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Effect of polymerization activation mode and accelerated aging in the degree of conversion of self-adhesive resin cements

Efeito do modo de ativação da polimerização e envelhecimento acelerado no grau de conversão de cimentos resinosos autoadesivos

Efecto del modo de activación de la polimerización y envejecimiento acelerado en el grado de conversión de cementos resinosos autoadhesivos

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Francisco Fernando Massola Filho
ORCID: https://orcid.org/0000-0002-1726-8252
Faculdade São Leopoldo Mandic, Brazil
E-mail: drfranciscomfilho@gmail.com
Joissi Ferrari Zaniboni
ORCID: https://orcid.org/0000-0003-0172-9891
São Paulo State University, Brazil
E-mail: joissizaniboni@gmail.com
Leticia Ferreira de Freitas Brianezzi
ORCID: https://orcid.org/0000-0002-9488-9140
Centro Universitário Sudoeste Paulista, Brazil
E-mail: leticia.brianezzi@hotmail.com
Marcus Vinicyus Manoel da Silva
ORCID: https://orcid.org/0000-0003-2896-2773
Universidade Estadual Paulista "Júlio de Mesquita Filho", Brasil
E-mail: mavinicyus@gmail.com
Edson Alves de Campos
ORCID: https://orcid.org/0000-0001-9120-4305
Universidade Estadual Paulista "Júlio de Mesquita Filho", Brasil
E-mail: edson.campos@unesp.br
Amanda Gonçalves Franco
ORCID: https://orcid.org/0000-0003-0983-7539
University of Itaúna, Brazil
E-mail: amandagfranco38@gmail.com
Thiago Amadei Pegoraro
ORCID: https://orcid.org/0000-0003-2867-1239
Centro Universitário Sagrado Coração, Brasil
E-mail: pegthiago@gmail.com

Abstract
Indirect adhesive restorations are increasingly being part of the oral rehabilitation procedures. With the purpose to reduce and minimize the difficulties and simplify the cementing technique with conventional adhesive resin cements, it was introduced a resin cement called self-adhesive for presenting chemical adhesion to dental tissues, ceramic and metal surfaces without the need for etching and adhesive systems aplications. Some studies point out to the problems of degradation of cement during storage. The inhibitors and peroxides presented in the cement composition are organic chemical compounds and, therefore, are susceptible to degradation phenomena under storage. For all of these factors, the resin cements have limited storage period and changes in the mechanism of polymerization may occur during this period. The present study has been prepared containing some variables to research. All these variables are seeking to investigate the consequences of different activation protocols of polymerization and the accelerated aging of self-adhesive dual cured resin cements on the degree of conversion. As well as test the null hypothesis that the activation protocol of polymerization and the accelerated aging do not cause alteration in the degree of conversion of the material to be tested.
Keywords: Resin cements; Aging; Degree of conversion.

Resumo
Restaurações adesivas indiretas estão cada vez mais fazendo parte dos procedimentos de Reabilitação Oral. Com o propósito de reduzir e minimizar as dificuldades e simplificar a técnica de cimentação com cimentos resinosos adesivos convencionais foi introduzido um cimento resinoso denominado de autoadesivo por apresentar adesão química aos tecidos dentais, superfícies cerâmicas e metálicas sem a necessidade de condicionamento ácido e emprego de sistemas

adesivos. Alguns estudos apontam para a problemática da degradação dos cimentos durante a armazenagem. Os inibidores e os peróxidos presentes na composição do cimento são componentes químicos orgânicos e, assim sendo, são susceptíveis aos fenômenos de degradação sob armazenagem. Por todos esses fatores, os cimentos resinosos têm prazo de armazenagem limitado e alterações no mecanismo de polimerização podem ocorrer durante esse período. O presente estudo foi elaborado contendo algumas variáveis em investigação. Todas essas variáveis buscaram investigar as consequências de diferentes protocolos de ativação da polimerização e do envelhecimento acelerado dos cimentos resinosos autoadesivos de polimerização dual no grau de conversão dos mesmos, bem como testar a hipótese nula de que o protocolo de ativação da polimerização e o envelhecimento acelerado não causaram alteração no grau de conversão dos materiais a serem testados.
Palavras-chave: Cimentos resinosos; Envelhecimento; Grau de conversão.

Resumen
Las restauraciones adhesivas indirectas son cada vez más parte de los procedimientos de rehabilitación oral. Con el propósito de reducir y minimizar las dificultades y simplificar la técnica de cementación con cementos de resina adhesivos convencionales, se introdujo un cemento de resina denominado autoadhesivo porque presenta adhesión química a los tejidos dentales, superficies cerámicas y metálicas sin necesidad de grabado ácido y el uso de sistemas especiales pegatinas. Algunos estudios apuntan al problema de la degradación del cemento durante el almacenamiento. Los inhibidores y peróxidos presentes en la composición del cemento son componentes químicos orgánicos y, por tanto, son susceptibles de sufrir fenómenos de degradación durante el almacenamiento. Debido a todos estos factores, los cementos de resina tienen una vida útil limitada y pueden ocurrir cambios en el mecanismo de polimerización durante este período. El presente estudio fue elaborado conteniendo algunas variables objeto de investigación. Todas estas variables buscaron investigar las consecuencias de los diferentes protocolos de activación de la polimerización y el envejecimiento acelerado de los cementos resinosos autoadhesivos de polimerización dual sobre su grado de conversión, así como probar la hipótesis nula de que el protocolo de activación de la polimerización y el envejecimiento acelerado no causaron cambios en la conversión. el grado de conversión de los materiales a ensayar.
Palabras clave: Cementos resinosos; Envejecimiento; Grado de conversión.

1. Introduction

Indirect adhesive restorations are increasingly becoming part of oral Rehabilitation procedures. Clinical procedures include complete crowns, aesthetic veneers, inlays and onlays, endodontic posts and fixed prostheses, which are regularly fixed to the dental substrate using adhesive resin cements, with dual polymerization (Arrais et al., 2007; Carvalho et al.,2004; Caughman et al., 2001; El-Badrawy & El-Mowafy, 1995; Ferrari et al., 2001; Manso et al., 2011; Pedreira et al., 2009; Pegoraro et al., 2007; Souza Jr et al., 2013; Tezvergil-Mutluay et al., 2007).

Resin cements have become popular clinically for their ability to adhere to both tooth structure and restoration. These materials are basically modified composites available in three variations in relation to their form of polymerization development: they can be photo-activated, chemically activated, and also double or dual activated, that is, both by light and by the chemical portion of the material. The success of using conventional resin cements depends on several aspects related to the frustration of union between dental tissues and restorations (Czasch & Ilie, 2013; Manso et al., 2011; Pegoraro et al., 2007).

Recent literature has shown several aspects related to this type of material that were previously unknown regarding the behavior of these cements, as there are many important factors in determining the reliability of cementation procedures with adhesive resin cements. One of these factors is related to the possibility of incompatibility phenomena between simplified adhesive systems and resin cements with chemical and/or dual polymerization, with deep inspiration in clinical practice. This occurs through two main buttons, which are acidity and, consequently, the phenomenon of permeability of simplified adhesive systems (Carvalho et al., 2004; Manso et al., 2011; Pegoraro et al., 2007; Tay et al., 2003; Tay et al., 2002; Svizero et al., 2013).

With the purpose of reducing and minimizing difficulties and simplifying the cementation technique with adhesive resin cements, a resin cement called self-adhesive was introduced on the market in 2002 because it presents chemical adhesion to dental tissues, ceramic and metallic surfaces without the need for acid etching and use of adhesive systems (Oliveira et al., 2012; Manso et al., 2011). According to the manufacturer, the material consists of a universal resin cement with dual polymerization, self-etching and self-adhesive, indicated for cementation of metallic and ceramic indirect restorations, composite resins and metallic and aesthetic intraradicular posts.

Among several factors, the final quality of cements is crucial for the success of adhesive cementation techniques in prostheses. As a general rule, cementation should be carried out in order to achieve the maximum properties of the cements and their union with the adhesive agents. Then, the dual-polymerization resin cements must be mixed, applied and then light cured. A fotoativação imediata assegura a estabilidade inicial necessária para suportar tensões clínicas, e a polimerização química deverá assegurar o alcance de suas propriedades máximas ao longo do tempo. Even if triggered independently, the two polymerization mechanisms initiate a dynamic of free radical formation and conversion of monomers that naturally overlap during the polymerization phase (Blackman et al., 19990; Floyd & Dickens, 2006; Pfeifer et al., 2009; Arrais et al., 2014; Pedreira et al., 2009). Ideally, both activation pathways should generate reactions that complete each other, providing maximum material properties. However, it is reported that the stiffening of polymer chains initiated by photoactivation, which is faster, can hinder their spatial movement, preventing the subsequent completion of chemical polymerization, which is slower, resulting in a lower degree of conversion and consequent change in physical properties (Asmussen & Peutzfeldt, 2004; Peutzfeldt & Asmussen &, 2005). While it is generally accepted that all dual cements must be light-cured to achieve maximum properties, questions exist demonstrating that light-curing can compromise the degree of conversion and flexural strength of some dual cements (Rueggeberg, 1999; Velarde et al., 2005). In this case, it would be interesting to allow the dual cements to polymerize freely through the chemical reaction for as long as possible, allowing the structural flexibility of the chains in formation and increasing the degree of conversion, to later be photoactivated. Delayed photoactivation could then enhance the physical properties through further conversion of monomers via light activation. Statements recently published in the literature, such as that not all dual-polymerization resin cements can or should be light-cured to achieve maximum properties, raise questions about the real need for light-curing cements (Miller, 2004). On the other hand, other studies point to the fact that some dual cements only reach a high degree of conversion and superior physical properties when exposed to light, suggesting an inefficiency of the chemical pathway of activation and polymerization (Fonseca et al., 2005; Velarde et al., 2005). Interesting findings demonstrated that some dual-polymerization resin cements, when photoactivated (as recommended by the manufacturers), had the chemical polymerization mechanism and final properties compromised (Sharp et al., 2005). In a material with a dual polymerization mechanism, it is possible for the reactions to overlap or even for the formation of a chain triggered by photoactivation to cause spatial impediments for the same chain to participate in the chemical reaction. Variations of this nature can determine the formation of a polymer that presents, for example, a high degree of conversion, but low cross-link density (Asmussen & Peutzfeldt, 2004). It is interesting to note, however, that changes in the degree of conversion caused by different polymerization modes do not necessarily affect the physical properties of some materials (Asmussen & Peutzfeldt, 2004). This is probably due to the lack of directly proportional relationships between the degree of conversion and the polymer's cross-link density. With the exception of the consequences on the physical properties of the cement and its clinical performance, dual-curing resin cements that have a deficient self-curing mechanism or a self-curing mechanism that is compromised by photoactivation may exhibit changes in their bonding characteristics. The answer to this and other questions about the polymerization of cements can generate a change in the concepts of adhesive cementation and, mainly, in the clinical conduct of their use.

Other studies point to the problem of cement degradation during storage. It has been demonstrated that, after accelerated aging, some cements may present changes in their working and setting times (Sharp et al., 2005). It is known that dual-polymerization resin cements have their self-polymerization mechanism based on the REDOX reaction (oxidation-reduction), which occurs between benzoyl peroxide (catalyst paste) and tertiary aromatic amines (base paste). Allied to this, one or both of the pastes contain a photosensitive component in their composition, responsible for the light activation mechanism. The speed of the self-polymerization reaction is controlled by the presence of inhibitors, which are responsible for allowing an adequate working time after mixing the pastes, and by the relative amount of peroxide and tertiary amines responsible for the setting time and consequent polymerization development.

The inhibitors and peroxides present in the cement composition are organic chemical components and, therefore, are susceptible to degradation phenomena during storage (Pegoraro et al., 2007; Malacarne et al., 2006; Santerre et al., 2001). Due to all these factors, resin cements have a limited shelf life and changes in the polymerization mechanism can occur during this period. The changes result in a possibly unstable resin cement, causing a consequent change in the material's working and hardening time.From the above, it is clear the problem involving these types of materials and the role that chemical interactions resulting from polymerization and accelerated aging play in determining the physical properties of dual-polymerization resin cements.

The present study was prepared containing some variables under investigation. All these variables sought to investigate the consequences of different polymerization activation protocols and accelerated aging of dual-polymerization self-adhesive resin cements on their degree of conversion.

The objetive of this article is to evaluate the effects of different polymerization activation protocols and accelerated aging on the degree of conversion of self-adhesive dual-activation resin cements. And test the null hypothesis that the polymerization activation protocol and accelerated aging did not change the degree of conversion of the materials to be tested.

2. Materials and Methods

In order to facilitate the understanding of the different experiments that were carried out, this chapter was divided into two parts described below:

PART A: Describes the different polymerization activation protocols. These protocols were applied as a variable to determine their effects on the degree of cement conversion.

PART B: Describes the accelerated aging procedure. This variable was applied in Part A, evaluating the degree of cement conversion before and after accelerated aging, according to the different activation protocols.

Materials used

Self-adhesive resin cements with dual polymerization and a conventional adhesive cement with exclusively chemical polymerization were selected as control material, which are commercially available and routinely used in daily clinical practice and in studies described in the literature (Table 1).

Table 1 – Selected materials for the study.

Resin Cement / Polymerization Mode	Composition *	Mixing procedure *	Manufacturer
Multilink Speed® / Dual	Self-adhesive resin cement with inorganic glass particles, ytterbium fluoride (40% by weight), dimethacrylate and HEMA	System for mixing base and catalyst pastes using a syringe with a self-mixing tip, provided by the manufacturer.	Ivoclar Vivadent
Maxcem Elite® / Dual	Self-adhesive resin cement with inert mineral particles and ytterbium fluoride (69.9% by weight), methacrylate-based monomers with phosphorylated esters, activators, stabilizers and dyes	System for mixing base and catalyst pastes using a syringe with a self-mixing tip, provided by the manufacturer.	Kerr, Corp
RelyX U200 ® / Dual	Self-adhesive resin cement with 43.0% filler by volume (72.0% by weight), with an average size of 12.5 μm	System for mixing base and catalyst pastes using a syringe with a self-mixing tip, provided by the manufacturer.	3M ESPE
Set PP ®/ Dual	Self-adhesive resin cement with 35.0% by weight of methacrylate ester and 65% by weight of inorganic particle	System for mixing base and catalyst pastes using a syringe with a self-mixing tip, provided by the manufacturer.	SDI, Australia

*According to manufacturers' information. ** Used as control material. Source: Own Authorship.

PART A

Methods description: polymerization activation protocol

The materials were divided into three experimental groups according to the protocol used to activate the polymerization reaction:

- Control Group: all materials were handled according to the time described in the manufacturer's instructions. In this group, the dual activation cements were photoactivated after the mixing time recommended by the manufacturer, according to the photoactivation time described by the manufacturer. All specimens were evaluated immediately after the photoactivation period, to analyze the degree of conversion.

- Experimental Group 1: the cements were manipulated according to the time described by the manufacturer and left undisturbed for 15 minutes in a dark environment. After this period, the cements were photoactivated and immediately tested to assess the degree of conversion.

- Experimental Group 2: the cements were manipulated according to the time described by the manufacturer and their photoactivation was suppressed. The specimens were kept for 15 minutes in a dark environment and submitted, immediately after this period, to the degree of conversion test.

All procedures for manipulating the cements and making the specimens were performed in a light-free environment so as not to compromise the activation of the polymerization reaction early.

The power density of the photoactivation source was determined using a radiometer (Model 100 OptiluxRadiometer, SDS Kerr) before photoactivation of each specimen. A photoactivator source with power density above 500mW/cm2 (V.I.P. Junior, Bisco) was used. It is noteworthy that the manipulation of resin cements and the preparation of specimens were performed by a single operator.

PART B

Description of methods: accelerated aging

The cement "kits" underwent an accelerated aging process. This aging was performed by storing the kits in an oven (37º C) for 12 weeks. This period represents approximately the simulation of a period of approximately 9-10 months of storage at ambient temperature (Malacarne et al., 2006).

Elevated temperature storage challenges the stability of the components and offers the opportunity to verify the possible effects of this change on the respective material properties. The cements used in this study were initially tested and then subjected to the accelerated aging method. After aging, the cements were used again to produce new specimens, which were then submitted to the degree of conversion test, under the same activation protocols.

3.2.2 Description of methods: Degree of conversion

The degree of conversion was determined by Fourier transform infrared spectrometry (FT-IR) (Figure 1) using the total attenuated reflection (ATR) method (Spectrum One; Perkin Elmer, Beaconsfield Bucks, England). The device operated with a resolution of 4cm-1 and 32 scans of each spectrum, recording spectra between the limits of 4000cm-1 (by convention) to 400cm-1 (defined by the equipment) (Figure 2).

After mixing the resin cement base and catalyst pastes, the material mass was immediately placed on the surface of the ZnSe crystal of the ATR, with a depth of penetration of the material of 1.6mm and pressed with a glass cover slip, for an intimate contact of the material with the ATR crystal (Figure 3). Intimate contact prevents possible interference from the environment, such as humidity, temperature, vibrations and possible presence of oxygen between the material and the crystal (Feng L & Suh, 2006). Portions of resin cements were then activated according to different polymerization activation protocols. 5 readings were performed for each material and for each group/condition. The percentage degree of conversion was calculated using the absorption bands from the aliphatic carbon (C=C) double bond peak, located at the 1638 cm^{-1} point of the spectrum, and the carbon double bond peak (C= C) aromatic, located at the 1608cm^{-1} point of the spectrum.The readings were performed on polymerized specimens at times predetermined by the groups and also on non-polymerized specimens so that it could be used as a basis for calculation using the following equation (Blank, 2000):

$$GC\% = \left(1 - \frac{\text{C aliphatic / C aromatic}}{\text{C aliphatic / C aromatic}} \right) X\ 100\%$$

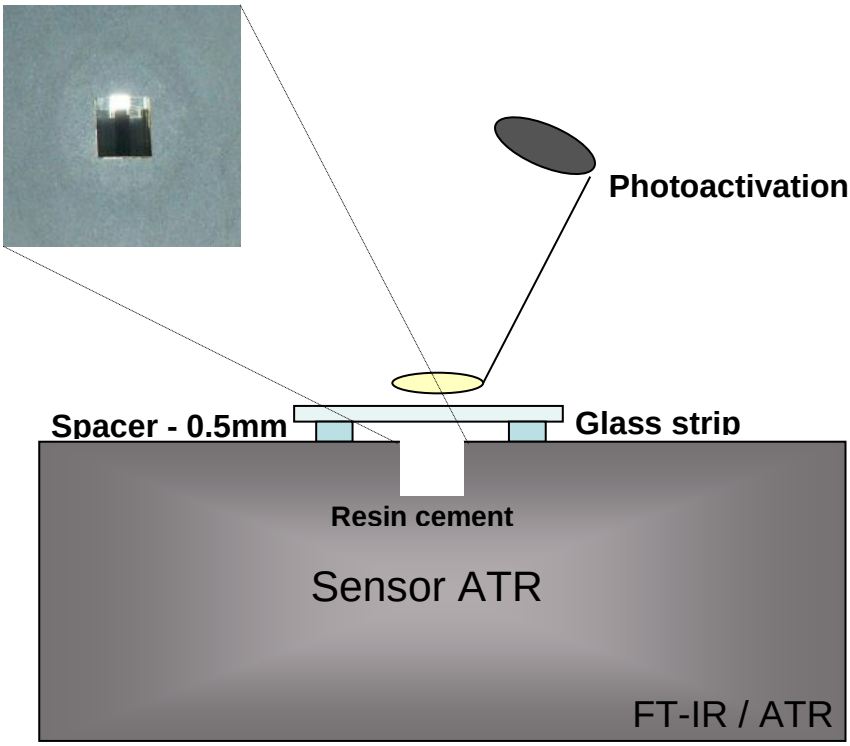
where:

Aliphatic C = absorption peak at 1638 cm^{-1} of the polymerized specimen.
Aromatic C = absorption peak at 1608 cm^{-1} of the polymerized specimen.
Aliphatic NC = absorption peak at 1638 cm^{-1} of the unpolymerized specimen.
Aromatic NC = absorption peak at 1608 cm^{-1} of the unpolymerized specimen.

Figure 1 - *Fourier-transform infrared spectroscopy* (FTIR).

Source: Own Authorship.

Figure 2 - ATR scheme.



Source: Own Authorship.

Figure 3 - Photograph of FT-IR ATR equipment.

Source: Own Authorship.

Data statistical treatment - Degree of Conversion

The mean values of the degree of conversion were described in percentage (%) and such values were tabulated through the total mean of each group/condition. Statistical analysis of the compiled data was investigated using two-way analysis of variance (ANOVA) and Tukey's test, in order to isolate each material used, to compare values of degree of conversion of different polymerization activation protocols with the condition aging. All statistical analyzes were performed using the Sigma Stat 2.5 statistical package (Jandel Scientific, USA) with a significance level of α= 5%.

3. Results and Discussion

The mean values of the degree of conversion were expressed in % ± SD and both factors, polymerization mode and aging significantly influenced the degree of conversion of the tested materials ($p<0.05$). The values of the degree of conversion varied, regardless of the material, time and mode of polymerization and aging, from 36.7 ± 2.7% to 80.0 ± 5.2% in the control group; from 59.3 ± 2.0% to 73.7 ± 5.3% in experimental group 1; and from 30.8 ± 2.2% to 70.6 ± 6.1% in experimental group 2. (Tables 1 and 2)

Em geral, o grau de conversão aumentou com o tempo após a ativação pela luz ($p<0.05$). O retarde da foto-ativação por 15 minutos causou alterações no grau de conversão final de alguns materiais testados, independentemente do envelhecimento ($p<0.05$). O processo de envelhecimento afetou a capacidade de polimerização química dos cimentos resinosos de polimerização dual ($p<0,05$). Portanto, o grau de conversão de cimentos resinosos de polimerização dual foi afetado significantemente pelo modo de polimerização e envelhecimento. A hipótese nula deve ser rejeitada. Alguns produtos não polimerizaram adequadamente sem a ativação pela luz (Experimental 2). (Table 3)

Table 1 - Averages and standard deviation of the tested materials (new) and their respective control and experimental groups.

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	83,7993483	75,4134409	71,604739
	84,6184802	76,0404259	60,7541082
Maxcem Elite®/ Dual	83,4660927	65,7655504	70,8795903
	70,6903651	71,4897707	77,3088612
	80,7993811	79,5890026	72,7358183
FINAL MEDIA	80,67473348	73,6596381	70,6566234
Standard deviation	5,153856638	5,266077367	6,074202909

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	40,47836273	72,71364758	65,67564976
	35,26547213	44,67631369	61,94586659
Set PP ®/ Dual	34,77410787	91,22517304	62,54725453
	33,38051796	61,50800177	41,17976369
	36,93841123	80,36473647	57,91168636
FINAL MEDIA	36,16737438	70,09757451	57,85204419
Standard deviation	2,724899371	17,87914381	9,72087548

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	59,2344481	60,48952562	25,91553378
	59,9185808	58,73399094	37,31625304
RelyX U200 ®/ Dual	60,49611421	62,15712042	42,12807746
	60,49611421	57,06452359	44,15225012
	53,6345514	58,2705295	21,08186553
FINAL MEDIA	58,75596174	59,34313801	34,11879599
Standard deviation	2,909713791	1,996643083	10,1525562

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	63,1175589	64,9933414	60,9818913
	64,0439211	66,1733454	45,7782061
Multilink Speed®/ Dual	63,4178438	67,5548389	38,5842202
	64,5028183	65,3590445	51,3398833
	65,1850618	69,7283356	63,3310137
FINAL MEDIA	64,05344078	66,76178116	52,00304292
Standard deviation	0,831031787	1,928126798	10,34644646

Source: Own Authorship.

Table 2 - Average and standard deviation of the tested materials (aged) and their respective control and experimental groups.

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	81,1117984	78,79652524	73,79075263
	82,19465171	82,06889241	58,65306501
Maxcem Elite®/ Dual	84,68395744	78,37049774	66,60118381
	76,2657465	86,5875042	53,4242827
	70,0956473	79,11461931	77,30886122
FINAL MEDIA	78,87036027	80,98760778	65,95562907
Standard deviation	5,780408098	3,452760122	10,0132895

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	27,9576834	37,4519023	50,07345623
	36,5469997	32,12782991	53,62146381
Set PP ®/ Dual	44,71325574	50,95551112	59,43293831
	37,35854785	47,87349542	53,56380991
	30,1290545	40,4219845	51,05768455
FINAL MEDIA	35,34110824	41,76614465	53,54987056
Standard deviation	6,615169161	7,665845585	3,637040479

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	59,2419382	59,2419382	31,00277128
	61,94115498	52,91611705	31,28959503
RelyX U200 ®/ Dual	61,59846152	57,85018057	33,68909389
	56,7654634	49,8645321	30,8723546
	51,9876345	45,8985746	27,4357123
FINAL MEDIA	58,30693052	53,1542685	30,85790542

Standard deviation	4,09956175	5,537045426	2,232408157

CEMENTS	CONTROL GROUP	EXPERIMENTAL 1	EXPERIMENTAL 2
	0	0	0
	0	0	0
Multilink N®/ Dual	0	0	0
	0	0	0
	0	0	0
FINAL MEDIA	0	0	0

Source: Own Authorship.

Table 3 - Results of degree of conversion % ± SD. **Control Group**: immediate photoactivation and CG reading; **Experimental Group 1:** CG reading after 15 minutes of activation of exclusively chemical polymerization and subsequent photoactivation; **Experimental Group 2**: CG reading after a total period of 15 minutes without further photoactivation;

Condition / Material	Control Group	Experimental Group 1	Experimental Group 2
MaxCem New	80,8 ± 5,2 A	73,7 ± 5,3 A	70,6 ± 6,1 A
MaxCem Aged	78,8 ± 5,9 A	80,9 ± 3,5 A	65,9 ± 10,1 A
Set PP New	36,7 ± 2,7 C	70,1 ± 17,9 A*	57,8 ± 9,7 B*
Set PP Aged	35,6 ± 6,6 C	41,7 ± 7,6 C	53,5 ± 3,6 B*
Rely X U200 Novo	58,7 ± 2,9 B	59,3 ± 2,0 B	34,1 ± 10,1 C*
Rely X U200 Aged	58,3 ± 4,1 B	53,1 ± 5,3 B	30,8 ± 2,2 C*
Multilink Speed New	64,1 ± 2,9 A	66,7 ± 1,9 A	52,0 ± 10,3 B
Multilink Speed Aged	0	0	0

Capital letters indicate statistically significant difference between new x aged conditions, by product; * (indicates statistically significant difference in the final polymerization of experimental groups x control polymerization condition). Source: Own Authorship.

It is described in the literature that an inadequate polymerization of resin cementing agents can be associated with clinical problems such as postoperative sensitivity, marginal infiltration and recurrent caries, susceptibility to degradation, discoloration and decrease in properties mechanics (Hofmann et al., 2001; Yap et al., 2000; Darr &Jacobsen, 1995; Van et al.,

1992; Bonfante et al., 2008; Cunha et al., 2007; Fonseca et al., 2004). An adequate degree of conversion of a luting agent is important for the overall clinical success, longevity and biocompatibility of the prosthetic restoration. Among the various ways to determine the polymerization quality of composites, Fourier transform infrared spectrometry (FT-IR) has proven to be a reliable technique to analyze the degree of conversion of monomers in dental composites and is widely used in studies of this nature (Feng L & Suh, 2006).

While some research suggests that the exclusively chemical polymerization pathway is not capable of producing the maximum hardness of dual cements other studies indicate that immediate photoactivation of dual cements may interfere with the chemical reaction pathway, preventing some cements from reaching their maximum properties if photoactivated (Velaverde et al., 2005; Sharp et al., 2005; El-Badrawy WA & El-Mowafy OM, 1995; Hasewaga et al., 1991).

The results of this study confirmed the behavior of the materials, which demonstrated, in general, constant conversion regardless of the mode of activation. Although the degree of conversion decreased significantly when the aged cement was polymerized exclusively by the chemical route (Experimental 2), suggesting alterations in the components of the REDOX reaction, it increased significantly in the control group where activation with light was used. This suggests that aging did not adversely affect the components responsible for the photoactivated reaction. Similar findings employing Raman spectrometry support this result. In our study, MaxCem cement showed the maximum degree of conversion when activated by light, regardless of aging. The degree of conversion significantly decreased after the aging process in experimental group 2, with exclusively chemical activation of the polymerization. This also suggests sensitivity of its components to the accelerated aging process, which must have compromised the REDOX reaction. The cement RelyX U200 showed a significant decrease in the degree of conversion after the final photoactivation in experimental group 1 and after the final time of exclusively chemical activation in experimental group 2, regardless of the storage condition. As with Multilink, the chemical route (REDOX) seems to be insufficient to produce minimally satisfactory polymerization in these materials, and the components appear to be highly sensitive to accelerated aging, probably at the stirring temperature of 37 ºC.

The polymerization potential of dual resin cements varies widely between products, and this variation was also confirmed in the present study (Hasegawa et al., 1991; Peutzfeldt, 1995; Rueggeberg &Craig, 1998). Incomplete conversion can result in unreacted residual monomers, which can lead to leaching into saliva, possibly producing allergic reactions or bacterial colonization around restorations (Carmichael et al., 1997; Hansel et al., 1998; Ilie & Hickel, 2009). They can also act as plasticizers, reducing the mechanical properties of the material (Pedreira et al., 2009).

Therefore, the degree of conversion was significantly affected by the activation mode of polymerization and aging of the dual resin cements, where some products tested did not cure sufficiently when photoactivation was omitted.

4. Final Considerations

The polymerization activation protocol and the accelerated aging caused significant alterations in the degree of conversion of the tested materials. The effects of the variables adopted were specific to each material and no systematic change in behavior was observed between them.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this article.

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