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Effect of different light sources and reinforcement fibers on the hardness of dual resin cements activated through an indirect composite resin

Efeito de diferentes unidades fotopolimerizadoras e fibras de reforço na dureza de cimentos resinosos duais ativados através de resina composta indireta

Gabriela dos Santos Kummer[a], Marcos Kenzo Takahashi[b], Luci Regina Panka Archegas[c], Rodrigo Nunes Rached[d], Evelise Machado de Souza[d]

[a] DDS, Private Practice, Curitiba, PR, Brazil.
[b] DDS, MDS, PhD, Private Practice, Curitiba, PR, Brazil.
[c] DDS, MDS, PhD, Adjunct Professor, Federal Institute of Technology of Paraná, Curitiba, PR, Brazil.
[d] DDS, MDS, PhD, Full Professor, Graduate Program in Dentistry, School of Health and Biosciences, Pontifícia Universidade Católica do Paraná, Curitiba, PR, Brazil.

Abstract

Objective: The aim of this study was to determine the hardness of dual-cured resin cements irradiated with quartz--tungsten-halogen (QTH) or light-emitting diode (LED) curing units through a dental composite reinforced with different types of fibers. **Material and methods:** Discs made with a laboratory composite were used to simulate indirect restorations reinforced with unidirectional and bidirectional glass fibers and triaxial woven and biaxial braid ultra-high-molecular-weight polyethylene fiber ribbons. Specimens were made in a teflon mold with dual-cured resin cements (Bifix QM, RelyX ARC, RelyX Unicem, and Variolink II) and cured through a non-reinforced (control) or fiber-reinforced (Ribbond, Vectris, Fibrex-Lab, and Connect) composite discs with QTH or LED curing unit for 40 sec. The specimens were stored under 100% humidity at 37°C for 24 h. The microhardness Knoop test was carried out under a 50 g load for 15 sec, with five indentations per specimen. Data were analyzed with three-way analysis of variance (ANOVA) and Games-Howell test ($p \leq 0.05$). **Results:** Bifix QM showed significantly higher hardness values, despite the fiber type used in the composite reinforcement ($p<0.05$). Ribbond reinforcement resulted in significantly higher hardness of the resin cements, with the exception of RelyX Unicem. Resin cements activated by a LED curing unit showed statistically higher values of hardness. **Conclusion:** The triaxial woven polyethylene fiber reinforcement allowed better curing of most of the dual-cured resin cements. The use of a LED curing unit with higher irradiance increased the hardness of the resin cements evaluated in this study.

Keywords: Fiber reinforcement. Hardness. Resin Cement. Light Curing Unit.

Resumo

Objetivo: *O objetivo deste estudo foi determinar a dureza de cimentos resinosos duais ativados com unidade fotopolimerizadora de quartzo-tungstênio-halogênio (QTH) ou diodo emissor de luz (LED) através de um compósito dental reforçado com diferentes tipos de fibras.* **Material e métodos:** *Discos feitos com um compósito de laboratório foram usados para simular restaurações indiretas reforçadas com fibras de vidro unidirecionais e bidirecionais, fibras trançadas triaxiais e fitas de fibra de polietileno trançado de ultra-alto peso molecular. As amostras foram feitas num molde de teflon com cimentos duais (Bifix QM, RelyX ARC, RelyX Unicem e Variolink II) e polimerizadas através de um disco de resina não-reforçada (controle) ou discos de compósitos reforçados com diferentes fibras (Ribbond, Vectris, Fibrex-Lab e Connect) com QTH ou LED durante 40 seg. As amostras foram armazenadas sob 100% de umidade a 37°C durante 24 h. O ensaio de microdureza de Knoop foi realizado sob uma carga de 50 g durante 15 s, com cinco indentações por espécime. Os dados foram analisados com a análise de variância de três critérios (ANOVA) e teste de Games-Howell (p≤0,05).* **Resultados:** *O cimento Bifix QM apresentou valores de dureza significativamente mais elevados, independente do tipo de fibra utilizado no reforço compósito (p<0,05). O reforço com a fibra Ribbond resultou em valores significativamente mais elevados de dureza dos cimentos resinosos, com exceção de RelyX Unicem. Os cimentos de resina ativada por uma unidade de LED mostraram maior dureza do que os ativados com QTH (p<0,05).* **Conclusão:** *O reforço fibra de polietileno permitiu uma melhor cura da maior parte dos cimentos resinosos duais. A utilização de uma unidade de cura a base de LED aumentou a dureza dos cimentos duais avaliados.*

Palavras-chave: *Reforço de fibra. Dureza. Cimento resinoso. Unidade fotopolimerizadora.*

Introduction

Laboratory processed composite materials have enlarged their clinical applications mainly because of mechanical properties improvement, such as hardness and wear resistance, achieved by secondary polymerization using high-intensity light, heat, pressure, a vacuum, and/or an inert-gas atmosphere. Indirect composites have a number of advantages compared with direct composites such as the use of new polymer formulations with improved filler particle distribution (1), better possibilities for anatomic shape and proximal contacts, thus less dependent on the operator's clinical skills (2), and lower polymerization shrinkage, since the composite polymerization is extra-orally performed (3). However, when located in high load-bearing areas, such as posterior crowns and bridges, indirect resin composites have some drawbacks (4).

Attempts have been made to reinforce dental polymers with several types of fibers aiming to improve their mechanical properties (4-7). Many parameters, such as type, volume fraction and orientation of the fiber (6-9), adhesion of the fiber to the resin matrix (10), and water sorption of the matrix (11), are known to influence the properties of fiber-reinforced composites. The fibers used to reinforce composites are mainly composed of polyethylene, carbon, and glass; they may or may not be pre-impregnated with a resin and they are disposed in different orientations such as unidirectionally, bidirectionally, or in a woven or braided pattern (1,10).

The most common clinical failure of direct and indirect posterior composite restorations is bulk fracture (12). However, the fracture resistance of indirect restoration composite may be more dependent upon the bond strength of the resin cement between the restoration and tooth substrate than upon the thickness of the restoration (13).

Dual-cured resin cements are recommended for cementation of nonmetallic inlays and onlays, since they provide better control during the cementation procedure and a delayed chemical reaction can complete the polymerization process in deep areas where the curing lights cannot penetrate the restorative material (14-15). Some studies demonstrated that dual-curing of resin cements produced better mechanical properties (flexural strength, elastic modulus, hardness, and degree of conversion) than self-curing or light--curing alone (15-17).

The method and intensity of the light source used for polymerization are important factors in the curing efficiency of dual resin cements when considering the attenuating effect of the indirect restorative materials (15,18-19). This attenuating effect may be higher when laboratory composites are used because these are less translucent than ceramics (15). Nevertheless, the effect of fiber reinforcement on light transmission through indirect composite restorations (FRC) is still unknown.

The aim of this study was to evaluate the effect of different reinforcement fibers and light sources on the polymerization efficiency of dual resin cements using a Knoop microhardness test.

The hypotheses to be tested in this study are: 1) that there is no difference in hardness when dual resin cements are cured with different light sources; and 2) that the hardness of dual resin cements is not affected by the presence of a laboratory composite reinforced with different types of fibers.

Material and methods

Laboratory Resin Composite Spacers

A stainless steel split mold was used to fabricate spacer discs (10 mm diameter x 2 mm thickness) with a laboratory composite (SR Adoro, Ivoclar Vivadent Inc., Schaan, Liechtenstein) to simulate fiber reinforced composite restorations. The first increment was made with deep dentin shade A3, followed by insertion of one of the reinforcement fibers listed in Table 1. The fibers were inserted in such a way that the whole mold was filled in. The non-impregnated fibers were covered with a layer of Bis-GMA bonding resin (Scotchbond Multi-Purpose Plus, 3M ESPE, St. Paul, MN) before insertion. The subsequent increments of composite were carried out with a body shade A2 and a translucent TS2 SR Adoro. The increments were light-cured by a quartz-tungsten--halogen (QTH) light curing unit (Optilight 600, Gnatus Medical and Dental Equipments Ltd., Ribeirão Preto, SP, Brazil) for 40 s each. Secondary polymerization was performed in Targis Power (Ivoclar Vivadent Inc., Schaan, Liechtenstein) equipment at a temperature of 104ºC for 25 min, as recommended by the manufacturer.

Controls were fabricated with the same composite and shades, but without any fibers being inserted.

Table 1 - Trade names, types, and manufacturers of the fibers used for the fiber reinforced composite spacers in the study

Trade name	Code	Fiber type	Manufacturer
Fibrex-Lab	FIB	Unidirectional glass fiber – pre-impregnated	Angelus Ltda., Londrina, PR, Brazil
Vectris Single	VEC	Bidirectional glass fiber – pre-impregnated	Ivoclar-Vivadent, Schaan, Liechtenstein
Ribbond	RIB	Woven high-molecular-weight polyethylene – non-impregnated	Ribbond, Seattle, WA, USA
Connect	CON	Braid-weave high-molecular-weight polyethylene – pre-impregnated	KerrLab, Orange, CA, USA

Dual-Resin Cement Specimens

Two hundred disc-shaped specimens (5 mm diameter x 0.5 mm thickness) were made with different dual--resin cements (Table 2). The cements were manipulated according to the manufacturers' recommendations, and the materials were placed in a teflon mold lined with a mylar sheet and a glass slide. A mylar strip and one of the composite spacers were placed over the cement surface. Fifty specimens of each cement were fabricated and divided into five groups, one for each fiber and one control without fiber. Half the specimens were light cured with a QTH curing unit (Optilight 600, Gnatus Medical and Dental Equipments Ltd., Ribeirão Preto, SP, Brazil) with a 520 mW/cm2 irradiance, and the other half were cured with a light-emitting diode (LED) curing unit (Radii, SDI Ltd., Bayswater, Victoria, Australia) with a 700 mW/cm2 irradiance for 40 seconds. The discs were kept in the mold for five minutes before removal. Excesses were removed with a scalpel, and then the discs were polished with a #800 silicon carbide paper.

Table 2 - Trade names, compositions, and manufacturers of the dual resin cements used in the study

Trade name	Composition	Manufacturer
RelyX ARC	Paste A: zirconia/silica, Bis-GMA*, TEGD-MA**, pigments, amina, and photoinitiator. Paste B: zirconia/silica, benzoyl peroxide.	3M-ESPE, St. Paul, MN, USA
RelyX Unicem Applicap	Liquid: methacrylated phosphoric acid, dimethacrylate, acetate, stabilizers, initiator. Powder: glass powder, initiator, silica, substitute pyrimidine, calcium hydroxide, peroxy compound, pigments.	3M-ESPE, St. Paul, MN, USA
Variolink II	Bis-GMA, UDMA***, TEGDMA, barium glass, ytterbium trifluoride, barium and aluminium fluorosilicate glass, spheroid mixed oxide, catalyst, stabilizers, pigments.	Ivoclar-Vivadent, Schaan, Liechtenstein
Bifix QM	Bis-GMA, benzoyl peroxide, amines, catalyst, aluminium, sodium and strontium silicate glass, fluoride.	VOCO, Cuxhaven, Germany

Bis-GMA* - Bisphenol A - glycidyl dimethacrylate; TEGDMA** - triethyleneglycol dimethacrylate; UDMA*** - Urethane dimetacrylate;.

Knoop Hardness Test

The specimens were stored at 37°C and 100% relative humidity in light-proof recipients for 24 hours. The Knoop hardness test was performed in a microindenter (HMV 2000, Shimadzu Corp., Tokyo, Japan) with a 50 g load for 15 seconds. Each specimen was indented at five locations.

Statistical Analysis

The mean Knoop hardness numbers (KHNs) of the five measurements for each specimen were tabulated. The normality of the samples was verified by the Kolmogorov-Smirnov test, and the homogeneity of variance by the Levene test. Data were submitted to a three-way analysis of variance (ANOVA) and to Games-Howell Multiple Comparison test at a significance level of 5%. The statistical software used was SPSS 17.0 (Statistical Package for the Social Science, for Windows, Chicago, IL).

Results

ANOVA detected statistically significant differences for the variables “light sources”, “cements”, and “fibers” and interactions among them ($p<0.05$). The mean and standard deviation of the Knoop hardness for all the groups tested are shown in Table 3, together with the significant differences.

Considering only the variable “light source”, the use of an LED curing unit resulted in significantly higher cement hardness values than did the use of QTH ($p<0.05$). The mean KHN values for Bifix QM were significantly higher than those for the other cements. Among the fibers tested, the use of Ribbond fiber resulted in harder cements, with statistically significant differences between the experimental and control groups.

The dual resin cements cured through a non-fiber reinforced composite spacer did not show statistically significant differences compared with the other groups ($p>0.05$), with the exception of RelyX ARC dual cement cured with LED through Vectris spacer and FibrexLab with both light-curing units.

The groups cured through spacers without fibers (control) and through spacers with Vectris had higher hardness values when LED curing was used, irrespective of the resin cement. Similarly, LED polymerization provided superior results for all the cements cured through spacers with Connect fiber, with the exception of RelyX Unicem self-adhesive resin cement. On the other hand, the type of light source did not affect the KHN of the cements when Ribbond and FibrexLab were used, with the exception of RelyX Unicem resin cement, for which LED curing resulted in higher hardness than curing with QTH.

Table 3 - Mean and standard deviation (SD) of Knoop hardness of the resin cements cured through laboratory composite spacers with or without fibers (WF)

Group	Light	Bifix QM	RelyX ARC	Variolink II	RelyX Unicem
WF	LED	45.96 (3.25) a A*	35.86 (2.54) b A	25.35 (4.36) c B	21.33 (1.74) d C
	QTH	41.69 (3.42) a BC	19.05 (4.11) b E	16.66 (4.51) bc C	11.95 (3.83) c D
RIB	LED	44.22 (3.91) a AB	36.09 (3.80) b A	26.46 (3.00) c B	28.09 (2.89) c A
	QTH	45.66 (3.07) a A	34.95 (8.13) b ABC	29.84 (6.83) b AB	16.15 (5.42) c D
CON	LED	44.34 (3.98) a AB	33.54 (3.48) b AB	31.13 (1.91) b A	25.20 (2.85) c AB
	QTH	33.20 (2.43) a D	18.00 (6.82) b E	24.08 (3.06) b B	22.10 (2.61) b BC
FIB	LED	43.92 (2.67) a AB	27.41 (3.64) b CD	24.12 (2.25) b c B	22.71 (3.32) c BC
	QTH	41.70 (2.70) a BC	25.38 (3.97) b D	24.60 (2.99) b B	13.88 (3.90) c D
VEC	LED	43.76 (3.13) a AB	31.99 (1.79) b B	25.24 (2.57) c B	21.95 (2.04) d C
	QTH	39.20 (3.50) a C	24.72 (7.83) b DE	17.30 (2.97) c C	14.36 (2.16) c D

* Groups connected by the same lowercase letter in a line or the same uppercase letter in a column are not statistically different (p>0.05).

Discussion

Dual resin cements are recommended for cementing ceramic and indirect composite restorations because they combine the most desirable properties of light-cured and self-cured materials. The chemical-curing component of dual resin cements ensures adequate polymerization in deeper areas of cavities where the curing light cannot penetrate (14,20), whereas photoactivation allows immediate finishing after exposure to the curing light (16).

The effectiveness of the polymerization of dual resin cements can be affected by several factors such as composition, thickness, shade and opacity of the restoration material; type, method, intensity and wavelength of the curing light; and the composition of the cement itself. In this study, a 2 mm thick laboratory composite resin spacer was used (with or without fiber reinforcement) to simulate an indirect restoration that was to be cemented in a preparation with adequate reduction of the dental structure. The thickness of indirect restorations can directly affect the properties of the resin cement (20,21). According to El-Mowafy and Rubo (20), the light intensity of the light curing unit decreases about 70% when a resin composite spacer 1 mm thick is used and continues to decrease gradually with increasing spacer thickness until it becomes totally obstructed at 4 mm. These findings support the use of dual curing resin cements for cementing indirect restorations, as the chemical curing component ensures complete polymerization in deep areas of the cavity where the curing light can only penetrate to a limited extent (16).

In the present study, the resin cements were activated by quartz tungsten halogen (QTH) or light emitting diode (LED) curing units through spacers that simulated different fiber-reinforced composite restorations. Light-polymerization with the LED curing unit was found to be more effective than polymerization with QTH curing light, thus leading us to reject the null hypothesis initially proposed. This can be explained by the different wavelength emission spectra of the light curing units. The LED lights have a narrower spectral emission with a peak at about 470 nm (22), corresponding to the absorption peak of camphorquinone (468 nm), the most commonly used photoinitiator for resin-based materials (23). Unlike LED lights, QTH units have a broad spectral emission that does not necessarily peak at this wavelength (22). According to Rueggeberg, Caughman, and Curtis (24), for a light curing unit to provide adequate polymerization of resin-based materials, its power density must be at least 400 mW/cm2 with an exposure time of 40 s. Although the curing units used in the present study had power densities higher than the recommended value, the discrepancy in hardness values of the resin cements could be attributed to the difference between the light intensity of the

LED and QTH curing lights (700 mW/cm2 and 520 mW/cm2, respectively). Previous studies that used different curing units to activate dual resin cements reported increased hardness with higher light intensities (15,25-26).

The mechanical properties of dual resin cements activated through spacers that simulate ceramic restorations have been widely investigated (18,26-30). However, few studies have investigated the polymerization effectiveness of these materials under indirect resin composite restorations (15,20). In addition, there is a dearth of literature regarding the influence of spacers that simulate fiber-reinforced composite restorations on the polymerization of resin cements.

The second hypothesis proposed in this study was rejected because the fibers included in the indirect composite spacers affected the hardness of the various dual resin cements differently. Ribbond polyethylene fiber weave resulted in higher hardness for most of the cements, probably because this fiber is the only one not impregnated by resin monomers. The monomers used to impregnate the other fibers could have affected light diffusion through the spacer, thereby reducing the degree of polymerization of the cements. Additionally, Ellakwa et al. (10) reported that the presence of fillers in an adhesive bonding resin creates a system that is radiopaque, resulting in further attenuation of the light.

A comparison of the experimental and control groups was expected to show better performance for the cements activated through spacers without fibers. However, the cements activated in this way were found to have similar or even reduced hardness compared with those in groups with fiber-reinforced spacers, in particular those polymerized with QTH curing light. Despite their increased thickness (35 μm) and their woven configuration, the use of Ribbond and Connect polyethylene fibers did not result in reduced hardness of the cements compared with the other groups. This might suggest that the presence of fibers with different thicknesses and configurations was not the determining factor for polymerization effectiveness of the dual resin cements evaluated in this study.

Bifix QM resin cement exhibited increased hardness compared with the other cements irrespective of the type of fiber or light source used. In contrast, RelyX Unicem cement had the lowest hardness values, although the difference was not always statistically significant from that for Variolink II. The discrepancies in hardness values are probably due to the differences in composition of the cements, as well as the concentrations of some components such as light initiators, chemical activators, filler particles, and monomers. Further studies are required to determine whether these parameters affect either directly or indirectly the degree of conversion of dual resin cements.

Insufficient polymerization of the cement may lead to post-operative sensitivity due to washout of the unset cement material, with consequent microleakage and recurrent caries (20). The dental clinician must therefore bear in mind the importance of using curing units with sufficient light intensity and long enough exposure times as well as adequate light activation at all accessible surfaces of the restoration to maximize the penetration of light through the restorative material.

Conclusions

Within the limitations of this study, it can be concluded that:

- The polymerization of dual resin cements with a LED curing unit was more efficient than activation with a quartz-halogen (QTH) curing unit;

- Bifix QM was found to have increased hardness compared with the other dual resin cements;

- Under most of the experimental conditions, the type of fiber did not have a negative effect on the hardness of the dual resin cements investigated.

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