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# Evaluation of the Knoop Microhardness of Resin Cements Photoactivated by Different Modulations

## *Avaliação da microdureza knoop de cimentos resinosos fotoativados por diferentes modulações*

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## ABSTRACT

**Introduction:** With the innovation of techniques and evolution of dental materials, the clinical indication of low thickness ceramics can be made with greater predictability and confidence. Currently, high strength ceramics have been made with thicknesses between 0.3 and 0.5 mm, which are called ceramic laminates or dental contact lenses. **Objective:** To evaluate the knoop microhardness of the top surface of two resin cements: NX3 Light Cure (Kerr) and RelyX Veneer (3M ESPE), used for the cementation of contact lenses, polymerized by two different photoactivation methods (High and Soft). **Method:** A disk (0.3 mm thick and 8 mm diameter) made of IPS Empress e-max ceramic in color B1 was made. A polyester strip was placed on a glass plate and a 6mm diameter, 1mm thick Teflon matrix was obtained, obtaining 40 specimens (n = 10). **Results:** Regardless of the mode of photoactivation tested, the Relyx Venner cement had significantly higher microhardness values than the NX3 cement. Another result presented is that, independent of the resin cement tested, the high photoactivation mode obtained higher values in relation to the Soft mode. In all analyzes the significance level of 5% was considered. **Conclusion:** Regardless of the mode of photoactivation tested, the RelyX Venner cement had significantly higher microhardness values than the NX3 cement. In addition, regardless of the resin cements tested, the high photoactivation mode obtained higher microhardness values than the Soft-Start mode.

**Keywords:** Resin Cements; Hardness Tests; Ceramics

## RESUMO

**Introdução:** Com a inovação de técnicas e evolução dos materiais odontológicos, a indicação clínica das cerâmicas de baixa espessura pode ser feita com maior previsibilidade e confiança. Atualmente, cerâmicas de alta resistência tem sido confeccionadas com espessuras entre 0,3 e 0,5 mm, que são chamados de laminados cerâmicos ou lentes de contatos dentais. **Objetivo:** Avaliar a microdureza knoop da superfície de topo de dois cimentos resinosos: NX3 Light Cure(Kerr) e RelyX Veneer (3M ESPE), utilizados para a cimentação de lentes de contato, polimerizados por dois diferentes métodos de fotoativação.(High e soft). **Método:** Foi confeccionado um disco (0,3mm de espessura e 8 mm de diâmetro) de cerâmica IPS Empress e-max na cor B1. Sobre uma placa de vidro, foi colocada uma tira de poliéster e, em seguida, foi posicionada uma matriz cilíndrica de teflon com 6mm de diâmetro e 1mm de espessura, obtendo 40 corpos de prova (n=10). **Resultados:** Independente do modo de fotoativação testado, o cimento Relyx Venner apresentou valores de microdureza significativamente maiores em relação ao cimento NX3. Outro resultado apresentado é que independente do cimento resinoso testado, o modo de fotoativação High obteve valores maiores em relação ao modo Soft. Em todas as análises foi considerado o nível de significância de 5%. **Conclusão:** Independente do modo de fotoativação testado, o cimento RelyX Venner apresentou valores de microdureza significativamente maiores em relação ao cimento NX3. Além disso, independente do cimento resinoso testado, o modo de fotoativação High obteve valores de microdureza maiores em relação ao modo Soft-Start.

**Palavras-chave:** Cimentos de Resina; Testes de Dureza; Cerâmica

## INTRODUCTION

With the evolution of materials and techniques in dentistry, as well as the advent of adhesive retention, more conservative preparations began to be performed, with minimal tooth wear, and in some cases it is even possible non-preparation[1].

The clinical indication of the low thickness ceramics can be made with greater predictability and confidence, due to the improvement of the mechanical properties, biological behavior coupled with the optical and aesthetic properties. Among the indications, we highlight the presence of anterior diastema and teeth with linguoversion, correction of small imperfections as discrete alterations of position, color, shape and length. They may be effective for masking class III, IV or V restorations[1,2,3].

Photopolymerizable cements are used for the cementation of facets and contact lenses, in which chemical polymerization is not a prime factor. However, the depth of effectiveness has decreased with the increase of the fouling thickness caused by light attenuation that has limited its use for laminated facets[4]. The main barrier of photoactivated cements consists in the dependence of the amount of photons that reaches it for excitation of the photoinitiator, which is usually camphorquinone. In addition, the activation is also dependent on the wavelength of the light reaching the material, the power by the irradiance of the photopolymerizer[5]. The nearer the absorption peak is the light emitted, the more effective the effect will be on this photoinitiator. However, as light passes through thicker ceramic restorations, less translucent and darker colors, a decrease in the incidence of light may occur and this would cause significant loss of part of the light that can be absorbed, dispersed or reflected and, consequently, attenuated, thereby compromising polymerization of the cement[6].

The new concepts of photoactivation, together with the different types of devices present in the market, allow the dentist numerous forms of procedures in relation to the photopolymerization of the materials. However, knowledge of the advantages and disadvantages of photoactivation methods and systems is essential, such as their influence on the marginal sealing of restorations, as well as the maintenance of the mechanical properties of restorative materials[7]. In order to attenuate the generation of stresses during the polymerization process, some studies have used different photoactivation approaches, such as low intensity in continuous light, high intensity in continuous light, photoactivation in steps (Soft-Start) and late pulse (Pulse-delay). The main objective of these methods is to prolong the pre-gel phase of the dental composites, allowing a longer time for the monomer flow and, consequently, a decrease in the tensions generated by the polymerization contraction[8,9].

Photopolymerization is characterized by the transformation of the monomer molecules into polymer molecules. In order to perform such a procedure with a high efficiency standard, new photoactivation concepts were created, together with the different apparatuses present in the market, making it imperative to know the advantages and disadvantages of each photopolymerization method, as well as the best method of photopolymerization, according to the materials that will be photoactivated[10,11]. In order to reduce the degree of tensions and possible formations of polymer chains considerably weak, studies have proposed different methods of photoactivation, as follows: continuous low intensity light (Low), continuous high intensity light (High), photoactivation in steps (Soft-Start) and late pulse (Pulse delay)[12].

In particular, the action of the Soft-Start method consists in extending the pre-gel phase of the dental composites allowing a longer flow time of the monomers. This technique will promote better marginal adaptation of the restoration, due to the relaxation of the tensions occurred in the composite during the initial polymerization phase. However, in this case, the rate of exposure to light is decreased, which may interfere with the degree of polymerization of the composite and, consequently, affect the hardness of the material, unlike the High method, where monomers become polymers uniformly and thus create more resistant polymer chains[12,13,14].

It is important to emphasize that the composites are influenced not only by the quality of photoactivating light but also by the type of material used, including resin cement composition and inorganic material content, which is responsible for the longevity of the material in the buccal cavity[15,16].

As photoactivation modulation and microhardness test are very controversial parameters in the literature, the aim of this study was to assess the Knoop microhardness of the top surface of two resin cements used for the cementation of contact lenses, polymerized by two different methods of photoactivation.

## MATERIAL AND METHODS

### Experimental Design

The factors under study were NX3 Nexus third generation light-cure resin cement (Kerr) and RelyX Veneer (3M ESPE), both in the Translucent color and the High and Soft Star photoactivation modes of the BluePhase Polywave (Ivoclar Vivadent). The response variable was the Knoop microhardness values on the top surface of each specimen.

### Preparation of test specimens

Prior to the preparation of the specimens, the dental laboratory was asked to prepare a disk (0.3 mm thick and 8 mm diameter) of IPS Empress e-max ceramic (lithium disilicate). First, the dental technician made a model of the disc by the lost-wax technique, followed by inclusion in a ring for coating and then placed in a kiln for coating processing, to be then taken to the oven for Emax injection. The chosen color of the ceramic color tablet was B1.

The specimens were made by a single operator, according to ISO 4049 specifications. All specimens were prepared under the same conditions of temperature, lighting and relative air humidity and then divided into four groups (n=10), taking into account the factors under study, which are the two trade marks of the cements and photoactivation modes (soft-start and high).

A polyester strip was placed on a glass plate and then a cylindrical Teflon matrix of 6mm diameter and 1mm thick was placed. The cement was inserted in a single increment in the matrix and on the set was placed another strip of polyester (Dentisply, Petrópolis, Rio de Janeiro, Brazil), under digital pressure, so that a material compression occurred and the excess could be extravasated.

After this step, the contact lens of 8mm diameter and 0.3mm thick was placed on the second polyester strip and photoactivated for 20s using the light curing device directly on the contact lens, according to the photoactivation method proposed.

The use of the polyester matrix was to allow that there was no adhesion of the ceramic to the resin cement, since the intention was to assess the cement property and not the quality of the adhesion of the cement to the ceramic.

After polymerization, the specimens were removed from the matrix and then stored in dark containers, so that no external light interference could alter the quality of the photoactivation until the tests were performed. The intensity of light in the two modes of photoactivation was measured through a digital radiometer (Hilux Led Max Curing Light Meter).

### Knoop Microhardness Test (KNH)

The protocol used was based on the study by Borges et al., 2010[17]. The evaluation of the KHN measurement was performed on the top surface of each specimen through the apparatus (HMV-2T E, Shimadzu Corporation, Tokyo, Japan). Five Knoop indentations were performed on the surface of all specimens: one central and the other four in the distance of approximately 200**µ**m from the central location, under load of 0.5Kg per 10s. The values of the five indentations for each surface were recorded and calculated the final mean of the surfaces of all the experimental units.

## RESULTS

### Statistical analysis

After the exploratory analysis of the data, analysis of variance (ANOVA) was applied in a factorial scheme (2X2) and multiple comparisons were performed by the Tukey test. In all analyzes the significance level of 5% was considered.

### Knoop microhardness

Table 1 presents the means and standard deviations of the Knoop microhardness values of the resin cements tested and **the** photoactivation modes used in the study. In this case, it was observed that interaction and statistical difference between the variables (photoactivation modes and resin cements) were found (p=0.03). Based on the results, it was observed that, regardless of the photoactivation mode tested, the RelyX Venner cement presented significantly higher microhardness values than the NX3 Light Cure cement. Another result presented in this table is that, independent of the resin cement tested, the High photoactivation mode obtained higher values of microhardness than the Soft-Start mode.

## DISCUSSION

The results of this study showed that, regardless of the mode of photoactivation tested, the RelyX Venner resin cement obtained better performance in relation to the NX3 cement. Therefore, because it is two photopolymerizable resin cements, a possible justification for the superior microhardness of RelyX Veneer cement can be explained by its chemical composition.

When analyzing the composition of the RelyX Veneer cement, this cement is composed of Bis-GMA monomers (bisphenol-A glycidyl methacrylate) and TEGDMA (triethylene glycol dimethacrylate) with zirconia and silica particles[18]. The presence of TEGDMA reduces cement viscosity significantly increasing the degree of conversion to the polymer. Bis-GMA is capable of promoting strong hydrogen bonding through the hydroxyl group, increasing molecular weight, providing little mobility and high stiffness. There is also a synergistic effect between Bis-GMA and TEGDMA which increases the cross--linking density, that is, crosslinks between linear molecules producing three-dimensional polymers with high molecular mass[19]. In this case, microhardness is an indirect measure of the degree of conversion of monomers into polymers, thus, it can be said that the higher presence of BIS-GMA and TEGDMA provides this cement a higher values of Knoop microhardness, independent of the photoactivation mode. On the other hand, the NX3 Light Cure cement has monomers of uncured methacrylate esters, inert mineral fillers, activators and stabilizers[20]. The manufacturer does not specify the methacrylate types and does not report the presence of Bis-GMA or TEGDMA, being this a possible hypothesis to justify the lower microhardness values of this cement.

Another factor studied in this study and that deserves to be understood is the difference that occurred for the two resin cements when photoactivated in different ways· The results showed that,

**Table 1.** Knoop microhardness (standard deviation) depending on the type of resin cement and the mode of photoactivation.

| Photoactivation Mode | Resin Cement | |
|---|---|---|
| | NX3 Light Cure | RelyX Veneer |
| Soft-Start | 58.32 (1.4) Aa | 60.45 (2.3) Ba |
| High | 63.23 (1.3) Ab | 67.98 (2.0) Bb |

Means followed by distinct letters (horizontal uppercase and vertical lowercase comparing distance within each surface) differ from each other (p≤0.05).

regardless of the cement tested, the High Power photoactivation mode showed higher Knoop microhardness values than the Soft-Start mode (gradual increase of light intensity with a reduced contraction voltage and lower temperature). Possibly, this can be explained because in the Soft-Start method the apparatus performs initial cycle of 5 seconds with low irradiance light (average of 601 mW0/cm$^2$) and then reaches a higher irradiance peak (average of 1079 mW/cm$^2$) for 15 seconds. Consequently, when the light was switched on, the apparatus did not reach the maximum intensity of light, the low intensity remaining at a first instant, and only reaching its maximum intensity shortly before the end of the cycle. Thus, the activating effectiveness of photopolymerization occurs only in the repetition of cycles, although the sum of the repeated intensity of the cycles is insufficient compared to the mode of activation of continuous light (High). Therefore, the specimens subjected to the Soft-Start method were subjected to lower light energy rates, causing a lower microhardness[14].

## CONCLUSION

From the results found in this study we can conclude that:

- Regardless of the photoactivation mode, the RelyX Veneer resin cement presented higher Knoop microhardness when compared to the NX3 Light Cure cement.
- High photoactivation mode presented higher Knoop microhardness when compared to Soft-Start mode, independent of resin cement.

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