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DOI: 10.1590/1807-3107bor-2019.vol33.0083

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Original Research
Dental Materials

Influence of activation mode, fatigue, and ceramic interposition on resin cements' diametral tensile strength

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Abstract: This study evaluated the influence of activation modes, on Diametral Tensile Strength (DTS) of dual cured resin cements subjected to a Mechanical Fatigue test (MF). Four dual-cured resin cements (RelyX UNICEM [U], RelyX ARC [A], ENFORCE [E] and Nexus 2 [N]) were activated by three different curing modes as follows: Self-Curing (SC), Dual Cure activation with photoactivation executed directly (DC) and Dual Cure activation with Photoactivation Through Porcelain (DCTP). After 24 hours, half of the sample was subjected to 30.000 fatigue cycles at 1 Hz frequency and 12 N load. Then, all specimens were subjected to DTS test in Instron Universal Testing Machine and data were analyzed by three-way ANOVA and Tukey's Test (5%). The results of DTS test means (MPa) and standard deviation, for each cement factor activated by SC, DC and DCTP was respectively: U (28.12 ± 5.29; 37.44 ± 6.49 and 40.10 ± 4.39), A (49.68 ± 8.42; 55.12 ± 5.16 and 63.43 ± 6.92), E (49.12 ± 3.89; 56.42 ± 8.88 and 56.96 ± 6.45) and N (61.89 ± 11.21; 59.26 ± 9.47 and 62.56 ± 10.93). Turkey's test indicated that DC is related to the highest DTS values; Nexus 2 DTS remained the same independently of activation mode and that the Porcelain disk interposition enhanced DTS only for RelyX ARC the ANOVA statistical test indicated that MF didn't alter the DTS values for all experimental groups. MF results clinical implication is that all cements tested exhibited, in an immediate loading, good cross linked bonds quality.

Keywords: Resin Cements; Materials Testing; Tensile Strength; Biomedical and Dental Materials; Self-Curing of Dental Resins.

Declaration of Interests: The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

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https://doi.org/10.1590/1807-3107bor-2019.vol33.0083

Submitted: January 17, 2019
Accepted for publication: July 2, 2019
Last revision: July 31, 2019

Introduction

Resin cements were introduced in the dental practice to bond fixed prostheses to abutments in the 1970s, after the success of attaching unfilled resins to acid etched enamel.[1] The concept of using resins to bond fixed prostheses came into existence due to the fact that this material has better properties, such as satisfactory adhesion to dental substrates and dental materials, low water solubility, easy manipulation, and esthetic compatibility with metal free systems, especially when compared with traditional acid-basic reaction cements.[2,3,4,5,6,7]

In dentistry, the use of resin cements has increased in the last years and currently, these are the first-choice materials for fixed esthetic inlays, onlays, crowns, and root posts.[3,4,7,8,9,10,11]

Resin cements can be indicated for different clinical situations according to their activation modes. In agreement with norm 4049 of the International Organization for Standardization,[12] resin cements (luting materials) are classified as Type 2 materials and according to their activation modes, they can be further divided into three classes: Class 1- Self-curing; Class 2- External-energy-activated (physical activation), and Class 3- Dual cure.

Class 3 cements represent the majority of resin cements currently used in clinical practices, owing to their ability to be activated by physical and/or chemical methods. These are indicated in dentistry mainly because of their activation system capability, despite the fact that they can be affected by light attenuation caused by fixed prostheses interposition.[3,4,5,6,13]

It is largely known that different activation modes can increase or decrease the mechanical properties of dual cure resin cements. Changes in hardness, solubility and flexural strength are also directly related with the degree of monomer conversion.[14] However, the relationships between the activation modes and diametral tensile strength (DTS) as well as fatigue strength (FS) remain unclear.[15]

The FS of resin cements is one of the main reasons for failure of indirect restorations and is characterized by the propagation of slow cracks inside the cement line.[16] When mechanical fatigue is associated with lower monomer conversion of the polymer matrix, it can help the deterioration of the dental restoration.[17,18] Since the activation mode may change the degree of conversion of the monomer, it may also possibly change the mechanical behavior of dual cure resin cements when under fatigue since induces stress of the polymer cross-linking bonds.

Consequently, it seems appropriate to study the influence of the three activation methods, self-curing, dual cure activation with photoactivation executed directly in the cement and dual cure activation with photoactivation through porcelain, on DTS of dual cure resin cements subjected to a mechanical fatigue test.

Methodology

The dual cure resin cements used in this study and their characteristics are described in Table 1.

A ceramic disc of Empress Esthetic (Ivoclar Vivadent, Schaan, Liechtenstein shade A3) was used to simulate an indirect restoration. This ceramic disc was finished and polished with 600 and 1,200 grit silicon carbide sandpaper (Norton S.A. São Paulo, Brazil) under continuous water cooling. Next, the ceramic disc reached its final shape: 10 mm in diameter and 0.7 mm in thickness.

The four types of dual-cured resin cements were activated in three curing modes with different protocols as follows: self-curing (no curing light exposure), dual cure activation through a polyester matrix tape (direct light exposure), and dual cure activation with light exposure through the porcelain disc.

For the DTS tests, 240 resin cement samples were prepared. Each experimental group (Figure 1) was composed of 10 specimens (n = 10), and the groups were randomly divided according to their activation mode (self-curing, dual cure, and dual cure through ceramic) and fatigue testing.

Cylindrical samples with a 4 mm diameter and a 2 mm thickness (Figure 2) were prepared to assess the diametral tensile strength.

All dual-cured resin cements were manipulated in agreement with each manufacturer's instructions. After proper manipulation of all cements, one

Table 1. Materials and compositions.

Cement (Batch)	Shade	Manufactures	Filer ontent	Monomer
RelyX ARC (GFHL)	A3	3M ESPE, Sant. Paul, EUA	67.5 wt%	TEGDMA Bis-GMA
Enforce (908521 and 878707)	A3	Dentsply Caulk, York, EUA	66 wt%	TEGDMA Bis-GMA BHT
RelyX UNICEM (307690)	A3 Opaque	3M, ESPE, Seefeld, Germany	50 vol%	Phosphoralated methacrylates
Nexus 2 (2878561)	A3	Kerr, Corp. Orange, EUA	68 wt%	Bis-GMA UDMA TEGDMA

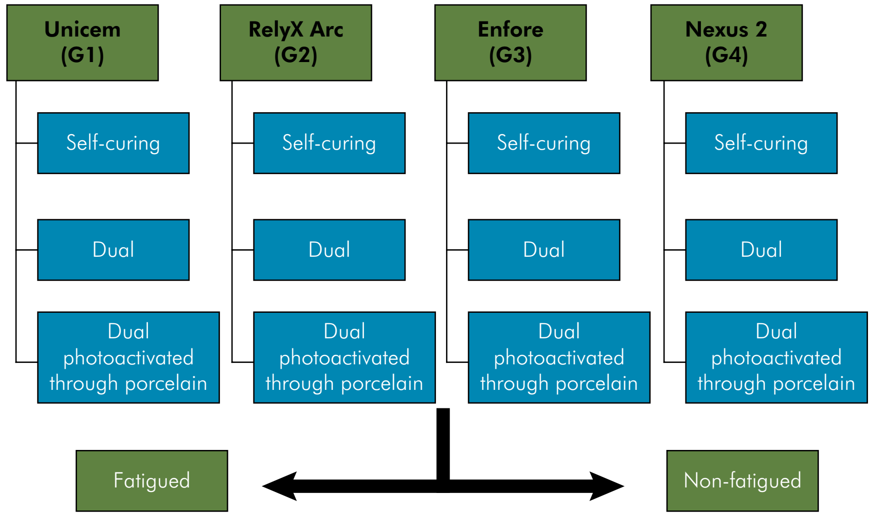

Figure 1. Experimental Groups where, G1 is Unicem, G2 is RelyX ARC, G3 is ENFORCE and G4 is Nexus 2; The second line is indicating activation mode Self-Curing, Dual Cure and Dual with photoactivation through porcelain (c).

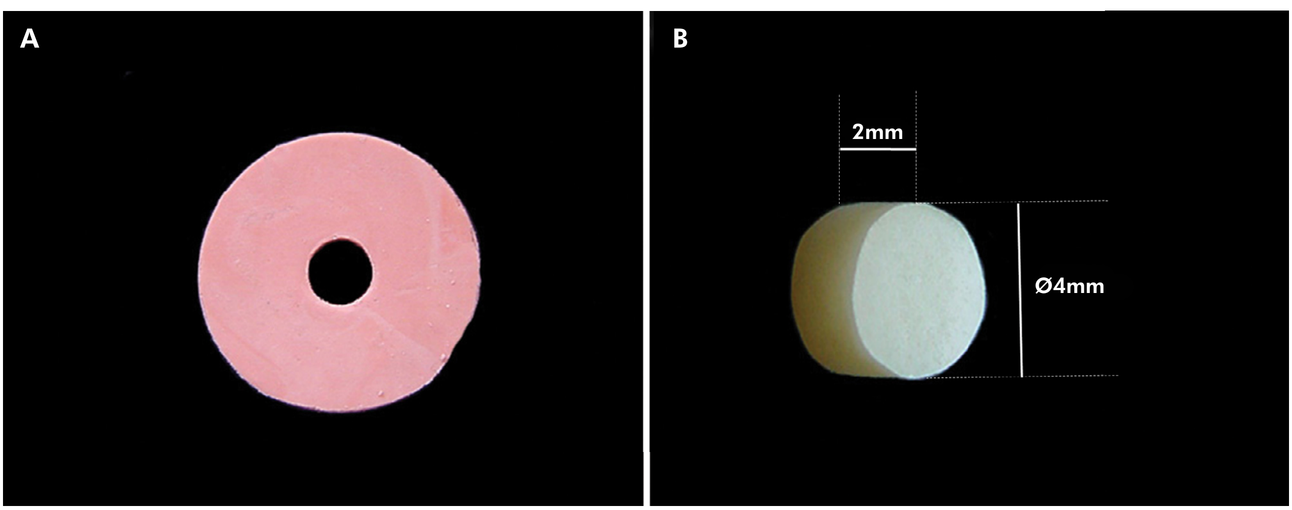

Figure 2. Rubber mold (a) and Test specimen geometry (b).

increment was inserted inside a rubber mold (Figure 3) that was previously placed on a glass plate covered with polyester matrix tape K Dent (Quimidrol, Joinvile, Brazil). Then, another polyester matrix tape and glass plate were placed on it in a sandwich format (Figure 3). For the subgroups in which only self-curing was required, the rubber mold, filled with the cement, remained without light exposure until the chemical cure time was terminated. For others, photoactivation was performed immediately after cement manipulation and both sides of the sample were light-cured (Figure 3).

Photoactivation was executed with the Ultra Lume Led 5 (Ultradent Products Inc., South Jordan, USA), with an irradiance of 1100 mW / cm^2, as specified by each cement manufacturer (Table 2).

One hour after the manipulation, all test specimens were removed from their rubber molds, finished with 1,200 grit silicon carbide sandpaper, and placed in distilled water for 23 hours at 37°C.

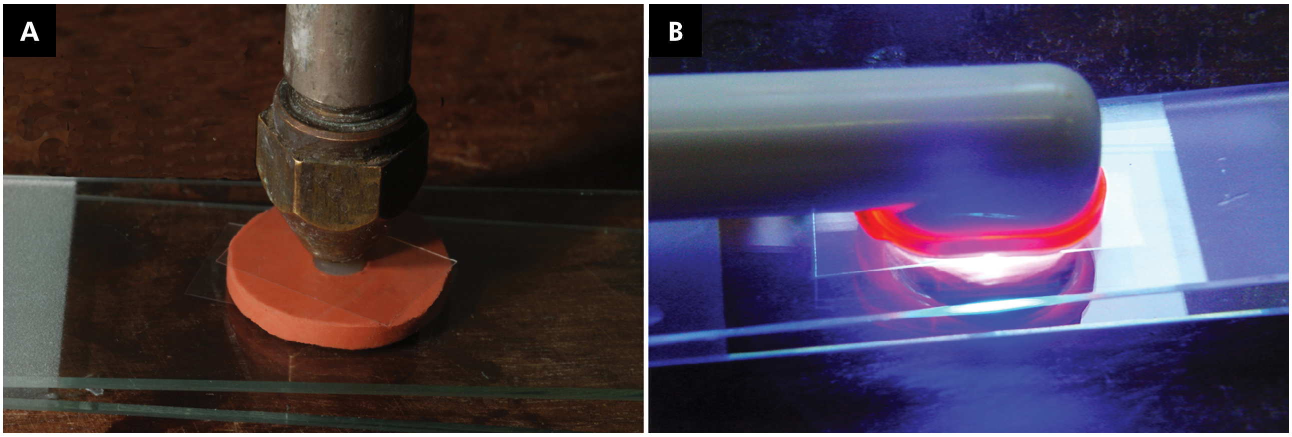

Figure 3. Rubber mold, filled with the resin cement (a) and Photoactivation (b).

Table 2. Activation modes for each resin cement.

Cement	Chemically-cured time (min)	Photo activation time (s)
RelyX ARC	10	40
Enforce	6	30
RelyX Unicem	10	20
Nexus 2	10	40

In summary, four groups were created according to each type of cement. Each group (n = 10) was subjected to three different activation modes, creating twelve subgroups.

The specimens of the twelve subgroups were randomly assigned to two fatigue conditions, creating two halves of the total number of specimens. One-half was subjected to a Mechanical Fatigue Test as showed in Figure 4. The other half (non-fatigued) remained continuously stored in distilled water at 37°C until the diametral tensile mechanical test.

The strength fatigue test (SFT) was performed by Mechanical Cycle Fatigue (ERIOS, São Paulo, Brazil). The specimens were subjected to 30,000 cycles with a 12 N load and 1 Hz frequency in distilled water at 37°C. The specimens that were not mechanically cycled remained in a lightproof container under water at 37°C in a kiln during all the SFT period. As a result, the Diametral Tensile Strength Test was performed at the same time for all specimens (fatigued and non-fatigued) after manipulation.

Before the diametral tensile strength test, the specimens' dimensions (diameter and thickness) were verified four times, and the average values were recorded and used to calculate the Mega Pascal (MPa) values from the Newton (N) data, as described in Equation 1.

$$\text{Diametral tensile strength (MPa)} = 2P / \pi DT \qquad \text{Equation 1}$$

Where, P is the maximum applied force (N); D is the diameter of the measured sample (mm); and T, the thickness of the sample (mm).

DTS testing was executed in an Instron, model 4411, Universal Testing Machine (Instron Inc. Canton, EUA). The compression force was employed with acrosshead speed of 0.5 mm/min and applied in a position parallel to the diameter of the specimens (specimens were positioned vertically) until failure, as shown in Figure 5. All DTS data in MPa were subjected to a factorial three-way analysis of variance (ANOVA) for cement type, activation mode, and mechanical fatigue (p = 0.05). The relationships and differences found between the groups were analyzed by a Tukey's Test at a 5% level of significance.

Results

The three-way ANOVA showed an interaction between the cement and activation modes (p = 0.00068). Mechanical cycling did not present a significant

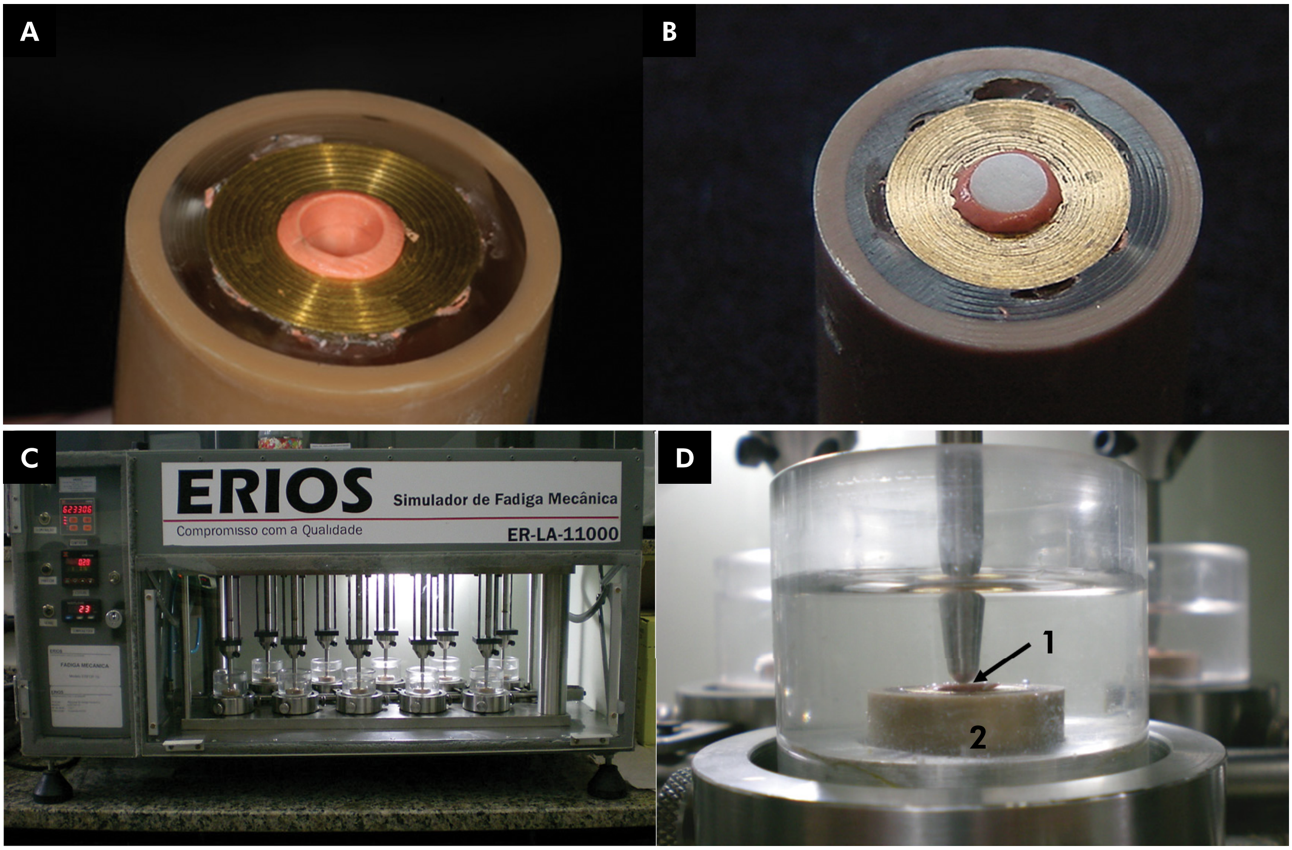

Figure 4. Joinable receptacle for strength fatigue simulator (a), positioned cement specimen at the receptacle (b), Erios mechanical cycle fatigue simulator (c) and specimen during mechanical fatigue. The black arrow indicated the cement disk position, 1 the receptacle is indicated by 2.

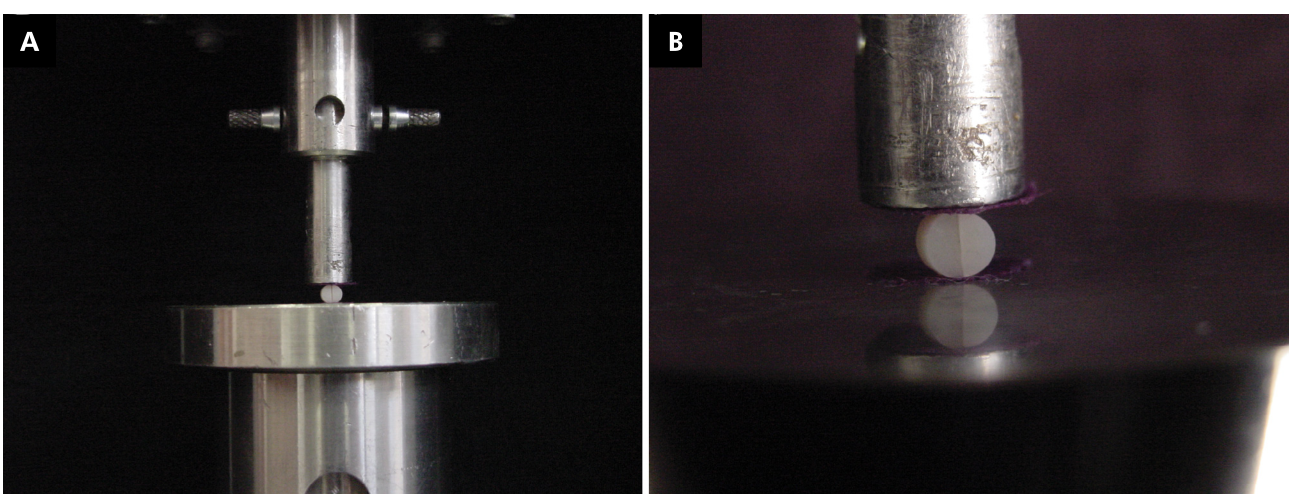

Figure 5. Diametral tensile strength test (a) and specimen after fracture, in detail (b)

interaction with the other factors (p = 0.64865). Likewise, the triple factor interaction was not significant (p = 0.15215).

Table 3 and Figure 6 show the results of statistical analysis; we concluded that the dual cure resin cement RelyX Unicem (3M, ESPE, Seefeld, Germany) had the

Table 3. Means of Diametral Tensile Strength (MPa) with standard deviations shown in parentheses.

Cements	Activation modes		
	Self-cured	Dual cure (Direct photoactivation)	Dual cure (photoactivated through porcelain disk)
Unicem	28.12 (5.29) cB	37.44 (6.49) bA	40.10 (4.39) cA
RelyX ARC	49.68 (8.42) bB	55.12 (5.16) aB	63.43 (6.92) aA
ENFORCE	49.12 (3.89) bB	56.42 (8.88) aA	56.96 (6.45) bA
Nexus 2	61.89 (11.21) aA	59.26 (9.47) aA	62.56 (10.93) abA

Means followed by distinct lowercase superscript letters at columns and distinct uppercase superscript letters at lines are statistically different at 95% level by Tukey's post hoc pairwise comparison test.

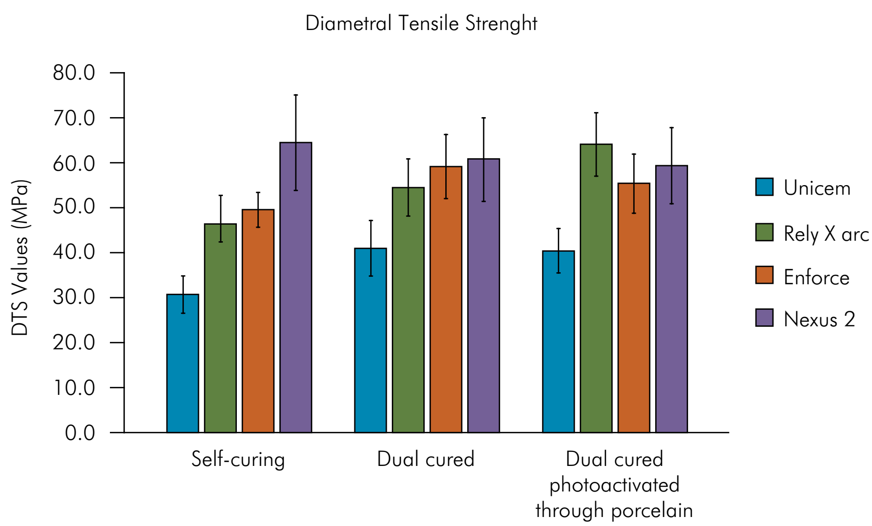

Figure 6. Mean diametral tensile strength.

lowest DTS value of all the activation modes. The Nexus 2 (Kerr, Corp. Orange, EUA) presented the highest DTS data, with no difference between the self-curing or dual cure activation. Additionally, no interference was observed from the porcelain disk interposition. The RelyX ARC (3M ESPE, Sant.Paul, EUA) and Enforce (Dentsply Caulk, York, EUA) had DTS values similar to that of Nexus 2 after the photoactivation was executed. The porcelain interposition (dual cure through porcelain disk) affected the DTS value of only RelyX ARC, which increased when the light was attenuated.

Discussion

Resin cement polymerization is the process of converting monomer molecules to form a polymeric network, thus improving its stiffness and consequently, its mechanical properties.[1]

Currently, the mechanical properties of resin cements are related to their monomer conversion degree (CD). However, other factors like inorganic particle types and amount, monomer types, diluent concentration, activation modes, type and number of inhibitors are also relevant, since these factors are related to the cross-linking density.[4,13,19]

Bragaet al.[20] investigated the influence of the curing method of dual-cured cements on other mechanical properties (flexural strength, flexural modulus and Knoop hardness) and no correlation was found between flexural strength and the Knoop hardness number, indicating "that other factors besides the degree of cure (*e.g.* filler content and monomer type) affect the flexural strength of composites."

Regardless of the fact that the DTS is not a reliable method for CD analysis, because a cement that has an organic resin matrix with a high ability to form cross-linking reactions will present a high DTS despite its low CD. That is because t cements with an organic resin matrix form polymers with a high mechanical strength compared to other cements formed by a less branched polymeric net.[21] DTS is a very reliable test for assessing the mechanical properties of dental composites.

Chung and Grener[22] found that composite materials with the same composition, when polymerized by different activation modes, result in different polymer chain conformations, with a shift in their mechanical properties (DTS and Knoop hardness number), which is independent of the conversion degree obtained.

The main aim of dual cure resin cements is to allow the effect of activation, despite the restoration light attenuation. The expected result of this study was to show that all cement types used behaved in a way similar to Nexus 2, which had the same DTS value in the three different activation modes. However, our results showed that Nexus 2 presented the highest DTS value. This can be explained by its organic matrix composed of the UDMA that, in accordance with Yoshida et al.,[23] results better mechanical properties. Therefore, our results are also in agreement with the higher Nexus 2 DTS values described by Meng et al.[24]

In our study, the dual cure resin cement RelyX Unicem had the lowest DTS value compared with all other cements in all three activation modes. This observation may be because of its composition since it was the only self-etching cement used and because part of its inorganic particles are not silanized, which helps the formation of stress concentration sites in these test specimens[25]. This self-etching cement is composed of only 50% inorganic particle content[26], which in accordance with Kim et al.,[27] is not enough to give an effective DTS to dental resins. Moreover, the organic matrix of this self-etching cement has been related to lower stiffness and strength of the final polymer.[28,29]

The RelyX Unicem cement has an organic matrix formed by acidic monomers that interact with the cement powder and dental structure, allowing buffering of the initial acidity of the material and chemical bonding to the tooth, respectively. Thus, in the chemically polymerized RelyX Unicem group, the lowest values of DTS were directly related to the lower power of its activation system (by oxidation-reduction reactions) in an acidic environment. Besides, according to Piwowarczyk and Lauer[28] and Holderegger et al.,[29] because of their unique characteristics and functions, the organic matrix of such self-etching cement seems to have lower stiffness compared to other resin cements.

The DTS values of Enforce and RelyX Unicem cements showed that chemical activation created a final polymer with structural characteristics such that maximum DTS could not be achieved. This may have resulted from the fact that chemical activation is slower than photoactivation and is dependent on the diffusion of molecules and activated free radicals. These reactions produce a lower density of crosslinks, resulting in a less resistant polymer dependent on a sufficient amount of self-curing activation system.[30,31,32,33]

The RelyX ARC high photoactivation potential probably was the main reason for the maximal values of DTS reached during indirect photoactivation and for its lower DTS when only self-curing or photoactivated without porcelain interposition. This could be justified by a negative interaction between the physical and the chemical activation systems.[30] Furthermore, photoactivation could have increased the cement viscosity during the polymeric chain propagation state, restring the self-curing activation system. This could be explained by the fact that in the RelyX ARC dual cure resin cement, the same tertiary aliphatic amine used during photoactivation is also used during its self-curing. Consequently, the high irradiance offered during direct photoactivation probably consumed a big amount of the tertiary aliphatic amines, leaving an insufficient amount of tertiary aliphatic amines for the self-curing activation system. Moreover, the high physical activation occurring during direct photoactivation had a role in the polymeric chain length. The larger number of photons delivered to the cement originated more activation centers that blocked polymeric chain growth, resulting in a polymer with shorter polymeric chains and inferior mechanical properties.[4,8,34,35]

Mechanical fatigue did not affect the DTS values for any cement in this study. Therefore, it can be concluded that under the fatigue conditions used, all the materials presented good quality cross-linked bonds resulting in an immediate Fatigue Strength.

Therefore, this study showed a significant difference between the mechanical behavior of the dual cure resin cements RelyX ARC, Enforce, Nexus 2, and RelyX Unicem due to their distinct chemical composition. Since we only evaluated two mechanical properties, the complete behavior of these cements over different activation modes could not be properly analyzed, demanding other properties to be studied to corroborate our findings.

Conclusions

The analysis of the present data allowed us to conclude the following:

a. For all cements tested, self-curing did not result in maximum DTS values, except in Nexus 2.
b. Porcelain disc interposition did not interfere with the DTS values for most of the cements evaluated. This resulted in changes only for the RelyX ARC cement, which showed an increase in DTS with disc interposition.
c. The mechanical fatigue cycle did not affect the DTS values under the current study conditions.

Acknowledgements

Based on a dissertation subjected to the Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, University of Campinas-UNICAMP, as partial fulfillment of the requirements for the M.S. degree. This study was possible thanks to the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) scholarship conceded to Dr. Paes.

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