

## UNIVERSIDADE ESTADUAL DE CAMPINAS



### FACULDADE DE ODONTOLOGIA DE PIRACICABA

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Effect of light-curing methods and indirect veneering materials in a resin cement Knoop hardness

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## Lista de abreviaturas e siglas

et al. = e outros

SiC = Carbeto de Silício

S.D. = Desvio padrão

**LED** = Luz emitida por diodos

PAC = Arco de plasma de xenônio

QHT = Quartzo halogênio tungstênio

KHN = Número de dureza Newton

MN = Desvio padrão

SP = São Paulo

USA = Estados Unidos da América

Corp. = Corporação

Equip. = Equipamentos

Ltda. = Limitada

mm² = milímetros quadrados

mm = milímetros

h = horas

s = segundos

% = por cento

#### Resumo:

Este estudo avaliou a dureza Knoop do cimento resinoso dual (Rely-x) ativado quimicamente (grupo controle) ou química/fisicamente (dual). Antes da ativação, uma faceta de 1,5mm de espessura da cerâmica (HeraCeram) ou compósito (Artglass) foi colocada sobre o cimento. No grupo dual, a fotoativação foi realizada com aparelho de luz de lâmpada halógena convencional (XL2500), por 40s (QHT); luz emitida por diodos (Ultrablue Is), por 40s (LED); e arco de plasma de Xenônio (Apollo 95E), por 3s (PAC). Um incisivo bovino teve sua face vestibular planificada e hibridizada. Sobre esta superfície, uma matriz de borracha (5mm de diâmetro e 1mm de altura) foi preenchida com cimento. Assentou-se uma tira de poliéster para fotoativação direta ou através de um disco dos materiais para faceta. Após armazenamento em ambiente seco e escuro (24h/37°C), as amostras (n=5) foram seccionadas para mensuração dos valores de dureza (KHN), obtidos em um aparelho microdurômetro (50gf/15s). Os dados foram submetidos à análise de variância e ao teste de Tukey ( $\alpha$ =,05). O cimento apresentou maiores valores de dureza Knoop com Artglass para QHT e LED, comparado ao HeraCeram. O grupo controle e grupo PAC/Artglass mostraram os valores de dureza mais baixos comparados aos grupos fotoativados com QHT e LED. A combinação PAC/HeraCeram resultou nos piores valores de dureza do cimento.

Descritores: cimento de resina, dureza, resina composta, porcelana dental

Abstract

This study evaluated the Knoop hardness of the dual-cured resin cement

(Rely-X) activated by solely chemical reaction (control group) or by chemical/

physical mode, light-cured through 1.5mm thick ceramic (HeraCeram) or composite

(Artglass). Light curing was carried out using conventional halogen light (XL2500), for

40s (QHT); light emitted by diodes (Ultrablue Is), for 40s (LED); and Xenon plasma

arc (Apollo 95E), for 3s (PAC). A bovine incisor had its buccal face flattened and

hybridized. On this surface a rubber mold (5mm in diameter and 1mm in height) was

bulk filled with cement. A polyester strip was seated for direct light curing or through a

disc of veneering material. After storage dry in dark (24h/37°C), samples (n=5) were

sectioned for hardness (KHN) measurements, taken in a microhardness tester (50gf

load/15s). Data were submitted to ANOVA and Tukey's test ( $\alpha$ =.05). The cement

presented higher Knoop hardness values with Artglass for QHT and LED, compared

to HeraCeram. The control group and PAC/Artglass group showed lower hardness

values compared to groups light-cured with QHT and LED. PAC/HeraCeram resulted

in the worst combination for cement hardness values.

Descriptors: resin cement, hardness, resin composite, dental porcelain

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#### Introduction

The use of resin cements has grown in the last few years due to a larger application of indirect restorative materials, as ceramics and resin composites. As advantages, these cements present adhesion to substrates, by silane agents and adhesive systems compatibility, low solubility, easy manipulation and favourable aesthetics when used with metal-free ceramic systems. The application of these cements can still result in higher fatigue compressive strength of all-ceramic crowns compared to glass ionomer cements and to zinc phosphate cements<sup>4</sup>.

In spite of the variety of available cements, there is not ideal cement for all the clinical situations. Therefore, the choice of the fixation agent must rely on its physical, biological and manipulation properties added to the characteristics of the remainder of prepared tooth and the prosthesis<sup>13</sup>.

Factors as light-curing method and exposure time, indirect restorative material and also the fixation agent can influence in the final quality of restoration 1,12. Inlays, onlays, laminated veneers and all-ceramic crowns are commonly fixated with dual-cured resin cements because light transmission through indirect restorative is critical and at this context, the chemical reaction theoretically would guarantee a satisfactory polymerization. It was verified that the light transmission spectrum through ceramic is influenced by its thickness, shade, and opacity 8,18. The influence of these factors can also be observed during the cementation of a laminated veneer of indirect resin composite 1. The application of longer light-curing times results in higher resin composite polymerization depth, higher conversion degree and higher hardness values 1,18, consequently, in improved mechanical and aesthetic properties 19. According to Tanoue *et al.* 17, the same can be applied to light-cured resin cements.

The hardness test is commonly used as a simple and reliable method to

indicate the degree of conversion of resin cements<sup>2</sup>. The degree of conversion in a

polymerization reaction is dependent of the energy supplied during light curing,

characterized as the product of the light intensity and exposure time14. In the same

brand, dual-cured resin cements when light-cured present higher hardness values

compared to light-cured solely1, and light-activated dual-cured resin cements present

higher hardness values compared to dual-cured solely chemically 14. Witzel et al. 20, in

2003, verified that the dual-cured resin cements, when not light-cured and associated

to one-bottle adhesive systems, resulted in about 51% and 64% lower values of bond

strength compared to that obtained with light-cured dual activated cements.

Light curing is usually performed with Quartz Tungsten Halogen light-

curing units. Other technologies as Xenon plasma arc (PAC) and light emitted by

diodes (LED) are also available. In spite of these systems are still developing, its

application is growing. Doubts about the effectiveness of light-activation of resin

cements with different methods using these light-curing units still exist. Thus, the null

hypotheses of this study are that similar resin cement hardness values would be

obtained with different veneering materials, light-curing units and cement activation

modes.

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#### **Materials and Methods**

For this study a disc-shaped specimen (1.5mm in thick and 7mm in diameter) was prepared for each material, a feldspathic ceramic (HeraCeram, Heraeus Kulzer, Wehrhein, Germany) and an indirect resin composite (Artglass, Heraeus Kulzer, Wehrhein, Germany).

To simulate cementation condition a bovine incisor was sectioned and its coronal portion was embedded in polystyrene resin maintaining the vestibular surface exposed. This surface was ground flat under water-cooling with SiC sandpapers with #200, 400 and 600 grit (Saint-Gobain, Recife, PE, Brazil), to obtain a dentine area of at least 25mm². On this surface a polyester strip was seated, and over this set a rubber mould (5mm in diameter and 1mm in height) was bulk filled with cement. The dual-cured resin cement Rely-X ARC (3M ESPE, Saint Paul, MN, USA), on shade A3, was manipulated according to manufacturer's instructions. A polyester strip was seated over this set, and with a disc of veneering material the cement was digitally compressed for excesses flow and removal. Light curing was carried out with conventional quartz halogen tungsten (QHT) light-curing unit (LCU) – XL 2500 (3M ESPE, Saint Paul, MN, USA) for 40s at 700mW/cm², light emitting diodes (LED) - Ultrablue Is (DMC Equip. Ltda., São Carlos, SP, Brazil) for 40s, at 440mW/cm², and with Xenon plasma arc (Apollo 95E, DMD Equip. Ltd., California, USA), for 3s at 1600mW/cm². As control group, the cement was set by solely chemical reaction.

After light curing, the samples were stored dry in dark at 37°C, for 24h. To perform resin cement Knoop hardness measurements, samples were embedded in self-cured acrylic resin, and sectioned longitudinally under water-cooling with a diamond saw (Extec model 12205, Extec corp., Enfield, USA). The surface obtained by sectioning was polished sequentially under water-cooling with SiC sandpapers with # 400, 600 and 1200 grit.

Indentations and micro-hardness measurements (KHN) were performed sequentially, in a micro-hardness tester machine HMV-2000 (Shimadzu, Tokyo, Japan). Three indentations were performed in each depth of 100, 500 and 900µm from the top surface, with load of 50gf for, 15s.

For each sample a mean hardness value was obtained from nine measurements, and data submitted to one-way ANOVA and to Tukey's test, both with  $\alpha$ =.05.

#### Results

ANOVA showed statistical significant differences among groups (p<.05). The Tukey's test showed that the resin cement presented higher Knoop hardness values with Artglass for QHT and LED, compared to HeraCeram. The control group and PAC/Artglass group showed lower hardness values compared to groups light-cured with QHT and LED. PAC/HeraCeram resulted in the worst combination for cement hardness values (Table1).

Table 1. Comparison of mean hardness values (KHN).

MEAN (S.D.)		
51.76 (5.01) a		
51.50 (4.11) a		
HeraCeram 45.35 (6.04) b		
LED/ HeraCeram 44.47 (4.90) b		
28.47 (2.99) c		
26.26 (4.93) c		
21.82 (3.81) d		

Different small letters in column represent statistical significant differences among groups (p<.05). Standard deviations are presented between ( ).

#### **Discussion**

The results (Table 1) showed that there was a negative influence of light-curing the resin cement Rely-X with PAC compared to QHT and LED, since lower hardness values were obtained with the former. Light curing with the last two ones resulted in similar hardness values. This behaviour might be due to the short exposure time during light-curing with PAC<sup>3,5</sup>, that resulted in low energy density supplied to resin cement. This low energy leads to low degree of conversion of the cement, determined indirectly by hardness values measurements<sup>15</sup>. It can be hypothesised that with an increase in light-curing exposure time similar hardness values to QHT and LED would be obtained. Light curing with PAC would be even better for resin cement because its high emitting light intensity, which would be less attenuated by the veneering materials compared to QHT and LED. The results of this study are in agreement with Rasetto *et al.*<sup>12</sup>. On the other hand, Ozyesil *et al.*<sup>9</sup> verified similar degree of conversion of resin cement Variolink II light-cured with conventional QHT and PAC.

The veneering material also showed to influence in resin cement hardness. Higher hardness values were verified with Artglass compared to HeraCeram (Table 1). It might be result of the different nature of these materials, which implies in distinct optical characteristics. It was not possible to compare these results with the literature, hence studies using this methodology were not found.

Light curing with PAC through HeraCeram resulted in the lowest hardness values, even compared to solely chemical-cured cement (Table 1). It can be supposed that the light scattering and refracting determined by HeraCeram would be higher compared to Artglass. The lower values of PAC could be result of the light attenuation by veneering materials, and by resin cement *per se*<sup>6</sup>. Another hypothesis was that light curing with PAC through HeraCeram, induced the initiation of

polymerization reaction characterised by cross-linking formation. It could have reduced the monomers mobility in the mass bulk decreasing the polymerization complementation by chemical-cure<sup>16</sup>. According to Soh & Yap<sup>11</sup>, in 2004, light curing with high intensity would lead to a high cross-linked polymer chain, and to higher hardness. In regions submitted to low energy density the polymer chain would be more linear with higher mobility, and lower hardness values.

The cementation using dual-cured resin cements has been indicated due the chemical initiators that would, theoretically, guarantee a reliable cement polymerization even with a deficient light-curing<sup>7</sup>. Peutzfeldt<sup>10</sup> observed that the best mechanical properties of the dual-cured cements were obtained with their light curing, avoiding the polymerization reaction by solely chemical curing.

Further studies are necessary about the veneering materials characteristics, and reasonable light-curing exposure times with PAC for dual-cured cement polymerization.

### Conclusions

Limited by the methodology used in this study and according to the results, it can be concluded that:

- 1. Higher hardness values were obtained with QHT and LED, compared to PAC and solely chemical-cured group.
- 2. Light curing through Artglass resulted in higher hardness values compared to HeraCeram.
- 3. Light curing with PAC for just 3s showed to influence negatively on resin cement hardness.

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