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The effect of shade and curing mode on cure efficiency of dual-cure resin cements

Efeito da cor e do modo de cura na eficiência de cura de cimentos resinosos de cura dual

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ABSTRACT

Objective: To evaluate the effect of shade and curing mode on cure efficiency of two dual-cure resin cements. **Material and Methods:** Two shades (A2 and TRANS) of two different dual-cure resin cements (G-CEM, GC Dental and SET PP, SDI) were submitted to different curing modes: light curing through a 2 mm thick ceramic disc (IPS Empress Esthetic, A2, Ivoclar Vivadent) for 20 s (16 J/cm2), 40 s (32 J/cm2) or 80 s (64 J/cm2) performed immediately or with 1 or 5 min of delay. Fourier transform infrared spectroscopy (FT-IR) was used to evaluate the degree of conversion (DC) after 48 h. For the statistical analysis, data were submitted to three-way analysis of variance, followed by Tukey's test for multiple comparisons ($p<0.05$). **Results:** Shade influenced DC (%) for the dual-cure resin cements tested ($p=0.00001$). TRANS shade showed lower DC (%) for both cements when there was no delay before light-curing ($p=0.00001$). Curing mode also influenced DC (%) for the dual-cure resin cements tested: radiant exposure greater than 32 J/cm2 and delaying light-curing for 1 to 5 min increased the DC (%) for both dual-cure resin cements evaluated. **Conclusions:** One minute delay prior to light-curing improved the cure efficiency and may be a more clinically acceptable approach to increase the degree of conversion of dual-cure resin cements.

KEYWORDS

Ceramics; Resin cements; Color; Polymerization.

RESUMO

Objetivo: Avaliar a influência da cor e do modo de polimerização no grau de conversão de dois cimentos resinosos duais. **Material e Métodos:** Duas cores (A2 e TRANS) de dois cimentos resinosos duais (G-CEM, GC Dental e SET PP, SDI) foram submetidos a diferentes modos de cura: fotoativação através de um disco de cerâmica de 2 mm de espessura (IPS Empress Esthetic, A2, Ivoclar Vivadent) por 20 s (16 J/cm2), 40 s (32 J/cm2) ou 80 s (64 J/cm2) realizada imediatamente ou após 1 ou 5 min de espera. Espectrometria com transformada de Fourier (FT-IR) foi utilizada para avaliação do grau de conversão (GC) após 48 h. Para a análise estatística, os dados foram submetidos à análise de variância com três fatores, seguida de teste de Tukey para comparações múltiplas ($p=0.05$). **Resultados:** A cor influenciou o GC (%) dos cimentos resinosos duais avaliados ($p=0.00001$). A cor TRANS resultou menor GC (%) para ambos os cimentos quando não houve espera antes da fotoativação ($p=0.00001$). O modo de cura também teve influência n o GC (%) dos cimentos resinosos duais avaliados: doses de energia superiores a 32 J/cm2 e espera de 1 ou 5 min antes da fotoativação aumentou o GC (%) dos dois cimentos resinosos duais avaliados. **Conclusão:** Um min de espera antes da fotoativação melhorou a eficiência de cura e pode ser uma abordagem clinicamente aceitável para aumentar o grau de conversão de cimentos resinosos duais.

PALAVRAS-CHAVE

Cerâmicas; Cimentos dentários, Cor; Polimerização.

INTRODUCTION

Dual-cure resin cements have been widely used in clinical practice [1], especially when photo-activation can be impaired by low light absorption due to the distance from the luting area to the light source, as in fiber posts cementation [2-4], or due to light scattering by the thickness and opacity of indirect restorations [5-8].

Effective light curing of resin cements depends on photon absorption in order to excite the photo-initiators and initiate polymerization; photo-absorption depends on the capacity of light flux through the overlying material [5-6]. The light flux is mainly inhibited by increased thickness and opacity of indirect restorations during cementation [5-7]. The color of resin cements also influences monomer conversion [8-10]. Low light absorption by light-cured resin cements can adversely affect degree of conversion and consequently, chemical-mechanical properties and marginal integrity leading to clinical failure [11]. The combination of light-cure and self-cure initiators in dual-cure resin cements can improve the polymerization in these cases, in that the chemical catalyst promotes polymerization irrespective of the photo-activation [2-7]. Dual-cure resin cements help to compensate for problems caused by light flux attenuation in photo-activation. However, an effective curing mode protocol to achieve satisfactory adhesive cementation is still not established [12].

Many methods have been used to improve monomer conversion. The greater the radiant exposure (J/cm2) and smaller the polymerization area (cm2), the greater is the radiant energy (J) increasing the degree of conversion up to the gel point of the polymer formed [13]. Delayed light-curing of dual-cure resin cements can also provide an increase in the degree of conversion due to a slower reaction to form the polymeric chains, thus delaying the gel point [14].

The degree of conversion analysis can be used as a parameter to predict better clinical approaches. Fourier transform infrared spectroscopy (FT-IR) and Raman vibrational spectroscopy are sensitive methods for providing detailed information about monomer conversion [15]. Thus, the aim of this study was to evaluate the effect of different shades and curing modes on degree of conversion of two dual-cure resin cements. The research hypotheses tested were that: (1) the degree of conversion of dual-cure resins depends on the shade of each resin material; (2) the radiant exposure and the delayed light-curing protocol will influence the degree of conversion of the dual-cure resin cements tested.

METHOD AND MATERIALS

Table 1 lists the two dual-cure resin cements used in this study: G-CEM (GC Dental, Bunkyo-ku, Tokyo, Japan) and SET PP (SDI, Bayswater, Victoria, Australia), in two different shades, A2 and transparent (TRANS). Light-curing was performed through a ceramic disc with 5 mm of diameter and 2 mm thick, IPS Empress Esthetic, A2 (Ivoclar Vivadent, Schaan, Liechtenstein) for 20 s (16 J/cm^2), 40 s (32 J/cm^2) or 80 s (64 J/cm^2), immediately or with 1 or 5 min delay, using the same dental light curing unit (Radii, SDI, Bayswater, Victoria, Australia) previously characterized with 800 mW/cm^2 using a radiometer (Demetron LED Radiometer, Kerr Corp, California, USA) in order to standardize the radiant exposure according to the different curing modes tested.

Cure efficiency analysis

Fourier transform infrared (FT-IR) spectroscopy (Spectrum 100, PerkinElmer, Waltham, MA, USA) coupled to an attenuated total reflectance (ATR) was used to measure the degree of conversion of the dual-cure cements tested according to the curing modes evaluated. Absorbance spectra included 16 scans at a resolution of 1 cm^{-1}. Non-polymerized materials

Table 1 - Commercial name (manufacturer), application mode, resin composition and filler content of each dual-cure resin cement

Commercial Name (Manufacturer)	Application Mode	Resin Composition	Filler content
G-Cem (GC Dental, Tokyo, Japan)	Auto-Mixture Syringe	Paste A: 13% urethanedimethacrylate; 13% dimethacrylate Paste B: 31% urethanedimethacrylate; 6% dimethacrylate; 4% phosphoric acid ester monomer; 1% initiator	71% fluoro-alumino-silicate glass
Set-PP (SDI, Victoria, Australia)	Auto-Mixture Syringe	<20% urethane dimethacrylate; <1% initiator; <20% acidic monomer	60-70% fluoroaluminosilicate glass

were scanned within a Teflon mold (= 4 mm, 0.1 mm thick) placed on the ATR. The cements were then photo-activated through a polyester strip and the ceramic disk with different curing modes, as previously described. The polymerized specimens were scanned 48 h later, and unconverted carbon double bonds were quantified by calculating the percentages derived from the aliphatic C = C (vinyl) absorption (1638 cm^{-1}) to the aromatic C = C absorption (1608 cm^{-1}) signals for both polymerized and non-polymerized specimens (n = 3). The degree of conversion (DC) was calculated according to the follow equation:

DC (%) = $\{1-(Xa/Ya)/(Xb/Yb)\}\times 100$, in which, Xa (polymerized) and Xb (non-polymerized) represent the bands of the polymerizable aliphatic double bonds, and Ya (polymerized) and Yb (non-polymerized) represent the bands of the aromatic double bond.

Statistical analysis

Shapito-Wilk normality and Levene's tests were performed and, then, data was submitted to three-way analysis of variance (ANOVA) followed by Tukey's test for multiple comparisons (= 0.05; = 0.2) using Stata/MP 13.0 (StataCorp, College Station, TX, USA).

RESULTS

The irradiance from the light-curing unit measured using a radiometer (Demetron LED Radiometer, Kerr Corp, Orange, CA, USA) resulted in an irradiance of 800 mW/cm^2 at the light tip and 100 mW/cm^2 through the ceramic disk. Table 2 shows the three-way analysis of variance for degree of conversion and Figures 1 and 2 show the mean standard deviation of the degree of conversion (%) for each shade and

Table 2 - Three-way analysis of variance (ANOVA) for degree of conversion (%).

Source of variation	df	SS	MS	F	P
Color	1	825,095,059	825,095,059	622,748	0.00001
Exposure	2	20,142,972,972	10,071,486,486	7,601,541	0.00001
Delay	2	5,930,356,909	2,965,178,454	2,237,994	0.00001
Color x Exposure	2	1,488,306,158	744,153,079	561,656	0.00001
Color x Delay	2	143,265,121	71,632,561	54,065	0.00001
Exposure x Delay	4	998,632,689	249,658,172	188,432	0.00001
Color x Exposure x Delay	4	725,260,133	181,315,033	136,849	0.00892
Residual	36	476,973,715	13,249,270		0.00001
Total	**53**	**30,730,862,756**			**0.00001**

Abbreviations: MS, mean squares; SS, sum of squares.

curing mode tested for the SET PP and G-CEM, respectively.

As can be observed in Table 2, the shade and the curing mode influenced DC (%) of the dual-cure resin cements tested (p = 0.00001). TRANS shade showed significantly lower DC (%) for both cements when there was no delay before light-curing (p = 0.00001).

Curing mode also influenced DC (%) of the dual-cure resin cements tested (p = 0.00001): radiant exposure greater than 32 J/cm^2 and delaying light-curing for 1 to 5 min increased the DC (%) for both dual-cure resin cements evaluated. Five minutes of delay before light curing significantly enhanced DC (%) to the maximum mean values for both A2 and TRANS shades for both resin cements tested; 64 J/cm^2 radiant exposure to light cure the A2 shade SET PP showed similar DC (%), regardless of the delay.

DISCUSSION

The first research hypothesis tested, that the degree of conversion of dual-cure resins depends on the shade of each resin material, was accepted. As observed in Table 2, the shade significantly influenced DC (%) of the dual-cure resin cements tested (p = 0.0001). The lower DC mean values for TRANS shade dual-cure resin cements compared to the A2 shade is most likely due to a lower camphorquinone concentration or the addition of a different photo-initiator in order to reduce the yellowish color caused by camphorquinone [17]. This may also explain the increase in degree of conversion with the higher radiant exposure or delayed light curing protocols regardless of the shade of the dual-cure resin cement tested (Figures 1 and 2). The absorption peak of these alternative photo-initiators are not in the same region of camphorquinone [18], thus a higher radiant exposure is necessary to improve monomer conversion and increase the degree of conversion as observed in this study.

The second research hypothesis tested, that the radiant exposure and the delayed light-curing protocol would influence the degree of conversion of the dual-cure resin cements

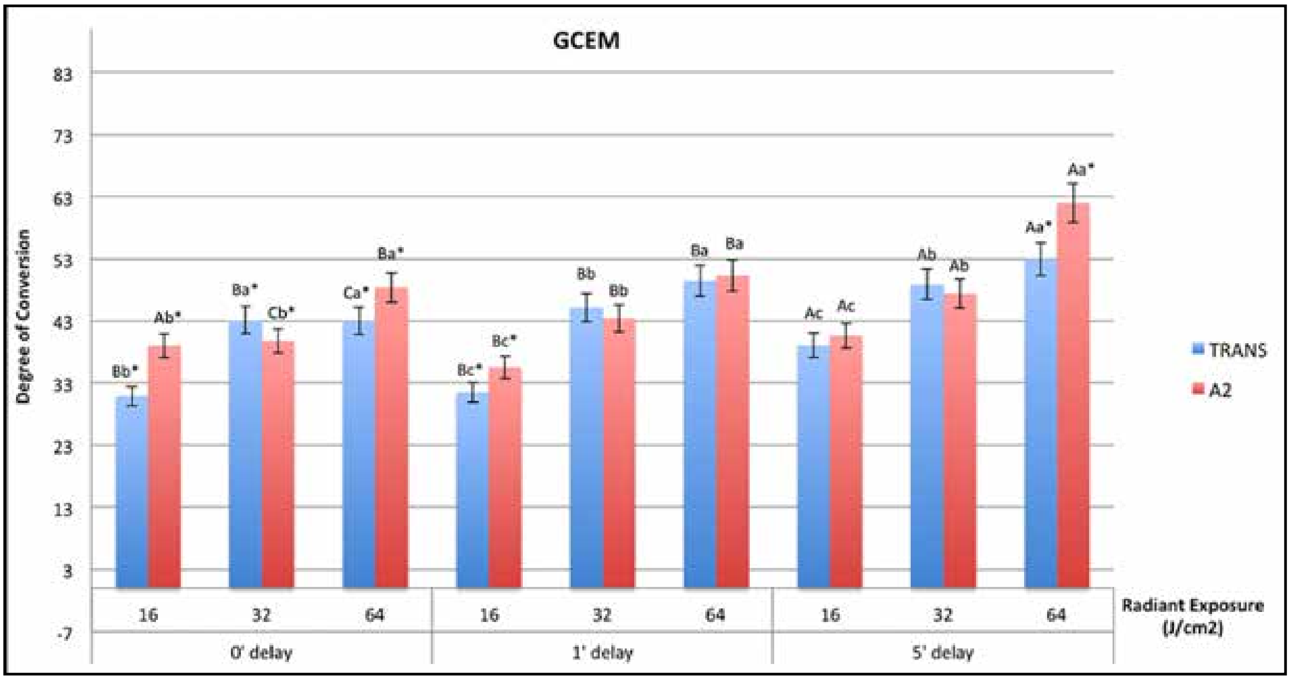

Figure 1 - For each of the curing modes of GCEM cement, the mean DC value (%) ± standard error is provided.

* Means followed by different capital letter show significant difference for the delay factor and small letters, for the radiant exposure and * for the color factor (p < 0.05).

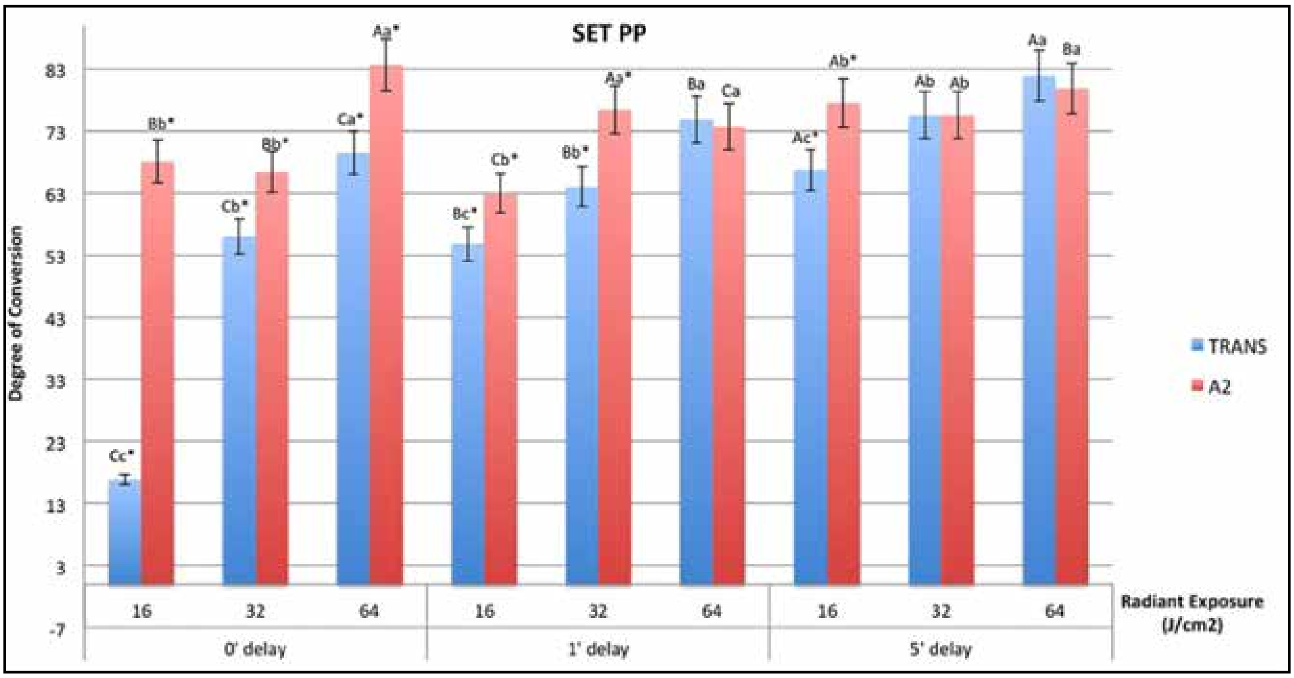

Figure 1 - For each of the curing modes of SET PP cement, the mean DC value (%) ± standard error is provided.

* Means followed by different capital letter show significant difference for the delay factor and small letters, for the radiant exposure and * for the color factor ($p < 0.05$).

tested was also accepted. When a higher radiant exposure or delayed light-curing protocols were applied, the degree of conversion was enhanced for both dual-cure resin cements tested, regardless of the shade (as previously observed in Figures 1 and 2). The irradiance from the light-curing unit resulted in an irradiance of 800 mW/cm2 at the light tip and 100 mW/cm2 through the ceramic disk. This light flux reduction resulted in only 12.5% of the light being transmitted through the 2 mm thick A2 IPS Empress ceramic disc.

Recent studies show that the main cause of reduction in degree of conversion is the thickness of ceramic [16]. As observed in several studies, light transmission is inversely related to ceramic thickness [6], to the extent that less than 2% of light is transmitted through 4mm thickness of ceramic [5]. As previously measured, the irradiance through the light tip was reduced from 800 mW/cm^2 to 100 mW/cm^2 when the 2 mm thick ceramic disc was placed at the tip: thus 12,5% of light was actually transmitted through the 2 mm A2 IPS Empress ceramic disc.

However, the increase in degree of conversion is restricted by the gel point of the polymer [13]. This would explain why delay prior to light-curing dual-cure resin cements provides greater degree of conversion compared to the conventional light curing protocol; as the reaction occurs slowly, the polymeric chains can more readily be formed or rearranged [14]. As observed in Table 2, the triple interaction between color x exposure x delay shows that all these factors can influence on the degree of conversion of a dual-cure resin cement.

As observed, five minutes delay prior to light curing significantly improved degree of conversion. This agrees with recent studies that also demonstrated delaying before light curing improves the degree of conversion of dual-cure resin cements [19-20]; One min delay prior to light curing also improved the degree of conversion compared to no delay and may be a more clinically acceptable approach to increase the degree of conversion.

CONCLUSION

Within the limitations imposed by the experimental design used in the current study, the following conclusions can be drawn: 1-Shade and curing mode can influence the cure efficiency of dual-cured resin cements. Higher energy density and delay before light curing are indicated to enhance the degree of conversion in the cementation of indirect ceramic restorations. 2- One min delay prior to light curing also improved the degree of conversion compared to no delay and may be a more clinically acceptable approach to increase the degree of conversion.

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