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Polymerization Eficiency of Dual-polymerized Resin Cements Light- Irradiated Through Ceramics and Laboratory-processed Resin Composite*

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Abstract - *The purpose of the study was to determine the influence of light-irradiation through two ceramic and one resin composite materials on the degree of remaining double carbon bonds in 3 dual-polymerized resin cements. After mixing, the cement was inserted into a 0.5mm deep recess in a silicon mold, covered with one ceramic or resin composite rectangular block and exposed through the block with the light from a halogen polymerization unit for 40s. Infrared spectroscopic analysis was used to record the degree of remaining double carbon bonds. Light irradiation through 2 mm–thick ceramic and resin composite materials increased the degree of remaining double carbon bonds relative to the direct photopolymerization analogues.*

KEY WORDS: Dual-polymerized, Resin cements, Polymerization efficiency, Remaining double bonds

INTRODUCTION

The increased use of aesthetic, non-metallic indirect restorations gave impetus to the development and use of resin cements. Resin cements are used to bond inlays, onlays, veneers, crowns, and fixed partial dentures made of ceramic and polymeric materials. Resin cements increase the fracture resistance and retention of etchable ceramics and may facilitate colour match for translucent materials[1].

Based on the polymerization system, resin cements can be classified as chemical-, photo- and dual-polymerized products. The photopolymerized resin cements are commonly light-irradiated through ceramic [2-10] or polymer [7,11-14] restorations. As a consequence, the light is attenuated resulting in a higher number of remaining double carbon bonds (RDB). This decreases the strength, stiffness, and colour stability, increases the solubility [15-18], and worsens the biologic behaviour of the resin cements[19]. Furthermore, it has been proposed that insufficient hardening of the cement may be a predisposing factor to postoperative sensitivity due to wash-out of the unset cement material with subsequent microleakage and recurrent caries[20].

Dual-polymerized cements have been designed to resolve the problem of insufficient polymerization in photopolymerized resin cements. Various characteristics concerning the polymerization performance of the dual-polymerized resin cements have been reported. It has been shown that without light exposure, a lower degree of polymerization is achieved for several dual-polymerized cements [12, 20-24]. Furthermore, despite the manufacturers' claims, there is no evidence for a substantial chemically-induced polymerization that occurs after light exposure is completed[25]. The influence of the chemical component on the final polymerization significantly differs among commercially available resin cements assessed[26-27].

The purpose of this in vitro study was to determine the polymerization efficiency of 3 dual-polymerized resin cements when light irradiated through 2 ceramic and 1 laboratory-processed resin composite material by means of measuring the degree of remaining double carbon bonds (RDB). The first null hypotheses of this study was that irradiation of 3 dual-polymerized resin cements through 3 different block materials had no significant effect on the degree of remaining double carbon bonds of the 3 cements. The second null hypothesis stated that the 3 dual-polymerized resin cements had no difference in the degree of remaining double carbon bonds.

MATERIAL AND METHODS

The lithium disilicate pressable core ceramic Empress 2 (Ivoclar, Vivadent, Schaan, Liechtenstein), the feldspathic dentin porcelain Vitadur Alpha (Vident, Brea, Calif) and the laboratory-processed dentin resin composite Sinfony (3M ESPE, St. Paul, Minn) were selected for the preparation of one rectangular block for each material with dimensions: 12 mm in length, 6 mm in width, and 2 mm in height. All materials were selected in the A3 shade. Three dual-polymerized resin cements were evaluated: Panavia F 2.0, tooth colour (Kuraray, Kurashiki, Japan), RelyX ARC, universal color (3M ESPE) and Nexus 2, light colour (Kerr, Orange, Calif). Three specimens were made for each cement – material combination. All combinations of the resin cements and polymerization conditions tested created 12

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groups with 3 specimens in each group. Additionally, one more group, with three specimens per cement was created as the control group, which was not combined with any block material.

The rectangular blocks precisely fit prepared silicon moulds having a 0.5 mm recess (Figure 1). After mixing, the cement was inserted into the mould, covered with a transparent matrix and with one of the blocks that was pressed to seat into the mould recess, allowing 0.5 mm space for the cement layer. Excess cement was removed. The cement was exposed to photopolymerization, for 40 seconds, through the block by a halogen light-polymerization unit (Optilux 540, Demetron; Kerr) (Figure 1) used in the standard mode with a power density of 650 mW/cm^2.

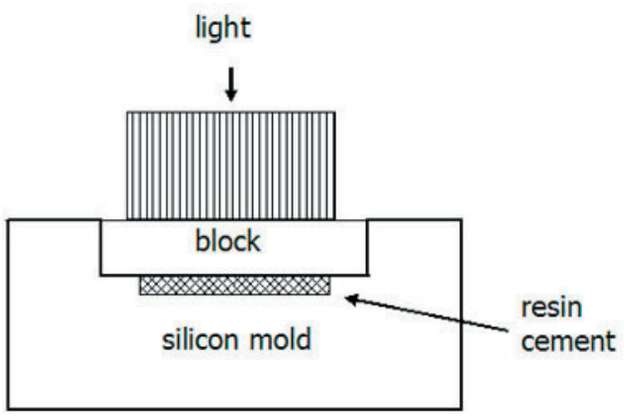

Figure 1. *Schematic presentation of set-up used for photopolymerization of dual-polymerized resin cements through block materials.*

For the control group, after mixing the cement was inserted into the mould, covered with a transparent glass slide cut at the exact dimensions of the mould and pressed to remove excess cement. Therefore, the standard 0.5 mm space for cement was assured and an O_2 inhibited surface was avoided. Care was taken to keep the tip of light-polymerization unit at 2 mm distance from the cement surface. Three specimens from each cement were prepared and remained with no photopolymerization (unset pastes), in order to calculate the amount of carbon carbon double bonds that each resin cement contained.

Micromultiple internal reflectance Fourier transform infrared spectroscopy (micro-MIR-FTIR) analysis was used, after 48-hour dry and dark storage of the specimens, to measure the degree of RDB, for all groups evaluated. Spectra of the unset pastes for each resin cement and of the top irradiated surfaces from all groups were acquired employing a micro-MIR cell attached on FTIR spectrometer (Micro-MIR accessory and Spectrum GX FTIR spectrometer; Perkin-Elmer, Norwalk, Conn) under the following conditions: 4000-400 cm^{-1} range, 4 cm^{-1} resolution, 45° para edge KRS-5 minicrystal of 7 internal reflections, 30 scans coaddition at 30 ±1° C. The cured specimens were pressed against the crystal with torque wrench device. The quantitative measurements were performed based on the 2-frequency technique. The stretching vibrations of the methacrylate C=C bonds (1638 cm^{-1}) were used as an analytical frequency, whereas the stretching vibrations of the aromatic bonds (1605 cm^{-1}) were used as the reference frequency. The net peak absorbance areas of these peaks were used to quantify the extent of C=C remaining (RDB) at the top resin-cement surfaces, relative to the corresponding unset pastes according to the following equation:

$$RDB = \frac{Ap(C=C) \bullet Am(C.C)}{Am(C=C) \bullet Ap(C.C)}$$

where:

Ap (C=C): the net peak absorbance area of the set material at 1638 cm^{-1},

Am (C..C): the net peak absorbance of area of the unset material at 1605 cm^{-1},

Am (C=C): the net peak absorbance area of the unset material at 1638 cm^{-1}, and

Ap (C..C): the net peak absorbance area of the set material at 1605 cm^{-1}

All results were analyzed by 2-way analysis of variance (ANOVA) and Ryan-Einot-Gabriel-Walsh (REGW) test for multiple comparisons of means (P<0.05). The independent variables were cement and block material and the dependent variable was RDB. These statistical tests were performed with a commercial software package (SPSS Inc, Chicago, Ill). A power analysis was performed on the experimental data, assuming α = 0.05, a power of 80% (β = 0.20), and the pooled standard deviation, to determine what statistical differences a sample size of N = 3 would show among RDB values for the several experimental groups.

RESULTS

The results of RDB, in the dual-polymerized resin cements, obtained through all block materials and after direct photopolymerization and the statistical analysis are presented in the Tables 1 and 2. The analysis showed a statistically significant difference among the 3 cements and the 3 block materials, and that there was interaction between the cement brand and the block material. Therefore, both null hypotheses were rejected.

The 3 resin cements selected for this investigation had no statistically significant difference in their RDB values when photopolymerized with no material intervention. No statistically significant differences were revealed among the resin cements when photopolymerized through Empress 2. Under Vitadur Alpha, RelyX ARC exhibited a statistically lower degree of RDB values in comparison with Panavia F 2.0 and Nexus 2. Under the laboratory-processed resin composite Sinfony, RelyX ARC exhibited lower RDB value than Nexus 2.

Panavia F 2.0 polymerized better through Empress 2 compared with the other 2 materials. For RelyX ARC, Empress 2 and Vitadur Alpha provided a lower RDB increase than Sinfony. It should be noted that for both Panavia F 2.0 and RelyX ARC, RDBs, when polymerized through Empress 2, were not significantly different than RDBs after direct polymerization without any material intervention. For Nexus 2, Empress 2 had less effect on RDB values than Vitadur Alpha and Sinfony.

Table 1. *Percentage of remaining double carbon bonds (RDB) (mean values and standard deviations) of dual-polymerized cements light-irradiated through the 3 block materials and after direct (no block) photopolymerization. Groups with different letters are statistically significantly different (p<0.05).*

Cement/Block	*No block*	*Empress 2*	*Vitadur Alpha*	*Sinfony*
Panavia F 2.0	39.90 (1.0) AB	45.03 (1.0) ABC	57.69 (4.8) EF	61.64 (3.5) EF
RelyX ARC	38.15 (0.6) A	44.50 (1.5) ABC	47.21 (3.5) BC	55.11 (1.5) DE
Nexus 2	40.52 (2.9) AB	48.53 (1.8) CD	58.82 (4.9) EF	63.56 (2.8)

Table 2. *Two-way ANOVA showing the effect of material and cement on the percentage of remaining double carbon bonds and the interaction between material and cement (p<0.05).*

Source	*Type III Sum of Squares*	*df*	*Mean Square*	*F*	*Sig.*	*Observed Power*
Corrected Model	2625,008	11	238,637	29,440	,000	1,000
Intercept	90230,653	1	90230,653	11131,327	,000	1,000
BLOCK MATERIAL	2225,746	3	741,915	91,527	,000	1,000
CEMENT	276,078	2	138,039	17,029	,000	,999
BLOCK MATERIAL * CEMENT	123,184	6	20,531	2,533	,048	,731
Error	194,544	24	8,106			
Total	93050,205	36				
Corrected Total	2819,552	35				

The power analysis performed on the experimental data, assuming $\alpha = 0.05$ and a power of 80% ($\beta = 0.20$), indicated that a sample size of N = 3 would show significant differences of 5.75 in the mean RDB value between sample groups. Although a larger sample size might have revealed statistically significant differences between some additional sample groups, compared to the results obtained with the current sample size, the general observations and clinical recommendations from this study would not have been substantially altered.

DISCUSSION

The results of the present study illustrate the polymerization state of 3 commercially available dual-polymerized resin cements used under clinically relevant conditions. The polymerization performance of the cements irradiated at a 2 mm-distance from the cement surface without the presence of a block material was examined to establish the maximum polymerization potential of the 3 cements. The 2 mm-thickness of the block used in this study corresponds to the mean thickness of the occlusal portion in an inlay, onlay, or crown restoration.

The polymerization efficiency of the dual-polymerized resin cements were determined recording the residual quantity of remaining double carbon (C=C) bonds (RDB) after 48 hours post-photopolymerization. Although RDB cannot fully indicate the clinical performance of a polymer material, it is an important factor that relates to the physic-mechanical properties and the biocompatibility of the material.[18-19]

Generally, dimethacrylate-based dental materials present RDB values ranging from 25 to 45% and the corresponding values for restorative resin composites fall into 40 to 50% [28]. The lower viscosity of the resin cements compared with the resin composites may be attributed to the relatively lower RDB values obtained for resin cements (37.3 to 42.3 %).[29] The results of the present study (38.15 to 40.42%) for the RDB values for resin cements light-irradiated directly with no material intervention agree with the previously published values.

The polymerization efficiency of the dual-polymerized resin cements were determined recording the residual quantity of remaining double carbon (C=C) bonds (RDB) after 48 hours post-photopolymerization. Although RDB cannot fully indicate the clinical performance of a polymer material, it is an important factor that relates to the physic-mechanical properties and the biocompatibility of the material[18-19].

The direct light exposure of a dual-polymerized resin cement achieves a rapid polymerization rate and forms a highly viscous gel, which hinders the monomer mobility and subsequently the polymerization process induced by the chemical catalyst [30,31]. Since, no significant differences were detected among the dual-polymerized resin cements under the direct irradiation condition, it can be assumed

that the monomer composition in combination with the light catalytic systems lead these products to a similar polymerization performance. The RDB values measured after post-polymerization period of 48 hours is believed to reveal the maximum polymerization potential of each cement. Within this period the photo and chemical mechanisms of the polymerization could be fully expressed and the influence of possible different initial reaction rates among the cements was excluded.

If the chemical catalyst system was equivalent and/or more efficient than the light-polymerization, absence of light activation of the cements through the spacers would not have any adverse effect on the polymerization extent. Many studies have already reported lower surface hardness and monomer conversion in dual-polymerized resin cements when the light-activation stage was skipped[12,13,25,26.]

In the present study, the RDB findings clearly presented a block-material effect. Photopolymerization through the laboratory-processed composite Sinfony caused a higher percentage RDB increase compared with the porcelain material Empress 2, independent to the dual-polymerized resin cement. It has been reported that even a 1 mm-thick resin composite decreases light intensity up to 70%.[20] El-Mowafy[11] has shown that above 4 mm, composite totally obstructs light. Furthermore, Myers et al.[7] detected that for light shades, the castable glass-ceramic Dicor (Dentsply International Inc, York, PA) transmitted light better that a photopolymerized composite (Herculite; Kerr) and a heat-pressed laboratory composite (Concept; Ivoclar / Williams).

Light intensity reduction through a resin composite is affected by the attenuation coefficient of the material, according to Lambert's exponential law of absorption, which is strongly influenced by the refractive index of the material [32]. High differences in refractive index between the inorganic fillers and the organic matrix of Sinfony may cause high light scattering and drop of the light energy[33,34].

It is well-documented that porcelain opacity has an important role on light obstruction[7,8] , although slightly higher opacity has been previously measured for Empress 2 than for the feldspathic porcelain Vitadur Alpha, in specimens 0.5 mm and 1.5 mm thick,[35,36] the first product demonstrated significantly lower RDB (combined with Panavia F 2.0 and Nexus 2) than the second ceramic. Thus, opacity differences between the 2 porcelains in thin layers may be reversed in 2 mm-thick specimens. Ceramic opacity is affected by various factors, such as crystalline structure, firing procedure, and glazing,[37] however, thickness seems to be the primary factor controlling opacity[6,38]. In addition to the thickness effect which is important for the same ceramic mass, different ceramic masses that have different opacities behave differently and cannot be compared in regards to thickness alone.

An interesting finding was the less negative effect of Vitadur Alpha and Sinfony on the cement RelyX ARC, compared to Panavia F 2.0 and Nexus 2. This finding may imply the incorporation of a stronger chemical-polymerization component in RelyX ARC. It is not easy to explain the Panavia F 2.0 performance that showed the lowest RDB increase of all the cements only when polymerized through Empress 2.

The results of the present study indicated that the dual-polymerized resin cements at the margins of indirect aesthetic restorations (the control group of the present study corresponds to this situation) that have received direct irradiation during polymerization may be under different polymerization state to that at areas that received light irradiation through 2 mm thick restorative material. Therefore, it is reasonable to hypothesize that different areas of the dual-cured resin cement of a restoration may not exhibit similar physic-mechanical properties, which subsequently may lead to non-uniform stress distribution through the restoration during function. However, the absolute relation between percentage of remaining double bonds (RDB) and clinical performance has not been established.

It was shown in this investigation that the incorporation of the chemical polymerization components in the dual-polymerized resin cements assessed cannot adequately compensate for the reduced light-polymerization activity due to the light attenuation of composite and ceramic materials. In particular, for inlays and onlays with deep interproximal boxes, the occlusal-cervical height is up to 7-8 mm and the light penetration is severely obstructed under these restorative materials. Thus, light-polymerization from buccal and lingual proximal walls may be desirable for Class II indirect restorations. Additionally, improvement of the light intensity reaching beneath the restorative material may be a key factor for sufficient polymerization, even for the dual-polymerized resin cements.

The ISO standard[39] does not report the minimally-accepted light intensity to polymerize composite resins and resin cements. It has been shown[40] that 300 mW/cm^2 is the lowest intensity, which appears to adequately polymerize most of the resin-based dental materials to a depth of 2 mm. In addition to the attenuation of light by the restorative material, keeping the tip of the photopolymerizing unit distant from the surface of the resin cement has proved to cause adverse effect in their polymerization potential[41,42] . High intensity halogen or plasma-arc light-polymerization units, specially designed light-guide tips, and longer irradiation times have been suggested for light polymerization of dual-polymerized cements through metal-free restorations[9,10,43-45] .

CONCLUSIONS

Within the limitations of the present study the following conclusions can be drawn: The 3 dual-polymerized resin cements examined showed no difference in remaining double carbon bonds (RDB) under direct light irradiation conditions. Direct light exposure of the dual-polymerized resin cements attained lower RDB values compared with the analogues obtained through 2 mm-thick porcelain Vitadur Alpha and laboratory-processed resin composite Sinfony. The laboratory-processed resin composite Sinfony provided higher RDB values than the ceramic Empress 2 for all the tested resin cements. Compared with Vitadur Alpha, Sinfony displayed higher values for the cement RelyX ARC. The resin cements Panavia F 2.0 and Nexus 2 demonstrated higher polymerization efficiency in combination with Empress 2 than with Vitadur Alpha. The resin cement RelyX ARC was less affected by the blocking effect of Vitadur Alpha than Panavia F 2.0 and Nexus 2.

ADDRESS FOR CORRESPONDENCE

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