Effect of activation mode on flexural strength in dual-polymerized resin cements

Efeito do modo de ativação na resistência a flexão de cimentos resinosos duais

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ABSTRACT

Objective
To compare he influence of two activation modes on two similar dual-polymerized resin cements.

Methods
Bars (2 × 2 × 12 mm) of RelyX ARC (3M ESPE, MN, USA) (ARC) and Enforce (Dentsply, Rio de Janeiro, Brazil) (ENF) were prepared, and three types of polymerization method were tested: (1) L: LED photoactivation/40 s; (2) H: halogen Light activations/40 s, and (3) C: chemical activation only (Complete absent of light). The groups were ARC-L; ARC-H, ARC-C, Enf-L, Enf-H, and Enf-C (n = 8). After 24 h, the specimens were tested using the 3-point bending method on a universal testing machine.

Results
The ARC cement showed superior flexural strength results. The mode of activation revealed similar results on both cements tested, but light-activated groups (with LED or Halogen light) showed higher flexural strength values than did the chemical-activation-only group.

Conclusion
(1) RelyX ARC cement showed superior flexural strength when compared with Enforce; and (2) light activation resulted in better flexural strength values when compared to chemical activation on dual-polymerized resin cements.

Indexing terms*: Material resistance. Resin cements. Self-curing of dental resins.*

RESUMO

Objetivo
Comparar a influência do modo de ativação de dois cimentos semelhantes, porém de fabricantes distintos.

Métodos
Barras (2 x 2 x 12 mm) dos cimentos RelyX ARC (3M ESPE, MN, USA) e Enforce (Dentsply, Rio de Janeiro, Brasil) (ENF) foram confeccionadas com auxílio de uma matriz de teflon bipartida, e três modos de fotopolimerização foram testados: (1) L: fotoativação por LED / 40 segundos; (2) H - fotoativação por luz halógena / 40 segundos; e (3) Q - apenas ativação química (ausência total de luz). Os grupos formados foram: ARC-L; ARC-H; ARC-Q; Enf-L; Enf-H e Enf-Q, com 8 amostras cada. Após 24 horas, o teste de miniflexão de três pontos foi aplicado em máquina universal de testes.

Resultados
O cimento ARC apresentou resultados de resistência superiores ao cimento Enf. O modo de ativação agiu de forma semelhante nos dois cimentos testados, sendo que a ativação por luz, seja L ou H, aumentou a resistência para ambos, comparada a ativação Q.

Conclusão
(1) O cimento RelyX ARC (3M ESPE, MN, USA) apresentou maior resistência à flexão de três pontos quando comparado ao cimento Enforce (Dentsply, Rio de Janeiro, Brasil); e (2) a ativação por luz aumentou a resistência para cimentos resinosos duais.

Termos de indexação: Resistência de materiais. Cimentos de resina. Autocura de resinas dentárias.

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INTRODUCTION

Resin cements are used for bonding of indirect restorations, due to their good physical and mechanical properties[1]. The clinical lifetime of these restorations is determined by the properties of the bonding agent, which are directly related to the degree of conversion of the resin cements; a higher conversion equates to improved physical and mechanical properties[2]. Variations in mechanical behavior of resin cements are also related to their chemical composition, the amount of and proportion between the contained monomers[3], and the type and amount of filler present in the cement[4].

Cements may be activated by light, chemical reaction, or both (dual curing). The limitation of light-cured resin cements is that when opaque or alumina restorations or intraradicular posts are cemented, the activation light may not reach the cement. Chemically activated resin cements allow the material to cure even in complete absence of light[5]. Thus, dual-cured resin cements would be the perfect choice in situations where light-cured cements are limited, allowing the finishing of restoration immediately after cementation[6].

The degree of conversion is significantly reduced when resin cements are light-activated through ceramic restorations[5]. The irradiation ability of light-curing devices is markedly affected (a decrease of 78% to 85%) when measured through indirect restorations[7], showing the important effect of chemical activation of resin cements. However, light activation is also important, since it presented a positive effect on bond strength between the composite resin and dentin which was not seen in the case of chemical activation only[8]. Fonseca et al.[9] showed that when the isolated chemical activation was employed only 86.2 % of the tensile bond strength achieved by dual activation (light + chemical) of RelyX ARC (3M ESPE, MN, USA) was reached. Also, a significant decrease in flexural strength and Young's modulus of the cement sample was observed when light activation was not used for the curing process, showing that chemical activation alone did not allow the material to realize the optimum mechanical properties[9].

During the curing process, carbon double bonds are broken and converted into single bonds between methacrylate monomers. Bonds that are not converted lead to a deterioration in the physical properties of the resin cement[3,11-12], enhance the cement's degradation[13], allow margin microleakage due to adhesive failures[7], and may cause pulp damage[14]. Cements may also present different behavior due to the activation mode[15-16]. The amount of catalyst and the intensity of absorbed light determine the rates of curing. Thus, the amount of light that reaches the material and its chemical composition directly affects the curing of resin cements[16].

The aim of this study was to evaluate the mechanical properties of two resin cements through a three point bending test according to three different activation modes: activation using LED (light-emitting diode), activation using halogen light, and chemical activation only. The tested hypotheses were (1) that resin cements would present similar behaviors when activated with the same source, and (2) light-activated samples would present better flexural strength.

METHODS

Chart 1 presents the composition and manufacturers' information of the two resin cements used in this study. Bar-shaped specimens (24 to each cement) were manufactured using a Teflon matrix. Similar length of the base and catalyst pastes were proportioned, mixed, and inserted into the matrix. A polyester strip (K Dent, Quimidrol, Joinville, Brazil) was positioned on the cement sample and was activated according to one of the following methods: L: immediate light activation of the cement sample with a LED (SmartLite PS, Dentsply, Milford, USA) having a light intensity of 1200 mW/cm2 during of 40 s; H: immediate light activation of the cement sample with a halogen light (CL-K50, Kondotech, SP, Brazil) having a light intensity of 500 mW/cm2 during of 40 s; C: chemical activation only. After insertion into the matrix, samples were stored in a completely dark compartment. The light intensity was controlled using a radiometer (Light Intensity Radiometer, model: 3K Spirith Health) during the use of all light sources. The final dimensions of the samples were 2 × 2 × 12 mm (width, thickness, and length, respectively). Chart 2 summarizes the experimental design and groups used in this study. After curing, the bars had their surface standardized with sandpaper under cooling (1200 grit size), and were stored in dark compartments for 24 h before the flexural test.

Flexural stregth test

A "micro" three-point bending test was used to measure the flexural strength of the bars. Each bar was positioned on two support rollers (1.5 mm in diameter) 7.5 mm from each other (span). A third roller with the same diameter applied the load on the central portion of the bar, until fracture occurred. The assembly was attached to a universal testing machine (DL 2000, Emic, Brazil) that recorded the maximum load applied (at 1 mm/min) until fracture (Figure 1).

The flexural strength (R) (MPa) was calculated according to the following equation:

$$R = 3*F*sp / 2*l*b^2 \qquad \text{Eq. (1)}$$

Where F is the maximum applied load (N), sp is the span of the test (7.5 mm), l is the bar width (mm), and b is the bar thickness (mm).

Chart 1 . Characteristic of materials used in this study (manufacturers' information).

Material	Manufacturer	Monomers
Enforce	Dentsply, Rio de Janeiro, Brazil	Bis-GMA[1] TEGDMA[2]
RelyX ARC	3M ESPE, MN, USA	Bis-GMA TEGDMA

Note: 1Bis-GMA: 2,2-bis[p-(2 -hydroxy-3 -methacryloxypropoxy)phenyl]propane.
2TEGDMA: triethylene glycol dimethacrylate

Chart 2 . Experimental design.

Dual-cured resin cement	Activation mode	Groups (n=8)
	LED	ARC-L
RelyX ARC (3M ESPE, MN, USA) (n = 24)	Hal	ARC-H
	Chem	ARC-C
	LED	Enf-L
Enforce (Dentsply, Rio de Janeiro, Brazil) (n = 24)	Hal	Enf-H
	Chem	Enf-C

Note: 1Bis-GMA: 2,2-bis[p-(2 -hydroxy-3 -methacryloxypropoxy)phenyl]propane.

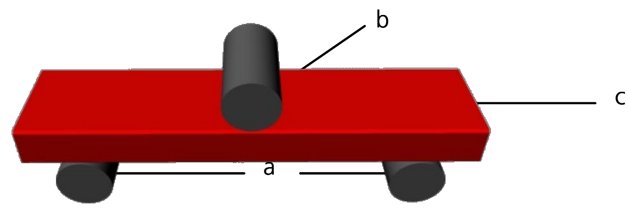

Figure 1. Design of Micro flexural test: (a) support rollers; (b) loading roller; (c) test specimens.

Statistical analysis

Obtained data presented normal distribution (Figure 2). A two-way analysis of variance (cement × activation mode) was carried out, followed by Tukey's test for means contrast ($\alpha = 0.05$).

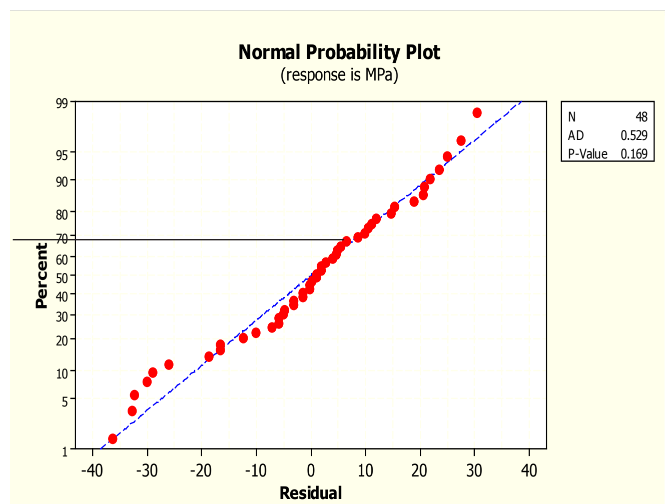

Figure 2. Adherence of data to normal curve.

RESULTS

The two-way analysis of variance showed that the resin cement RelyX ARC had higher values of flexural strength than Enforce (p = 0.0006). The activation mode also affected the flexural strength of dual-cure resin cements (p = 0.0003). The interaction of both factor was not significant (p = 0.721), meaning that activation mode affected both cements in the same way.

Table 1 presents the statistical details comparing all tested groups (Tukey's test), and Figure 3 presents flexural strength values according to activation mode for each tested cement.

Table 1. Mean flexural strength (MPa), standard deviation, and statistical significance of tested groups.

Activation \ Cement	RelyX ARC (3M ESPE, MN, USA)	Enforce (Dentsply, Rio de Janeiro, Brazil)	Total#
LED	110.5 (±21.3) A	86.32 (±18.9) ABC	98.43 (±23.1) a
Halogen light	95.92 (±18.3) AB	81.70 (±14.6) BC	88.81(±17.6) a
Chemical	80.15 (±20.4) BC	61.85 (±7.86) C	71.00 (±17.7) b
Total #	95.53 (±22.9) a	76.63 (±17.6) b	

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Same capital/lowercase letters indicate statistical similarity; Different capital/ lowercase letters indicate statistical difference.

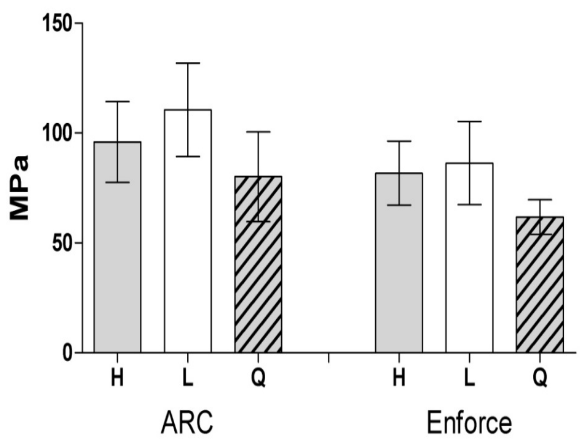

Figure 3. Adherence of data to normal curve.

DISCUSSION

This study evaluated the mechanical behavior of two dual-cure resin cements based on the effect of three different activation modes. The RelyX ARC resin cement presented higher values of flexural strength than the Enforce resin cement (Table 1). The three point bending test was carried out 24 h after cement handling.

The conversion of the monomers is directly related to the mechanical properties of the resin materials. When a resin material undergoes a change in its physical state due to curing, polymeric chains arise into its structure. In these cases, however large the conversion is, it is never complete (i.e., 100% conversion is not achieved).

The products of incomplete conversion are monomers and carbon double bonds. Noronha Filho et al.[5] used FT-IR analysis (Fourier transform infrared spectroscopy), and found higher conversion values for the resin cement RelyX ARC than for the resin cement Enforce, after dual curing (75.2 % and 59.5 %, respectively) and after chemical cure (38 % and 21.6 %, respectively). This result explains why in our study, RelyX ARC presented higher strength values than Enforce, in every tested activation mode. RelyX ARC also presented higher Knoop microhardness values after both dual and chemical cure than the resin cements Enforce, Bistec II DC (Tokuyama, Japan), and Fill Magic Dual Cement (Vigodent, Brazil)[15].

The activation mode also affected the mechanical properties of the cements (Table 1). Light activation (with either LED or halogen light) improved flexural strength to a greater extent than isolated chemical activation did, similar to the results showed by Bernardo et al.[15] and Lee & Um[16]. The absence of light activation also resulted in lower values of Vickers' hardness[17]. Two factors may be responsible by the differences in conversion rates: the material's composition (determined by manufacturer) and the intensity of the light that reaches the material[16]. Compared to chemical-cured cements, dual-cured resin cements have a limitation related to the addition of agents that promote chemical cure, i.e., the amine from the base paste associated with catalyst peroxide from catalyst past, which, when combined, initiate the chemical cure. The addition of these components has to be limited in order to provide a reasonable work time.

The components responsible for the chemical activation in dual-cured resin cements are not able to compensate the total absence of light and polymerize the cement completely. Resin cements were less effective when not activated by light[4,11,18]. Thus, light activation is fundamental to realize the adequate conversion[6] and better mechanical properties of dual-cured resin cements[19-20]. Moreover, the polymeric structure formed after the curing process is another important factor in obtaining better mechanical properties[21], and it may vary according to the composition determined by the manufacturer.

The type of light used had no effect on the flexural strength of the resin cements. Both sources, halogen light with an intensity of 500 mW/cm^2 and LED with an intensity of 1200 mW/cm^2, presented light intensities above 400 mW/cm^2 with 40 s of exposure. These parameters may have lead to similar values of flexural strength for tested cements.

The evaluated cements presented different mechanical properties (flexural strength). However, they were affected in the same way by the activation modes investigated in this study. Chemical activation resulted in the worst results in terms of strength for both cements.

CONCLUSION

The resin cement RelyX ARC (3M ESPE, MN, USA) presented higher flexural strength than Enforce (Dentsply, Rio de Janeiro, Brazil). In addition, light activation promoted higher values of flexural strength for both tested cements.

Collaborators

M AMARAL conducted the experiments and wrote the manuscript. RD NICOLÓ, JC ROCHA, RM ARAÚJO contributed to writing and reviewing of the paper. MAM ARAÚJO coordinated the research group, enabled the research, and gave substantial intellectual contribution to the writing of the manuscript.

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