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Research Article

Nanohardness and Elastic Modulus of Different Resin Cements After Accelerated Aging

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Abstract

Background

The properties of resin cements used to lute prosthesis to dental structures should be investigated by simulating the oral environment.

Objective

The aim of this study was to evaluate the mechanical properties of resin cements of chemical and dual sets by immediate hardness and after accelerated aging by thermocycling.

Method

Five Disc shaped specimens were prepared for each resin cement evaluated using a metallic mold (10mm diameter X 1mm thick). The resin cements evaluated in the present study were: Multilink (Ivoclar-Vivadent), Relyx ARC (3M/ESPE), All Cem (FGM), Enforce (Caulk/Dentsply), Variolink II (Ivoclar-Vivadent), and Relyx U-200 (3M/ESPE). The specimens were subjected to indentations before and after accelerated aging by thermocycling to determine mechanical properties such as hardness and elastic modulus.

Results

Statistical analysis revealed significant differences in hardness and elastic modulus between the moments (before and after accelerated aging). Multilink presented the lowest elastic modulus and hardness (8.51GPa before aging, 8.31GPa after aging, and 0.33 before aging, 0.32 after aging) and Relyx U-200 presented high elastic modulus and hardness (11.61GPa before aging, 12.84GPa after aging, and 0.53 before aging, 0.66 after aging), respectively.

Conclusion

Water induce to hydrolysis and degradation on resin cements, which in association with the temperature variations can cause alterations in materials properties.

Introduction

The indirect restorations (crowns, laminated, inlays, onlays, and fixed prosthesis) and and root posts are often present in the daily routine of clinicians [1,2]. The patient's expectations for dental rehabilitations that mimics the natural dentition accelerated the aesthetic dental materials evolution and resulted in the need for the development of resin cements to lute prosthesis to dental structure. According to the curing mode, three possible alternatives can be chosen: chemical, photo or dual-curing resin cements. The use of auto-cured resin cements offers chemical cure with luting of base and catalyst pastes, worse handling characteristics due to the absence of control of the setting reaction. Photoactivated allow setting control greater than chemical cements. Dual-curing resin cements are expected to combine favorable properties of both, auto and photoactivated systems [3]. Regarding resin cements, the curing mode can also influence of shrinkage produced after polymerization. The shrinkage is dependent to unfavorable factor of cavity configuration (factor-C). The high factor-C can restrict the flow of resin cement affecting the integrity of the adhesive interface [1].

Adequate polymerization is a crucial factor for obtaining optimal mechanical properties and satisfactory clinical performance of dental resin material like Young's modulus and hardness [4]. Good mechanical properties, high degree of conversion and high cross-linking density may be advantageous not only from the point of view of certain mechanical properties, but also because of reduced susceptibility to the softening action of food substances and to enzymatic attack [5].

Water degradation is a phenomenon that can cause several alterations in dental resins, from physical changes, such as plasticization and softening to chemical ones like oxidation or hydrolysis [6]. Negative effects in the physical properties (tensile and flexural strength, Young's modulus, wear resistance) of the composite materials caused by water exposure have been reported by Sorderholm *et al.* [7]; Oysaed *et al.* [8]; Scarret *et al.* [9], respectively.

The cementation procedure is necessary to clinical success of the prosthesis because it provides the interface sealing and reduction of marginal infiltration, avoiding pulp sensitivity and caries [2,4].

Recently, the photoactivated have been used less than auto-cured and dual-cured resin cements because the visible light doesn't get act effectiveness in deep regions or over thick prosthetic devices [10].

Choose of a cementation agent is dependent of clinical condition combining with physical, biological and handling properties [11].

One of the most advantage of nanoindentation is the possibility of testing the cements in situ [3], this method has been applied in dentistry because of less material is needed to produce specimens in reduced dimensions and it's possible to obtain faithful results.

The aim of this study was to evaluate the mechanical properties of resin cements chemically and dual cured by nanoindentation test done immediately and after accelerated aging.

Materials and Methods

The resin cement evaluated in the present study were Multilink (Ivoclar-Vivadent Schaan, Liechenstein) (M), Relyx ARC (3M/ESPE, St. Paul, MN, USA) (ARC), All Cem (FGM, Joinville, SC, BR) (AC), Enforce (Caulk/Dentsply, Milford, DE, USA) (EN), Variolink II (Ivoclar-Vivadent Schaan, Liechenstein) (VR) and Relyx U-200 (3M/ESPE, St. Paul, MN, USA) (U-200). The resin cements were manipulated according the manufacturer instructions (Table 1).

Table 1: *Product, compositions, and manufacturers of resin cements used in this study*

Product	Composition	Manufacter
Multilink	Autopolymerized resin cement. Monomer matrix: ethoxylated Bis-EMA, UDMA, Bis-GMA, HEMA. Inorganic fillers: barium glass, itterbium trifluoride, spheroid mixed oxide.	Ivoclar-Vivadent Schaan, Liechenstein.

Relyx ARC	Dual-curing or autopolymerized resin composite cement; contains adhesive resin cement. Resin: Bis-GMA and TEGDMA polymer, zirconia/silica filler and photoinitiator. Paste A: zirconia/silica filler, pigments, amine and photoinitiator system. Paste B: zirconia/silica filler, peroxide, benzoyl peroxide.	3M/ESPE, St. Paul, MN, USA.
All Cem	Dual-curing resin cement. Base paste: methacrilic monomers such as TEGDMA, Bis-EMA e Bis-GMA, camphorquinone, co-initiators, barium aluminum-silicate glass microparticles, silicium dioxide nanoparticles, inorganic pigments and preservatives. Catalytic paste: methacrylic monomers, dibenzoyl peroxide and stabilizers, barium-aluminum-silicate glass microparticles.	FGM, Joinville, SC, BR.
Enforce	Dual-curing resin cement. Base paste: TEGDMA, Boron glass, Aluminum Silicate and Silanized Barium, Silanized Pyrolytic Silica, CQ, EDAB, BHT, Mineral Pigments, DHEPT. Catalyzing Paste: Titanium Dioxide, Silanized Pyrolytic Silica, Mineral Pigment, Bis-GMA, BHT, EDAB TEGDMA, Benzoyl peroxide.	Caulk/Dentsply, Milford, DE, USA.
Variolink II	Dual-curing or atopolymerizing resin composite cement; contains adhesive resin cement. Paste of Bis-GMA, inorganic fillers, itterbiumtrifluoride, initiators, stabilizers and pigments. Bis-GMA, triethylene glycoldimethacrylate (TEGDMA), urethane dimethacrilate (UDMA) and benzoil peroxide.	Ivoclar-Vivadent Schaan, Liechenstein.
Relyx U-200	Dual-curing or autopolymerizing resin composite cement; contains self-adhesive resin cement. Base paste: Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, Silanated fillers, Initiator components, Stabilizers, Rheological additives. Catalyst paste: Methacrylate monomers, Alka.	3M/ESPE, St. Paul, MN, USA.

Five-disc shaped specimens were prepared for each resin cement evaluated using a metallic mold, 10mm in diameter and 1mm thick. The molds were placed on a glass plate and the materials were inserted into the molds with Hu-Friedy instrument. After insertion, the mold was covered with a polyester strip and a glass microscope slide.

The Multilink resin cement was allowed to auto-cure for 5 min, while the dual resin cements mixture was photoactivated with an Optilux 500 device (Demetron/ Kerr, Danbury, CT, USA) with a light output not less than 600mW/cm^2 for 40s.

After 10 min, specimens were removed from the molds and stored in darkness and distilled water until evaluation at 37°C for 24 h. Samples were luted on a metallic supports and surfaces were polished with alum in a suspension slurry of 1μm and felt metallographic discs of 1μm for 2 min each.

Nine indentations were made on the disc shaped specimens. Young's modulus and hardness of the cements were determined from the load-displacement curves. The indentations were made with Berkovich point, 400mN (40gf) greatest load, peak hold time of 30 seconds and time to load of 5s.

Specimens were subjected to thermocycling in water (500 X, 5°C/55°C dwell time of 15 seconds) to simulate aging condition. Subsequently, the specimens were evaluated again according described before.

To compare nanohardness values and Young's modulus before and after aging accelerated was used Kolmogorov-Smirnov test, ANOVA repeated measures and Tukey's test in SPSS software.

Results

The Komolgorov-Smirnov revealed symmetric amounts distribution. The descriptive analysis is present in Table 2. ANOVA repeated measures to analysis of Young's modulus showed significant differences between resin cements, between moments (before and after accelerated aging) and revealed interactions between moment-cement. Tukey HSD test was used to multiple comparisons and showed significant differences in Young's modulus between AC and ARC, AC and M, AC and U-200, ARC and EN, ARC and M, ARC and U-200, ARC and VR, EN and M, EN and U-200, M and U-200, M and VR, VR and U-200 resin cements. Tukey HSD test revealed significant differences between moments before and after accelerated aging ($p<0.05$). ANOVA repeated measures to hardness analysis showed significant differences between resin cements and revealed interactions between moment-cement, but no significant differences were obtained between moments. Tukey HSD test showed significant differences in hardness between ARC and AC, AC and M, ARC and M, ARC and U-200, EN and M, EN and U-200, M and U-200, M and VR, U-200 and VR resin cements. M presented the lowest elastic modulus and hardness (8.51GPa before aging, 8.31GPa after aging and 0.33 before aging, 0.32 after aging), respectively. U-200 presented high Young's modulus and hardness (11.61GPa before aging, 12.84GPa after aging and 0.53 before aging, 0.66 after aging), respectively.

Table 2: *Mean (standard deviation) of hardness (GPa) and elastic modulus (GPa)*

Properties	Hardness (GPa)		Elastic modulus (GPa)	
	Before	After	Before	After
All Cem	$0.54\pm0.02^{A,ade}$	$0.53\pm0.03^{A,a,d}$	$11.05\pm0.68^{A,a}$	$11.03\pm0.51^{A,a}$
Relyx ARC	$0.47\pm0.03^{B,ac}$	$0.44\pm0.02^{B,bd}$	$10.44\pm0.49^{B,a}$	$8.91\pm1.1^{B,b}$
Enforce	$0.50\pm0.02^{C,bf}$	$0.50\pm0.04^{C,acd}$	$10.61\pm0.92^{C,a}$	$10.85\pm0.84^{C,ab}$
Multilink	$0.33\pm0.17^{D,c}$	$0.32\pm0.16^{D,cd}$	$8.51\pm2.05^{D,a}$	$8.31\pm1.90^{D,b}$
Relyx U-200	$0.53\pm0.03^{E,de}$	$0.66\pm0.43^{E,d}$	$11.61\pm0.62^{E,ab}$	$12.84\pm3.94^{E,ab}$
Variolink II	$0.54\pm0.03^{F,ef}$	$0.49\pm0.05^{F,abcd}$	$12.62\pm0.45^{F,b}$	$10.15\pm3.00^{F,ab}$

*Different capital letters reveal significant differences in the line for each property (hardness and elastic modulus). Different small letters reveal significant differences in the column.

Discussion

This study agrees with Ceballos *et al.* [3] results. The chemically cured resin cement (Multilink), presented the lowest values of stiffness and hardness, although is ability to being deformed without damage is superior to other cements, but disagrees with Hofmann *et al.* [12], where properties of self-curing resin cement ranged between those of the dual-cure materials.

Dual resin cements seem to possess the best combination of properties. The dual resin cements presented intermediate values and self-adhesive resin cement Relyx U-200 revealed high stiffness and hardness values in accordance with other studies [2]. Moosavi *et al.* [13] studied a dual self-adhesive resin cement (Clearfil SA cement) and showed hardness values ranging from 0.43 to 0.51. Attar, Tam and McComb [11] studying mechanical and physical properties of dental luting agents such as zinc phosphate cement, glass ionomer, resin-modified glass ionomer, resin cements of dual and chemical sets, obtained in flexural test that de zinc phosphate presented highest Young's modulus which has contributed to its clinical success for many years. The low Young's modulus for resin-modified glass ionomer suggested that these materials should be limited for use to low stress and single prosthetic units. They related that resin luting agents displayed a good combination of high flexural strength and high Young's modulus. The good mechanical properties, coupled with adhesive capabilities, make the use of resin cements an excellent choice for difficult retention situations. When they tested the storage for 24 h and 3 months, they found no significant differences in modulus of elasticity for zinc phosphate and resin cements groups, there was an increase in modulus of elasticity for both conventional and the resin-modified glass ionomer groups. These results disagree to the present study that obtained significant differences between immediate and thermocycled specimens ($p < 0.05$), maybe because the immersion in different temperatures (5 and 55°C) that can affect hardness of the materials. Irie *et al.* [14] related that the thermal stress increased flexural strength and Young's modulus for all luting materials (Compolute, Permacem, Fuji Plus and Panavia 21). This study showed that the properties increased to U-200 and EN, but to other resin cements the properties decreased after termocycling. Water that has entered the polymer by sorption can hydrolyze the covalent bonds in the resin matrix, filler-matrix interface, or filler. The hydrolysis can cause loss of mass, filler debonding and degradation of mechanical properties such hardness and Young's modulus [2].

The properties of resin cements are influenced by the nature of the matrix, type of filler, filler volume, filler-matrix interfacial bond, filler load and polymerization mode [2].

Surface hardness of materials characterizes their outer surface properties and is important to determine their capacity to be polished and their abrasive wear rate [15].

Dental materials that have properties similar to dental structures have good behavior [2,16-20]. All resin cements presented Young's modulus closer than dentine (17,5 ± 3,8 GPa), according Plotino *et al.* [21], but self-adhesive resin cement was more similar than the other resin cements.

The *in vitro* nature of the present study is considered a limitation. Future studies should focus on clinical research.

Conclusion

Considering the limits of this study, the following conclusions can be formulated.

Significant differences in hardness and elastic modulus between moments (before and after thermocycling) were found.

The resin cement of chemical set Multilink presented the lowest elastic modulus and hardness.

The dual self-adhesive resin cement Relyx U-200 presented high elastic modulus and hardness.

Water induce to hydrolysis and degradation on resin cements, which in association with the temperature variations can cause alterations in materials properties.

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Conflicts of Interest

The authors declare no conflict of interest, financial or otherwise.

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