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Degree of conversion and cohesive strength of conventional dual resin and self-adhesive cements by using of different forms of activation

Grau de conversão e resistência coesiva de cimentos resinosos duais convencionais e autoadesivos através de diferentes formas de ativação

Grado de conversión y fuerza cohesiva de cementos de resina dual convencionales y autoadhesivos a través de diferentes formas de activación

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Abstract

The aim of this study was to evaluate the degree of conversion (DC) and cohesive strength (CS) of two conventional resin cements and a self-adhesive resin cement using different forms of activation. Two conventional dual resin cements (Variolink II and RelyX ARC) and a self-adhesive resin cement were polymerized in three different ways of activation, namely Chemically Activated (CA), Light Activated with Interposition of a resin disc (LI) and Light-Activated (L). The DC was evaluated using Fourier Transformed Infrared Spectroscopy - FTIR (Spectrum 100). In the CS test, the samples were tested until fracture in a universal testing machine (Instron 4411). The mean values obtained in the DC and CS tests were evaluated using two-way analysis of variance (ANOVA) (cement and activation) and Tukey test ($p \leq 0.05$). Irrespective of the activation mode, RelyX ARC showed the highest DC values, RelyX U200, the lowest values, and Variolink II intermediate values. Group L always had higher for DC and CS values than group CA. Among the resin cements, irrespective of the activation mode, there were no differences in CS values. The chemical activation mode of dual resin cements led to lower DC and CS values. RelyX ARC cement had the highest DC values and RelyX U200 cement the lowest values, however, in relation to CS, there were no differences.

Keywords: Resin cements; Polymerization; Physical properties.

Resumo

O objetivo deste estudo foi avaliar o grau de conversão (GC) e a resistência coesiva a tração (RC) de dois cimentos resinosos convencionais duais e um cimento resinoso autoadesivo usando diferentes formas de ativação. Dois cimentos resinosos convencionais (Variolink II e RelyX ARC) e um cimento resinoso autoadesivo foram polimerizados sob três diferentes formas de ativação nomeados como Quimicamente Ativado (QA), Fotoativado com Interposição de um disco de resina (FI) e Fotoativado (F). O GC foi avaliado usando espectroscopia infravermelho transformada de Fourier - FTIR (Spectrum 100). No teste de RC, as amostras foram testadas até a fratura em uma máquina de ensaio universal (Instron 4411). Os valores médios obtidos nos testes de GC e RC foram avaliados utilizando análise de variância (ANOVA) de dois fatores (cimento e ativação) e teste de Tukey ($p \leq 0,05$). Independentemente do modo de ativação, RelyX ARC obteve os maiores valores de GC e RelyX U200 os menores valores e o Variolink II valores intermediários. O grupo dos F sempre obtiveram maiores valores que o grupo dos QA

para GC e RC. Entre os cimentos resinosos, independentemente do modo de ativação, não existiu diferenças nos valores de RC. O modo ativação química dos cimentos resinosos duais levou a menores valores de GC e RC. O cimento RelyX ARC obteve os maiores valores de GC e o cimento RelyX U200 os menores valores, entretanto, em relação a RC, não houve diferenças.
Palavras-chave: Cimentos de resina; Polimerização; Propriedades físicas.

Resumen
El objetivo de este estudio fue evaluar el grado de conversión (GC) y la resistencia a la tracción cohesiva (RC) de dos cementos de resina dual convencionales y un cemento de resina autoadhesivo utilizando diferentes formas de activación. Se polimerizaron dos cementos de resina convencionales (Variolink II y RelyX ARC) y un cemento de resina autoadhesivo bajo tres formas de activación diferentes denominadas Químicamente Activadas (QA), Fotoactivadas con Interposición de un disco de resina (FI) y Fotoactivadas (F). La GC se evaluó utilizando espectroscopia infrarroja por transformada de Fourier - FTIR (Spectrum 100). En la prueba RC, las muestras se probaron para fracturar en una máquina de prueba universal (Instron 4411). Los valores medios obtenidos en las pruebas GC y RC se evaluaron mediante análisis de varianza de dos factores (ANOVA) (cemento y activación) y prueba de Tukey ($p \leq 0.05$). Independientemente del modo de activación, RelyX ARC obtuvo los valores GC más altos y RelyX U200 los valores más bajos y los valores intermedios Variolink II. El grupo F siempre obtuvo valores más altos que el grupo QA para GC y RC. Entre los cementos de resina, independientemente del modo de activación, no hubo diferencias en los valores de RC. El modo de activación química de los cementos de resina dual condujo a valores de GC y RC más bajos. El cemento RelyX ARC tuvo los valores más altos de GC y el cemento RelyX U200 los valores más bajos, sin embargo, en relación al RC, no hubo diferencias.
Palabras clave: Cementos de resina; Polimerización; Propiedades físicas.

1. Introduction

Resin cements are classified according to their activation mode into chemically activated, light activated and dual activated (light and chemically activated types) (El-Mowafy et al., 1999).

The chemically activated systems are presented in a paste-paste system, and are indicted for the cementation of thick and meta restorations) (Braga et al., 1999).

Light activated resin cements require photoinitiators such as camphorquinone to enable polymerization to occur. Their advantage is the prolonged working time and color stability (El-Mowafy et al., 1999). Their use is limited to situations in which the light is capable of crossing the restorative material and light activate the resin cement, such as laminated veneers, and thin inlays and onlays.

However, the resin cement most frequently used in clinical practice is the dual polymerization type (light and/or chemically activated). In this type of resin cement, the polymerization reaction occurs in the presence of light, or by means of the mechanism of self-polymerization without the presence of light (De Munck et al., 2004). These cements are indicated for cementation of esthetic intraradicular posts, inlays, onlays and total crowns both made of ceramics and of composites. The aim of the mechanism of dual polymerization is to compensate an eventual attenuation of light due to the thickness of the prosthetic part (Hofmann et al., 2001).

The dual activation cements were developed seeking the advantages of the light activated cements such as greater color stability and control of working time; with the possibility of achieving polymerization even when there is deficiency or absence of light, the main advantage of chemically activated cements. However, the majority of dual activation cements depend on the presence of light in order to achieve an adequate degree of conversion (El-Badrawy & El-Mowafy, 1995).

In addition to the possibility of using different forms of activation of dual resin cements, some years ago the composition of these cements was changed with the aim of reducing the quantity of application steps with the development of self-adhesive cements (Radovic et al., 2008). The ease of the application technique is the main characteristic of this system, as it dispenses with acid etching and thus making it easier for the clinician, because the conventional resin cements need various steps for the cementation of ceramic and indirect composite restorations: the use of acid etching, primer, adhesives and

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application of resin cement (De Munck et al., 2004).

Considering this scenario of great versatility with the use of conventional dual and self-adhesive systems, the importance of the process of conversion of the material in the quality of polymerization, because an inadequate conversion could lead to a low resistance to microleakage, greater sorption, solubility and color instability, thereby leading to a greater chance of failures in cementation and loss of the restoration (Ferracane, 1985). The aim of this study was to evaluate the degree of conversion (DC), cohesive and tensile strength (CS) of two conventional dual resin cements and a self-adhesive resin cement with different forms of activation: Chemically Activated (CA), Light Activated with Interposition of a resin disc (LI) and Light Activated (L).

The hypothesis tested was that there would be no significant differences in the DC and CS of the materials tested, irrespective of the form of activation.

2. Methodology

The materials used in this study were two dual resin cements (RelyX ARC 3M ESPE and Variolink II Ivoclar Vivadent) and a self-adhesive resin cement (RelyX U200 3M ESPE).

2.1 Interposition of indirect resin composite

An indirect resin composite disc was made of - Sinfony (3M ESPE, St. Paul, Minnesota, USA), shade A3, and used as interposition between the light and resin cement. A disc, 11 mm in diameter was obtained according to the manufacturer's instructions. After this, the specimen was polished with humidified SiC carbonate abrasive papers in the granulations of 400, 600, and 1200 until a thickness of 2 millimeters was obtained.

2.2 Light Source

A light source of the light emitting diode (LED) type, Freelight 2 (3M ESPE, St. Paul, Minn., USA) was used in this study. For the light activation procedures, it was connected to a tension stabilizer, and the level of irradiance was verified with a digital power meter (Ophir Optronics, Danvers, Massachusetts, USA). The value of irradiance transmitted through the indirect composite resin disc was also verified. Furthermore, the spectral distribution of the light transmitted through the indirect resin composite sample was obtained by using a spectrometer (USB2000, Ocean Optics, Dunedin, Fl., USA) and compared with the profile of control (light source without interposition of the resin disc).

2.3 Analysis of Degree of Conversion (DC)

The degree of conversion (DC) of the resin cements were evaluated using Fourier Transform Infrared Spectroscopy (Spectrum 100 Optica; Perkin Elmer, MA, USA). In total 36 DC readouts were made (n=4). Equal volumes of base paste and catalyzer paste of the cementation agents were mixed for 20 seconds, and afterwards inserted into a circular mold (internal diameter 7 mm and 1 mm high) then covered with a strip of transparent polyester. The indirect composite resin disc was placed on the polyester strip and light activated for 40 seconds with the tip of the light guide placed in contact with the disc. This group was denominated Light Activated with Interposition (LI). Light activation was also performed without interposition of the resin disc, and this group was denominated Light Activated (L). Lastly, no light activation was performed, allowing the sample to react chemically only. This group was denominated Chemically Activated (CA).

2.4 Cohesive and Tensile Strength Test

In order to obtain specimens for the cohesive and tensile strength test, a silicone mold in the shape of an ampule was produced. This mold measured 11 mm long, 2 mm wide, 1 mm high and the narrowest region had a width of 1 mm. The dual resin cements were inserted directly into the mold with a spatula, so that the entire mold was filled. A Mylar strip was used to cover the resin, and afterwards compressed, discarding the excess resin with a spatula. For group L, light activation was performed for 40 seconds, using the light activator tip placed directly onto the Mylar strip and resin cement. For group LI, the light activator tip was placed directly onto the resin disc, and for group CA, no light activation was performed. A total of 90 samples were fabricated, so that for each group, 10 samples (n=10) were made. After polymerization, each ampule-shaped resin composite sample was carefully removed from the mold, smoothly polished with a strip of SiC abrasive paper with granulation 2000, to remove some rough edges and then stored in distilled water at 37°C for 24h. For performing the cohesive strength test, a Universal test machine (Instron, Model 4411, Instron, Canton, MA, USA), equipped with a load cell of 500 N, was used at a speed of 0.5 mm/min. All the samples were tested until they fractured. The cohesive strength to traction, in MPa, was calculated by dividing the value of force (Kgf) obtained in the Universal test machine by the area of the region that fractured (cross section of the narrowed region), previously determined by using a digital pachymeter.

The mean DC and CS values were evaluated statistically using the 2-way (Cement and Activation) analysis of variance (ANOVA). Comparisons were made by the Tukey test ($p \leq 0,05$).

3. Results

Table 1. Means (standard deviation) of the degree of conversion - DC (%) of dual resin cements when chemically activated and light activated with or without the interposition of an indirect resin composite disc.

Dual resin cement	Chemically Activated (CA)	Light Activated with Interposition (LI)	Light Activated (L)
RelyX ARC	65.4 (1.0) A, b	68.3 (0.5) A, ab	71.1 (1.5) A, a
Variolink II	56.2 (2.0) B, b	57.8 (3.4) B, b	63.8 (2.5) B, a
RelyX U200	38.7 (1.5) C, c	46.0 (2.4) C, b	54.7 (0.4) C, a

Means followed by different capital letters in the same column and lower-case letters on the same line, differ significantly ($p < 0.05$). Source: Authors.

Among the resin cements, irrespective of the mode of activation, RelyX ARC obtained the highest DC values, and RelyX U200, the lowest values. Variolink II showed intermediate values. The Light Activated group always obtained higher values than the Chemically Activated group. The Variolink II and RelyX U200 cements obtained the highest values in the Light Activated group (Table 1).

Table 2. Means (standard deviation) of cohesive and tensile strength - CS (MPa) of dual resin cements when chemically activated and light activated with or without the interposition of an indirect resin composite disc.

Dual resin cement	Chemically Activated (CA)	Light Activated with Interposition (LI)	Light Activated (L)
RelyX ARC	17.5 (12.2) A, b	19.9 (2.8) A, b	45.4 (1.7) A, a
Variolink II	12.3 (2.6) A, b	30.7 (10.5) A, b	50.7 (18.9) A, a
RelyX U200	5.7 (4.8) A, b	13.8 (5.1) A, ab	25.9 (6.7) B, a

Means followed by different capital letters in the same column and lower-case letters on the same line, differed significantly ($p < 0.05$). Source: Authors.

Among the resin cements, irrespective of the mode of activation, there were no differences in the CS values, with the exception of the RelyX U200 cement, in which the Light Activated group showed the lowest CS values (Table 2).

The RelyX ARC and Variolink II cements obtained the highest CS values in the Light Activated group and the lowest values in the Chemically Activated group, and Light Activated with Interposition group. The RelyX U200 cement obtained the highest CS values in the Light Activated group and the lowest values in the Chemically Activated group and showed intermediate values in the group Light Activated with Interposition (Table 2).

4. Discussion

The results of this study showed that dual polymerization of the resin cements used resulted in an increase in the degree of conversion (DC) and cohesive strength (CS). Therefore, the hypothesis of the study was rejected.

This showed that chemical activation is not sufficient to provided the cement with adequate properties (Chen et al., 2016; El-Mowafy et al., 1999; Jang et al., 2017; Kim et al., 2017; Majumder et al., 2019; Niemi et al., 2020; Shim et al., 2017) because, as its polymerization reaction is slower (Inokoshi et al., 2021), it influences the formation of the polymeric chain by generating long, linear chains. This generates inadequate conversion, interfering in the mechanical properties (Braga et al., 2002), because to obtain appropriate polymerization, even in areas where there is a scarcity of light, a dual resin cement needs to attain similar vales of DC by both chemical activation and light polymerization (El-Mowafy et al., 1999) contrary to one of our expectations that chemical polymerization would be capable of compensating the attenuating effect of light, by chemically polymerizing regions that were remote from the preparation, where the light was deficient (Chen et al., 2016; Hofmann et al., 2001).

Moreover, RelyX ARC and Variolink II were observed to obtain higher DC values than RelyX U200, irrespective of the mode of activation. RelyX U200 is a self-adhesive cement and has hydrophilic acid monomer in its composition, which play an important role in control of the chemical reaction of polymerization (Pegoraro, 2010). The acid monomers have been shown to negatively affect the DC of dual materials, in both the chemical mode and dual activation mode (Vrochary et al., 2009). In their dual polymerization mode, they appear to interact chemically with the co-initiator (amine) present in the dual cements (Frasseto et al., 2012). To compensate for this incompatibility, an activator/initiator system has been included in their composition, such as sodium sulphate or aryl borate salts (Suh et al., 2003). However, this different system of initiation resulted in a different polymerization behavior, involving low initial values of conversion (Vrochary et al., 2009).

Resin Cements such as RelyX U200 have a high content of filler particles, therefore, their viscosity interferes with the mobility of the polymeric chain preventing its propagation. This leads to diminishing the reactivity of the radicals, so that they become trapped in the viscous chain of polymers, thereby slowing down the polymerization reaction. This may also explain the lower value of conversion. Retarded light activation may lead to a more satisfactory reactivity, allowing the mobility of the free radicals until the end of the polymerization process (D´Alpino et al., 2015).

Among the conventional cements, RelyX ARC showed a higher DC than that of Variolink II. The higher DC results obtained by the resin cement RelyX ARC occurred due to the fact that it has TEGDMA in its composition, which is a monomer with a low molecular mass and high mobility, thus allowing higher efficiency of the polymerization reaction and allowing the formation of cross links between the polymeric chains, thereby influencing the increase in the DC of this cement (Hardy et al., 2018; Peutzfeldt, 1997). However, more flexible monomers such as TEGDMA increase the DC but leave the polymeric chain less rigid and susceptible to factors of degradation (Watts, 2005). Whereas Bis-GMA, a monomer that is also a constituent of RelyX ARC, due to its high rigidity, increases the density of the cross links of the polymer chain in formation, and these are important in the formation of more resistant and homogeneous polymers.

However, these differences in DC among the different cements evaluated were not capable of influencing the results of CS. This probably occurred due to the differences in composition of these cements. The resin cement Variolink II has a higher filler particle content than RelyX ARC, and the greater viscosity of this cement affects the mobility of the polymeric chains, interfering in the DC. However, this filler content promotes a lower rate of shrinkage and improves the strength (Spinell et al., 2009).

The exception only occurred in group L, in which RelyX U200 had the lowest CS values. As the light activation in this group occurred without any interference, all the cements probably achieved the maximum polymerization reaction and, in this context, RelyX U200 showed the lowest CS values. This proves the need for retarding light polymerization of self-adhesive cements in which the different systems of initiation result in polymerization behaviors with low values of initial conversion (Vrochary et al., 2009). Because the cement has a high filler content, it reacts slowly to the polymerization reaction, and requires prolonged polymerization (D´Alpino et al., 2015). However, this increase in polymerization time could lead to the displacement of the part, generating maladaptation and increasing the risk of pulp toxicity by the acid monomers released (Schmid-Schwap et al., 2009; Spinell et al., 2009). Further to the polymerization of resin cements, Tosco et al. (2021) obtained similar DC values when they compared two different polymerization protocols. In the first, the samples were polymerized for 40 s, and in the second, the samples were polymerized for 5 s, and after 20 s, they were again polymerized for another 40 seconds, concluding that the clinician can safely use the protocol of step luting (5s + 40s) for cementing indirect restorations. This simplifies the removal of excess cement, particularly in the interdental space. In fact, we have seen the use of this protocol in several case reports (Ferro et al., 2021).

When the different modes of activation were tested, in group CA, the DC of the cements used in this research were shown to be lower than those in group L, showing the need for exposure to light. In fact, the result obtained in this research corroborated the findings of various studies (Alovisi et al., 2018; Arrais et al., 2008; Chen et al., 2016; El-Mowafy et al.,1999; Inokoshi et al., 2021; Jang et al., 2017; Kim et al., 2017; Majumder et al., 2019; Niemi et al., 2020; Shim et al., 2017) and demonstrated the dependence on light activation of dual cements, and this is probably related to the differences in the proportion between the initiator and accelerator. The type of amine used as accelerator may also have influenced polymerization (Caughman et al., 2001). Moreover, Variolink II and RelyX U200 showed lower DC values in the LI mode of activation in comparison with L, and once again the dependence on exposure to light of these cements was demonstrated, in order to achieve high DC values. Therefore, care must be taken with the distance between the light source and the cement, thickness (Hardy et al., 2018; Jang et al., 2017; Lise et al., 2018; Martins et al., 2019; Rizzante et al., 2018; Tafur-Zelada et al., 2021; Turp et al., 2018), color (Tafur-Zelada et al., 2021) , translucency (Liporoni et al., 2020; Shim et al., 2017) and type of restorative material of the prosthetic part (Lise et al., 2018; Majumder et al., 2019; Rizzante et al., 2018; Shim et al., 2017; Turp et al., 2018) to be cemented, since they could interfere in the DC, leading to low chemical stability, resulting in color instability and compromised mechanical properties.

In the CS test, group L was observed to have the highest values in all the cements tested. This proved that attenuation of the light intensity by the resin disc was an interfering factor in the final conversion (Watts & Cash, 1994), confirming the dependence on exposure to light of the dual cements. In fact, Bragança et al. (2020) demonstrated that the interposition of 0.5 mm of ceramic reduced the irradiance received by the resin cement by approximately 50%.

The resin cement RelyX U200 had an acceptable behavior of CS and therefore, is an alternative to the conventional cements. A higher value was achieved by the dual activation in relation to chemical activation, since the double polymerization is preferable, whenever possible.

The clinical significance of this study is in agreement with the studies of various authors and allow us to affirm that chemical polymerization is insufficient to enable conventional dual resin cements to attain complete polymerization, resulting in lower DC (Alovisi et al., 2018; Arrais et al., 2008; Chen et al., 2016; El-Mowafy et al., 1999; Inokoshi et al., 2021; Jang et al., 2017; Kim et al., 2017; Majumder et al., 2019; Martins et al., 2019; Niemi et al., 2020; Shim et al., 2017) and CS (Chen et al., 2016; Jang et al., 2017; Kim et al., 2017; Shim et al., 2017). Special clinical steps must be taken, such as increasing the time of polymerization to compensate the attenuated intensity of light during cementation of resin and ceramic restorations (Almeida et al., 2018; Hardy et al., 2018; Jang et al., 2017; Majumder et al., 2019; Shim et al., 2017; Shim et al., 2018; Turp et al., 2018). However, continuous polymerization with high irradiance for long periods will produce a higher temperature, so that pauses in polymerization are recommended in order to avoid an accumulation of energy (Shim et al., 2018).

5. Conclusion

The mode of chemical activation of dual resin cements produced the lowest DC and CS values.

The RelyX ARC cement obtained the highest DC values and the cement RelyX U200, the lowest values, however, in relation to the CS values, there were no differences between the groups.

Moreover, the data obtained for a given brand cannot be extrapolated for an entire material's category. Thus, further laboratory studies are recommended with other brands.

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