Light-activation through indirect ceramic restorations: does the overexposure compensate for the attenuation in light intensity during resin cement polymerization?

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ABSTRACT

O bjectives: This study evaluated the effects of light exposure through simulated indirect ceramic restorations (SICR) on hardness (KHN) of dual-cured resin cements (RCs), immediately after light-activation and 24 h later. Material and Methods: Three dual-cured RCs were evaluated: eco-Link (Ivoclar Vivadent), Rely X ARC (3M eSPe), and Panavia F (Kuraray Medical Inc.). The RCs were manipulated in accordance to the manufacturers’ instructions and were placed into cylindrical acrylic matrixes (1-mm-thick and 4-mm diameter). The RC light-activation (Optilux 501; Demetron Kerr) was performed through a glass slide for 120 s (control group), or through 2-mm or 4-mm thick SICRs (IPS empress II; Ivoclar Vivadent). The specimens were submitted to KHN analysis immediately and 24 h after light-activation. The data obtained at the 2 evaluation intervals were submitted to 2-way ANOVA repeated measures and post-hoc Tukey’s test (pre-set alpha of 5%). Results: Lower KHN was observed when light-activation was performed through SICRs for eco-Link at all evaluation intervals and for Rely X ARC 24 h later. For Panavia F, no significant difference in KHN was observed between control and experimental groups, regardless of evaluation interval. Most groups exhibited higher KHN after 24 h than immediately after light-activation, with the exception of Rely X ARC light-activated through SICR, as no significant difference in KHN was found between evaluation intervals. Conclusions: Light overexposure did not compensate for light intensity attenuation due to the presence of SICR when Rely X and Eco-Link were used. Although hardness of such RCs increased over a 24-h interval, the RCs subjected to light overexposure did not reach the hardness values exhibited after direct light exposure.

K ey words: Dual-curing of resin cements. Hardness. Ceramics.

INTRODUCTION

Indirect ceramic restorations have been chosen as better options than direct resin composite restorations to restore wide dental cavities, such as large inlay and onlay restorations. The main advantages of indirect ceramic over direct resin composite restorations are the better wear resistance, lower stain susceptibility and the ability to simulate enamel aesthetical features. For this reason, indirect ceramic restorations became one of the most important and popular clinical procedures in the last few years. Adhesion of most types of glass ceramic restorations to tooth structure can be obtained by the use of resin cements (RCs), as they show a reliable
bonding to the tooth substrates, low solubility, and optimal aesthetics\textsuperscript{13,18,22,23}. Furthermore, when compared to glass-ionomer and zinc phosphate cements, resinous materials provide higher fracture strength values to fatigue when used to bond metal-free ceramic crowns, ceramic inlays and onlays to the prepared tooth\textsuperscript{14}.

Dual-cured RCs were developed in an attempt to combine the desirable physical properties obtained from chemical and light polymerization\textsuperscript{6}, and to allow proper monomer conversion at deep areas where the activation light is attenuated or totally absent\textsuperscript{20}. Ceballos, et al.\textsuperscript{6} (2007) demonstrated that dual-cured RCs present better mechanical properties than self- and light-cured RCs. However, some authors have reported that dual-cured RCs cannot achieve proper polymerization and acceptable mechanical properties when they rely solely on the self-curing mode\textsuperscript{15,28}.

An optimal monomer conversion of RCs is crucial to assure the ideal physical properties and durable clinical performance of the indirect ceramic restoration on the tooth\textsuperscript{19}. Conversely, poor polymerization impairs the cement physical properties, so low bond strength, high water absorption and compromised shade stability are expected as a consequence\textsuperscript{19}. In addition, low monomer conversion allows the release of toxic substances from the polymer to the pulp due to the poor polymeric chain formation\textsuperscript{8,10,20}.

Low monomer conversion of dual-cured RCs has also been observed when activation light is attenuated by the presence of indirect ceramic restorations as the self-curing components are not able to compensate for the low light intensity that reaches the resinous material\textsuperscript{11,19}. For this reason, light-activation of dual-cured RCs through ceramic discs with thickness ranging from 1 to 5 mm results in lower cement hardness, which has been considered an indirect assessment of monomer conversion\textsuperscript{5,8,10}. The authors also observed that the decrease in hardness was more evident when the ceramic restorations were thicker than 4 mm. Therefore, the authors concluded that the thickness of the indirect ceramic restoration is related to the decrease in cement hardness.

Polymerization in deep cavities located on mesial and distal areas, where indirect ceramic restorations are thicker, deserves more concern as the activation light is usually transmitted through at least 4 mm of indirect restoration to reach the RC layer, so the RC relies mostly upon the self-curing mode\textsuperscript{1}. Based on such issue, manufacturers recommend to light-activate the RC not only from the occlusal surface, but also from buccal and lingual surfaces. However, most studies that evaluated the effect of simulating indirect restorations on the hardness of dual-cured RCs were based on 40-s light-activation through the indirect restoration\textsuperscript{8,9,15}. Therefore, it is important to evaluate the hardness of dual-cured RCs under simulated indirect ceramic restorations when light-activation is performed from all sides of the indirect restoration. Moreover, it is also important to evaluate the hardness of RCs just after light-activation, when the indirect restoration is subjected to the stress generated by the occlusal adjusts and polishing procedures. Thus, the aim of the present study was to evaluate the hardness of three dual-cured RCs under simulated indirect ceramic restorations with different thicknesses when light-activation was performed on the occlusal, vestibular and lingual surfaces. The research hypothesis was that light overexposure does not compensate for the attenuation in light intensity promoted by the presence of ceramic restorations after light-activation.

**MATERIAL AND METHODS**

**Specimen preparation**

Three dual-cured RCs were evaluated in the current study, Eco-Link (Ivoclar Vivadent, Schaan, Liechtenstein), Rely X ARC (3M ESPE, St. Paul, MN, USA), and Panavia F (Kuraray Medical Inc., Kurashiki, Okayama, Japan). The RC compositions are shown in Figure 1.

The RCs were manipulated as recommended by the manufacturers and were placed into acrylic matrices to create specimens measuring 1 mm in thickness and 4 mm in diameter. Each specimen was covered with Mylar strip, which was then covered with a glass slide. A 500-g weight was placed over the glass slide for 30 s to create a flat surface. Although the RCs evaluated in the current study require the use of their respective adhesive systems, no bonding agents were associated with the RCs.

In an attempt to simulate the clinical situation, 2- and 4-mm-thick A3-shade IPS Empress II (Ivoclar Vivadent, Schaan, Liechtenstein) ceramic discs were made to simulate indirect restorations with different thickness. The shape of the indirect restoration covered not only the top of the specimen but also the specimen sides (Figures 2A and 2B). However, the thickness of the indirect restoration around the specimen was approximately 1.6 mm (Figures 2A and 2B).

The RCs were then exposed to the activation light (power density: 700 mW/cm\textsuperscript{2}, Optilux 501; Demetron Kerr; Danbury, CA, USA) for 40 s on the top and on both sides of the indirect restoration to simulate light-activation on the occlusal, lingual, and buccal surfaces (Figure 2), so each specimen was exposed to 120-s light exposure. In the control groups, the light exposure of RCs was performed through a glass slide for 120 s. Light from the curing unit had its intensity constantly evaluated with the radiometer attached to the curing unit device. Therefore, this study comprised 6 (three products
and two indirect restorations with different thickness) experimental groups with 10 specimens (RC) each (n=10).

**Microhardness analysis**

After the indirect restorations were removed from the top of the RC layer, the top surface of the cured RCs was submitted to Knoop hardness analysis (KHN) (Pantec, Panambr Ind. e Técnica SA, São Paulo, SP, Brazil) immediately and 24 h after light-activation, under 25 g load with a dual time of 5 s. Five indentations distant 100 mm from each other were performed to determine the average KHN for each specimen (Figure 1). Afterwards, the specimens were dark stored in relative humidity at 37°C for 24 h and another KHN analysis was performed. The data obtained from the RCs immediately and 24 h after light-activation were submitted to 2-way ANOVA repeated measures ("ceramic thickness" and "evaluation interval" factors) for each product followed by post-hoc Tukey’s test at a pre-set alpha of 5%, with "evaluation interval" factor as the dependent variable. As the current study aimed only to evaluate the effects of light attenuation and overexposure on KHN of RCs, no comparison were performed among products.

**RESULTS**

The KHN values are displayed in Table 1. For Eco-Link, lower KHN values were observed when light-activation was performed through indirect ceramic restoration (p=0.00126), regardless of its Product (Manufacturer) Composition (Batch#)

| Product (Manufacturer) | Composition (Batch#) |
|------------------------|-----------------------|
| Eco-Link (Ivoclar Vivadent) | Paste of dimethacrylic, inorganic fillers, ytterbium trifluoride, initiators, stabilizers and pigments Bis-GMA; TEGDMA; urethane dimethacrylate; benzoyl peroxide (lot H29208) |
| Panavia F (Kuraray) | Paste A: 10-Methacryloyloxydecyl dihydrogen phosphate; Hydrophobic aromatic dimethacrylate; Hydrophilic aliphatic dimethacrylate; Silanated silica filler; Silanated colloidal silica; dl-Camphorquinone; Catalysts Paste B: sodium fluoride; Hydrophobic aromatic dimethacrylate; Hydrophobic aliphatic dimethacrylate; Hydrophilic aliphatic dimethacrylate; Silanated barium glass filler; Catalysts; Accelerators; Pigments (lot 51185) |
| Rely X ARC (3M ESPE) | Paste A: Silane treated ceramic;TEGDMA*; Bis-GMA**; Silane treated silica; Functionalized dimethacrylate polymer; Paste B: Silane treated ceramic; TEGDMA; Bis-GMA; Silane treated silica; Functionalized dimethacrylate polymer (lot FBFM) |

*TEGDMA: Triethylene glycol dimethacrylate, **Bis-GMA: Bisphenol A diglycidyl ether methacrylate
thickness and evaluation interval. No significant difference in KHN values was observed when 2- or 4-mm-thick indirect restorations were used, and all KHN values obtained after 24 h were higher than those obtained immediately after light-activation.

For Rely X ARC, no significant difference in KHN values was observed between the control group and groups with indirect restorations immediately after light-activation. On the other hand, the control group exhibited higher KHN values than the other experimental groups after 24 h (p=0.02731). No significant difference in KHN values was observed between groups with indirect restorations, regardless of analysis interval. The 24-h KHN values were significantly higher than the values obtained immediately after light-activation only in the control group (p=0.00836).

When Panavia F was evaluated, no significant difference in KHN values was observed between control and experimental groups, regardless of time interval. All groups exhibited higher KHN values after 24 h than immediately after light-activation (p=0.00022).

**DISCUSSION**

The results of the current study demonstrated that the effects of curing light attenuation and overexposure on KHN of dual-cured RCs were product and time dependent. The use of 2- or 4-mm thick ceramic discs between light curing unit tip and the RC resulted in lower KHN values for Eco-Link, regardless of time, and for RelyX only at 24-h evaluation interval. Therefore, the research hypothesis was accepted for Eco-Link and was only partially accepted for RelyX. Such results corroborate with the results from other studies that evaluated the effects of 40-s light-activation through ceramic discs, so the longer light exposure used in the current study did not provide enough energy to compensate for the light attenuation when these products were used.

The lower KHN values exhibited by Eco-Link immediately after light-activation demonstrate that light attenuation may have resulted in slower polymerization kinetics when compared to that observed in the control group, despite the long time of light exposure (120 s). El-Mowafy and Rubo (2000) showed that the intensity of light transmitted through 2-mm-thick ceramic indirect restorations decreases from 700 mw/cm² to approximately 100 mw/cm². Thus, while a regular 40-s light-activation through a glass slide delivers approximately 283/cm² to the RC surface to ensure its highest potential for polymerization, the 120-s light-activation through a 2-mm-thick ceramic disk delivers approximately 12 J/cm² to the RC. As immediate KHN values are closely related to the effects of light-activation rather than to the slow self-curing mechanism, such slower kinetics of polymerization observed in Eco-Link may be attributed to a low photoinitiator content. However, only studies that evaluate the relationship between light intensity and photoinitiator content in dual-cured RCs could confirm such speculation.

On the other hand, no significant difference in KHN values was observed among groups immediately after light-activation when Rely X ARC was used. One could state that the attenuated curing light that reaches the RC with interposed ceramic discs had enough intensity to harden the material similarly to the control group. However, the control group exhibited a significant increase in KHN between the two evaluation periods, immediately after light-activation and 24 h later. Based on such an unexpected difference, it is possible to speculate that the monomer conversion of the control group of Rely X ARC immediately after light-activation was lower than its optimal polymerization, despite the 120-s light exposure. Moreover, it should be emphasized that some mechanical properties of RCs are not only influenced by the degree of conversion, but also by the crosslinking and network formation after an initial stage of polymer chain propagation. Therefore, it is possible that the higher energy delivered to the RC in the control group for 120 s produced a faster polymerization rate, which in turn promoted an average polymer chain length lower than that when slower polymerization rate is promoted. As a consequence, the increase in
KHN values after 24-h interval might be attributed to a delayed increase in polymer chain and cross-linking formation.

The results exhibited by Rely X ARC groups using 2- and 4-mm-thick ceramic discs may confirm such assumption, as no significant increase in KHN values was observed in these groups 24 h after light-activation through ceramic restorations. On the other hand, the lack of increase in KHN values over time may be attributed to the low effectiveness of the cement self-curing components, which were not able to provide further polymerization when activation light was attenuated. Such lower effectiveness of self-curing components in Rely X ARC was confirmed in other studies, which demonstrates that Rely X ARC depends upon photoactivation to improve its mechanical properties5,12,28.

Among all products evaluated in the present study, Panavia F exhibited the most clinically acceptable results when activation light was attenuated by the presence of an indirect restoration and KHN values depended on chemical activation, as no significant difference in KHN was observed among groups. Such result was unexpected, since some studies have shown that Panavia F should not depend solely on its self-curing mode17,27. Therefore, it seems that overexposure to light compensated for the light attenuation when ceramic discs with different thickness were used. Thus, the research hypothesis was rejected for Panavia F.

The effects of curing light attenuation on monomer conversion deserves more concern when a 4-mm-thick ceramic disk is used, as monomer conversion depends upon the self-curing mode to occur7. However, no significant differences in KHN values were observed between groups with 2- and 4-mm-thick ceramic restorations, regardless of product and evaluation interval. Therefore, it is possible to assume that light exposure for 120 s allowed light to excite the photoinitiators and start the polymerization even when 4-mm-thick ceramic discs were used.

A significant increase in KHN values was observed in groups with interposed ceramic discs or in the control groups after 24 h when Panavia F and Eco-Link were used. Such result was expected since polymerization still continues for 24 h after light-activation26. However, the increase in KHN values did not result in KHN values as high as those observed in the control group for Eco-Link. Interestingly, the increase in KHN values in groups with 2-mm and 4-mm-thick ceramic restorations (4.2% and 5.2%, respectively) was apparently higher than that observed in the control group (1%) when Eco-Link was used. This result may be attributed to the low initial monomer conversion of the RC after light-activation through the ceramic restorations, as lower monomer conversion results in low initial viscosity, which has allowed the radicals to migrate and facilitate the self-curing components to react for some hours after initial polymerization26. Further analyses of the polymerization kinetics of dual-cured RCs polymerized under ceramic restorations may confirm such speculation.

Besides the differences in polymerization features shown by the evaluated RCs, differences in the chemical structure of such products may have also played an important role in their water sorption and solubility. Therefore, the presence of hydrolytically susceptible groups, such as ester, urethane, ether and hydroxyl groups at different concentrations may also determine different solubility patterns for the tested RCs11,21. The plasticizing effect in the resin matrix promoted by water penetration into the resin matrix results in decreased hardness11,21, so the increase in hardness promoted by further 24-h polymerization could be less evident in RCs with higher solubility to water. Thus, it is possible to speculate based on the 24-h KHN values that Panavia F showed lower solubility after 24 h of immersion in distilled water than the other RCs.

In the present study, specimens of RCs were prepared without any bonding agent. It is important to emphasize that the mechanical properties of RCs in the clinical situation may change when they are coupled with their respective bonding agents. One could state that previous light activation of the adhesive layer would avoid the mixture between bonding agent and RC to occur. However, some manufacturers recommend that the adhesive layer should be left in the uncured state when the RC is applied to the prepared tooth, so a combined layer composed of bonding agent and RC is created in that situation2. Therefore, such new combined layer could present different mechanical properties when compared to the RC layer solely since the mixture between bonding agent and RC would change composition and polymerization kinetics1. Further studies are required to evaluate the differences in hardness and solubility of such combined layer in comparison to RCs.

Light overexposure was able to compensate for the light attenuation caused by ceramic restorations in only one RC, while the other RCs showed lower KHN values when ceramic restorations were interposed between the curing unit tip and the material surface. The low KHN values observed immediately after light-activation deserve some concern, as stress is generated by the occlusal adjusts and polishing procedures at that time.
CONCLUSION

Based on the obtained results, it was possible to conclude that light overexposure did not compensate for the light intensity attenuation due to the presence of ceramic restorations for most of the evaluated dual-cured RCs. Although an increase in hardness was observed over time, the RCs exposed to attenuated light did not reach the hardness values exhibited after direct light exposure.

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