The effect of viscosity and activation mode on biaxial flexure strength and modulus of dual resin cements

Efeito da viscosidade e modo de ativação na resistência flexural biaxial e módulo em cimentos resinosos duais

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Abstract

Purpose: The aim of this study was to evaluate the effects of curing mode and viscosity on the biaxial flexural strength (FS) and modulus (FM) of dual resin cements.

Methods: Eight experimental groups were created (n=12) according to the dual-cured resin cements (Nexus 2/Kerr Corp. and Variolink II/IvoclarVivadent), curing modes (dual or self-cure), and viscosities (low and high). Forty-eight cement discs of each product (0.5 mm thick by 6.0 mm diameter) were fabricated. Half specimens were light – activated for 40 seconds and half were allowed to self-cure. After 10 days, the biaxial flexure test was performed using a universal testing machine (1.27 mm/min, Instron 5844). Data were statistically analyzed by three-way ANOVA and Tukey's test (5%).

Results: Light-activation increased FS and FM of resin cements at both viscosities in comparison with self-curing mode. The high viscosity version of light-activated resin cements exhibited higher FS than low viscosity versions. The viscosity of resin and the type of cement did not influence the FM. Light-activation of dual-polymerizing resin cements provided higher FS and FM for both resin cements and viscosities.

Conclusion: The use of different resin cements with different viscosities may change the biomechanical behavior of these luting materials.

Key words: Dual cure resin cement; materials testing; viscosity

Resumo

Objetivo: O objetivo do estudo é avaliar o efeito do modo de ativação na resistência flexual biaxial (RF) e módulo (MF) em cimentos resinosos duais.

Metodologia: Foram formados oito grupos experientais (n=12) de acordo com cimento resinos (Nexus 2/Kerr Corp. and Variolink II/IvoclarVivadent), modo de ativação (dual ou auto-polimerizado) e viscosidade (baixa e alta). Quarenta e oito discos de cada cimento foram fabridados (0,5 mm espessura por 6,0 mm diametro). Metade dos espécimes foram foto-ativados e a outra metade foi deixado para auto-polimerização. Após dez dias, o teste biaxial foi realizado em máquina de ensaio universal (1,27 mm/min, Instron 5844). Os dados foram analisados estatisticamente por ANOVA e teste Tukey (5%).

Resultados: A foto-ativação aumentou a RF e RM para os cimentos em ambas as viscosidades em comparação com os grupos auto-polimerizados. A versão alta viscosidade dos cimentos foto-ativados apresentou maior RF que os cimentos em baixa viscosidade. A viscosidade e o tipo de cimento não influenciam o MF. A foto-ativação aumenta os valores de RF e MF para ambos os cimentos e viscosidades.

Conclusão: O uso de diferentes cimentos com diferentes viscosidades pode influenciar o comportamento biomecânico de cimentos resinoso.

Palavras-chave: Cimento resinoso dual; teste de material; viscosidade

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Introduction

The dual-polymerizing resin cements present characteristics that combine both auto- and light-polymerization to provide cure even in the attenuation of light produced by thickness and opacity of restorations or the absence of light in dark zones at the apical region and deep interproximal areas. Studies have shown that attenuation or absence of light reduces the degree of polymerization for most of the cements (1-3), which can affect some mechanical properties and the solubility of the material. According to Hofmann et al. (4), when the resin cements were lighted-cured the values of flexural strength, modulus and surface hardness were higher than those observed for self-curing without photo-activation groups.

The mechanical properties of resin luting cements are also determined by the type of resin monomers and the content of inorganic particles (4,5). Manufacturers have changed material composition to produce versions of the same commercial product with different viscosities (6). Filled composites are usually highly-viscous materials; however, the resin cements need to present low viscosity, in order to facilitate the cementation of indirect restoration, with minimal thickness of the cement layer (4-7).

The main alteration is observed in the proportion between resin matrix and filler particle content, which results in a low- or high-viscosity material. The manufacturers have indicated these resin cements for specific clinical situations, which are related to marginal and internal fit between the indirect restoration and the dental structure (7). Hahn et al. (6) evaluated the Influence of resin cement viscosity on microleakage of ceramic inlays and recommended highly viscous luting cements for cementing inlays in larger luting spaces.

The practice of fixed prosthodontic has changed with the use of resin cements, which provides resistance to most dental ceramics, standing the occlusal loading without deformation and bonding the indirect restoration to tooth (8,9). The influence viscosity and monomeric conversion of resin cements on physical properties and marginal sealing needs be investigated, in attempt to show the dentists the better luting material for each clinical scenario (6-10, 13).

In this study, biaxial flexure strength test was used to evaluate high and low viscosity versions of two resin cements, when they were tested in the dual polymerization (light- and auto- polymerization) and only auto-polymerization modes. The null hypothesis tested was that curing mode and viscosity do not affect flexural strength (FS) and modulus (FM) of resin cements.

Methods

The compositions of two resin cements tested are presented in Table 1. Two dual-cured resin cements were evaluated in two viscosities: Variolink II High and Low (IvoclarVivadent, Schaan, Liechtenstein) and Nexus 2 High and Low (KerrCo., Orange, CA, USA). The resin cements were manipulated according to manufacturers' instructions in the proportion of 1:1 (catalyst and base paste by volume) and were applied into an elastomeric impression material mold (Aquasil Ultra, Dentsply Caulk, Milford, DE, USA) to create disc-shaped specimens with dimensions of approximately 0.5 mm-thick and 6.0 mm in diameter. The specimen was covered with a Mylar strip and a microscope glass slide. Manual pressure was applied to force the material to flow into the mold and the excess material was removed after the specimen is removed from the mold. The specimens were either exposed to light for 40 seconds from a halogen light curing unit (XL 3000, 3M ESPE, St. Paul, MN, USA) or were allowed to self-cure. When light-activation was performed, light intensity was constantly monitored with a radiometer (Curing Radiometer Mode 100, Demetron Research Corporation, Danbury, CT, USA) and maintained between 550 and 600 mW/cm^2. Afterwards, specimens were stored at 37°C in the dark for 24 hours. Eight experimental groups were created according to product, viscosity, and activation mode, and twelve specimens (n=12) were prepared per group.

Table 1. Compositions of the resin cements used in this study

Resin cement (Manufacturer)	Composition	Lote number (L - low; H - high)
Nexus 2 (KerrCorp., Orange, CA, USA)	*Base:* Bis-GMA, camphoroquinone, barium aluminoborosilicate glass. *Catalyst:* Bis-GMA, TEGDMA, barium aluminoborosilicate glass *(70% byweightand 53% by volume offillersparticles)*.	438681 452344 (L) and 452365 (H)
Variolink II (IvoclarVivadent, Schaan, Liechtenstein)	*Base:* Bis-GMA, urethane dimethacrylate, TEGDMA, inorganic filler, ytterbium trifluoride, initiator, stabilizer *Catalyst:* Bis-GMA, urethane dimethacrylate, TEGDMA, inorganic filler, ytterbium trifluoride, benzoyl peroxide, stabilizer *Low-viscosity:* 27.9% byweightofmonomersand 71.2% byweightor 43.6% by volume offillersparticles. *High viscosity:* 22% byweightofmonomersand 77% byweightor 52% by volume offillersparticles.	J24363 J19103 (L) and J19730 (H)

Abbreviations: Bis-GMA: bisphenol-A-diglycidyletherdimethacrylate; TEGDMA: triethylene glycol dimethacrylate.

The discs were individually placed into a custom-made testing jig and tested for biaxial flexure strength on a universal testing machine (Instron 5844, Instron Corp, Canton, MA, USA) at 1.27 mm/min until failure occurred (14). The maximum load was recorded for each specimen and the elastic modulus was determined from the linear portion of each stress/strain curve. Formula for biaxial flexural strength (σ):

$$\sigma = -0.238 * \frac{7P(X-Y)}{b^2},$$

where:

σ is the maximum center tensile stress (megapascals);
P is the total load causing fracture (Newtons);
$X = (1+\nu)\ln(r_2/r_3)^2+[(1-\nu)/2](r_2/r_3)^2$;
$Y = (1+\nu)[1+\ln(r_1/r_3)^2]+[(1-\nu)(r_1/r_3)^2$;
b is the specimen thickness at fracture origin (millimeters);

in which:

ν is Poisson's ratio (used $\nu = 0.25$);
r_1 is radius of support circle (millimeters);
r_2 is radius of loaded area (millimeters);
r_3 is radius of specimen (millimeters).

Flexural strength (FS) and modulus (FM) were calculated by SRS Biaxial Testing Software (Instron Corp., Canton, MA, USA). FS and FM data were normal and homocedastic. They were analyzed by three-way (viscosity, resin cement, and polymerization mode factors) analysis of variance (ANOVA) and Tukey's post-hoc test (pre-set alpha of 0.05).

Results

Mean FS and FM values are presented in Tables 2 and 3, respectively. For FS analysis (Table 2), three-way ANOVA revealed statistically significant differences for the factors "viscosity" (P=0.0272) and "polymerization mode" (P<0.0001). No significant difference was revealed for the factor "resin cement" (P=0.5020). The double interaction "viscosity" × "polymerization mode" was also statistically significant (P=0.05). For the FM analysis (Table 3), three-way ANOVA revealed no statistically significant differences for the factors "viscosity" (P=0.4790) and "resin cement" (P=0.6197). However, it was observed significant difference for the factor "polymerization mode" (P<0.0001). No double or triple interaction among factors were significant (P>0.05).

Light-activation increased FS (134.7±27.4 MPa for Nexus 2/low viscosity; 146.9±18.6 MPa for Nexus 2/high viscosity; 137.6±22.1 MPa for Variolink II/low viscosity and 159.0±22.0 MPaVariolink II/high viscosity) and FM values (7.2±2.1 GPa for Nexus 2/low viscosity; 7.6±1.4 GPa for Nexus 2/high viscosity; 7.1±1.5 GPa for Variolink II/low viscosity and 7.6±2.0 GPaVariolink II/high viscosity) for both resin cements and in both viscosities (P<0.05). The high viscosityversion of resin cements presented higher FS than low-viscosity version only in light-activated groups (P<0.05). The self-curing mode of resin cements resulted in similar FS when resin cements with low viscosity (120.9± 11.0 MPa for Nexus 2 and 117.1±24.3 MPa for Variolink II) were compared to those with high viscosity (120.2±20.5 MPa for Nexus 2 and 120.0±12.0 MPa for Variolink II) (P>0.05). The FMwas not influenced by type of resin cement and its viscosities (Low viscosity: 7.2±2.1 GPa for Nexus 2/Light-activated; 5.5±8.6 GPa for Nexus 2/Self-cured; 7.1±1.5 GPa for Variolink II/Light-activated; 5.4±8.9 GPa for Variolink II/Self-cured and High viscosity: 7.6±1.4 GPa for Nexus 2/Light-activated; 5.6±1.9GPa for Nexus 2/Self-cured;7.6±2.0 GPa for Variolink II/Light-activated; 5.3±9.2 for Variolink II/Self-cured) (P>0.05).

Discussion

The results showed that the dual-polymerizing mode (light-activating plus self-curing) led to higher FS and FM than self-curing mode alone. Moreover, the viscosity

Table 2. Means values (SD) of flexural strength (MPa)

Viscosity	Nexus 2		Variolink II	
	Light-activated	Self-cured	Light-activated	Self-cured
Low	134.7 (27.4) Ab	120.9 (11.0) Ba	137.6 (22.1) Ab	117.1 (24.3) Ba
High	146.9 (18.6) Aa	120.2 (20.5) Ba	159.0 (22.0) Aa	120.0 (12.0) Ba

Means having similar letters (lower case within column; upper case within row) are not significantly different.

Table 3. Means values (SD) of flexural modulus (GPa)

Cement	Low viscosity		High viscosity	
	Light-activated	Self-cured	Light-activated	Self-cured
Nexus 2	7.2 (2.1) Aa	5.5 (8.6) Ba	7.6 (1.4) Aa	5.6 (1.9) Ba
Variolink II	7.1 (1.5) Aa	5.4 (8.9) Ba	7.6 (2.0) Aa	5.3 (9.2) Ba

Means having similar letters (lower case within column; upper case within row) are not significantly different.

affected FS values for both resin cements in light-activated groups with no influence on FM. Hence, the null hypothesis that curing mode does not affect FS and FM of resin cements was rejected, while the null hypothesis that viscosity does not affect FM was accepted.

Light-activation combined with chemical polymerization in dual-curing resin cements reduces the quantity of remaining double bonds, which increases the monomeric conversion of the material and consequently the hardness of the resin cement (15). Studies have demonstrated that only the chemical polymerization of dual-cured resin cements was not enough to promote hardness (16,17) and degree of conversion values (1,3) as high as those obtained with the light-activation.

Clinically, the light intensity reaching the resin cement is strongly attenuated by the distance from the light source, absorbing characteristics of the indirect restorative material or during post cementation. This attenuation may result in low degree of conversion if self-curing components would not be capable of promoting proper polymerization in the absence of light. El-Mowafy and Rubo (18) and Hofmann et al. (4) found that the self-curing components of most dual-cured resin cements were not capable of compensating the light attenuation and reported reduction in hardness and flexural strength values. In this study, the chemical curing mechanism alone resulted in lower FS and FM than the light-activated reaction when dual-cured resin cements were used.

For Nexus 2 (Kerr Corp.) and Variolink II (Ivoclar Vivadent), the cement viscosity changes with the use of a catalyst paste (high or low). The manufacturer of Variolink II has informed the percentage of the monomeric components and filler content: the catalyst fluid (low viscosity) paste contains 27.9% by weight of monomers and 71.2% by weight or 43.6% by volume of fillers particles. The dense catalyst (high viscosity) presents 22% by weight of monomers and 77% by weight and 52% by volume of fillers particles, indicating that the changing of their monomeric and inorganic composition is significant. The manufacturer of Nexus 2 resin cement does not supply these data and only shows in average, 70% by weight and 53% by volume of fillers particles, not specifying the viscosities.

Milleding et al. (12) demonstrated that specimens made with high-viscosity materials had a significantly higher microhardness compared to those made with low-viscosity materials. On the other hand, Ferrari et al. (13) showed that higherfiller content increased polymerization stress in luting cements, decreased push-out bond strength, and increased interfacial nanoleakage expression. In this study, when the resin cements were light-activated, the high viscosity materials exhibited higher FS than low viscosity versions. In the self-curing mode, no difference on FS was observed when comparing the viscosities and products. Also, the FM was not influence neither by the viscosity nor the products. Thus, the increasing for the concentration of filler particles (barium aluminoborosilicate glass, ytterbium trifluoride and other inorganic fillers) in approximately 16% by volume and the light-activation mode led to FS increasing for both resin cements.

The light-activation of the dual-cured cements showed essential in order to ensure high FM and, along with that, better mechanical properties, in the same way that happens with FS. When the increase in viscosity did not affect FM and FS, similar results were observed because the hardness provided by the polymerization of the monomers was enough to compensate the reduction of the inorganic content of the low-viscosity cement.

The flexural strength is a mechanical property that shows the maximum stress before fracture. If the load exceeds the maximum value of flexural strength of the cement, it can fracture and compromise the durability of the indirect restoration (9,19,20). The biaxial test provided another data about an important property related to the clinical behavior of the dental materials. This property is called flexural modulus, which is the ratio between stress and strain in the elastic regimen, in other words, how much stress is necessary to deform the material before the proportional limit (9,21)**.** In this study, the FM did not differ between the resin cements, similar to what happened with the FS. Also, no statistical difference in FM was observed between high and low viscosities. However, when the cements were dual polymerized (light-cured), the FM average values were 20% higher than those observed in auto polymerized cements (Tables 2 and 3).

Authors have suggested that an intermediate FM value is important in order to prevent microinfiltration (21) and transferring stress from the loaded restoration to the underlying tooth structure (22)**.** The intermediate FM values should be between the dentin value that is around 18 GPa and the indirect restoration, which varies according to the type of the material used (9). The FM of the metal alloy restoration is between 88 and 220 GPa, while for ceramic restorations is around 55 and 236 GPa (9). Thus, the results of the FM for the resin cements tested in this study were lower than the dentin and the restorative materials, however, other properties of these luting materials have been considered for cementation of indirect restorations (11, 22-25).

Conclusions

In conclusion, the results suggested that the light-activation of dual resin cements, independently from their viscosity, may improve the biomechanical features (FS and FM) of these luting materials. The high viscosity version of light-activated resin cements produced higher FS than those observed in low viscosity versions. The viscosity and the type of resin cement did not influence the FM.

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