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Dual-Curing, Self-Adhesive Resin Cement: Influence of the Polymerization Modes on the Degree of Conversion and Microhardness

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RelyX™ Unicem, dual-curing self-adhesive resin cement, was tested in order to evaluate the degree of conversion (DC) and Vickers microhardness (VMH). The specimens were prepared into 4 groups (GR). For GR1, the specimens were irradiated with 500 mW.cm^{-2} during 20 seconds while for GR3 the same intensity was used but the irradiation time was 40 seconds. The GR2 followed the same polymerization mode of the GR1 but a ceramic disk was applied as a light barrier. For the last group, only the chemical polymerization was carried out. The DC and VMH were measured immediately after the irradiation time and again after 24 hours. Dual-polymerization (light and chemical polymerization) showed higher DC and VMH than the chemical polymerization (dark cure). The presence of a ceramic disk did not affect the DC but reduced the VMH. Increasing the exposure to light, the DC remained almost constant whereas the VMH raised slightly indicating that the VMH is quite dependent on the incident energy. Additionally, the powder/liquid portions of the cement were also characterized. The TG showed that the solid fraction was constituted of 96% of inorganic matter and 4% of vinyl-silane, a coupling agent. Regarding the liquid part, it was found that 84% was related to the mixture of monomers. The SEM revealed that the size particle of the inorganic fillers were between 400 and 11.000 nm. Factors such as energy density, size and distribution of inorganic fillers and the low effectiveness of the chemical cure contributed to the low values of the DC and VMH.

Keywords: *dual-curing cement, self-adhesive cement, degree of conversion, vickers microhardness*

1. Introduction

The use of restoration cements has been growing jointly with the increase of ceramics and composite resins for repairing teeth. Although these materials show high mechanical strength and remarkable esthetic appeal, the filling of the tooth cavity requires multiple steps and consumes a lot of time[1]. Self-adhesive resin cements have been developed to eliminate pre-treatment steps such as etching, priming and bonding when compared to multi-step composite cements. However, they need clinical and in vitro studies in the long-term, dimensional stability, their risks and benefits and so on. An important aspect to be considered is the monomer's degree of conversion which affects the physico-chemical properties including strength, stiffness, water absorption, biocompatibility as well as color stability. Post-operative sensitivity has also been reported as a possible consequence of inadequate polymerization of the resin cement[2-13]. The use of dual-curing resin cements intends to combine chemical and light-polymerization and at the same time allowing polymerization to take place in deeper areas of the tooth cavity applying shorter light irradiation time. Several studies reported that when photoactivation is omitted i.e. dark-curing took place and the material was unable to attain a high degree of monomer conversion[14-24]. Indeed, the degree of polymerization is still influenced when a light barrier simulating a porcelain indirect restoration is placed between the light source and the cement. Several studies showed adverse effects on the degree of conversion and microhardness of the dual-curing and light-polymerized resin cements with the increase of the thickness of the light barrier material[25-31].

The purpose of this study was to evaluate the degree of conversion and microhardness of one commercial dual-curing self-adhesive resin cement, named RelyX™, polymerized using different protocols, considering the polymerization mode, irradiation time and the use of a ceramic disk as a light barrier. Additionally, the powder/liquid portions of the cement were characterized in order to help the evaluation of the cement's behavior with regard to the polymerization conditions.

2. Experimental

2.1. Material and sample preparation

Cylindrical specimens (0.5 mm height and 8.0 mm diameter) were prepared in a Teflon® mold with RelyX™ Unicem Aplicap (shade A2, 3M® ESPE Dental Products, St Paul, MN, USA). The cement constituted two portions – one liquid and the other solid. The specimens were separated into 4 groups according to different preparation protocols. The degree of conversion and the Vickers

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microhardness were measured immediately after the irradiation time. After that, the specimens were kept in the light-proof box, at 37 °C, and after 24 hours those properties were also evaluated.

A ceramic disk similar to the microscopic glass slide (2.0 mm height and 10.0 mm diameter) was molded using IPS Empress 2 (shade A2, Ivoclar® Vivadent, Schan, Liechtenstein) following the manufacturer's instructions in order to be used as a light barrier simulating a porcelain indirect restoration.

For Group 1 (GR1), the specimen preparation followed strictly the manufacturer's instructions by placing the resin cement into the mold, covered with a Mylar strip and a microscope glass slide. After that, the cement was polymerized using a light curing unit (Optilux 400, Demetron, Kerr Corp, Orange, CA,USA) with 500 mW.cm^{-2} of light intensity, with an irradiation time of 20 seconds.

The specimens of Group 2 (GR2) were polymerized under the same conditions but the microscope glass slide was replaced by a ceramic disk in order to simulate a porcelain indirect restoration. The specimens of Group 3 (GR3) were obtained under the same conditions as those of Group 2 but the irradiation time was higher (40 seconds) with the purpose of assessing its influence on the degree of polymerization.

For Group 4 (GR4), the cement was inserted into the mold and then put into a light-proof box, kept for five minutes, then for 24 hours in order to ensure only chemical/dark curing took place, as the maker had suggested. For both reaction times, the degree of conversion and microhardness were measured.

2.2. Fourier transform infrared spectroscopy (FT-IR)

The degree of conversion was measured using Varian equipment model Excalibur 3100 equipped with an attenuated total reflectance assembly, with 100 scans and 4 cm^{-1} of resolution. The calculation of the degree of conversion was based on the ratio between the absorbances at 1637 and 1610 cm^{-1}, according to the previous work[32]. The results represented the average of five measurements.

2.3. Vickers microhardness (VHM)

Hardness is a general term which describes a combination of properties, such as the resistance to penetration, abrasion and scratching. It is an important parameter concerning the mastication and teeth' cleaning and they are the reasons for it large application in physical characterization of dental restoration. The Vickers microhardness test was carried out with a Carl Zeiss® Neophot 2 microdurometer, with load of 0.1 Kgf for 30 seconds. The results represented the average of five measurements.

2.4. Thermogravimetry (TG)

The thermogravimetry was carried out in a TA thermogravimetric analyzer model Q500 in the range of 30 – 700 °C, at 10 °C/min, under nitrogen atmosphere.

2.5. ^{13}C and ^{31}P nuclear magnetic resonance (NMR)

The nuclear magnetic resonance of carbon-13 (^{13}C NMR) of the solid and liquid portions of the cement was performed in Varian equipment model Mercury 300, at a frequency of 75.4 MHz. The portions were dissolved in deutered chloroform, analyzed at 25 °C, in order to estimate the chemical composition of the cement[32].

The nuclear magnetic resonance of phosphorus 31(^{31}P NMR) was also used to confirm the presence of combined phosphorus in the liquid portion of the cement.

2.6. Scanning electron microscopy (SEM)

The scanning electron microscopy was performed using JEOL® equipment, model JSM 5600, in order to observe the size and distribution of inorganic filler in the cement. A post-polymerized fragment was metallized with gold from which a photograph was taken.

3. Results and Discussion

3.1. Fourier transform infrared spectroscopy (FT-IR)

The solid portion of the cement indicated infrared absorptions at 1100 – 1000 and 800 cm^{-1} which were attributed to Si-O-C, S i-O-Si and Si-OH linkages. These absorptions are related to the presence of silica as one of the fillers and possibly vinyl-silane, used as a coating of the fillers in this portion, in view of the increase in the interfacial adhesion between the organic and inorganic phases. Relating to the liquid phase, the infrared absorptions were noticed at 1716, 1637, 1620, 1582 and 1510 cm^{-1} and in the interval between 1300 – 1000 and 900 – 650 cm^{-1}. These are related to the symetric and assymetric stretching of the ester carbonyl, double linkage of the vinyl monomer and stretching and bending of the C = C and C – H links of the aromatic ring. Thus, the liquid phase seems to contain monomers of the unsaturated ester type, and considering the absorptions with reference to the aromatic ring, one may conclude that the monomer Bisphenol-A diglycidylether methacrylate – Bis-GMA, usually employed as the main vinylic monomer in the formulation of dental restoration composites, is one of the monomers employed in the formulation of the cement[33-35].

3.2. ^{13}C and ^{31}P nuclear magnetic resonance (NMR)

In Figures 1 and 2, respectively present the nuclear magnetic resonance spectra of carbon-13 of the solid and liquid portions of the cement. In Table 1 are listed the main chemical displacements. In relation to the solid fraction, the chemical displacements could be attributed to the unsaturated carbon-carbon linkage and would correspond to vinyl-silane, confirming the FT-IR results. With reference to the liquid phase, the ^{13}C NMR revealed inumerous chemical displacementes. Comparing it with spectra of known samples of monomers usually employed in dental composite formulae, it was verified that the displacements – 18.0; 30.9; 41.5; 61.9; 65.3; 113.6; 126.2; 126.4; 127.5; 136.0; 156.4; 166.3 and 167.2 ppm – would be related to the monomer Bis-GMA while those at 18.0; 63.7; 125.6; 136.0 and 166.8 ppm characterize the presence of the monomer triethyleneglycol dimethacrylate – TEGDMA. The relative percentual of each monomer was calculated considering the peak areas of chemical displacements at 65.3 ppm (Bis-GMA) and 63.7 ppm (TEGDMA), according to the previously described procedure[32]. Thus, the monomeric fraction of the liquid fraction of the cement is 18.6% of Bis-GMA and 81.4% of TEGDMA. The higher amount of TEGDMA, almost 4 times higher than Bis-GMA, plays a role as diluent owing to its low viscosity[33-34]. Thus, a greater quantity of diluent in the monomeric composition aimed to facilitate the diffusion of monomers in the reactional medium and permit their penetration in deeper areas of the tooth cavity.

Further, in the liquid phase a nuclear magnetic resonance analysis of phosphorus-31 was performed, in an attempt to characterize the presence of organic phosphate in the cement (Figure 3). The analysis showed a broad peak where the maximum displacement occurred at 1.727 ppm, which could be attributed to the presence of a phosphorus-ester of the methacrylate monomer, which the finality would be to promote anchorage of the vinylic monomer to the tooth surface.

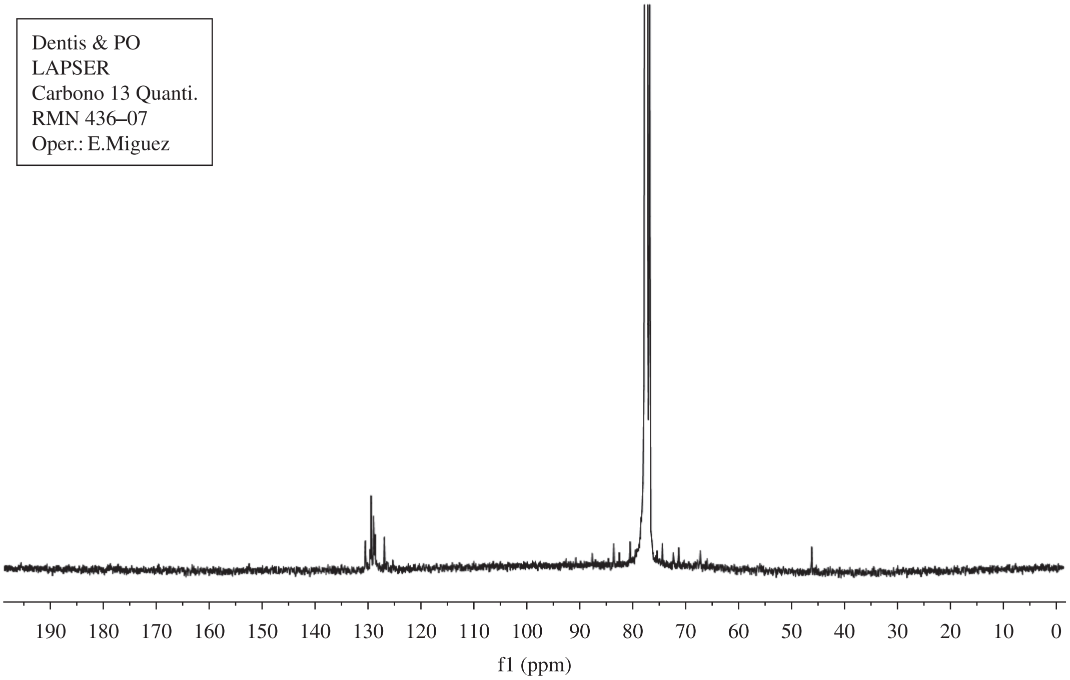

Figure 1. ^{13}C NMR spectrum of the cement solid portion.

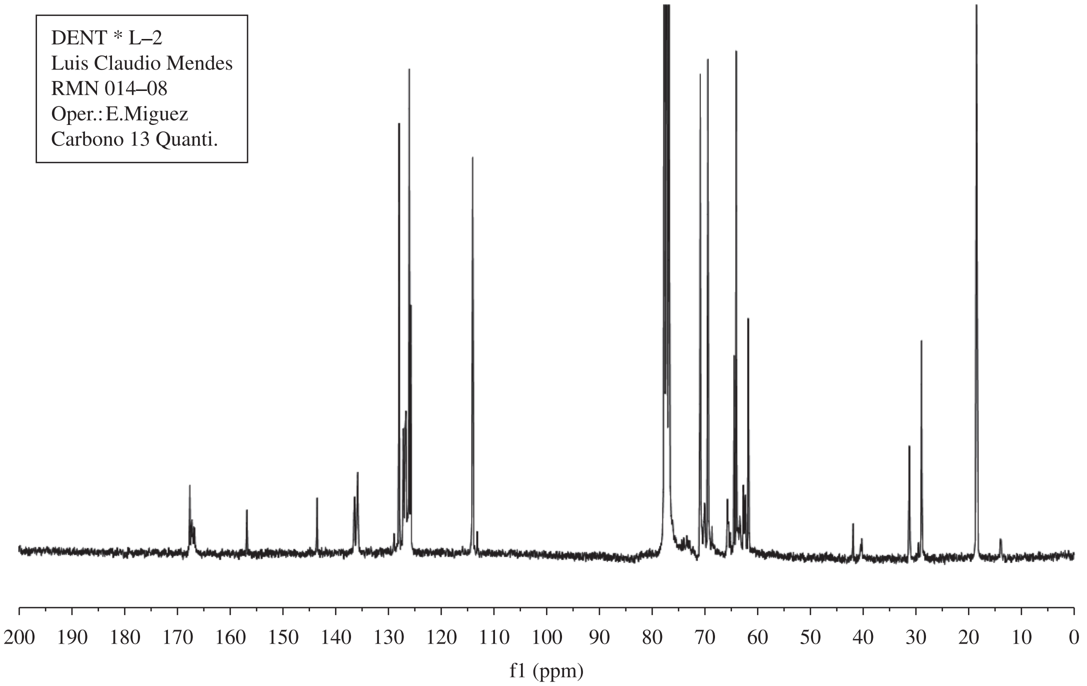

Figure 2. ^{13}C NMR spectrum of the cement liquid portion.

3.3. Thermogravimetry (TG)

Thermogravimetry indicated that the solid fraction constituted 96% of the inorganic volume and 4% of the organic material which was related to vinyl-silane, used as a coating with the view of improving the interfacial adhesion of the composite. Regarding the liquid fraction, the mass loss was 84% further revealing an incombustable portion of 16% which could be related to the phosphorus pentoxide residue and incomplete burning of organic material due to the non-oxidizing nitrogen atmosphere.

Table 1. ^{13}C NMR chemical displacements of the cement solid and liquid portions.

Portion	Chemical displacement (ppm)
Solid	46.1; 126-130
Liquid	13.6; 18.0; 28.5; 30.9; 39.9; 41.5; 61.4; 61.9; 62.1; 62.3; 63.0; 63.7; 64.1; 64.8; 65.3; 68.2; 69.0; 69.7; 112.7; 113.6; 125.3; 125.6; 126.2; 126.4; 126.8; 127.5; 128.5; 135.4; 136.0; 136.1; 143.1; 156.4; 166.3; 166.5; 166.8; 167.2

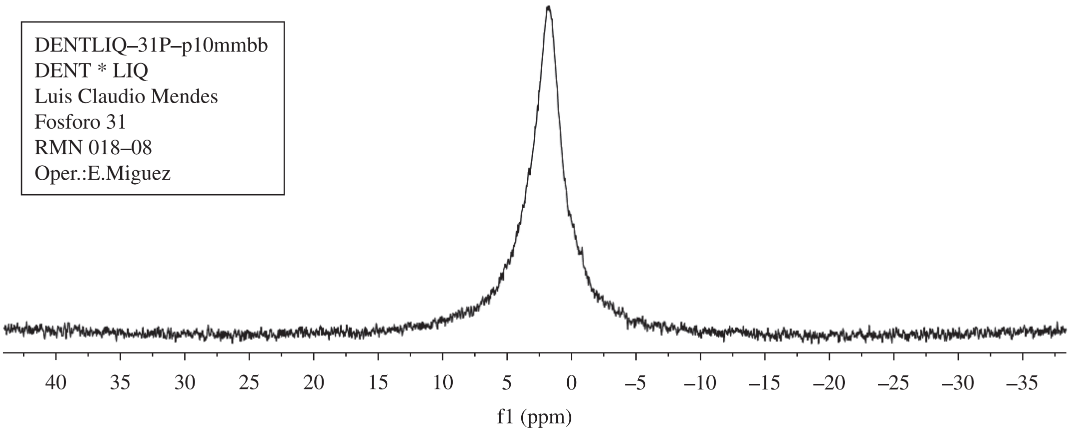

Figure 3. ^{31}P NMR spectrum of the cement liquid portion.

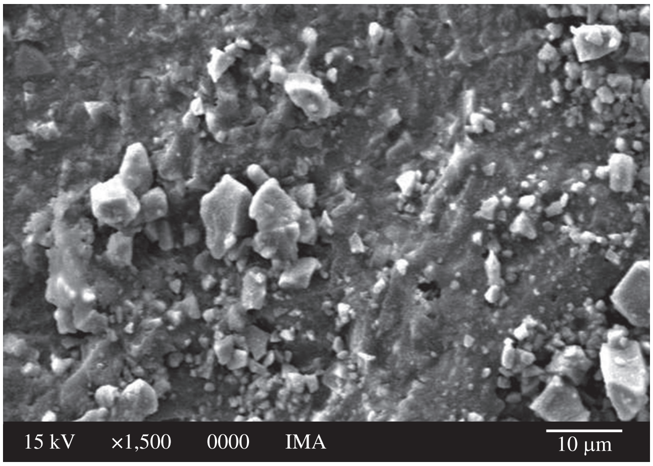

Figure 4. SEM photomicrograph of the polymerized composite.

3.4. Scanning electron microscopy (SEM)

A photomicrograph of the test specimen, after polymerization (Figure 4), indicated there is a great variation of size and form of the particles of the inorganic portion. Particle size varied between 0.4 – 11.2 microns (400 – 11.200 nm).

3.5. Degree of conversion (DC)

In Table 2 are listed the degree of conversion for the different groups. The specimens polymerized in the presence of light showed values of DC almost 4 times superior to that of the group whose polymerization occurred in the absence of light. After 24 hours, it was verified that the test specimens polymerized by light had an increase in the degree of conversion of around 10% while for those polymerized chemically an increase of around 150% was observed. Although being a considerable percentage, the DC value was lower (~60%) to those observed for GR1, GR2 and GR3. Regarding the presence of the ceramic disk and the increase in light exposure time on the cement, it appears these did not influence the degree of conversion.

3.6. Vickers microhardness

In Table 3 are listed the Vickers microhardness values of the different groups. It was observed that this property was greatly dependent on the polymerization conditions. With respect to the test specimens of groups GR1, GR2 and GR3, the VMH values varied due to the ceramic disk and with light exposure time. After 24 hours, the VMH values were around twice as high to those determined at the end of light irradiation. It was not possible to measure the VMH of group GR4 after five minutes reaction time because the composite was not sufficiently rigid. Although the test specimens of GR4 resulted in an average value of DC inferior to those activated by light, the average of microhardness was in the same order of magnitude after 24 hours of reaction.

The presence of a ceramic disk influenced the microhardness of the composite. Lesser values of this property were found for groups GR2 and GR3 in relation to GR1 (glass slide). The average VMH value of group GR3 was sharply superior to group GR2, indicating that increase of light exposure time also affected this property. Although it did not alter the DC value, the presence of the ceramic disk significantly reduced the VMH, indicating that this property is very dependent on the quantity of incident light in the polymerization system. Thus, microhardness is related as much to the DC as to the degree of crosslinking.

With the light irradiation, the initiation step occurs instantaneously due to the production of free radicals. The propagation step proceeds

Table 2. Degree of conversion of the different specimen groups.

Group	Degree of conversion (%)		
	Polymerization mode	Immediately after light exposition	24 hours after light exposition
GR1	light-curing	29.6 ± 4.0	32.6 ± 3.0
GR2	light-curing	27.1 ± 2.4	28.9 ± 3.9
GR3	light-curing	28.2 ± 3.6	32.3 ± 4.0
-	-	After 5 minutes	After 24 hours
GR4	dark-curing	7.9 ± 2.4	20.4 ± 4.9

Table 3. Vickers microhardness of the different specimen groups.

Group	Vickers microhardness		
	Polymerization mode	Immediately after light exposition	24 hours after light exposition
GR1	light-curing	8.0 ± 1.8	14.9 ± 2.3
GR2	light-curing	4.8 ± 0.1	9.1 ± 1.6
GR3	light-curing	6.5 ± 2.5	11.4 ± 2.0
-	-	After 5 minutes	After 24 hours
GR4	dark-curing	-	10.7 ± 0.9

and there is an increase of the viscosity of the composite making difficult the monomer diffusion to the propagation centers of the polymeric chain. This is one of the reasons that could be attributed to the low values of the DC and VHM. Further factors such as energy density (light intensity × exposition time), size and distribution of the inorganic fillers could be also considered. Braga, Ozyesil, Santos Jr. and Rasetto[7-8, 17, 20] reported that the increase of the energy density led to the increase of the DC in their studies on dual resin cements. In fact, Kumbuloglu et al.[16] mentioned that 56% of DC was achieved when 32 $J.cm^{-2}$ of the energy density was applied to dual-curing cement. The energy density irradiated to the RelyX™ was 10 and 20 $J.cm^{-2}$. It is 2-3 times lower than that used by Kumbuloglu and then, it would be not enough to attain higher values of DC and VMH. The size and distribution of the inorganic fillers could be also mentioned. The SEM analysis revealed that the size particles were between 400 and 11.000 nm. Considering that the wavelength of the incident light used in this work was 470 nm, there was a great scattering of the light and its transmission to the liquid portion was unable to lead to higher values of degree of conversion and crosslinking, both straight related to the microhardness.

There is not influence of the ceramic disk which simulated a porcelain indirect restoration on the properties measured herein. The result is in agreement with Prinsloo's[25] study. He mentioned that only disk with thickness upper than 2mm modified significantly the DC.

The chemical curing (without light), in both polymerization modes, seems to have low effectiveness. Its activation was extremely slow making the chemical polymerization system a little inefficient and in the presence of light its contribution on the composite properties would be neglected.

4. Conclusion

Dual-polymerization mode showed higher DC and VMH than the chemical polymerization one. The ceramic disk did not affect the DC but reduced the VMH. The VHM seems to be more dependent on the incident energy than the DC. The energy density, size and distribution of the inorganic fillers and the low effectiveness of the chemical initiation led to the low values of the monomeric conversion and microhardness.

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