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Dual Resin Cement Knoop Hardness After Different Activation Modes Through Dental Ceramics

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This study investigated the influence of ceramic compositions on Knoop Hardness Number (KHN) immediately and 24 h after polymerization and the effect of activation modes on the KHN of a resin cement. Ten Panavia F 2.0 resin cement discs were activated either directly using curing light, or chemically without light, or through 1.2-thick ceramic discs. The following ceramics were evaluated: Duceram, Cergogold, IPS Empress, IPS Empress 2, Procera, Cercon, In Ceram Alumina and In Ceram Zirconia. The KHN was obtained immediately and after 24-h testing time. Two-way ANOVA and Tukey's test were performed for statistical analysis ($p<0.05$). Direct activation showed higher KHN than activation through ceramics and chemical activation for both immediate and 24-h post activation. The KHN for 24-h post activation time was higher than that of the immediate post activation time except for the direct activation mode. The glass and di-silicate based ceramics showed higher KHN than alumina- and zirconia-based ceramics, immediately and after 24-h. The reinforced and opaque ceramics had the lowest KHN. The ceramic composition resulted in light attenuation, lower polymerization and lower KHN, and the 24-h testing time promoted an improvement of KHN except for direct activation mode.

Key Words: dental ceramic, hardness, luting agent, polymerization.

INTRODUCTION

Dental ceramics are appreciated as highly esthetic restorative materials with optimal esthetic properties that simulate the natural dentition appearance. Other desirable characteristics include translucence, fluorescence, chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to tooth structure (1). In spite of their many advantages, ceramics are fragile under tensile (2).

Luting materials are vitally important for the longevity of dental restorative materials (3,4). Resin cements offer distinct advantages, such as adhesion to both ceramic and dental structure substrates, as well as low solubility, easy handling, and favorable esthetics (5). Higher fatigue and compressive strength of feldspathic ceramic restorations is observed in these cements when compared to glass ionomer cements (5). However, in order to increase the restoration retention to the tooth structure and allow efficient polymerization of the resin cement, the luting agent has to flow smoothly and fill the interfacial gap completely (6,7). Survival of these restorations also depends on the degree of conversion of these luting agents, which influences properties such as hardness, wear resistance, water absorption, residual monomer, and biocompatibility (6,8).

Dual cure resin cements joins the advantages of both chemical cure and light cure materials (9,10). Al-

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though the materials undergo a dual polymerization mode, an adequate amount of light is required to initiate the polymerization process (11). The composition, thickness, opacity, and shade of the ceramic may attenuate the light from the curing unit used to polymerize the resin cement under the ceramic restoration (10,12). Light intensity and the distance of the curing unit tip during polymerization are other important factors that should be observed during luting of ceramic restorations (7,13). Ceramics from different manufacturers have different compositions and crystal content, which may impact the amount of light that passes through them for cement activation (6). As crystalline ceramics are opaque, they would be expected to attenuate more light (13). Limited information is available on the effect of composition, opacity, and thickness of ceramic materials on light attenuation.

The light attenuation from curing unit to polymerize the resin cement under different ceramics has not been adequately studied. Therefore, the purposes of this study were to evaluate: (a) the influence of ceramic composition on KHN of a resin cement, immediately and 24 h after polymerization; (b) the effect of different activation modes (direct light-activation, light activation through ceramic and chemical activation) on the KHN of a resin cement. The null hypotheses were that: (I) ceramics do not affect the KHN of the resin cement; (II) time conditions do not affect the KHN of the resin cement.

MATERIAL AND METHODS

Materials, brand names, manufacturers, composition and batch numbers are listed in Table 1.

Ceramic Specimen Fabrication

The mode of fabrication of specimens of the tested materials are summarized below.

Porcelain Duceram Plus: shade dentin A3 was condensed in a metallic mold to form a cylinder of 8 (± 0.01) mm that was sintered in a ceramic furnace (Austromat M; Dekema Austromat-Keramiköfen, Freilassing, Germany), according to manufacturer's instructions. Next, the cylinder was sectioned under water cooling with a diamond disc at low speed to obtain 1.2-mm-thick discs, which were then finished and glaze fired.

Cergogold: a wax pattern of 8 mm diameter and 1.3 mm thickness was sprued and invested using Cergofit investment (Degussa Dental, Hanau, Germany). The pattern was then placed in a burnout furnace (7000-5P; EDG Equipments Ltda, São Carlos, SP, Brazil) to eliminate the wax and the Cergogold ingot (shade A3) was pressed in an automatic press furnace (Cerampress Qex; Ney Dental Inc, Bloomfield, CT, 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 and finally with 100-µm aluminum oxide at 1-bar pressure. It was then sectioned under water cooling with a diamond disc at low speed to obtain 3 1.2-mm-thick discs, which were finished and stain fired.

IPS Empress: wax patterns of 8 mm diameter and 1.3 mm in thickness were sprued and invested in IPS Empress investment (Ivoclar-Vivadent, Schaan, Liechtenstein) and then eliminated in a burnout furnace (7000-5P; EDG Equipments Ltda) by heating the refractory die. Simultaneously, the IPS Empress ingots (shade A3) and the alumina plunger were heated at an increase of 3°C *per* min to 850°C and held for 90 min. After completion of this procedure, the investment, plunger, and ingot were transferred to a furnace (EP 500; Ivoclar-Vivadent) that increased the temperature to 1180°C. After pressing the melted ingot into the mold and slowly allowing to cool at room temperature, the ceramic was divested with air abrasion using 50 µm glass beads at 2-bar pressure, then ultrasonically cleaned in a special liquid (Invex Liquid; Ivoclar-Vivadent) for 10 min, washed in running water, and dried. The ceramic disc was then treated with 100 µm aluminum oxide at 1-bar pressure. They were then sectioned under water with a diamond disc at low speed to obtain 1.2 mm thickness. The discs were finished and stain fired.

IPS Empress 2: wax patterns of 8 mm diameter and 0.7 mm thickness were sprued and invested in IPS Empress 2 Speed investment (Ivoclar-Vivadent). The wax was eliminated in a burnout furnace (700-5P; EDG Equipments Ltda) and then the investment, plunger, and 2 ingots of IPS Empress 2 (shade 300) were transferred to a furnace (EP 500; Ivoclar-Vivadent) and automatically pressed in accordance with manufacturer's instructions. After cooling to room temperature, the ingots were divested with air particle abrasion 50-µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex Liquid; Ivoclar-Vivadent), 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 brass plate of 8 mm diameter and 0.5 mm thickness was fabricated on a lathe (Nardini ND 250 BE, São Paulo, SP, Brazil). The plate was measured after finishing by using a precision electronic micrometer (Electronic Micrometer; LS Starrett, Athol, MA,

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,CeO2, 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	SiO2, K2O, ZnO, ZrO2, Li2O, CaO, Na2O, Al2O3)	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
Zirconia ceramic	In Ceram Zirconia	Vita Zanfabrik, Seefeld, Germany	Al2O3 (62%), ZnO (20%), La2O3 (12%), SiO2 (4.5%), CaO (0.8%), other oxides (0.7%)	22470
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	Panavia F 2.0	Kuraray, Osaka, Japan	Paste A: BPEDMA, MDP, DMA, silica, barium sulfate, dibenzoylperoxide. Paste B: N,N-diethanol-p-toluidine, silica sodiumfluoride, Polyethyleneglycol, glycerine, sodium benzenesulfinate cont. gel.	51581

*$^{\#}$ Manufactures information

USA) with an accuracy of 0.002 mm. It was then 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 and In-Ceram Zirconia: a stainless steel mold (20 x 20 x 5 mm) with a central depression 8 mm diameter and 0.5 mm thickness was obtained. An impression of this model was made with polyvinyl siloxane, and then duplicated in a plaster (Special Plaster; Vita Zahnfabrik, Bad Sackingen, Germany). The aluminum oxide powder or the aluminum zirconia powders were 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^{o}C in the furnace (Inceramat II; Vita Zahnfabrik) for 10 h. Glass infiltration was achieved by coating the aluminum oxide frameworks with glass powder (silicate-aluminum-lanthanum) mixed with distilled water, and fired for 4 h at 1100^{o}C. Excess glass was removed using a fine-grained diamond (Renfert, Hilzingen, Germany). Subsequently, the specimens were air abraded using 100-µm aluminum oxide at 3-bar. Porcelain VM7 shade dentin A3 (Vita Zahnfabrik) was applied and fired over the infiltrated alumina and zirconia 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 of 8 mm diameter and 0.4 mm thickness was obtained. The wax model was placed in the Cercon brain (DeguDent, Hanau, Germany) unit for scanning. The confocal laser system measured the wax to an absolute precision of 10 µm and reproducibility of <2 µm. Scanning was accomplished in 4 min. A Cercon base blank of pre-sintered zirconia was milled and then sintered to a fully dense structure in the Cercon Heat (DeguDent) at 1350^{o}C for 6 h. The specimens were finished using 100-µm aluminum oxide at 3-bar pressure. Cercon Ceram S shade dentin A3 (DeguDent) 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 Panavia F 2.0 (Kuraray Co. Ltd, Osaka, Japan), shade A3, was mixed according to manufacturer's directions and inserted in a nylon mold having a centered hole with 5.0 (±0.01) mm diameter and 1.0 (±0.01) mm thickness. The nylon mold was pre-coated with black paint (Colorgin Spray; Sherwin-Wiliams, Taboão da Serra, SP, Brazil) to limit light transmission through the ceramic and the resin cement only. A polyester film (25 µm thickness) was placed above the mold. The cement was mixed under controlled temperature 23°C (±1) and relative humidity (higher than 30%), according to the ISO 4049 standard.

Regarding the activation modes, the resin cement was chemically activated (chemical activation mode) and activated by 2 modes (photo/chemical): directly photo-activated (direct activation mode) and photo activated through the discs fabricated for 8 different ceramics (activated through ceramic mode). Two post-cure times were investigated: immediately and 24 h after cure. In the immediate post-cure time, the specimens were evaluated between 10-20 min after light-activation or between 20-30 min for the chemically activated groups. The specimens were then stored under dry, dark conditions at 37°C. In the 24-h post-cure time, the specimens were stored under dry, dark conditions at 37°C for approximately 24 h.

In the specimens in which the cement was light-activated through different ceramic discs, the discs were interposed between the tip of the light source unit and the polyester film that covered the resin cement before irradiation. The resin cement was light activated for 40 s with a quartz-tungsten-halogen light unit (XL 2500; 3M ESPE, St. Paul, MN, USA) with light irradiance of 650 mW/cm^{2}, as measured with a handheld curing radiometer (model 100; Demetron/Kerr, Danbury, CT, USA).

Knoop Hardness Testing

Ten resin cement discs for each group were finished with 1,200 silicon carbide paper discs and subjected to a universal indenter tester (HMV 2; Shimadzu, Tokyo, Japan) for Knoop Hardness testing (KHN). Measurements were obtained at 40× magnification and values were obtained at 100 µm from the irradiated surface application of a load of 50 g for 15 s. Three indentations were made in each specimen (n=30) at a distance of 1 mm between them, and the mean was calculated for each specimen. The KHN was calculated automatically by the hardness tester's software. Data were analyzed statistically by two-way ANOVA and multiple comparisons were done by Tukey's test. All tests were performed at $p<0.05$.

RESULTS

The KHN means and standard deviations for each group are shown in Table 2. The KHN of the resin cement was affected not only by the ceramic composition, but also by the post activation testing time ($p=0.00001$).

The direct activation mode showed significantly higher KHN than the activation through ceramic and chemical activation for both testing times (immediate and 24 h).

For both testing times, immediately and 24-h, Duceram, Cergogold, IPS Empress and IPS Empress 2 showed significantly higher KHN than Procera, Cercon, In Ceram Alumina and In Ceram Zirconia.

For the group activated through ceramics and the chemically activated group, the 24-h testing time showed statistically significant higher KHN than the immediate testing time, but no statistically significant difference was found for the direct activation mode.

For the immediate testing time, the direct activation mode was statistically superior to the mode of activation through ceramic and the chemical activation mode. Duceram and Cergogold showed statistically significant higher KHN than IPS Impress 2, and IPS Empress showed intermediate KHN. Procera and Cercon showed lower KHN than IPS Empress 2, but higher than the chemical activation mode. On the other hand, the KHN for In Ceram Alumina and In Ceram Zirconia groups did not show statistically significant difference than the chemical mode activation.

Table 2. Knoop hardness number (KHN) of Panavia F 2.0 resin cement as a function of activation mode and testing time (mean ± SD; n=30).

Activation Mode	Testing time	
	Immediate	24 hours
Direct	51.56 (2.93) Aa	52.03 (3.63) Aa
Duceram	22.26 (1.85) Bb	31.33 (2.84) Ba
Cergogold	22.09 (3.83) Bb	32.20 (3.07) Ba
IPS Empress	18.09 (2.02) BCb	30.19 (3.91) Bca
IPS Empress 2/Eris	15.85 (3.15) Cb	26.96 (3.30) Ca
Procera/Allceram	12.77 (1.54) Db	20.10 (2.61) Da
Cercon/CerconCeram	11.20 (1.79) Db	17.79 (2.06) Da
In Ceram/VM7 D	9.19 (0.73) DEb	18.90 (1.54)Da
In Ceram/Zirconia	8.78 (0.90) DEb	18.71 (2.06) Da
Chemical	8.63 (1.29) Eb	14.98 (2.09) Ea

Different uppercases letters indicate significant differences between the activation modes and lowercases letter indicate significant differences between testing times (Tukey's test; $p\leq0.05$).

For the 24-h testing time, the groups activated through ceramic showed intermediate KHN between the direct activation mode (higher values) and the chemical activation mode (lower values). Duceram and Cergogold showed statistically significant higher KHN than IPS Empress 2, and IPS Empress showed intermediate values. Procera, Cercon, In Ceram Alumina and In Ceram Zirconia showed statistically significant lower KHN within the groups activated through ceramic.

DISCUSSION

The direct mode resulted in a statistically significant higher KHN than the activated through the ceramics groups and chemical mode groups for both testing time. The KHN for 24-h post activation was always statistically superior to the immediately post activation time except for direct activation mode. The glass and di-silicate based ceramics showed significantly higher KHN than the alumina- and zirconia-based ceramics in both testing times (Table 2). Therefore, the null hypotheses evaluated in this study were rejected.

The findings of the present study suggest that the ceramic type and composition are critical factors for the hardness development in the tested indirectly activated dual-cured resin luting agent. Furthermore, it may be assumed that the degree of polymerization depends on the interaction between ceramic composition and light attenuation, and that the indirect activation decreases the level of irradiance reaching the luting material. As a result, the development of the polymer network could be affected by diminishing the monomer conversion, interfering with the type and degree of cross-linking (1). The present results also confirm that the most translucent ceramics (Duceram, Cergogold, IPS Empress and IPS Empress 2) have significantly higher KHN than most opaque ones (Procera, Cercon, In Ceram Alumina and In Ceram Zirconia) (Table 2).

The direct activation mode showed higher KHN than the activated through ceramics groups and chemical activation mode groups for both testing times, immediately and 24-h (Table 2). Therefore, the self-curing component *per se* might not be sufficient to ensure the adequate polymerization (10). In addition, the results showed that the luting agent formulation is a critical factor for the development of hardness in indirectly activated dual cure cements. Although it is difficult to predict whether different clinical performances are likely to occur for restorations luted under similar conditions to those tested in the present study, the use of a light source with high intensity or the increase of the light exposure time is advisable when cementing more opaque ceramic restoration (1). In the present study, the KHN was measured immediately and 24 h after mixing. Further research to study the effect of time on the KHN over a long period of time are encouraged.

Since it has been shown that even well polymerized resin cements can release residual monomers, it is reasonable to assume that more substances would elute from poorly polymerized resin cement. These substances have the potential to irritate soft tissues and pulp, stimulate the growth of bacteria and promote allergic reactions (14). Furthermore in a real clinical situation, dentists frequently confront with a cement line that stays in direct contact with the gingiva in the intrasulcular margin of the crown preparation (9,15,16). Therefore, those monomers released from the partially cured cement would potentiate the gingival irritation, and lead to adverse clinical consequences such as microleakage, postoperative sensitivity, discoloration, and secondary caries (10,12-17).

The immediate testing time showed lower KHN than the 24-h testing time. The immediate testing time showed lower KHN than the 24-h testing time. Similar findings have been observed in previous studies evaluating different cements (7,12,18-20). Restorations are unstable immediately after placing and could be dislocated during the mastication process. Thus, it is recommended that a clinical protocol may be created for a cementation process that includes additional time to allow adequate polymerization. Moreover, this protocol should include an advice to the patients to avoid chewing hard-based diet for at least 24 h.

In conclusion, within the limitations of this study, it may be concluded that polymerization through ceramic affected the Knoop microhardness of the dual resin cement, ceramic composition affected the polymerization of the dual resin cement, an improvement in microhardness was found after 24-h testing time except for the directly activated mode, and alumina- and zirconia-based ceramics had lower Knoop hardness than silica- and di-sicate-based ceramics.

RESUMO

Este estudo investigou a influência de composições de cerâmica no número de dureza Knoop (KHN), imediatamente e 24 h após a polimerização; e o efeito de modos de ativação na KHN de um cimento resinoso. Dez discos de cimento resinoso Panavia F 2.0 foram ativados diretamente usando uma fonte de luz, ou quimicamente sem luz, ou através de discos de cerâmica com espessura de 1,2 mm. As cerâmicas avaliadas foram Duceram, Cergogold, IPS Empress, IPS Empress 2, Procera, Cercon, In Ceram Alumina e In Ceram Zirconia. O KHN foi obtido nos tempos imediatamente e após 24 h. Os testes ANOVA two-way e Tukey foram realizados para a análise estatística (p<0,05). A ativação direta apresentou maiores KHN que a ativação através das cerâmicas e ativação química, para ambos os tempos testados imediatamente e após 24 h. O KHN para o tempo de ativação testado após 24 h foi superior que o tempo imediatamente, exceto para o modo de ativação direto. As cerâmicas à base de vidro e di-silicato apresentaram KHN superiores que as cerâmicas à base de alumina e zircônia, imediatamente e após 24 h. As cerâmicas mais reforçadas e opacas resultaram um dos menores KHN. A composição da cerâmica resulta em atenuação de luz, menor polimerização e menor KHN; e o tempo de 24 h testado promove uma melhora no KHN, exceto para o modo de ativação direto.

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