

INFLUENCE OF PHOTOACTIVATION PROTOCOL AND CERAMIC THICKNESS ON MECHANICAL PROPERTIES OF CEMENTATION AGENTS

ABSTRACT

The objective of this work was to evaluate the influence of photoactivation protocol and ceramic thickness on the Knoop microhardness (KHN) and the diametrical tensile strength (DTS) of a dual resin cement (Rely X ARC 3M ESPE (RL)) and a low viscosity composite resin (Filtek Z350 Flow 3M ESPE (Z)). Thereunto, cementation agents were photoactivated under ceramic discs of lithium disilicate with thickness of 1 and 2mm, and a glass slide (control group). The photoactivation was performed with halogen light-curing unit in continuous mode (XL 3000 3M Espe,) (XL) 500 mW/cm² X 38 s, and with third generation LED light-curing unit (Valo Ultradent) in Standard mode (S) 1000 mW/cm² X 19 s and Plasma Emulation mode (PE) 3200 mW/cm² X 6 s, all them providing approximately 19 J/cm². For KHN measurement, samples (5mm X 1mm) were made (n=8) and submitted to the KHN test on the top surface under a load of 10g applied for 10 seconds. For the DTS (n=8), samples (5mm X 3mm) were made and submitted to a compressive load of 0.5 mm/s until fracture. The data obtained were submitted to the statistical analysis. For KHN, there was statistically significant effect for interactions cementation agent X ceramic thickness, where RL presented higher values of KHN than Z. Comparing inside of cementation agent, RL presented higher values of KHN when photoactivated under 1 mm of ceramic thickness, different from the others. For Z, there was no statistically significant difference between different thickness and the control. The interaction cementation agent X photoactivation protocol was also significant, where for all the photoactivation protocols RL presented higher values of KHN than Z. For the cementation agent there was no significant difference in the values of KHN in all the photoactivation protocols for RL. For Z, the higher values of KHN were obtained by XL protocol and the lower values by S. For DTS, there was significant effect for the interaction cementation agent X photoactivation protocol, where in the photoactivation protocol factor, RL presented higher DTS when photoactivated by XL protocol. For cementation agent factor, the higher values of DTS for RL were obtained with XL, and the lower ones with S. For Z, similar values of DTS were obtained among photoactivation protocols evaluated. It was possible conclude that ceramic thickness and the photoactivation protocols exerted influence on the mechanical properties of cementation agents evaluated.

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INTRODUCTION

The use of resin cements has increased considerably in recent years¹ due to the higher search for aesthetic restorations, specially ceramic restorations.^{2,3} Most commonly used materials are resin cements, and they may be chemically activated, physically by light or duals. The greatest advantage of photoactivated resin cements in relation to the chemical ones and duals is in the color stability and time of work.⁴ In order to achieve the advantages of photopolymerizable materials, and because they present low cost, low viscosity composite resins have been used as cementing, especially for cementation of ceramic laminates.⁵

Halogen and LED light-curing units (LCUs) have been used for photoactivation of resin materials. However, in order to occur an effective polymerization of photoactivatable resin cement is necessary that the light overtake the barrier of the indirect restoration and achieve the material with enough energy to stimulate the photoinitiators⁶ and, in this way, initiate the polymerization process. Indirect restoration provides absorption, refraction and reflection when the light focuses on it and these phenomena decreases the quantity of photons which will reach the resin material to be polymerized. The photons are responsible by the sensitization of the photo initiator, generally camphorquinone (CQ), raising their energy for the named triplet state.

Next, the CQ will react with tertiary amine (co-initiator), promoting the generation of free radicals which will be responsible by the conversion of monomers into polymers.⁷ For materials in which the activation is chemical or dual, the chemical activator used is the benzoyl peroxide; it will react with tertiary amine increasing the concentration of free radicals⁷ in areas where the light may enters in insufficient quantity. The effective polymerization of the material is a predominant factor for good mechanical properties presented by the resin material, what will favor a greater longevity for the restoration carried out. However, for materials with exclusive physical activation (photoactivated), the degree of conversion may be reduced, and a decrease in its properties could be observed.⁵

Superficial hardness is one of the most important physical characteristics in a material, and it is defined as the strength presented by the material to the indenter penetration. It is a test used for an indirect evaluation of the degree of conversion. Another test also used to study dental cements is the diametrical tensile strength, determined by the compressive strength of the material and that became popular because of its easy execution and reproducibility of results.⁸

Therefore, the objective of this work was to evaluate the microhardness and diametrical tensile strength of cementation

agents, photoactivated through ceramics restorations with different thickness.

MATERIAL AND METHODS

To perform this study, a low viscosity composite resin Filtek Z350 Flow and dual resin

cement Rely X ARC were used, both from 3M ESPE (Frame 1). An halogen light-curing unit XL 3000 (3M Espe) in Continuous mode (XL) and the third generation LED LCU Valo (Ultradent) in the Standard mode (S) and Plasma Emulation (PE) were used.

Frame 1: Composition of Cementing Agents used in this study.

Cementation Agent (Manufacturer) Color	Composition
Rely X ARC (3M Espe) A3	Paste A – Bis-GMA, TEGDMA, pigments, dimethacrylate polymer, amine and photo initiator. Load of Silica/Zirconia (68% in weight) Paste B – Bis-GMA, TEGDMA, dimethacrylate polymer, Benzoyl peroxide. Load of Silica/Zirconia (67% in weight)
Filtek Z350 Flow (3M Espe) A3	Bis-GMA, Bis-EMA, UDMA Nanoparticles of zirconia and silica and nanoagglomerated of zirconia/silica (65% in weight)

SAMPLES

Prepare of ceramic discs:

Cylindrical ceramic lithium disilicate discs in 5mm of diameter (IPS e.max Ivoclar-Vivadent, color A1), were confectioned through the lost wax technique in two different thickness, 1 and 2mm. The ceramic discs was basted with aluminum oxide (particles of 50µm) and next they were submitted to the bath with distilled water in ultrasonic vat to remove the residues.

Prepare of samples (n=8):

Cementation agent was inserted in a cylindrical cavity (5mm of diameter and 1mm of thickness) of a bipartite Teflon matrix. A polyester matrix strip was positioned on the material inserted, and on it was positioned the ceramic disc

(Figure 1). Photoactivation was performed positioning the light-cutting tip on the ceramic disc, following one of the protocols below: XL: 500 mW/cm² during 38 seconds (19 J/cm²); S: 1000 mW/cm² during 19 seconds (19 J/cm²); PE: 3200 mW/cm² during 2 cycles of 3 seconds (19.2 J/cm²).

For the control group, the confection of samples occurred in the same mode previously described; however, the photoactivation was carried out through a glass slide.

KNOOP MICROHARDNESS TEST

After the confection, the samples were removed from the Teflon matrix and they were taken to the microdurometer HMV Shimadzu for evaluation of Knoop microhardness. The measure of Knoop microhardness was performed on the top surface, in 5 points, and in order to perform it, a

load of 10g during 10 seconds was applied (Figure 2).

Figure 1: Illustrative scheme of the sample preparation.

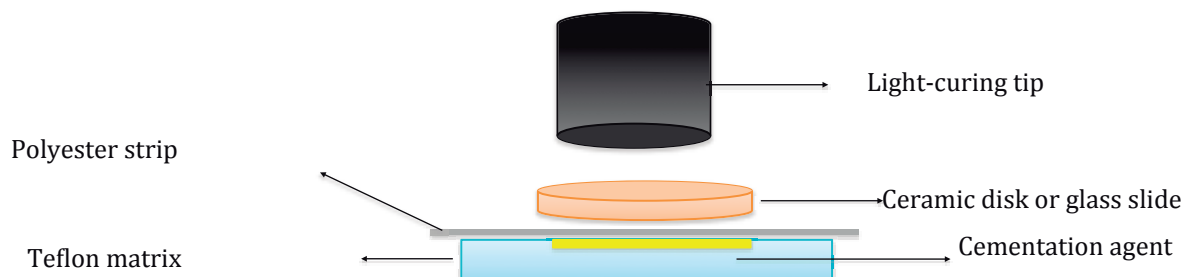
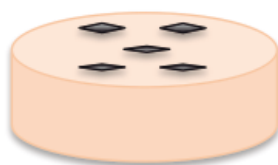


Figure 2: Illustrative scheme of indentations to study Knoop microhardness.



The average of the 5 indentations by surface was calculated and the value was transformed in the Knoop Hardness Number (KHN) using the following formula: $KHN = L/I^2 \cdot CP$, where L correspond the load applied, I to the greater penetration diagonal and CP to the constant of the projected area 14229.

DIAMETRICAL TENSILE STRENGTH TEST

The samples were confectioned in bipartite Teflon matrix containing a cylindrical cavity (5mm diameter and 3 mm thickness), according to the experimental groups and methods of prepare described before for microhardness test.

After the confection of samples, they were taken to the universal test machine Instron model 4411 (Instron Inc. Canton, MA, EUA) in a device to

the diametrical tensile strength test. The compressive tensile was applied in parallel to the diameter of the sample with constant speed of 0.5 mm/min until occur the fracture of the sample (Figure 3).

The values obtained were inserted in the formula: $R = 2L/\pi \cdot D \cdot h$, where R = resistance; L = Load, D = diameter, h = height.

RESULTS

Knoop microhardness:

After the exploratory analysis, it was observed normality of data and the Analysis of Variance was applied in factorial scheme 2 X 3 X 3 (cementation agent X ceramic thickness X photoactivation protocol) and Tukey Test.

There was significant effect for the interaction cementation agent X ceramic thickness, and for the interaction cementation agent X photoactivation protocol.

The results of the interaction cementation agent X ceramic thickness are presented in the Table 1, where the higher averages of Knoop microhardness were observed for dual resin

cement in each one of evaluated thickness. Inside the same cementation agent was observed that the dual resin cement presented higher average of microhardness when activated through 1mm of ceramic thickness. The low viscosity composite resin presented similar behavior for the three evaluated ceramics.

Figure 3: Diagram of diametrical tensile strength.

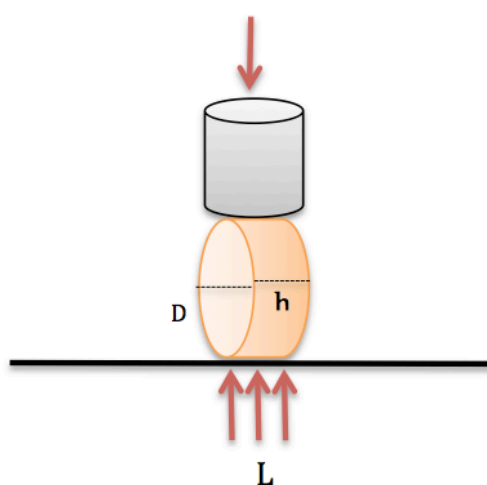


Table 1: Averages for Knoop Microhardness test.

Cementation agent	Thickness of ceramic		
	0 mm	1 mm	2 mm
Rely X ARC	22.01 aB	25.43 aA	22.34 aB
Filtek Z350 Flow	18.60 bA	17.27 bA	17.82 bA

Averages followed by distinct letters (capital letters in horizontal and lower case in vertical) are different between them ($p < 0.05$).

The results of interaction cementation agent X photoactivation protocol are presented in the Table 2, where is observed that in the same photoactivation protocol, higher microhardness values were obtained by dual resin cement. When isolated the cementation factor, it is observed that there was no

difference in the Knoop microhardness resulting from different photoactivation protocols. For the low viscosity composite resin was observed that the higher value of microhardness was obtained with the protocol XL and the lower average with the photoactivation protocol S; intermediate

values were obtained with protocol XL and the lower average with photoactivation protocol S, and the intermediate values were obtained for the photoactivation protocol PE, which was not different from others.

Diametrical tensile strength:

After the exploratory analysis, it was observed normality of data and the Analysis of Variance was applied in factorial scheme 2 X 3 X 3 (cementing agent X ceramic thickness X photoactivation protocol) and Tukey Test.

There was significant effect for the interaction cementation agent X photoactivation protocol. The results are presented in the Table 3.

For dual resin cement, the higher value of diametrical tensile strength was obtained

when protoactivated by the protocol XL, and lower by the protocol S; the protocol PE presented intermediate values without difference for the others. For the low viscosity composite resin there was no statistically significant difference for the values obtained for each photoactivation protocol.

When isolated the photoactivation protocol, it was observed for the protocol XL, the higher value of DTS was obtained for dual resin cement. For the protocols S and PE there was not statistically significant difference between the cementation agents.

The LCUs irradiance were measured through a portable radiometer with and without interposition of ceramic discs and of glass slide (control) used in this study (Frame 3).

Table 2: Average for Knoop microhardness test.

Cementation agent	Photoactivation protocol		
	XL	S	PE
Rely X ARC	22.90 aA	24.30 aA	22.58 aA
Filtek Z350 Flow	19.11 bA	16.67 bB	17.92 bAB

Averages followed by distinct letters (capital letters in horizontal and lower case in vertical) are different between them ($p < 0.05$).

Table 3: Average (MPa) for the diametric tensile strength test.

Cementation agent	Photoactivation protocol		
	XL	S	PE
Rely X ARC	92.73 aA	79.21 aB	81.80 aAB
Filtek Z350 Flow	75.16 bA	86.13 aA	82.93 aA

Averages followed by distinct letters (capital letters in horizontal and lower case in vertical) are different between them ($p < 0.05$).

Frame 3: Irradiance of LCUs used in this study.

Photoactivation protocol	Ceramic thickness	Irradiance (mW/cm ²)
XL	-	500
S	-	1000
PE	-	>1000* (3200**)
XL	1	120
S	1	210
PE	1	500
XL	2	70
S	2	100
PE	2	200
XL	Glass slide	320
S	Glass slide	560
PE	Glass slide	>1000

* Maximum value achieved by the radiometer used. ** According to the manufacturer's information.

DISCUSSION

Cementation in indirect restorations represents a step of great clinical importance, because cementation agents are responsible by the union between the indirect restorer material and the tooth structure.

In most cases, the choice of cementation agent has been by dual resin cements introduced in the market in order to conciliate favorable characteristics of self-polymerizable and photopolymerizable materials, obtaining better degrees of conversion.⁸ However, in clinical cases such cementation of extensive ceramic restorations, a significant reduction of the light intensity could occur. In these cases, the extension in which each polymerization will occur may vary, and may result in a material with reduced mechanic characteristics. On the other hand, the cementation of ceramic veneers requires that

their cementation is performed with a material which presents adhesion with the tooth structure and color stability.⁹ In this sense, low viscosity composite resins have been used because they are chemically more stable than dual and self-polymerable materials that present higher content of amines as initiator, causing color changes along the time.⁵

Microhardness test is very used because it is a simple and reliable method to indicate the degree of conversion of resin cements.

In this study, higher averages of Knoop microhardness were observed for dual resin cement in all the ceramic thickness evaluated (control, 1 and 2 mm), the results are shown in the Table 1. This finding may be justified by the dual activation (chemical and physical) of the polymerization reaction of the material. The dual activation allows the material achieves higher values of degree of conversion and

microhardness, and may result in better physical properties of the polymer formed. Beyond the degree of conversion and crosslinked density, the content of load particles also influences in the material hardness. Dual resin cement used in this study presents higher content of load particles (67 to 68% in weight) when compared to the low viscosity composite resin (65% in weight).

In the same cementation agent was observed that dual resin cement presented higher average of microhardness when photoactivated through 1mm of ceramic thickness. When the light overtook the ceramic disk of 1mm thickness presented a reduction of intensity in 70%, allowing a slower chemical polymerization, what provides an additional conversion. The control group that had as interposed material a glass slider of 2mm thickness presented light attenuation in only 20%, and higher quantity of light energy reaches the molecules of camphorquinone. It triggers the reaction of polymerization by physical activation, rapidly increasing the stiffness of the material, what does not allow that the chemical activation increase the degree of conversion of the material. In other words, this process rapidly decreases the molecular mobility, what reduces the possibility of reactions by chemical activation.

These factors reflected in the results of microhardness without statistically significant differences among the groups. For the low

viscosity composite resin was observed similar microhardness values among the two ceramic thicknesses evaluated and the control, what indicates that the quantity of photons that reaches the material resulted in a polymer with similar characteristics. The distance between the LCU and the resin material, as the color and the opacity of the ceramic result in decrease of the number of photons that effectively reaches the molecules of photoinitiator and consequently initiates the reactions of polymerization.

The results found by dismemberment of the interaction cementation agent X photoactivation protocol are described on the Table 2. For each photoactivation protocol, the values of microhardness of dual resin cement were higher than of low viscosity composite resin. It may be justified by the composition of each material, both for content of load particles (previously mentioned) and also by the interaction of load particles and the organic matrix.

For the factor cementation agent there was no statistically significant differences between the values of microhardness for dual resin cement, what indicates the benefits of chemical polymerization acting in a complimentary way to the physical polymerization. On the other hand, for the low viscosity composite resin, the higher values of microhardness were found when the material

was photoactivated by the protocol XL, and the lower ones by the protocol S. Notwithstanding, all the protocols have provided approximately the same density of energy (exposure time X irradiation), and statistical differences in the microhardness were observed. The photoactivation protocol XL used 500 mW/cm² during 38 seconds, and in this way probably resulted in a speed reaction of polymerization lower than the protocol S (1000 mW/cm² X 19s). Polymerization kinetics is a complex event, because a simple reciprocal relationship between irradiation and exposure time do not occur. In fact, the degree of conversion depends on the product of the exposure time elevated to 1 and of the irradiation elevated to 0.5-0.6.¹⁰ It means that, for a given density of energy, higher exposure time in low irradiation leads to higher degrees of conversion than lower exposure time to higher irradiation.¹¹ The results of the present study are according with this affirmation.

An alternative method to evaluate resin materials strength is the diametrical tensile strength, a test determined by a compressive load and known to be easy to execute and reproduce.⁷

The results of diametrical tensile strength test (Table 3) indicated a significant effect for the interaction cementation agent X photoactivation protocol. About the cementation agent factor, the behaviors found

for the low viscosity composite resin were similar between them. However, for dual resin cement, the higher values were obtained through the protocol XL, and the lower ones through the protocol S. Here it is possible use the same explanation previously provided and that it is based on the kinetic complexity of polymerization.

In the photoactivation protocol factor, there was statistically significant difference only for the protocol XL, where the higher values of DTS were found by dual resin cement. Higher time of irradiation used by the protocol XL allowed that the chemical polymerization of dual resin cement influenced positively in the mechanical properties of the material. Photoactivation protocol PE provided highest irradiation in short exposure time (3200 mW/cm² X 6s) could result in worst values of DTS when compared to the others. This possibility would follow the example of curing unit sources based on arc plasma (PACs), where high irradiation used in short time would result in poorly formed polymers, presenting deficient mechanical properties.¹² However, the protocol PE resulted in strength values similar both for XL and S; it is possible conclude that this protocol does not result in prejudice the material properties, both about DTS and microhardness. This fact is probably explained by the attenuation that occurs in light intensity when it crosses a material, due

to the phenomenon of absorption and scattering.

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