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## Laboratory Research

# Influence of Different Ceramics on Resin Cement Knoop Hardness Number

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### Clinical Relevance

Polymerization of a dual resin cement is significantly affected by type of ceramic, activation modes and post-activation times.

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### SUMMARY

**This study evaluated: 1) the effect of different ceramics on light attenuation that could affect microhardness, measured as the Knoop Hardness Number (KHN), of a resin cement immediately and 24 hours after polymerization and 2) the effect of different activation modes (direct light-activation, light activation through ceramics and chemical activation) on the KHN of a resin cement.**

**Resin cement Rely X ARC (3M ESPE) specimens 5.0 mm in diameter and 1.0 mm thick were made in a Teflon mold covered with a polyester film. The cement was directly light activated for 40 seconds with an XL 2500 curing unit (3M ESPE) with 650 mW/cm², light activated through ceramic discs of Duceram Plus (DeguDent), Cergogold (DeguDent), IPS Empress (Ivoclar), IPS Empress 2 (Ivoclar), Procera (NobelBiocare), In Ceram Alumina (Vita) and Cercon (DeguDent), having a 1.2 mm thickness or chemically activated without light application. The resin cement speci-**

**mens were flattened, and KHN was obtained using an HMV 2 microhardness tester (Shimadzu) with a load of 50 g applied for 15 seconds 100 µm from the irradiated surface immediately and after storage at 37°C for 24 hours. Ten measurements were made for each specimen, with three specimens for each group at each time. The data were submitted to ANOVA and Tukey's test (*p*=0.05). The KHN of the resin cement was not only affected by the mode of activation, but also by the post-activation testing time. The mean KHN of the resin cement for chemical activation and through all ceramics showed statistically significant lower values compared to direct activation immediately and at 24 hours. The KHN for 24 hours post-activation was always superior to the immediate post-activation test except with direct activation. The most opaque ceramics resulted in the lowest KHN values.**

## INTRODUCTION

The materials used in this study are described in Table 1. The methods of preparing the ceramic specimens are explained by material.

Improvements in adhesive bonding techniques and all-ceramic systems have contributed significantly to esthetic dentistry.[1-2] The continuous development of ceramic materials and refinements in fabrication techniques have been instrumental in overcoming some of the problems seen in earlier ceramic materials. The use of ceramic materials has increased exponentially, because of their lifelike appearance, fluorescence, biocompatibility, durability, chemical stability, high compressive resistance and their thermal expansion being similar to tooth structure. However, brittleness and susceptibility to fracture are major disadvantages to their use.[3]

Luting materials are of vital importance to the longevity of dental restorative materials.[4] Some all-ceramic restorations may be cemented with zinc phosphate, glass ionomer or resin composite cements, and the success of the cementation may depend on the mechanical properties of the cement.[5] When zinc phosphate or glass ionomer cements are used, adequate retention of the preparation is necessary. When this is compromised, adhesive luting systems are recommended. Dual-cure adhesive luting cements have shown higher bond strength values than chemically-activated adhesive luting cements, perhaps because the latter have fewer initiators and/or more inhibitors of polymerization to extend the working time.[6]

Furthermore, the degree of conversion of cement affects hardness, wear resistance, water absorption, residual monomer and biocompatibility of adhesive luting resin cements and these properties affect the survival of the restoration.[7] Dual cure resin cements have the advantage of both chemical-cure and light-cure materials.[8-11] Even though the materials are dual cured, an adequate quantity of light is required to initiate the polymerization process.[12] The composition, thickness, opacity and shade of the ceramic material may attenuate light from the curing unit that is used to polymerize the resin cement under the ceramic restoration.[11]

Many different ceramics are available on the market. They have dissimilar compositions and crystal content, which could impact the quantity of light that passes through them for activation of the resin-luting cement. Since more crystalline ceramics are opaque, it could be expected that they would attenuate more light. Among the current reinforced ceramics that are on the market, In-Ceram Alumina and In-Ceram Zirconia (Vita Zahnfabrik, Seefeld, Germany), IPS Empress and IPS Empress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein), Cergogold (Degussa Dental, Hanau, Germany), Procera (Nobel BioCare, Gothenburg, Sweden) and Cercon (Degussa Dental, Hanau, Germany) are commonly used.

In-Ceram Alumina is an aluminous ceramic with 82% by weight alumina infiltrated by glass.[13] It is indicated for anterior and posterior full crowns and anterior three-unit fixed partial dentures. In-Ceram Zirconia is 67% by weight alumina and 13% zirconia. It is indicated for posterior full crowns and posterior three-unit fixed partial dentures. IPS Empress and Cergogold are glass-ceramic materials containing leucite crystals[4] indicated only for fabricating single-unit crowns. IPS Empress 2 is a multiphase glass ceramic with 60% by volume of two crystal phases: lithia disilicate based-($Li_2O{\cdot}SiO_2$) crystals as the main phase and lithium orthophosphate crystals as the second phase.[14] This material is used to fabricate anterior and posterior full crowns and anterior three-unit fixed partial dentures (FPDs).[15] Procera is a high density alumina containing 99.5% aluminum oxide and is indicated for full crowns and laminates. Cercon is a zirconia-based ceramic that contains 94% $ZrO_2$ stabilized by 5% $Y_2O_3$, and it is indicated for the fabrication of crowns and up to four-unit posterior fixed partial dentures (FPDs). Despite the differences among these ceramic materials, similar procedures for photopolymerization have been used to activate adhesive resin cements when under these restorations. It should be noted that porcelain materials, such as Duceram, cergogold and IPS Empress, recommend adhesive resin cement, while ceramics, such as IPS Empress 2, In Ceram, Procera and Cercon, claim that their restorations can be cemented with either resin adhesive cement or conventional cements, such as glass ionomer. Furthermore, some zirconia-based ceramic systems recommend the use of the chemically-activated resin cement Panavia 21.

There is limited information available in the literature on the effect of the composition, opacity and thickness of ceramic materials on light attenuation from curing units to polymerize resin cement under a ceramic restoration. This study evaluated: 1) the effect of different ceramics on microhardness, measured as the Knoop Hardness Number (KHN) of a resin cement immediately and 24 hours after polymerization and 2) the effect of different activation modes (direct, chemical and through the ceramic) on the KHN of a resin cement. The null hypotheses were that the different activation modes and different post-activation times would not influence the microhardness of a dual-cured resin cement.

## METHODS AND MATERIALS

### Ceramic Specimen Fabrication

Porcelain Duceram Plus (Degussa Dental, Hanau, Germany): Shade dentin A3 was condensed in a metallic mold to form a cylindrical specimen that was fired in a ceramic furnace (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany) according to the manufacturer's instructions. One cylinder of 8.0 (± 0.01) mm diameter was fabricated. This specimen was sectioned under water with a diamond disc at low speed to obtain a disc 1.2 mm thick that was then finished and glaze fired.

Cergogold: A wax pattern, 8 mm in diameter and 1.3 mm thick, was sprued and invested (Cergofit investment; Degussa Dental) and allowed to set. It was then placed in a burnout furnace to eliminate the wax. The Cergogold ingot (shade A3) was pressed in an automatic press furnace (Cerampress Qex, Ney Dental Inc, Bloomfield, CN, USA). After cooling, the specimen was divested with air abrasion using 50-µm glass beads at 4-bar pressure, followed by 100-µm aluminum oxide at 2-bar pressure to remove the refractory material. Finally, the specimen was treated with 100-µm aluminum oxide at 1-bar pressure. This specimen was ground under water with a diamond disc at low speed to obtain a 1.2 mm thick disc, which was finished and stain fired.

Table 1: *Materials, Brand Names, Manufacturers, Composition and Batch # Used*

| Materials | Brand Name | Manufacturer | Composition* | Batch # |
|---|---|---|---|---|
| Feldspatic porcelain | Duceram Plus | Degussa Dental, Hanau, Germany | $K_2O_2$, $Al_2O_3$, $SiO_2$, SnO, ZrO, $Na_2O$, CaO, pigments | 0122/5 |
| Feldpatic ceramic | Cergogold | Degussa Dental, Hanau, Germany | $SiO_2$, $Al_2O_2$, $K_2O$, $Na_2O$, CaO | 2018/12 |
| Feldspatic porcelain | Duceragold | Degussa Dental, Hanau, Germany | $SiO_2$,$Al_2O_3$,$K_2O$,$Na_2O$,CaO,BaO, $SnO_2$,$Li_2O$,F,$Sb_2O_3$,$CeO_2$,$B_2O_3$,$TiO_2$ | 0230/4 |
| Leucite ceramic | IPS Empress | Ivoclar,Vivadent, Schaan, Liechtenstein | $SiO_2$, $Al_2O_3$, $K_2O$, $Na_2O$, $CeO_2$, $B_2O_3$, CaO, BaO, $TiO_2$ | F68542 |
| Lithium di-silicate ceramic | IPS Empress 2 | Ivoclar-Vivadent, Schaan, Liechtenstein | $SiO_2$, $Al_2O_3$, $La_2O_3$, MgO, ZnO, $K_2O$, $Li_2O$, $P_2O_5$ | G02567 |
| Feldspatic porcelain | Eris | Ivoclar-Vivadent, Schaan, Liechtenstein | $SiO_2$, $K_2O$, ZnO, $ZrO_2$, $Li_2O$, CaO, $Na_2O$, $Al_2O_3$) | F69117 |
| Alumina high content ceramic | Procera | Nobel Biocare, Gothenburg, Sweden | $Al_2O_3$ | 03/2003 |
| Feldspatic porcelain | AllCeram | Degussa Dental, Hanau, Germany | $SiO_2$,$Al_2O_3$,$K_2O$,$Na_2O$,CaO, $Y_2O_3$,$SnO_2$,$Li_2O$,$ZrO_2$ | 0182/1 |
| Alumina ceramic | In Ceram Alumina | Vita Zanfabrik, Seefeld, Germany | $Al_2O_3$, $La_2O_3$, $SiO_2$, CaO, other oxides | 10780 |
| Feldspatic porcelain | VM7 | Vita Zanfabrik, Seefeld, Germany | $SiO_2$,$Al_2O_3$,$B_2O_3$,$Na_2O$, $K_2O$,CaO and $TiO_2$ | 62530 |
| Zirconia ceramic | Cercon | DeguDent, Hanau, Germany | $ZrO_2$, $Y_2O_3$, Hf $O_2$, $SiO_2$,$Al_2O_3$ | 20018669 |
| Feldspatic porcelain | Cercon Ceram S | DeguDent, Hanau, Germany | $SiO_2$,$Al_2O_3$,$K_2O$,$Na_2O$ and silicate glasses | 30240 |
| Resin cement | Rely x ARC | 3M ESPE, St Paul, MN, USA | Paste A: Bis-GMA, tri-ethylene glycol dimethacrylate, zircon/silica filler (68 wt%), photoinitiators, amine, pigments Paste B: Bis-GMA, tri-ethylene glycol dimethacrylate, benzoic peroxide, zircon/silica filler (67 wt%) Ceramic Primer: gamma-methacryloxypropyl trimethoxysilane, ethanol, water | EMFM 08/07 |

**Manufacturers' information*

IPS Empress: A wax pattern, 8 mm in diameter and 1.3 mm thick, was sprued and invested in IPS Empress investment and eliminated in a burnout furnace (7000-5P; EDG Equipments Ltda, Sao Carlos, Brazil) by heating the refractory die. At the same time, the IPS Empress ingots (color A3) and the alumina plunger were heated at increased increments of 3°C per minute to 850°C and held for 90 minutes. After the above procedure was completed, the investment, plunger and ingot were transferred to a furnace (EP 500; Ivoclar-Vivadent, Schaan, Liechtenstein) that increased the temperature to 1180°C and automatically pressed the melted ingot to the mold. After pressing and cooling to room temperature, the ceramic was divested with air abrasion using 50 µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid; Ivoclar-Vivadent) for 10 minutes, washed in running water and dried. The ceramic disc was then treated with 100 µm aluminum oxide at 1-bar pressure and ground under water with a diamond disc at low speed to obtain a 1.2 mm thickness. The disc was finished and stain fired.

IPS Empress 2: A wax pattern, 8 mm in diameter and 0.7 mm thick, was sprued and invested in IPS Empress 2 Speed investment. The wax was eliminated in a burnout furnace (700-5P; EDG Equipments Ltda, São Carlos, Brazil). The investment, plunger and 2 ingots of IPS Empress 2 (shade 300) were then transferred to a furnace (EP 500) and automatically pressed in accordance with the manufacturer's instructions. After cooling to room temperature, the specimen was divested with air particle abrasion 50-µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid), washed in running water and dried. It was then treated with 100 µm aluminum oxide at 1-bar pressure. Porcelain Eris shade dentin A3 (Ivoclar-Vivadent) was applied and fired over the di-silicate disc. The porcelain was ground and submitted to finishing and glaze firing to achieve 0.5 mm, providing a total disc thickness of 1.2 mm.

Procera: A plate of brass, 8 mm in diameter and 0.5 mm thick, was fabricated on a lathe (Nardini ND 250 BE, Sao Paulo, Brazil). The plate was measured after finishing by using a precision electronic micrometer (Electronic Micrometer; LS Starrett, Athol, MA, USA) with an accuracy of 0.002 mm. This plate of brass was sent to Gothenburg, Sweden, and a ceramic plate of sintered high-purity aluminum-oxide ceramic was fabricated following the CAD/CAM technique used by Nobel Biocare (Gothenburg, Sweden). Porcelain AllCeram shade dentin A3 (Degussa Dental) was applied and fired over the alumina disc. The porcelain was ground and submitted to finishing and glaze firing to achieve 0.7 mm. Thus, a 1.2 mm thick disc was obtained.

In-Ceram Alumina: A mold of stainless steel (20 x 20 x 5 mm) with a central depression, 8 mm in diameter and 0.5 mm thick, was obtained. An impression of this model was made with polyvinyl siloxane and then duplicated in a plaster die (Special plaster; Vita Zahnfabrik, Bad Sackingen, Germany). The aluminum oxide powder was mixed with a special liquid, as instructed by the manufacturer. The slurry mixture was then painted into the depression in the special plaster die and fired at 1120°C in a furnace (Inceramat II; Vita Zahnfabrik) for 10 hours. Glass infiltration was achieved by coating the aluminum oxide frameworks with glass powder (silicate-aluminum-lanthanum) mixed with distilled water and fired for four hours at 1100°C. The excess glass was then removed by using a fine-grained diamond (Renfert, Hilzingen, Germany). Subsequently, the specimen was air abraded using 100 µm aluminum oxide at a pressure of 3-bar. Porcelain VM7 shade dentin A3 (Vita Zahnfabrik, Seefeld, Germany) was applied and fired over the infiltrated alumina disc. The porcelain was ground and submitted to finishing and glaze firing to achieve a total disc thickness of 1.2 mm.

Cercon: A wax pattern 8 mm in diameter and 0.4 mm thick was obtained. The wax model was placed in the Cercon brain (DeguDent) unit for scanning. The confocal laser system measured the wax to an absolute precision of 10 µm and a reproducibility of <2 µm. Scanning was accomplished in four minutes. A Cercon base blank of presintered zirconia was milled, then sintered to a fully dense structure in the Cercon Heat (DeguDent) at 1350°C for six hours. The specimen was finished under water, followed by using 100 µm aluminum oxide at a pressure of 3-bar. Cercon Ceram S shade dentin A3 (DeguDent, Hanau, Germany) was applied and fired on the zirconia disc. The porcelain was ground, finished and glazed to achieve a total disc thickness of 1.2 mm.

### Resin Cement Activation

The resin cement RelyX ARC shade A3 (3M ESPE, St Paul, MN, USA) was mixed according to the manufacturer's directions and inserted in a nylon mold with a centered hole 5.0 (± 0.01) mm in diameter and 1.0 (± 0.01) mm thick. The nylon mold was previously coated with black paint (Colorgin Spray, Sherwin-Williams do Brasil Ind Com Ltda, São Bernardo do Campo, SP, Brazil). The aim of this procedure was to limit light transmission through the ceramic and the resin cement only.[12] A polyester film (± 25 µm thick) was placed above the mold and resin cement. The cement was mixed under controlled temperature (23°C ± 1) and relative humidity (higher than 30%), according to ISO 4049.[16] The resin cement was chemically activated (self-cured) or photo/chemically activated (dual-cured). When photo/ chemical activation was used, the resin cement was light irradiated by two modes: direct light-activation (Dir) or photo-activation through the ceramic (Pcer). In the Pcer groups, the ceramic discs were interposed

Table 2: *Results of Two-way ANOVA for Knoop Hardness Number of Rely X ARC Resin Cement*

| Source | Df | Sum of Squares | Meam Square | *F* | *p*-value |
|---|---|---|---|---|---|
| Treatment | 9 | 66200.9235874 | 7355.65581764 | 644.9030 | 0.00001 |
| Error | 90 | 1026.5252220 | 11.4058358 | --- | --- |

Table 3: *Knoop Hardness Number (KHN)) of Rely X ARC Resin Cement as a Function of Activation Mode and Testing Time (Mean ± Sd; n=30)*

| | | Testing Time | |
|---|---|---|---|
| **Activation Mode** | **Code** | **Immediate** | **24 Hours** |
| Direct | DI | 49.54(6.68) A | 48.10(6.23) A |
| Duceram | DU | 20.70(2.44) a | 32.54(3.23) a |
| Cergogold | CE | 18.07(2.46) ab | 31.37(5.79) a |
| IPS Empress | I1 | 15.47(3.08) b | 29.89(2.26) a |
| IPS Empress 2/Eris | I2 | 15.24(2.99) b | 27.44(4.33) b |
| Procera/Allceram | PR | 12.00(1.53) c | 17.80(1.82) c |
| In Ceram/VM7 | IC | 9.14(1.10) c | 16.13(2.08) c |
| Cercon/CerconCeram | CN | 8.59(0.81) c | 15.68(1.46) c |
| Chemical | CH | 8.87(0.71) c | 14.03(0.95) c |

*Capital letters denote no significant differences between testing times (p≤0.05); Groups in columns with the same lower case letter are not statistically different (p≤0.05); (Tukey's test).*

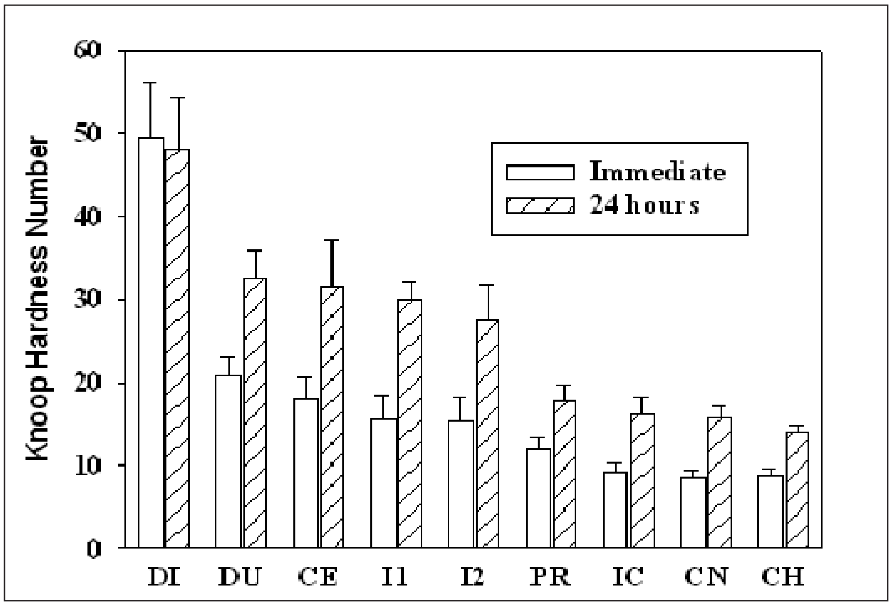


Figure 1: *Knoop Hardness Number (KHN) of Rely X ARC resin cement as a function of activation mode and testing time (Mean ± Sd; n=30).*

between the tip of the light guide of the light-activation unit (LPU) and the polyester film that covered the resin cement before irradiation. The resin cement was light-activated by a quartz-tungsten-halogen light unit XL 2500 (3M ESPE) with an irradiance of 650 mW/cm$^2$ for 40 seconds, which was in contact with the ceramic material or polyester. The irradiance (light intensity) of the curing units was measured with a hand-held radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT, USA). The light-activation unit was connected to an electrical voltage stabilizer and supported by an apparatus to allow standardization in light guide placement for irradiation. Two post-cure times were investigated. Specimens were evaluated 1) between 10-20 minutes after light-activation or 2) between 20-30 minutes for the chemically-activated groups (self-cured mode). The specimens were stored in dry, dark conditions at 37°C. Two 24-hour post-cure specimens were stored in dry, dark conditions at 37°C for approximately 24 hours. Nine groups (n=10) were tested.

The specimens were stabilized in acrylic and sectioned longitudinally. Each test surface was ground and finished through 1200 SiC using a decreasing grit sequence.

## Indentation

A universal indenter tester (HMV-2, Shimadzu, Tokyo, Japan) was used for Knoop Hardness testing (KH). The tester was set for an automatic mode of 50 grams-force for 15 seconds. Knoop Hardness values were obtained at 100 µm from the surface irradiated. Three indentations were made for each specimen (10 specimens for each mode of activation), with a 1 mm distance between them and the means were calculated for each specimen. Measurements were made with 40x magnification. The KHN was provided automatically by the tester's software. To maximize standardization, the testing was conducted by one experienced operator. The data were analyzed statistically using two-way analysis of variance (ANOVA) and Tukey's test. All tests were performed at $p$=0.00001.

## RESULTS

The results of the Knoop Hardness Number (KHN) are shown in Tables 2 and 3 and Figure 1. Table 2 represents the results of the two-way ANOVA and showed significant differences among the ceramics and mode of activation ($p$=0.00001). Table 3 and Figure 1 indicate the mean and standard deviation of KHN for each group. The KHN of the resin cement was not only affected by the ceramic composition, but also by the post-activation testing time. The mean KHN of the resin cement for chemical activation and through all ceramics showed statistically significant lower values compared to direct activation for both immediate and 24 hours. The KHN for 24 hours post-activation was always superior to the immediate post-activation test except for direct activation.

For immediate testing time, Duceram was statistically inferior to the direct group; however, it was superior to all other groups except for Cergogold. Cergogold, IPS Empress and IPS Empress 2 did not differ statistically, but they were superior to the remaining groups. Cercon, In Ceram Alumina, Procera and Chemical resulted in the lowest KHN without any statistical difference among them.

For the 24-hour testing time, Duceram was inferior to the direct group but was not statistically different compared to Cergogold and IPS Empress. However, Duceram showed superior KHN values in relation to the remaining groups. IPS Empress 2 was inferior to Direct, Duceram, Cergogold and IPS Empress, but it was higher than the In Ceram Alumina, In Ceram Zirconia, Procera and Chemical groups. Chemical, Cercon, In Ceram Alumina and Procera showed the lowest KHN, but they were not statistically different among themselves.

## DISCUSSION

According to the conditions tested (post-irradiation time and mode of activation), the null hypotheses evaluated in this study were rejected. The results showed statistically significant differences in microhardness means.

The results of the current study suggest that the type (composition) of ceramic influences the degree of polymerization, especially for denser ceramics, such as alumina and zirconia-based ceramics (Table 3 and Figure 1). The thickness of the ceramics used in the current study was designed to be as close as possible to that used clinically. Other studies evaluating various ceramics have shown that the degree of cure and hardness decreases with the interposition of a thicker ceramic restoration.[10,12,17] The KHN of light-activated Rely X ARC under opaque ceramics (Procera, Cercon and In Ceram) did not differ statistically from chemical activation, showing that almost no light passed through the ceramic structures (Table 3 and Figure 1). Therefore, polymerization under those ceramics relies only on the self-curing component of the resin.[9,18] The KHN results were low and the self-curing component did not improve the KHN even after 24 hours (Table 3 and Figure 1). These results are in agreement with studies conducted by Hasegawa and others[18] and el-Badrawy and others,[19] who reported that the self-curing component is not sufficient to induce conversion of dual-cured cement and achieve a high hardness number. This observation may help to explain why some zirconia- and alumina-based ceramic systems recommend the use of a chemically-activated resin cement (Panavia 21).

Even though the cement KHN under IPS Empress, IPS Empress 2, Cergogold and Duceram demonstrated statistically significant differences, each was statistically superior to the other ceramics and the chemical activation mode (Table 3 and Figure 1). Although IPS Empress, IPS Empress 2, Cergogold and Duceram have shown lower KHN when compared with direct activation, these ceramics appear to allow more light to pass through them and activate the cement better. This could result in improved properties of the resin cement, especially after 24 hours. The dual-cured cement may be more reliable for more translucent ceramics than for opaque ones.

According to ISO 4049,[16] the setting time for dual-cure resin cements should be no more than 10 minutes. The current study shows that only direct light polymerization of a resin cement layer has maximum hardening to receive immediate loads. At 10 minutes after mixing, the resin cement should have good mechanical properties, such as satisfactory elastic modulus and microhardness. This will protect bonding and increase fracture toughness of some ceramic restorations due to an inherent brittleness and limited flexural strength, especially of silica-based ceramics.[20]

The polymerization of dual-cured resin cement depends upon the light-activation element as well as the quantity and efficiency of the chemical component.[21-22] The self-curing chemical component can play an important role in polymerization, especially in areas that are inaccessible to curing light.[19] This may explain differences in KHN values between the more translucent and more opaque ceramics. The behavior of the cement used in this study seems to depend more on light activation. Therefore, in an effort to try to maximize the degree of conversion as much as possible, light curing all clinically-accessible margins would be indicated.

In the current study, the KHN value of the cement was noted to be related to both time and activation modes (Table 3 and Figure 1). Similar findings have been shown in previous studies evaluating different cements.[26-27] In the current study, the KHN was measured immediately after mixing and at 24 hours. Further research needs to be done to study the effect of a longer period of time on the KHN value of the cement.

## CONCLUSIONS

Within the scope of this study, the following conclusions may be drawn:

1. The ceramics of the thickness tested reduced the Knoop Hardness of dual-cured resin cement.
2. Alumina and zirconia ceramics demonstrated a significantly greater decrease in Knoop Hardness values than silica-based ceramics.
3. An improvement in microhardness was found after 24 hours of storage, except for directly-activated cement.

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