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# INFLUENCE OF CERAMIC THICKNESS ON MECHANICAL PROPERTIES OF DUAL LUTING AGENTS

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## Abstract

**Aim:** The influence of the ceramic thickness (0.7 mm, 1.2 mm and 2.0 mm) in the Union resistance to the Microshear and the elasticity module of two cements Resin (RelyX ARC and RelyX U200), by means of the Knoop hardness test.
**Materials and methods:** 36 Ceramic discs reinforced by di-silicate Lithium diameter 12 mm Were Prepared and separated in 2 Groups (n = 18) according to resin cements. Each group of the resinous cement was subdivided into 3 groups (n = 6) According to the thickness thereof (0,7 mm, 1.2 mm and 2.0 mm). The Union resistance to the Microshear was performed. The Union resistance values were calculated in MPa. For each combination thickness of ceramic-resin cement, 6 specimens were tested and the average values of the three cylinders were recorded as values of union resistance to the microshear for each specimen. Then the specimens were subjected to the Knoop hardness test later, the resin cement elasticity modulus was obtained by means of the hardness measurements Knoop. The data were submitted Variance analysis of two factors then to the test of Tukey.
**Results:** For union resistance the Relyx ARC cement when used with ceramics of 1, 2mm thick obtained the lowest value of union resistance. All other cement groups with their due ceramic thickness were statistically similar to each other and to the three groups previously mentioned. For the elasticity module no difference was found between the ceramic thicknesses. The difference was only given between the cements, so that the RelyX ARC presented The highest values were statistically different from RelyX U100. The same characteristic of results were Found for Knoop hardness. In the analysis of the fracture pattern of specimens In the RelyX ARC groups And RelyX U100, showed PREdominantemente fractures.
**Conclusions** PResistance of Union the group that presented the smallest result was the RelyX ARC photoactivated cement through the ceramic disc of 1,2mm. For the modulus of elasticity and hardness Knoop the groups of behaved similarly differing statistically only for the type of cement, being the RelyX ARC Different from RelyX U200.

**Keywords:** dental luting agente, hardness Knoop, resin cement

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## INTRODUCTION

Indirect restorations in ceramics have advantages over direct restorations in composite resin as; Increased resistance to wear, lower susceptibility to pigmentation and better ability to mimic dental enamel [1]. The union between the ceramic restorations and the dental structure can be obtained by the association of ceramic surface treatments and the use of resin cements [2, 3, 4].

In turn, the success of ceramic restorations depends largely on the type of resinous cement used, to ensure effective union between restorative material and dental structure, providing good marginal adaptation [5]. Ceramic restorations allow bonding to the dental substrate through adhesive cementation, using silane, adhesive system and resin cement [6]. Resin cements can be classified according to the type of conditioning: Total conditioning, adhesive and autoconditioning. Resin cements that employ total conditioning require the use of phosphoric acid followed by multiple or 2-step adhesives before the use of resin cement. Resin Cements Auto Adhesives are able to attach to the dental tissues without the prior need of the use of a adhesive. Already the auto cements Conditioners initially use a Primer Resin acid, which is not washed immediately, for the purpose of modifying the surface of the dental tissue before the fixation procedure [7.8].

On the other hand, resin cements can be classified according to the activation mode: photoactivated, chemically activated and dual-activation. Resinous cements activated only by light exposure offer advantages such as longer working time and better color stability and chemically activated resin cements present the advantage of being used for cementation in places where the poly lightMerizadora cannot have access [6]. Thus, when the photoactivation of the resinous cement is carried out indirectly, some aspects must be taken into consideration: as the thickness of the indirect restorative material increases, the absorption and dispersion of the light increases, reducing the amount of Energy supplied by the Photoactivator device on the resin cement 912. Some studies have shown that there is an attenuator effect proportional to the thickness of the ceramic and the opacity of the indirect restorative material by reducing the mechanical properties of the resin cements and may compromise the union between the cement resInoso and the restorative material [13]. In this way, it is important to optimize the methods of Photoactivation of resin cements for the purpose of improving the clinical performance of these materials, because greater conversion of monomers is indispensable for the best performance of these materials 14, because the inadequate polymerization of the resin cement may be associated with lower mechanical property, high water absorption and solubility, in addition to the color instability [6].

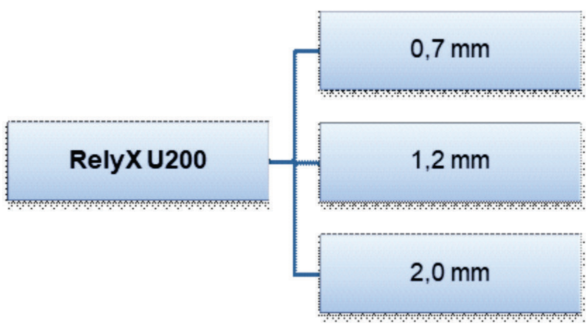
However, DúLives remain on the effectiveness of indirect photoactivation in the mechanical properties of Photoresin cements photoactivated through different thicknesses of the ceramics. Therefore, the objective of this study was to aThe influence of the ceramic thickness (0.7 mm, 1.2 mm and 2.0 mm) in the Union resistance to the Microshear and the elasticity module of two cements Resin (RelyX ARC and RelyX U200), by means of the Knoop hardness test.

## MATERIALS AND METHODS

### PREPARATION OF SPECIMENS FOR THE UNION RESISTANCE TEST

Double-resin cements were used, being a self adhesive, RelyX U200 (3m Espe, St. Paul, MN, USA) and two conventional, RelyX ARC (3m Espe, St. Paul, MN, USA).

36 ceramic discs reinforced by lithium di-silicate with 12 mm diameter (IPS E. Max Press, Ivoclar-Vivadent, Schaan, Liechtestein) were Prepared and separated in 2 Groups (n = 18) according to resin cements. Each group of the resinous cement was subdivided into 3 groups (n = 6) According to the thickness thereof (0,7 mm, 1.2 mm and 2.0 mm), Figure 1 and 2.



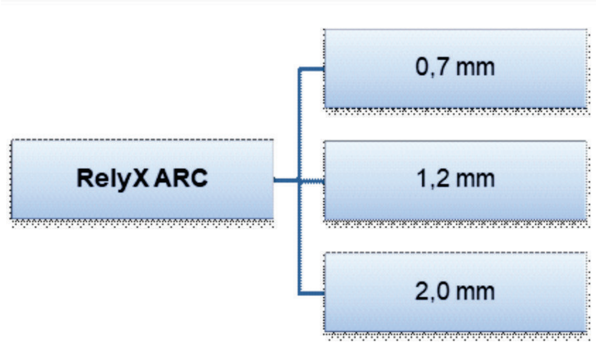



Figure 1 – Division of resin cement groups by thickness of ceramics.

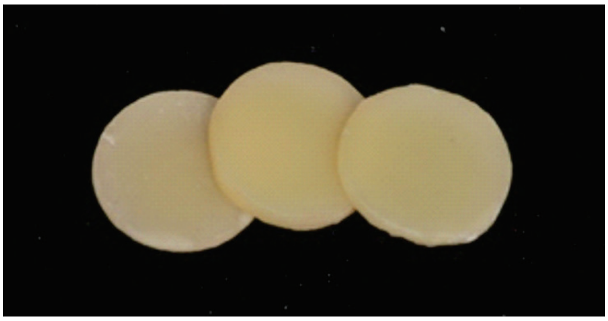


Figure 2 -Ceramic discs.

After the preparation, the surface of the ceramic discs was conditioned by 20s, using hydrofluoric acid gel 10% (Dentsply), following recommendations from the manufacturer. The surface conditioning of the disc was carried out as follows: The gel was applied and distributed over the surface of the ceramic disc using the tip of theRinga containing acid (Figure 3). After application, the discs were washed with water/ air jet by 60 s, cleaned in ultrasound for 5 min in distilled water followed by air-jet drying for 30s. After cleaning and drying, the active application of a layer of the Ceramic Primer (3m ESPE) was made using Microbrush for 20s and, after 1 min, the surface was dry With air jet per 30s (Figure 4).

Figure 3 – Application of the hydrofluoric acid gel 10% (Dentsply) on the ceramic disc, which is wrapped by a silicone matrix so that the acid does not reach the opposite side of the disc.

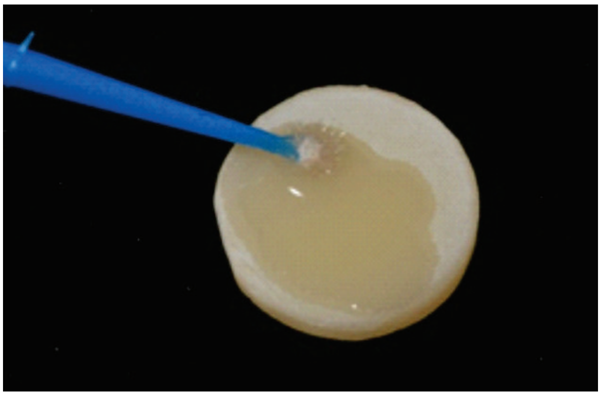


Figure 4 – Active application of the Ceramic Primer Signaling Agent (3m ESPE) in Ceramic disc.

After preparation of the discs, the resin cements were manipulated according to the recommendations of each manufacturer and applied within 3 Tygon tubes with 0.7 mm diameter by 0.5 mm positioned on the ceramic discs covered with A polyester strip (Figure 5). After removal of the excess of the cementing agent using Microbrush, each ceramic disc was photoactivated by the opposite side, by 40s with the apparatus Bluephase G2 (Ivoclar-Vivadent, Schaan, Liechtestein), with irradiance of 1000 mW/cm2 (Figure 6). 50 and fourth specimens were obtained for each resinous cement. After the preparation, the samples were stored in humidity relative to 37 º C for 24 hours, in a greenhouse.

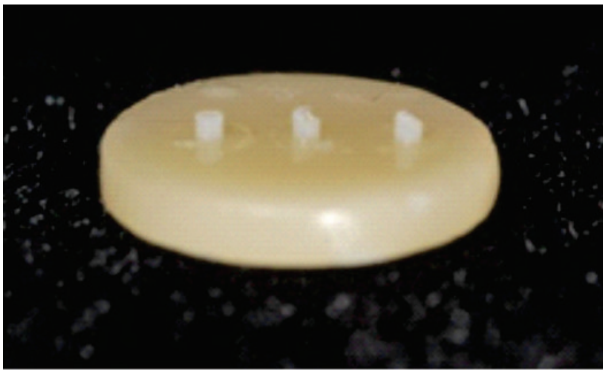


Figure 5 – Resin cements were manipulated and positioned on the discs of Ceramics, by means of Tygon tubes.

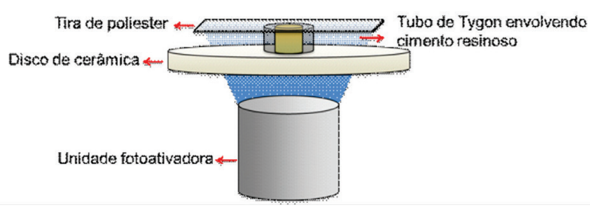



Figure 6 – schematic design of the photoactivation stage of the resinous cement specimens through the ceramic disc.

After storage, the Tygon tubes were carefully removed with knife blade aid. Then the opposite side of the ceramic discs were fixed on the test device (Bencor-Multi-T, Danville engineering Co, San Ramon, CA, USA), with Cyanoacrylate-based adhesive (Superbonder, Loctite, Itapeví, SP), which will be engaged in the test machine Universal. Then a stainless steel wire with 0.2 mm diameter was placed round the resinous cement cylinder and aligned with the Union interface. The Union resistance to the microshear was performed at the speed of 0.5 mm/min until the fault occurred (Figure 7). The Union resistance values were calculated in MPa. For each combination thickness of ceramic-resin cement, 6 specimens were tested and The average values of the three cylinders were recorded as values of union resistance to the microshear for each specimen.

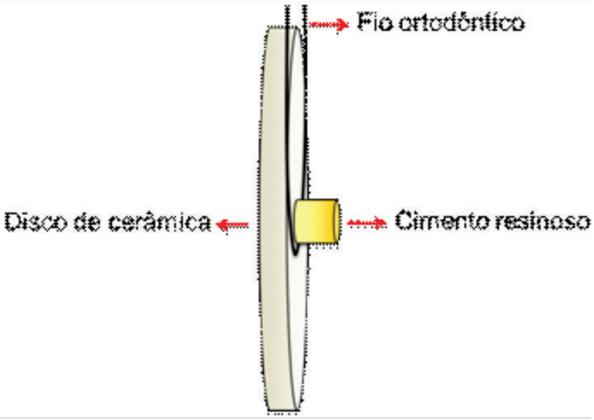



Figure 7 – schematic design of the positioning of the resinous cement specimen adhered to the ceramic disc for the microshear test.

## ANALYSIS OF MECHANICAL PROPERTIES (HARDNESS AND MODULUS OF ELASTICITY)

Tests were carried out to verify the mechanical properties of the resin cements according to the photoactivation through the different ceramic thicknesses. For this, the cements resin Rely U100, RelyX ARC and Variolink II, were manipulated according to the recommendations of each manufacturer and inserted in holes in a polycarbonate matrix with 2.0 mm diameter by 2.0 mm thick and the photoactivation was performed Through the ceramic discs, separated by polyester strip and glass coverslipping (n = 5).

The specimens were then subjected to the test of Knoop hardness in the apparatus HMV – 2 (Shimadzu, Tokyo, Japan) (Figure 08), calibrated for load of 200 GF, acting for 20 seconds. Ten penetrations were made in each specimen, for each type of resin cement and ceramic thickness, totaling 90 measurements. Subsequently, the elasticity modulus of the resin cements was obtained by means of the Knoop hardness measurements using the following formula: KHN = 0.45 x E2/(0.140647-D/d) x 100, where and is the modulus of elasticity, D is the diagonal minor and D is the largest diagonal of the tip Penetrating specimens. After that, the data was tabulated and submitted to statistical analysis.

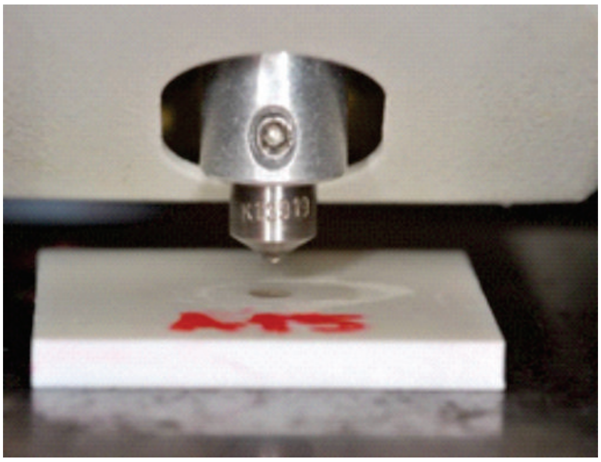



Figure 08 – Specimens subjected to the test of Knoop hardness in the apparatus HMV – 2.

## STATISTICAL ANALYSIS

The data obtained in the Union resistance test were subjected to the Kruskal Wallis test and Tukey for Check Differences Between The Groups Tested, In significance level 5%. Already in the analysis of the mechanical properties (hardness and modulus of elasticity). Analysis of variance (ANOVA) of two factors, then the test of Tukey to verify differences between the groups tested, in significance level 5%. All tests were carried out in the SigmaStat 3.5 program.

## ANALYSIS OF THE FRACTURE PATTERN OF SPECIMENS SUBJECTED TO THE MICROSHEAR TEST

After the procedures for fixation and dehydration of specimens, they were fixed in

Stub Aluminium with double-sided carbon tape aid (Electron Microscopy Sciences, Washington, DC, USA). Then all specimens received metallic cover with a layer of gold/Palladium in metallized (Denton vacuum Desk II sputtering, Denton vacuum, Cherry Hill, NJ, USA). The Stub Containing the specimens was positioned in the electronic high-vacuum scanning microscope (LEO 435 VP; LEO Electron Microscopy Ltd., Cambridge, UK) with 15 KV voltage acceleration, 15 mm working distance and Spotize Ranging from 25 PA to 100 PA, for evaluation of surface topography after Microshear test. The fault modes were analyzed in Measurement Microscope (STM – Olympus Optical Co. LTDA, Japan), With 40 X increase, and fault modes will be classified as follows: Adhesive failure (mode 1), cohesive failure in resinous cement (Mode 2) and mixed failure involving resin and ceramic cement (mode 3).

## RESULTS

The average and standard deviations of union resistance, modulus of elasticity and Knoop hardness are described in tables 1, 2 and 3 RespeCtivamente. For the resistance of the Relyx ARC cement when used with ceramics of 1, 2mm thick obtained the lowest value of union resistance. All other cement groups with their due ceramic thickness were statistically similar to each other and to the three groups previously mentioned.

For the elasticity module no difference was found between the ceramic thicknesses. The difference was only given between the cements, so that the RelyX ARC presented The highest values were statistically different from RelyX U100. The same characteristic of results were found for Knoop hardness as can be seen in table 3.

In the analysis of the fracture pattern of specimens subjected to the microshear test, all evidence bodies Tested in the RelyX ARC groups And RelyX U100, showed predominantly mixed fractures (mode 3), presenting both cohesive in cement and adhesive in the ceramic-cement interface.

Table 1-Union resistance.

| Groups | Union Resistance (MPA) |
|---|---|
| ARC 0,7 | 27,60 ± 11,14 a |
| ARC 1,2 | 16,91 ± 6,40 b |
| ARC 2,0 | 23,57 ± 7,09 a |
| U200 0,7 | 21,01 ± 3,70 a |
| U200 1,2 | 20,88 ± 4,01 ab |
| U200 2,0 | 27,69 ± 1,86 a |

Table 2-Modulus of elasticity.

| | Ceramic thicknesses | | |
|---|---|---|---|
| **Cements** | **0,7mm A** | **1,2mm A** | **2,0mm A** |
| **Relyx ARC a** | 14,33±4,60 | 16,25±3,02 | 11,81±1,05 |
| **Relyx U200 b** | 8,20±0,76 | 7,68±1,25 | 7,11±1,54 |

Table 3-Hardness Knoop.

| | Ceramic thicknesses | | |
|---|---|---|---|
| **Cements** | **0,7mm A** | **1,2mm A** | **2,0mm A** |
| **Relyx ARC a** | 75,16±3,01 | 72,64±3,50 | 70,74±2,64 |
| **Relyx U200 b** | 59,20±6,55 | 54,74±8,67 | 50,86±2,37 |

## DISCUSSION

Among the cementing agents currently available for cementation of indirect restorations, resin cements can be considered as options due to their physical properties. The type of physical and chemical polymerization (dual) is commonly found in current commercial formulations. However, there are several clinical situations where it is not possible to use the photoactivation. In these cases, the chemical polymerization process is necessary for a minimum conversionBe assured [15].

Resin cements, used for cementing crowns, onlays and inlays, cannot depend solely on photoactivation to achieve maximum mechanical properties, due to the fact that the intensity of light that reaches the cement layer is or totally eliminated due to the distance between the layer of the Cementing agent and the source of light or by the absorption characteristics of indirect restorative materials onjacentes [15, 16]. Therefore, the mechanical properties of composite resin-based cementation agents have been evaluated, with photoactivation through ceramic bulkheads or indirect composite resin, to make the search conditions as close to the practice Clinic. The objective of this study was to aThe influence of the ceramic thickness (0.7 mm, 1.2 mm and 2.0 mm) in the Union resistance to the Microshear and the elasticity module of two resin cements (RelyX ARC and RelyX U100), by means of the Knoop hardness test.

The clinical success of the ceramic restorations depends on the quality and durability of the union established between the ceramics and the resinous cement. The Union of the resinous cement to the ceramics is controlled primarily by the treatment of the surface of the ceramic, the ceramics with the highest concentration of silica more susceptible to silanization and the surface conditioning with acid HidrofluorídRico [17] In addition to grade Dand conversion of resin cement [18]. After the treatment of the surface of the ceramic, the resin cement applied penetrates the microretenções of the surface and the polymerization of this material is responsible for the mechanical implantation and ConseqüeNTE retention [19]. However, when polymerization is insufficient it can cause a decrease in restoration longevity, among other factors (Della Bona & Van Noort, 1995). In the present study, there was no statistical difference in the results of microshear, except for the group RelyX ARC 1.2, which presented the lowest value of union resistance, this, may be because the chemical and physical phases were not effective in Polymerization of this cement with this thickness.

The Knoop microhardness test is capable of detecting differences between polymeric chains, which are not detected by the evaluation of the degree of conversion (Price et al., 2004). The smallest microhardness value can be synonymous with an incomplete polymerization of the CompósitThe resin for cementation [20].

El-Mowafy, Rubo & el-Codrawy in 1999, evaluated the hardness of dual resin cements with the interposition of ceramic inlays with thickness from 1 to 6mm and observed significant reduction of microhardness with the interposition of ceramics with thickness of 3mm or More 14. These same authors, They showed that composite resin inlays/onlays with thickness greater than 4mm decreased the hardness of the dual cements by 50% or more.

The elasticity modulus (E), it exerts great importance on mechanical properties, since it represents the inherent rigidity of a material within the elastic phase, besides having a linear relationship between the tension and DTraining [21]. In this way, the efficient methods for evaluating E Of these materials becomes very important. Different mechanical tests can be used to measure the modulus of elasticity of resin materials, such as nanoindentation 22MiCrodureza [23], and three-or four-point flexion tests [24]. Measuring the dimensions of the short and long diagonals obtained with the Knoop indentation test (KH), the elasticity modulus (GPa), can be determined by an empirical relationship, resulting in a simple method and Low cost, in this sense, [25 and 26]. Due to this range of tests, it is not possible to compare results with other jobs.

Therefore, in the present study, both in the values of microhardness, as in the values of the modulus of elasticity, there was no statistical difference between the different ceramic thicknesses (0.7; 1.2 and 2.0), however, the Difference was only given between the cements, so That the RelyX ARC presented The highest values and were statistically different from the RelyX U100, although all are resin cements of dual polymerization, there is difference in the composition of the organic matrix and quantity/type of content INorgânico [27].

## CONCLUSION

From the results obtained in this study, it was possible to conclude that for union resistance the group that presented the lowest result was that of the RelyX ARC photoactivated cement through the ceramic disc of 1, 2mm. For the modulus of elasticity and hardness Knoop the groups of behaved similarly differing statistically only for the type of cement, being the RelyX ARC Different of RelyX U100.

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