

### **UNIVERSIDADE ESTADUAL DE CAMPINAS**



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Resin cement polymerisation depth:

Effect of light-curing methods in

cement Knoop hardness.

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## Sumário

Lista de tabelas	5
Lista de abreviaturas e siglas	6
Resumo	7
Summary	8
Introduction	9
Materials and methods	11
Results	13
Discussion	14
References	16

## Listas de tabelas

Figure1 – Schematic illustration of indentations performing sequence.......13

## 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

mm<sup>2</sup> = milímetros quadrados

mm = milímetros

h = horas

**s** = segundos

% = por cento

#### **RESUMO**

Este estudo avaliou a profundidade de polimerização do cimento ativado (grupo controle) quimicamente resinoso (Rely-X) química/fisicamente (dual), através de teste de dureza Knoop. Antes da ativação, uma faceta de 1,5mm de espessura da cerâmica (HeraCeram) 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 do material 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) em três diferentes profundidades, 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 QHT e LED, comparados ao grupo controle e PAC. A fotoativação com PAC resultou em valores de dureza inferiores ao grupo controle. A dureza do cimento foi menor em regiões mais profundas.

SUMMARY

This study evaluated the polymerisation depth 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),

by Knoop hardness test. 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

surface flattened and hybridised. On this surface a rubber mould (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 in three different depths, 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 for QHT and LED, compared to control

group and PAC. Light curing with PAC resulted in lower hardness compared to

control group. The cement hardness decreases in deeper regions.

KEY WORDS: resin cement, hardness, dental porcelain, polymerisation

8

#### INTRODUCTION

The use of resin cements has grown in the last few years due to a larger application of indirect restorative materials, such as ceramics. 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<sup>1,12</sup>. 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 polymerisation. It was verified that the light transmission spectrum through ceramic is influenced by its thickness, shade, and opacity<sup>8,18</sup>. The application of longer light-curing times results in higher resin composite polymerisation depth, higher conversion degree and higher hardness values<sup>1,18</sup>, consequently, in improved mechanical and aesthetic properties<sup>19</sup>. According to Tanoue *et al.*<sup>17</sup>, the same can be applied to light-cured resin

cements.

to indicate the degree of conversion of resin cements<sup>2</sup>. The degree of conversion in a polymerisation reaction is dependent of the energy supplied during light curing, characterised as the product of the light intensity and exposure time<sup>14</sup>. In the same brand, dual-cured resin cements when light-cured present higher hardness values compared to light-cured solely<sup>1</sup>, and light-activated dual-cured resin cements present higher hardness values compared to dual-cured solely

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

5 m

chemically<sup>14</sup>. Witzel et al.<sup>20</sup>, 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 light-curing units and cement activation modes.

10

### **MATERIALS AND METHODS**

For this study a disc-shaped specimen (1.5mm in thick and 7mm in diameter) was prepared with a feldspathic ceramic (HeraCeram, 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/cm<sup>2</sup>, and with Xenon plasma arc (Apollo 95E, DMD Equip. Ltd., California, USA), for 3s at 1600mW/cm<sup>2</sup>. As control group, the cement was set by solely chemical reaction.

After light curing, the samples (n=5) 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 waffering blade (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 (Figure 1).

For each sample a mean hardness value was obtained for each depth, and data submitted to ANOVA split-splot design 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 for QHT and LED, compared to PAC and to control group, in all tested regions. The PAC group showed lower hardness values compared to control group, except to the center region (Table1). For QHT and LED, just the bottom region showed lower hardness values. The control group presented similar hardness values in all regions. For PAC, the center region showed higher hardness values compared to the bottom region, and the top surface showed intermediate values.

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

TOP	TOP	CENTER	ВОТТОМ
		MEAN (S.D.)	
QHT/HeraCeram	49.22 (5.41) A, a	46.21 (4.13) A, a	40.63 (5.84) A, b
LED/ HeraCeram	47.52 (3.30) A, a	46.02 (4.41) A, a	39.87 (3.58) A, b
Chemical	28.76 (2.99) B, a	31.28 (2.88) B, a	25.38 (4,27) B, a
PAC/HeraCeram	21.93 (2.30) C, ab	25.36 (2.11) B, a	18.16 (2.93) C, b

Different capital letters in the column and small letters in the row 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, LED, and control group, since lower hardness values were obtained with the former. Light curing with the last two ones resulted in similar hardness values, in all tested regions. 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 material 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.

Light curing with PAC resulted in the lowest hardness values, even compared to solely chemical-cured cement (Table 1). 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 polymerisation 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 polymerisation reaction by solely chemical curing. This behaviour was also observed in this study. In deeper regions, where less light is available, lower hardness values were obtained (Table 1).

Further studies are necessary about the light curing through indirect prosthetic materials, and about which would be a reasonable light-curing exposure time with PAC for dual-cured cement polymerisation.

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