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Additional chemical polymerization of dual resin cements: reality or a goal to be achieved?

Polimerização química adicional de cimentos resinosos duais: realidade ou um objetivo a ser atingido?

Luzia Sakaguti UMETSUBO[a], Karen Cristina Kazue YUI[a*], Alessandra Bühler BORGES[a], Daphne Camara BARCELLOS[a], Sérgio Eduardo de Paiva GONÇALVES[a]

[a]Instituto de Ciência e Tecnologia, UNESP – Universidade Estadual Paulista, São José dos Campos, SP, Brasil

Resumo

Introdução: Este trabalho serve como um alerta para dentistas e pesquisadores, que alguns cimentos resinosos duais podem não polimerizar completamente sob alguns tipos de coroas protéticas. **Objetivo:** Analisar o grau de polimerização de cimentos resinos duais, sob barreiras protéticas, por teste de microdureza. **Material e método:** Três cimentos: Bistite II, RelyX ARC e Variolink II, foram fotopolimerizados através da interposição de vários tipos de barreiras, interpostas entre o cimento e a fonte de luz, formando os grupos: G1: sem barreira; G2: Resina composta Cesead; G3: Inceram alumina/Allceram; G4: IPS Empress; G5: Inceram zircônia/Allceram; G6: fragmento dental. Utilizou-se a luz halógena (650 mW/cm^2) para fotoativação e a microdureza foi avaliada: 50gf durante 15s (MicrohardnessTester FM 700), em dois períodos (30min e 24h) pós ativação. Os dados foram submetidos à ANOVA e teste de Tukey (5%). As interposições de Inceram alumina e Inceram zircônia resultaram na polimerização deficiente dos cimentos, impossibilitando a realização dos ensaios. **Resultado:** Para o fator cimento, o Bistite revelou a maior microdureza, seguido do RelyX e do Variolink ($p<0,05$). A barreira influenciou a microdureza, sendo que os maiores valores foram obtidos sem barreira, seguida do dente. Empress e Cesead proporcionaram os menores valores de microdureza e não diferiram entre si. **Conclusão:** A barreira afetou negativamente a microdureza dos cimentos resinosos duais; o período de avaliação não afetou os valores de microdureza para quase todas as condições testadas; existe um fator limitante do ativador químico na polimerização de alguns cimentos resinosos duais, e seu desempenho é dependente do produto.

Descritores: Cimentos dentários; microdureza; cimentação; polimerização.

Abstract

Introduction: This study serves as a warning to dentists and researchers that dual-cured resin cements may not polymerize completely under some prosthetic crowns. **Objective:** The aim of this study was to analyse the polymerization degree of dual-cured resin cements under prosthetic barrier, by microhardness test. **Material and method:** Three cements (Bistite II, RelyX ARC and Variolink II) were light-cured through different barriers, placed between the cement and the light source: G1: without barrier; G2: composite resin (Cesead); G3: Inceram alumina; G4: IPS Empress; G5: Inceram zirconia; G6: tooth fragment. Photopolymerization was carried out using a halogen light unit (650 mW/cm^2); microhardness was evaluated using the Microhardness Tester FM 700, under a load of 50gf with a dwell time of 15s, at two evaluation times (30min and 24h). **Result:** The results were submitted to ANOVA and Tukey tests (5%). Both Inceram alumina and Inceram zirconia ceramic barriers hindered polymerization. Bistite, followed by RelyX and Variolink, exhibited the highest microhardness values ($p<0.05$). As the highest values were obtained without a barrier, it was determined that the barrier, followed by the tooth, influenced microhardness. Both Empress and Cesead had the smallest microhardness values but with no statistically significant difference between them. **Conclusion:** The barrier negatively affected the microhardness of dual-cured resin cements; evaluation time did not affect microhardness values for most of the conditions tested. There is a limited effect of the chemical activator on the polymerization of some dual-cured cements, and their performance is product specific.

Descriptors: Dental cements; microhardness; cementation; polymerization.

INTRODUCTION

This study serves as a warning to dentists and researchers that dual-cured resin cements routinely used in dental clinics may not polymerize completely under materials commonly used for prosthetic crowns.

Resin cements are associated with aesthetic indirect restorative materials. These aesthetic materials include resins and ceramics and are used to mimic the tooth's translucency and color, mask the presence of metallic posts and/or tooth darkening, provide a favorable aesthetic outcome (typically for anterior restorations), and provide long-term retention of the prosthesis.

Resin cements can be classified based on their polymerization mode into chemically-cured, light-cured, or dual-cured. Chemically-cured resin cements exhibit a shorter working time, but their polymerization is not influenced by the thickness of the indirect restoration. Light-cured resin cements present ideal working characteristics, although they require an adequate amount of light to achieve polymerization[1]. The polymerization of a light-cured cement may be compromised by insufficient light intensity emitted by the light-curing unit, wavelengths emitted outside the spectrum of the photoinitiator within the resin material, and the thickness of the indirect restoration[2]. Dual-cured resin cements were developed to combine the desirable properties of chemical polymerization with those of light curing to assure enough working time and adequate polymerization, or degree of conversion, even in the deepest area of the preparation[3,4].

Ideally, the resin materials would have their monomers completely converted into polymers during the polymerization reaction, to optimize their mechanical properties and adhesive capacity. However, all polymers exhibit a considerable amount of residual monomer with degrees of conversion ranging from 55% to 75% under conventional irradiation conditions[5]. The residual monomers present high toxicity and may induce adverse biological reactions in the tissues, mainly in the pulp, in addition to increasing the potential for marginal leakage and development of secondary caries as a consequence of insufficient cement polymerization[4] if the light intensity was insufficient.

Thus, inadequate polymerization of the resin cement compromises its physical properties by interfering with its resistance, hardness, water absorption, adhesivity, and color stability.

Studies have demonstrated that the color, thickness and type of either restorative material or tooth structure through which the emitted light passes, as well as the time of exposure to the curing light, may influence the amount of light that reaches the resin cement, interfering with the degree of conversion and mechanical properties[6-9].

When considering dual-cured resin cement, the degree of polymerization is affected by a reduction or lack of light, since polymerization is not totally initiated by the chemical component of the reaction[7].

The aim of this in vitro study was to evaluate the microhardness of three commercially available, dual-cured resin cements by interposing five different barriers at two evaluation times. The hypotheses tested were that: 1) the placement of barriers does not interfere in the curing of the resin cements; 2) the evaluation times do not affect the microhardness of the resin cements.

MATERIAL AND METHOD

Three commercially available, dual-cured resin cements were used: Bistite II (Tokuyama Dental Corp, Tokyo, Japan), Rely X ARC (3M ESPE, St. Paul, MN, USA) and Variolink II (IvoclarVivadent, Schaan, Liechtenstein).

Five types of barriers were constructed, consisting of a tooth fragment and four indirect restorative materials. These rectangular A3 shade barriers (7mm × 7mm × 2 mm of thickness) were placed between the resin cement and the light source, composing the following groups: G1(control)- no barrier was used; G2- indirect composite resin (Cesead, Kuraray Dental America, New York, NY); G3-Inceram Alumina (Vita, Bad Säckingen, Germany) with AllCeram (Degussa Dental, DegudentInd and Com Ltda, SP, Brazil); G4- glass ceramic (IPS Empress, IvoclarVivadent, Schaan, Liechtenstein); G5-Inceram Zirconia (Vita, Bad Säckingen, Germany) with AllCeram (Degussa Dental, Degudent Ind and Com Ltda, SP, Brazil); G6- tooth fragment (enamel and dentin) from the buccal crown portion of a sound mandibular third molar that had been obtained from a tooth bank.

The specimens were fabricated using a three-part matrix: one block of acrylic resin, a bipartite Teflon intermediary piece, and a metallic aluminum rod which linked the other two parts. The black acrylic resin block was rectangular (30mm × 15mm × 10mm of thickness). Each block had four equidistant round cavities that were 3mm in diameter and 2mm deep. The Teflon bipartite matrix was 2mm thick and was constructed to cover the acrylic resin block. The junction of the two Teflon parts created cavities coinciding with those of the resin block where the dual-cured resin cement was inserted and light-cured.

All materials were weighed and mixed according to the manufacturers' instructions. The cement was mixed and inserted in two increments. The first increment was light-cured, without the barrier to retain the cement in the acrylic block. The second increment was inserted and a polyester strip and a microscope lamina were used to cover it. To standardize the surface, the experimental barrier was then placed on the polyester strip and the cement was light-cured through this barrier.

The samples were polymerized for 40s using a conventional halogen lamp (Optilux401, Demetron, Kerr Corp Danbury, CT, USA) with an intensity of 650 mW/cm^2. During the polymerization of one sample, the others were protected from light irradiation by a black cardboard device. After polymerization, the acrylic blocks with the resin cement were removed from the metallic and Teflon matrixes, identified and submitted to the first microhardness test (immediate).

To evaluate microhardness, two perpendicular lines were traced on the top of each sample using a n. 11 scalpel blade, dividing the surface into four quadrants. Then, the specimens were submitted to microhardness testing (Microhardness Tester FM – 700, Future-Tech Corp., Tokyo, Japan) with load of 50gf for

15s to determine the Vickers Hardness (VH). Four readings were obtained for each sample, one from each quadrant (immediate group=30min after polymerization), to calculate the mean hardness for statistical analysis.

After the immediate test, the samples were stored in artificial saliva, without light, in an incubator at 37 °C, until testing after 24 hours[8].

The data were submitted to ANOVA-RM (time as repetitive factor) and Tukey tests (α=0.05). For each cement, a two-way ANOVA followed by the Tukey test was applied using STATISTIX for Windows (Analytical Software, Inc., version 8.0, 2003), MINITAB (Minitab, version 14.12, 2004) and STATISTICA (StatSoft, version 5.5, 2000).

RESULT

In the immediate assessment (Table 1), Bistite II exhibited the highest microhardness means under the following conditions: without barrier and tooth fragment. Notwithstanding, Bistite II demonstrated unstable behavior under the other experimental conditions, with great variability in the hardness values (from 69.91 to 28.47, for Cesead barrier) (Table 1).

Variolink presented the smallest mean values (Table 2), yet with median variability for the first and second evaluations (Table 1).

RelyX ARC displayed the smallest general standard deviation, that is, it had the most stable behavior in the immediate assessment. Moreover, it exhibited the highest means under the following conditions: Cesead and Empress; and, it exhibited the second highest for the tooth fragment (Table 1).

In the first evaluation, the resin composite Cesead (for Bistite barrier) was the factor with the most influence on reducing microhardness when compared with the control group (without barrier).

In the second evaluation, after 24h, again the highest values were found for Bistite and the lowest for Variolink (Table 1).

After the statistical analysis, it was observed that:

RM ANOVA (statistic $F_{df(6;132)}$=0.78; p-value=0.0001) for the three factors (cement, barrier and time) was significant. The Tukey test for the factor "cement" revealed significantly higher microhardness means for Bistite, followed by Rely X ARC and Variolink (Table 2).

The barrier significantly influenced the microhardness means. Table 3 shows that the highest values were obtained for the groups without a barrier, followed by the tooth fragment. Cesead and Empress presented the lowest microhardness values.

Two-way ANOVA was applied for each cement and significant differences were obtained for all groups (p<0.05). The results of the Tukey test for the different cements, using distinct barriers and tested after 30min and 24h, are shown in Table 1. The presence of different barriers significantly reduced the microhardness values for Bistite, and the lowest means were obtained with the Cesead barrier. The means obtained after 24h for Bistite were higher for all conditions, but were significantly different only associated with the Cesead barrier.

The Rely X ARC groups showed similar results for all conditions tested, for both barrier and time factors. In the groups where Variolink II was used, the presence of different barriers significantly reduced the microhardness values. There were no significant differences for the time of measurement.

DISCUSSION

Aesthetic indirect restorations are used to minimize many of the deficiencies associated with resin composite direct restorations: longevity, proximal contacts, polymerization shrinkage, and post-operative sensitivity, among others. The resin cement plays a fundamental role in the clinical success of this approach because inadequate polymerization can reduce the mechanical properties and cause failures, including: displacement or fracture of restoration[9], increased cement cytotoxicity, microleakage, caries and degradation of the interface of the restoration within the oral cavity.

Czasch, Ilie[10] found a correlation between mechanical properties and degree of polymerization. For the same material, they found the highest reliability, highest degree of conversion and highest micromechanical properties (indentation modulus and Vickers hardness).

Since indirect restorations provide true obstacles for the penetration of the light used for polymerization, studies on both the dual capacity of the cements and the light blockage of different barriers are of fundamental importance. Some clinical situations in which dual-cured cements are indicated expect that the chemical

Table 1. Microhardness means (±SD) and results of Tukey test for the cements tested, according to barriers and times

Barrier	Without		Tooth		Cesead		Empress	
Time	30min	24h	30min	24h	30min	24h	30min	24h
Bistite	69.91^{A} ±3.59	73.89^{A} ±2.85	51.73 ^{B} ±7.52	54.73^{B} ±7.39	28.47 ^{E} ±10.68	36.07 ^{D} ±5.58	35.33^{CD} ±8.39	40.46 ^{C} ±5.41
Rely X	39.03^{A} ±2.67	38.42^{A} ±1.91	42.19^{A} ±3.31	39.35^{A} ±4.85	39.20^{A} ±3.29	36.90^{A} ±2.62	37.68^{A} ±3.49	36.83^{A} ±4.18
Variolink II	41.73 ^{B} ±3.07	43.60 ^{B} ±5.13	35.52^{A} ±2.69	35.69^{A} ±3.36	30.54^{A} ±2.83	33.00^{A} ±2.01	30.33^{A} ±5.44	35.63^{A} ±3.12

Means (SD) of the cements for the experimental groups. Different letters denote significant difference in rows.

Table 2. Results of Tukey test for the cements tested

Cement	Mean (HV)	Homogeneous Groups*		
Bistite	48.42	A		
Rely X ARC	38.70		B	
Variolink II	35.75			C

*Different letters denote significant differences.

Table 3. Results of Tukey test for barrier tested

Barrier	Mean (HV)	Homogeneous Groups*		
Without	51.78	A		
Tooth	42.52		B	
Empress	36.03			C
Cesead	34.02			C

*Different letters denote significant differences.

factor of polymerization is enough, such as in deep gingival walls and proximal boxes[7].

The above notwithstanding, according to Price et al.[11], as the thickness of the restoration material increases to greater than 2.5mm, the transmitted light exponentially reduces and can become insufficient to cure the dual-cured resin cement properly. In a study by Kilinc et al.[12], a thickness greater than or equal to 3mm adversely affected the polymerization of resin cements. The results of the present study demonstrated that, even when using a 2mm thick barrier, only Rely X obtained microhardness values similar to when no barrier was used (Table 1).

Ilie, Simon[13] compared eight dual-cured, self-adhesive resin cements with two conventional dual-cured resin cements and found that the influence of light on the polymerization process was material dependent.

Darr, Jacobsen[14] affirmed that resin cements are not properly cured at the initial stages of the cementation and may lead to the displacement of the restoration during the finishing procedures if performed immediately after cementation. According to those authors, it is likely that the photopolymerization of the cement onto the adjacent etched enamel accounts for the stabilization of the restoration, while chemical or dual polymerization would occur in the deepest areas. However, the restorations can be vulnerable during the first 24h.

Immediate microhardness evaluation was performed to evaluate the amount of polymerization of the dual-cured resin cements, after light curing only. The 24h assessment time aimed to observe the behavior of the dual-cured resin cements following continued chemical polymerization[15]. A 24-hour storage condition may be sufficient, irrespective of the curing modes, for primary evaluation of the mechanical and chemical properties or bonding behavior of dental resin cements[16].

After the microhardness tests, the samples obtained by the interposition of InCeram alumina/AllCeram and InCeram zirconia/AllCeram were found not to exhibit enough superficial hardness. Therefore, they did not allow for indentation at the immediate reading. These samples were submitted to the second reading to determine whether subsequent chemical polymerization would provide effective total polymerization. However, it was not possible to conduct the second reading, demonstrating that the tested cements required initial photopolymerization. In an additional study, conducted with InCeram alumina/AllCeram and InCeram zirconia/AllCeram barriers that used a radiometer to evaluate similar results, these barriers were found to have totally blocked the light emitted by both the conventional halogen (650 mW/cm^2) and the high intensity LED light-curing units (>1100 mW/cm^2).

Chemical- and dual-cured resin cements, as well as light-cured resin cements, appear to be cured within the first 24 hours post-mix or post-light activation, without further significant changes in the degree of conversion or microhardness[16]. The authors verified that Rely X ARC reached a maximum microhardness value at 15 min, which was stable after 7 days, indicating that the chemical component did not continue acting 15 minutes after mixing.

Kumbuloglu et al.[17] compared dual- and self-cured Rely X ARC and verified high values for degree of conversion, 81% and 61%, respectively. They concluded that light activation improved the degree of conversion of this material.

In the present study, RelyX ARC exhibited the highest mean microhardness values for the Cesead (39.20 HV) and Empress (37.68 HV) groups and the median values for the tooth fragment (39.03 HV) group at the immediate readings (Table 1). The light attenuation of the barriers did not affect the RelyX ARC microhardness, suggesting a strong chemical polymerization of this cement. On the other hand, Attar et al.[18] found that, when photopolymerization was not performed, chemical activation alone was not sufficient to obtain the maximum mechanical properties of materials, like RelyX ARC, which presented significant reduction in flexural strength and modulus of elasticity.

The results of the present study demonstrated high microhardness values for Bistite II when no barrier was used (Table 1), which can be related to the photoinitiators. However, those values decreased markedly when a barrier was used, with the composite resin Cesead, the ceramic Empress and the tooth fragment significantly influencing the microhardness for Bistite II. This indicates the need for light-curing with units having higher intensity and suggests that its polymerization is based more on light curing than on chemical curing. This is in agreement with the findings of El-Mowafy, Rubo[3]. Thus, Bistite II would be more indicated for clinical situations in which there is a thinner barrier, as with anterior aesthetic veneers[19].

Jung et al.[20] demonstrated that increasing the ceramic thickness from 1mm to 2mm negatively affected the polymerization depth and Vickers microhardness of the resin cement when using several light sources. The use of a chemical catalyst, instead of photopolymerization alone, was recommended because it produced higher hardness and greater polymerization depth with all sources used. On the other hand, Rueggeberg, Caughman[21] observed that the polymerization induced by chemical activation after light activation was not significant for the dual-cured resins evaluated. According to those authors, the degree of polymerization observed 10min after light activation was not significantly different from that observed after 24h, which is similar to the results of the present study. Additionally, Hasegawa et al.[22] studied dual-cured cements under resin composite inlays and verified that chemical polymerization did not promote maximum curing of the cements when the light was attenuated by the tooth and restorative material. The self-curing component was not found to be sufficient to induce conversion of dual-cured cement[15,23].

The self-curing chemical component can play an important role in polymerization, especially in areas that are inaccessible to the curing light. This may explain differences in KHN values between more translucent and more opaque ceramics[15]. Moreover, the KHN of light-activated Rely X ARC under opaque ceramics (Procera, Cercon and In Ceram) did not differ statistically from that of chemical activation, showing that almost no light passed through the ceramic structures[15].

For Variolink II, the interposition of all barriers statistically reduced its microhardness with the light intensity employed. Additionally, there was no variation in microhardness when evaluated both immediately and after 24h, indicating that the chemical component of this cement was not effective for continuing the polymerization.

The hypotheses tested were not accepted, although the barrier interposition interfered with the polymerization of the resin cements and the assessment times interfered with the microhardness values for Bistite.

Moreover, studies regarding the performance of dental materials should be noted since it is possible that product information provided by the manufacturer might not be reliable, as observed in the present study regarding to the Inceram alumina/AllCeram and Inceram zirconia/AllCeram barriers. On the other hand, Kim et al.[24] found that the chemical polymerization mechanism of the dual-cured resins tested (Duo Link and Panavia F 2.0) worked effectively when photo-polymerization was impaired by insufficient light reaching the material. However, the suggest that a proper light-curing technique is still important for maximizing the conversion from monomer to polymer.

Giráldez et al.[25] observed that dual-curing resin cements should always be irradiated for longer periods than those recommended by the manufacturers.

Therefore, both the indication of the aesthetic restoration and the cementation technique should be carefully and judiciously executed because they play an essential role in the clinical success of an indirect restoration.

The results of the current study indicate that: placement of a barrier between the curing light and resin cement negatively affected the microhardness of dual-cured resin cements; evaluation time did not affect the microhardness values for most of the conditions tested; there is a limited effect of the chemical activator on the polymerization of some dual-cured cements and their performance is product specific.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

*CORRESPONDING AUTHOR

Karen Cristina Kazue Yui, Instituto de Ciência e Tecnologia, UNESP – Universidade Estadual Paulista, Av. Engenheiro Francisco José Longo, 777, Jardim São Dimas, 12245-000 São José dos Campos - SP, Brasil, e-mail: karenyui@fosjc.unesp.br; karenyui@hotmail.com

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