Cimentos resinosos.

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