10.5005/jp-journals-10024-1274
ORIGINAL RESEARCH

Degree of Conversion of Polymer-matrix Composite assessed by FTIR Analysis

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ABSTRACT

Aims and objectives: The behavior of polymer-matrix composite is dependent on the degree of conversion. The aim of this study was to evaluate the degree of conversion of two resin cements following storage at 37°C immediately, 24 and 48 hours, and 7 days after light-curing by FTIR analysis.

Materials and methods: The specimens were made in a metallic mold and cured with blue LED with power density of 500 mW/cm^2 for 30 seconds. The specimens were pulverized, pressed with KBr and analyzed with FTIR following storage times.

Statistical analysis used: ANOVA (two-way) and Tukey's post hoc.

Results: To the polymer-matrix composites between 24 and 48 hours does not show a significant increase ($p > 0.05$), however, the highest values were found after 7 days.

Conclusion: The polymer-matrix composites used in this study showed similarity on the degree of conversion and increased of according to the time of storage.

Keywords: Polymers, Degree of conversion, FTIR analysis.

How to cite this article: Tonetto MR, Pinto SCS, de Nara Souza Rastelli A, Borges AH, Saad JRC, Pedro FLM, de Andrade MF, Bandéca MC. Degree of Conversion of Polymer-matrix Composite assessed by FTIR Analysis. J Contemp Dent Pract 2013;14(1):76-79.

Source of support: Nil

Conflict of interest: None declared

INTRODUCTION

The polymer-matrix composite has been widely in many areas of engineering and health. Within in dentistry, these materials can be used for cementation of indirect restorations, which they have been demonstrated to be effective although of clinical studies which reported a survival rate for single crowns up to 8 years by current resin cements.[1-3] Successful bonding of polymer-matrix composite is essential for decrease marginal leakage with improved apical seal, increases failure resistance compared with conventional cementation and retention of material bonded of indirect materials.[4-7]

Ideal polymer-matrix composite should provide an efficient bond of the indirect material to tooth for increasing the fracture resistance and reinforce the remaining tooth structure.[8] Within polymer-matrix composite in Dentistry, the resin cements are the first choice to achieve such conditions.[9]

The resin cements are polymerized via chemical, light or the both mechanisms of cure (dual-cure).[9,10] They can be found into self-adhesive, combined with self-ecthing or etch-and-rise systems. The self-adhesive resin cement does not require any pretreatment of dentin. The simplification of the technique application with these new materials is simplifying the cementation procedure and operator-sensitive than when using etch-and-rise systems.[11-15]

The cure of the resin cement is established by conversion the carbon double bonds in carbon single bonds that can be measure through degree of conversion. The extent to which monomers react to form polymers during the polymerization reaction is expected to affect the physical properties of dental resins.[10]

The purpose of this study was to evaluate the degree of conversion of two resin cements following storage at 37°C immediately, 24 and 48 hours and 7 days after light-curing by FTIR analysis. The null hypotheses tested were: (1) Degree of conversion would be different for the cements to the same storage times; and (2) degree of conversion would increase of according to the time of storage.

MATERIALS AND METHODS

The resin cements were prepared with auto-mix and placed in a stainless steel mold (diameter 8.0 ± 0.1 mm, thickness 1.0 ± 0.1 mm) (Table 1).

Table 1: Materials used in the study

Materials	*Composition*	*Manufacturer*	*Bach number*
Breeze® self-adhesive	Bis-GMA, UDMA, TEGDMA, HEMA, 4-MET, Silane treated barium glass, silica (amorphous), minor additives, BIOcl, curing system, Ca-Al-F-silicate	Pentral Clinical Technologies, Wallingford, EUA	161489
Nexus® third generation	Uncured methacrylate ester monomers, non-hazardous inert mineral fillers, non-hazardous activators and stabilizers and radiopaque agent	Kerr Corporation, Orange, CA, EUA	C037E8

The polyethylene film was covered each side of the mold and a glass slide was placed on the top of the mold. To standardize the top surface of the specimens a circular weight (1 kg) with an orifice to pass the light tip of the light-curing unit was placed on the top surface.

The light-curing unit based on blue LED (LEC 1000, MMOptics, São Carlos, Brazil) was used to irradiate (λ = ±470 nm, 500 mW/cm^2, ϕ = 8 mm). The specimens from one side were polymerized along the whole extension during 30 seconds. The power density was checked using Fieldmaster (Sensor LM-3 HTD, Coherent Commercial Products Division, model number FM, set n° WX65, part number 33-0506, USA). The glass slide thickness standardized the distance from the light tip to the resin cement and provided a smooth surface.

Eighty specimens were made for all groups (n = 10). The groups were divided according with rate of conversion determined in different times: Immediately, 24 and 48 hours and 7 days after light-curing process.

The resin cements were pulverized into a fine powder and maintained in a dark room until the moment of the FTIR analysis. Ten milligrams of the ground powder was thoroughly mixed with 100 mg of KBr powder salt. This mixture was placed into a pelleting device and then pressed in a press with a load of 10 tons during 1 minute to obtain a pellet.

The number of double carbon bonds, which are converted in single bonds, provides the degree of conversion (%DC) of dual-cure dental resin cements.

To measure the %DC the pellet was then place into a holder attachment into the spectrophotometer (Nexus 470, Thermo Nicolet). For this technique, the specimens were made and analyzed immediately after light-curing process, 24 and 48 hours and 7 days. Fourier transform infra-red spectroscopy (FTIR) spectra of both uncured and cured specimens were analyzed using an accessory of reflectance diffuse. The measurements were recorded in absorbance operating under the following conditions: 32 scans, 4 cm^{-1} resolution, 300 to 4000 cm^{-1} wavelength.

The percentage of unreacted carbon-carbon double bonds (% C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak at 1638 cm^{-1}) against internal standard before and after curing of the specimen: Aromatic C-C (peak at 1608 cm^{-1}). The rate of conversion was determined by subtracting the % C=C from 100%, according to the formula:

$$RC = \frac{1–(1638\ cm^{-1}/1608\ cm^{-1})\ cured}{(1638\ cm^{-1}/1608\ cm^{-1})\ uncured} \times 100\%$$

RESULTS

The representative FTIR spectra of the dual-cure dental resin cements before and after setting are shown in Figure 1.

The ANOVA (two-way) and Tukey's post hoc showed significant differences between the RC immediately and 24 or 48 hours and 7 days after light-curing, however no significant difference between 24 and 48 hours ($p > 0.05$). The dental resin cements showed similarity on DC ($p < 0.05$) (Table 2).

DISCUSSION

The FTIR measurement has been widely used to evaluate the conversion monomers of the resin cements,[16] as it detects the C=C stretching vibrations directly before and after curing of materials.[17] This method cannot determine depth of cure due the pulverization of the specimens into a powder.

The Figure 1 reports the spectrum in the 1660 and 1600 cm^{-1} region showing the bands of 1638 and 1608 cm^{-1}

Table 2: Mean values (%) and standard deviations (SD) of each group

Materials		*Mean*	*SD*	*Tukey's test (p < 0.05)**
Breeze® self-adhesive	Immediately	29.5	0.8	A
	24 hours	36.2	2.6	B
	48 hours	39.5	1.7	B
	7 days	58.2	2.9	C
Nexus® third generation	Immediately	25.8	1.0	A
	24 hours	39.7	1.3	B
	48 hours	39.2	2.8	B
	7 days	74.2	3.8	C

*Same letter indicates statistical similarity

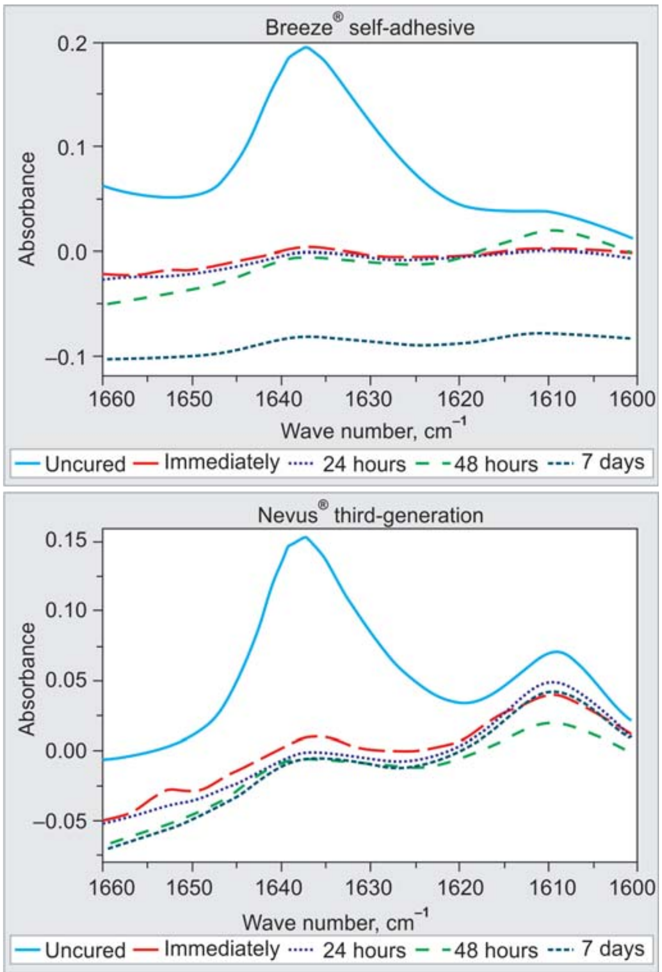

Fig. 1: FTIR spectra of uncured and cured dual-cure dental resin cements in different storage times

stretching vibrations of the resin cements to be calculated the rate of conversion.

The both resin cements are not still found in the literature. The degree of conversion did not show difference significant between the resin cements, which the fist null hypothesis was not confirmed. The materials are dual-cure resin cement, which the monomers are polymerized by self and light activation. The self-activation starts with the reaction between benzoyl peroxide and tertiary amine, which generates free radicals that will break the aliphatic carbon double bonds to begin the polymerization process.[18] The initiator system of light activation is based on camphoroquinone that absorbs energy when exposed to the visible light energy in the wavelength between 400 and 500 nm, and combines with tertiary amine to form a state complex that breaks down into reactive free radicals. Although these two modes of activation are independent, light activation is required for some dual-cured resin cements to increase the rate of conversion.[18]

The Breeze® is a self-adhesive resin cement that does not require any pretreatment of dentin. The own resin cement is responsible to provide the bond between tooth structure and dental material by cross-linked in the cement matrix.

The Nexus® Third Generation shall use the adhesive system to tooth bonding. The adhesive system can be used two types of according with manufacturer's instructions: Etch-and-rise (OptiBond® Solo Plus, Kerr Corporation) or self-etch (OptiBond® All-In-One, Kerr Corporation) adhesives.

The Breeze is self-adhesive resin cement, such as The RelyX™ Unicem, which is widely used in dentistry community. It consists of specially designed multifunctional, phosphoric acid modified methacrylate monomers.[10] These monomers form a highly cross-linked cement matrix during radical polymerization and the phosphoric acid groups contribute to self-adhesion. The carbon double bonds cause a high reactivity of the methacrylate monomers with each other that shows a high degree of conversion.

Therefore, the dental resin cements depend on the physical and mechanical properties, monomer conversion, the filler and the coupling agent used, the concentration, the particle size and size distribution of the filler, and the light-curing process including the nature of the photoinitiator and the activator, their concentration, the power density of light curing unit, exposure time and especially, the type of light curing unit.[19-22]

The use of LED is more appropriate to cure of resin materials due the narrow spectral range with a peak around 470 nm, which matches the optimum absorption wavelength for the activation of the camphorquinone photoinitiator.[23,24]

The storage times analyzed in this study were due to incompleteness of the polymerization reaction in the processing of the resin cement; this study showed the lowest degree of conversion was found immediately after light-curing and the highest rate of conversion after 7 days after light-curing, which the second hypothesis was confirmed. There are some unreacted monomers before 7 days that was reported by Bandéca et al.[10]

CONCLUSION

The polymer-matrix composites used in this study showed similarity on the degree of conversion; the degree of conversion increased of according to the time of storage.

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