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A critical review on the conversion degree of resin monomers by direct analyses

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LITERATURE REVIEW

A critical review on the conversion degree of resin monomers by direct analyses

Revisão crítica sobre o grau de conversão de monômeros resinosos detectados por métodos de análise direta

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ABSTRACT

Objectives: The aim of this study was to carry out a review on the methods of direct detection of the conversion degree of composite resins. Materials & **methods:** The authors searched the Cochrane Library, Embase, PubMed, and the Web of Science (ISI) for papers dated from January 1991 to November 2011. The search was complemented by manual searches of the reference lists from each identified relevant paper. The main search terms were: "degree of conversion", "monomers", "polymers", "composite", "pre-gel", "post-gel", "residual monomers" and "double bond". The inclusion criteria were studies that evaluated consolidated methods in the literature to directly evaluate the degree of conversion. Excluded studies were those considered to be unrelated to the question addressed, such as mechanical and physical properties and also monomers from adhesive systems and compomers. The selected papers were assigned to a score (A, B or C), according to the predetermined criteria. Results: A total of 45 papers were selected and 15 papers were included in the critical appraisal. Two articles were labeled as grade A, nine articles were labeled as grade B, and four articles were labeled as grade C. Conclusions: According to the definitions of evidence levels, the current article study showed a strong evidence of review. Clinical significance: The degree of conversion predicts the quality of polymers because the higher degree of conversion, the higher strength of the resin matrix.

KEYWORDS

Degree of conversion; Double bond; Residual monomers.

RESUMO

Objetivos: O objetivo deste estudo foi revisar os métodos de detecção direta do grau de conversão de resinas compostas. Material e Métodos: Os autores pesquisaram artigos publicados no período de Janeiro de 1991 a novembro de 2011, nas bases de dados Cochrane Library, Embase, PubMed e Web of Science (ISI). A pesquisa foi complementada por pesquisa manual das listas de referências de cada artigo identificado como relevante sobre o tema. Os principais termos pesquisados foram: "grau de conversão", "monômeros", "polímeros", "compósito", "pré-gel", "pós-gel", "monômeros residuais" e "dupla ligação". Os critérios de inclusão foram estudos que avaliaram diretamente o grau de conversão utilizando métodos consolidados na literatura. Os estudos excluídos foram aqueles considerados não relacionados à questão abordada, como estudos sobre propriedades físicas e mecânicas e também sobre monômeros de sistemas de união e compômeros. Os artigos selecionados foram divididos na classificação A, B ou C, de acordo com critérios pré-determinados. Resultados: Quarenta e cinco artigos foram selecionados e quinze artigos foram incluídos na avaliação. Dois artigos foram classificados como grade A, nove artigos como grade B e quatro artigos como grade C. Conclusões: De acordo com as definições dos níveis de evidência, o presente estudo apresentou forte evidência de revisão. Significância Clínica: O grau de conversão prediz a qualidade dos polímeros formados, uma vez que quanto maior o grau de conversão, mais resistente é a matriz resinosa.

PALAVRAS-CHAVE

Grau de conversão; Dupla ligação; Monômeros residuais.

INTRODUCTION

Residual monomers are defined as the unreacted monomers in resin composites [1]. Ideally, it is desirable for dental composites that all monomers would be converted into polymers during the polymerization reaction [1], but this does not occur. Residual monomers can be detected by the degree of conversion (DC), the determination of amount of carbon double bonds (C = C) that are converted into carbon single bonds (C - C) [1,2].

The efficiency of polymerization is influenced by the degree of conversion, (the greater number of double bonds converted into single bonds), resulting in a more resistant polymer network [3,2]. Studies have shown that direct correlations with mechanical properties such as hardness, tensile strength and compressive strength are almost a consensus when the degree of conversion is evaluated [2-5]. Insufficient degree of conversion results in hydrolytic degradation of monomers present in between fillers and the organic matrix [6].

The methods to measure the degree of conversion vary greatly from report to report [7]. Some studies indirectly evaluate the DC, independent of leaching out tests, which consisted of an extraction of unreacted monomers in order to detect the quantity of reacted monomers [4,8]. Methods that directly evaluate DC by means of spectroscopy have been successfully established [9,10]. These studies verify the absorption peak of C = C bonds before and after polymerization, showing the amount the remaining C = C not converted into C - C [7]. The baseline is the aromatic absorption peaks at 1609 or 1590 cm⁻¹ 1 , being the internal C = C reference peak at 1638 cm⁻¹ [11,12]. The most commonly used DC calculations are based on the proportion of cured and uncured materials [1,3-5,7,13,14,15,]. Since a variety of method parameters is used, it would be useful to know how quick and reliable the method of determining the degree of conversion of dental resins is. The purpose of this article is to present a systematic review about the methods of direct detection of degree of conversion of composite resins.

REVIEW

• Question addressed by this review:

What is the most reliable methodology to directly evaluate the degree of conversion of resin composites?

• Literature search

The authors searched for papers on Cochrane Library, Embase, PubMed, and Web of Science (ISI) databases from January 1991 to November 2011. The search was supplemented by manual searches of the reference lists of each relevant paper identified. The search terms were "degree of conversion", "monomers", "polymers", "composite", "pre-gel", "post-gel", "residual monomers", and "double bond". The search was limited to in vitro studies. Only original papers written in English were included. Interim reports, abstracts, letters, short communications, reviews, and chapters in textbooks were excluded. First, the papers identified in databases were printed as abstracts, or full-text articles, if the abstract was missing. In a second step, three authors independently selected relevant papers, and those considered of interest for this review were ordered in fulltext versions. During the evaluation process, reference lists were manually searched.

• Inclusion and exclusion criteria

After the appraisal, papers were included in this review when they showed consolidate methods in the literature to directly evaluate the degree of conversion. Excluded studies were those considered to be unrelated to the question addressed, such as mechanical and physical properties and also monomers from adhesive systems and compomers.

• Evaluation of scientific papers and levels of evidence

The papers that met the inclusion criteria were subjected to critical appraisals, which were carried out independently by at least two reviewers. Data was extracted using a pilottested form, and each paper was given a score of A, B or C, according to predetermined criteria for methodology and performance, as presented in Table 1. Based on the literature evaluated and

on the scores, the final level of evidence was judged according to the protocol of the Swedish Council on Technology Assessment in Health Care [16] and modified by Kantovitz et al. [17] as described in Table 3.

Table 1 - Topics from each article includes in this review

First Author	Year	Method	Material	Light Intensity/ Curing	Specimens Description	Laser / Wavelength	Peak Vibration Reference	N	DC Calcula- tion	Spectra Exposi- tion	Grade
Yoshida	1993	FTIR	Experimental proportion of TEGDMA/UDMA	Halogen curing unit	3 mg of the resin powder pressed into a transparent pellet with a pellet- making apparatus	standard	Aliphatic C = C peak 1638 cm ⁻¹ . Carbonyl absorption peak (C=0) at 1730 cm ⁻¹ . Urethane absorption peak (N-H) at 3350 cm ⁻¹	5	Ratio of the reference absorption peaks	yes	Α
Shin	1993	FT Raman Scatter- ing and Raman spectros- copy	Experimental proportion of Bis-GMA, TEG- DMA, UDMA, HDDM, EBPADMA.	light-cured (130 mW of a filtered tung- sten source) and heat oven at 100°C heat-cured	3 mm-thick NMR tubes	FT Raman: InGaAs detector atached on FiTIR equipment with near IR optics. Raman: 30 mW from the 514.5 nm line of a 5-watt Coherent Argon ion laser. Wavelength between 1500 and 1800 cm ⁻¹	C = C peak 1640 cm ⁻¹ for all monomers. C=0 peak 1718 cm ⁻¹ for Bis-GMA, ABPADMA, EGDMA; 1714 cm ⁻¹ for TEGDMA, 1,6-HDDM; 1738 cm ⁻¹ for EGDA.	-	Ratio of the reference absorption peaks	yes	В
Peutzfeldt	1994	IR	Experimental proportion of BISGMA, TEGDMA, UEDMA, HEMA	light curing resins for 80 s	Monomer: film of thickness less than 50 μm. Polymer: 25 mL of monomer com- pressed between two glass plates	standard	C = C band at 1640 cm- 1, and the aromatic C - C band at 1610 cm -1	3	Ratios of polymer peaks to monomers peaks	no	В
Peutzfeldt	1994	Light- intensity tester (CL-Tester) and IR espectros- copy	Experimental proportion of BISGMA, TEGDMA	Halogen light and LED light	thin film between two KBr-disks	standard	C = C band at 1640 cm ⁻¹ , and the aromatic C - C band at 1610 cm ⁻¹	3	Ratios of polymer peaks to monomers peaks	no	В
Jancar	2000	FTIR	Experimental proportion of Bis-GMA, TEGDMA	vacuum oven at 120° C for 1h and then post-cured at 250 C for 1h.	specimens with 25 x 10 x 0,5 mm ³	standard	the C = C peak at 1634 cm ⁻¹ and peak at 1716 cm ⁻¹	-	-	yes	С
Peutzfeldt	2000	IR	Comercial composites with BisGMA/ TEGDMA. Shade A3 and A 3,5.	Halogen ligth and heat oven	approximately 60 µm thick, 10 mg paste had been pressed into a thin film between two KBr disks.	standard	aliphatic(C= C)† band at 1637 cm ⁻¹ and the aromatic(C - C)† band at 1608 cm ⁻¹	3	Ratios of poly- mer peaks to monomers peaks	no	В

First Author	Year	Method	Material	Light Intensity/ Curing	Specimens Description	Laser / Wavelength	Peak Vibration Reference	N	DC Calcula- tion	Spectra Exposi- tion	Grade
Jan	2001	FTIR	Experimental proportion of UDMA, TEGDMA	Halogen light for 40 s	specimens with 6 mm of diameter and 3 mm thick	standard	peak of aliphatic C = C at 1650 cm ⁻¹ and a carbonyl group COO at 1730 cm ⁻¹	-	Ratios of polymer peaks to monomers peaks	yes	В
Tarle	2002	FTIR	Comercial composites. Do not specified the monomers. Shade: A3	Three different light sources. 1. LED (9 mW cm ⁻²); 2. PLASMA(1370 mW cm ⁻²); 3. HALOGEN lamp (560 mW ⁻²)	sample pressed into KBr pellets with 1 cm of diameter	standard	aliphatic C = C peak at 1637 cm ⁻¹ and aromatic C = C at 1608 cm ⁻¹	20 scans	Ratios of polymer peaks to monomers peaks	yes	В
Yoon	2002	FTIR	Comercial composites. Do not specified the monomers. Shade: A3	Three different light sources: 1. LED(400 mW cm ⁻²); 2. PLASMA(1472 mW cm ⁻²); 3. HALOGEN lamp(400 mW cm ⁻²)	sample pressed into KBr pellets with 50 - 70 µm thickness	standard	The aliphatic C = C group has a peak around 1636 cm ⁻¹ . The aromatic C = C peak around 1609 cm ⁻¹	3	Ratios of polymer peaks to monomers peaks	yes	В
Mendes	2005	FTIR	Comercial composites. Do not specified the monomers. Shade: B	-	-	standard	peak height absorbances at 1637 and 1610 cm ⁻¹	-	Ratios of polymer peaks to monomers peaks	no	С
Nomoto	2006	FTIR	Comercial composites. Do not specified the monomers. Shade: B	LED/Halogen ligth	specimens with 4 mm of diameter and 8 mm of depth	standard	C = C absorbance peak at 1637cm ⁻¹ ; aromatic absorbance peak at 1608cm ⁻¹	3	Ratios of polymer peaks to monomers peaks	yes	В
Väkiparta	2006	FTIR spectroscopy	Experimental proportion of BISGMA, TEGDMA, POLI AMIDE	Halogen light (wavelength 480 nm, light intensity ca 650 mW/cm²) for 40 s	powder from specimens form of 2x2x25 mm ³	standard	aliphatic C = C peak at 1637 cm ⁻¹ , and the aromatic C = C peak at 1609 cm ⁻¹	3	Ratios of polymer peaks to monomers peaks	no	В
Filho	2008	FTIR	Experimental proportion of Bis-GMA, TEGDMA, UDMA	Halogen ligth (850 mW/cm² for 20 s) and (1000 mW/cm² for 10 s)	Films in 3 mm of diameter and 01 thick	standard	aliphatic C = C peak at 1638 cm ⁻¹ and C = O peak at 1720cm ⁻¹	5	Ratios of polymer peaks to monomers peaks	yes	А
Goncalves	2009	FT- Raman spectros- copy	Experimental proportion of BISGMA, TEGDMA, BIS-EMA	Halogen ligth (420 mW cm²)	specimens with 5 mm of diameter and 1 mm of thick	Nd-YAG laser, emitting radiation at 1.064 nm at an intensity of 100 mW	aliphatic (1.640 cm ⁻¹) and aromatic (1.610 cm ⁻¹) carbon double bond absorption	-	Ratios of polymer peaks to monomers peaks	no	С
Galvão	2010	FTIR	Comercial compos- ites with Bis-GMA, Bis-EMA, TEGDMA	LED for 40s	5 mg ground powder	300 to 4000 cm ⁻¹ wavelength	aliphatic C = C peak at 1637 cm ⁻¹ , and the aromatic C = C peak at 1610 cm ⁻¹	-	Ratios of polymer peaks to monomers peaks	no	С

Table 2 – Grades used to classify the articles included

GRADE A	Direct method of detection of degree of conversion of composite resin			
All criteria states on the right side should be met	Representative study Defined methods for analysis of degree of conversion The reliability of evaluation methods described All criteria from table 1			
GRADE B	Direct method of detection of degree of conversion of composite resin			
All criteria states on the right side should be met	Representative study Defined methods for analysis of degree of conversion The reliability of evaluation methods described One criterium from table 1 absent			
GRADE C	Direct method of detection of degree of conversion of composite resin			
All criteria states on the right side should be met	Representative study Defined methods for analysis of degree of conversion The reliability of evaluation methods described Two or more criteria from table 1 absents			

RESULTS

A total of 115 papers were originally identified based on the main search terms. Initially, 70 papers were excluded after evaluation. A total of 45 papers were selected. Fifteen papers were included in the critical appraisal project based on the inclusion and exclusion criteria. The papers were labeled as grade A if all criteria were described. Papers were labeled as grade B if one criterion was not described. Papers were labeled as grade C if more than one criterion was not described (Table 1). The grades B and C are concerned methodologies not completely described. The criteria of this classification are described in Table 2. Two articles were labeled as grade A, nine articles were labeled as grade B, and four articles were labeled as grade C. According to the definitions of evidence level, modified by Kantovitz et al. [17], the current article was classified with strong evidence of review (Table 2). The thirty papers that indirectly verified the degree of conversion evaluated the degree of conversion of adhesive systems or evaluated the degree of compomer conversions were not submitted to grade classification (Table 4).

Table 3 - Definitions of evidence level*

1. Strong evidence	At least two studies with high level of evidence (grade A)
2. Moderate evidence	One study with high level of evidence (grade A) and at least two studies with a moderate level of evidence (grade B)
3. Limited evidence	At least two studies with a moderate level of evidence (grade B)
4. Inconclusive evidence	Fewer than two studies with a moderate level of evidence (grade B)

^{*}Kantowitz et al [18]

Table 4 – Articles excluded of this review

AUTOR	YEAR	TITLE
Simitzis, J	1984	Influence of Backbone Rigidity on the Curing and the Dielectric Relaxations of Unsaturated Polyesters
J. W. Stans- bury	1992	Evaluation of methylene lactone monomers in dental resins
C. P. Hsu	1993	Free-radical crosslinking copolymerization of styrene/unsaturated polyester resins 3. Kinetics-gelation mechanism
Oguz Okay	1995	Free-radical crosslinking copolymerization: effect of cyclization on diffusion-controlled termination at low conversion
M. C. SUN	2000	Microgelation of Unsaturated Polyester Resins in the Presence of Poly(vinyl acetate) by Static and Dynamic Light Scattering
Munksgaard EC	2000	Elution of TEGDMA and BisGMA from a resin and a resin composite cured with halogen or plasma light
Xuehao He	2001	Monte Carlo Simulation of Hyperbranched Copolymerizations in the Presence of a Multifunctional Initiator
C. Sarmoria	2001	Spanning-tree models for Af homopoly- merizations with intramolecular reactions
Paula Bosch	2002	New Fluorescent Probes for Monitoring polymerization Reactions: Photocuring of Acrylic Adhesives, 2
Nilgun Ozturk	2005	Degree of conversion and surface hard- ness of resin cement cured with different curing units
Cadenar	2005	Degree of conversion and permeability of dental adhesives
Wan-Yu Tseng	2006	Effects on Microstrain and conversion of Flowable Resin Composite Using Different Curing Modes and Units

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AUTOR	YEAR	TITLE
Marju Vaki- parta	2006	Residual monomers and degree of conversion of partially bioresorbable fiber- reinforced composite
Murat Cavit cehreli	2007	Impact of similarity in chemical composi- tion of light-polymerized resin composites on post-gel strains and interface integrity
D Skrtic	2007	Effect of chemical strcture and composition of the resin phase on vinyl conversion of amorphous calcium phosphate-filled composites
Francisco López-Sue- vos	2008	Degree of cure and fracture properties of experimental acid-resin modified composites under wet and dry conditions
Navara CO	2009	Degree of conversion and interfacial nonaleakage expression of three one-step self-etch adhesives
J. Simitzis	2010	Influence of Backbone Rigidity on the Curing and the Dielectric Relaxations of Unsaturated Polyesters
Navara CO	2009	Degree of conversion and interfacial nonaleakage expression of three one-step self-etch adhesives
J. Simitzis	2010	Influence of Backbone Rigidity on the Curing and the Dielectric Relaxations of Unsaturated Polyesters
Baojiao Gao	2010	Studies on Preparation of Composite Chelating Material Poly(amidoxime)/SiO2 with Grafting-Type
Vesna Miletic	2010	Micro-Raman Assessment of the Ratio of Carbon-Carbon Double Bonds of Two Ad- hesive Systems Cured with LED or Halogen Light-curing Units
Hideaki Tokuyama	2010	Preparation of poly(N-isopropylacryl- amide) hydrogel beads by circulation polymerization
M. R. Galvão	2010	Influence of Light Guide Tip Used in the Photo Activation on Degree of Conversion and Hardness of One Nanofilled Dental Composite
Letícia Cris- tina Cidreira Boaro	2010	Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites
Maurizio Ferrante	2010	Thermal analysis of light-curing composites
Sarah L. Rolland	2010	Use of micro-Raman spectroscopy to investigate hybrid layer quality in demineralized root dentine
D. Tantbirojn	2011	Do Low-shrink Composites Reduce Polymerization Shrinkage Effects?

AUTOR	YEAR	TITLE
Tantbirojn	2011	Do Low-shrink Composites Reduce Polymerization Shrinkage Effects?
Gao, BJ	Gao, BJ	Studies on Preparation of Composite Chelating Material Poly(amidoxime)/SiO2 with Grafting-Type
Miletic, V	2011	Micro-Raman Assessment of the Ratio of Carbon-Carbon Double Bonds of Two Adhesive Systems Cured with LED or Halogen Light-curing Units

DISCUSSION

This review searched and provided a comprehensive and contemporary appraisal of a substantial amount of literature, using a suitable methodology. This review is, as such, fundamentally different from traditional review articles due to the comprehensive nature of the literature search and data extraction. Moreover, subsequent quality assessments of the included papers in this study are now well-established measures for evidence-based dentistry.

The basic requirements for classification of the studies reviewed were the methods, materials tested, light curing/ intensity, specimens description, laser /wavelength, peak vibration reference, the number of specimens (N), degree of conversion (DC) calculation, and exposition of acquired spectra (Table 1).

The main methods shown in this review were: the Fourier Transform Infrared (FTIR), the Raman and the FT-Raman (Fourier Transform Raman) spectroscopy. From 15 articles reviewed, 14 evaluated the Degree of Conversion (DC) of resin composite monomers using an Infrared technique [2-5,7,13,14,18-22]. During the IR absorption (or reflection) experiments, the technique probes the monomer characteristics by measuring the absorbed radiation in the infrared range. The process is very efficient, since it occurs when resonance takes place between the incident radiation and the vibrational frequency characteristic of a specific arrangement of atoms or molecules [23]. FTIR, or Fourier transform IR, refers to the use of an interferometer (to separate the spectral components) and a mathematical algorithm to accelerate the measurement

[24]. While the FTIR technique is based on the absorption (or reflection) of radiation, Raman spectroscopy probes the light scattering processes [23,24]. Within this context, it is important to remark that [23,24]: (a) the absorption of radiation is much more efficient (~ 109 times) than scattering, (b) IR absorption measurements are suitable for polar systems (in which there is a large transfer between atoms or molecules), as the chemical bonds formed by polymerization of resin monomers, and (c) Raman scattering is best suited for systems in which the electron cloud may be deformed. More specifically, during the Raman measurements, when laser light strikes a sample, it acts by deforming the electron cloud. which will cause scattering. Based on these aspects, the use of FTIR absorption spectroscopy is the easiest and simplest method [25]. FT-Raman scattering provided an alternate method of obtaining degrees of conversion for these systems and was particularly useful for measuring spectra of materials without any sample preparation [5].

A study of DC should specify what monomer system is evaluated, even if the resin composite studied is commercially available, because it could influence the values of DC. The total replacement of TEGDMA by BisEMA decreases the DC in the Bis-GMA based resin matrix [25]. Some articles that used the DC analysis did not specify the monomer system in the methodology [1,14,15,20].

The quality of polymerization is influenced by the light cure type, and by its wavelength and intensity. Some studies specified what type of light cure was used, if halogen or LED, and their wavelengths and intensities [1,4,5,13,15,18,25]. Other studies only cited the type of light and the time of irradiation [2,3,7,13,14,20,22,26] . The specimens greatly varied among the studies reviewed. Some studies did films [3,7,22]; while others studies did circular specimens [7,8,10,12,16,24,20] and some studies powdered the polymers [2,22,25]. The description of procedures included citing the dimensions of a mold used as a standard for specimens' fabrication, until the weight of monomers and polymers used are obtained. One study did not describe the specimens [20]. However, all studies inspected the specimens before and after polymerization. This is a basic requirement in order to obtain the DC of monomers directly from

IR and/or Raman spectroscopy, and identifying the wavenumbers of the aliphatic and aromatic bands [1,4,5,13,14,15].

The DC calculations from all the articles reviewed were obtained from the spectrum of the monomer absorbance of the aliphatic (C = C) band at approximately 1637 cm-1 and the absorbance of the aromatic (C - C) band at approximately 1608 cm-1 [5,14,15,18,19,20,23,25,26]. The band peak variations up to ~ 5 cm-1, as obtained by different authors, is not significant because they can be associated to variations in composition and/or geometry of the structures [23,24]. The ratio between C = C/C - C of polymers and C =C/C – C of monomers are taken as the fraction of unreacted double bonds in the polymer and it is important to directly compare the spectra to follow the conversion process [23,24]. If they are not subject to problems of background and do not experience changes in their weights (which may suggest the absence of vibration modes), the fraction of material converted is directly proportional to the peak intensity or area. Seven reviewed articles did not show the spectra acquired [3,7,13,19,20,22,25].

Whereas the IR absorption methods are carried out under standard conditions (broad band and low-intensity IR radiation sources) [23], Raman measurements can be performed following many different experimental approaches. Considering that the conversion degree can be influenced by the laser radiation characteristics (photon energy, power, probed area), it is fundamental to specify all the experimental details involved in the Raman analyses [24]. The two studies based on the Raman technique described only the photon energy or laser wavelength [13,5].

Finally, the number of specimens also greatly varied. Some studies did not cite [4,5,13,19,20,26] while other studies evaluated 3 specimens [1,3,7,14,22,25] and yet other studies evaluated 5 specimens [2,18]. One study associated the number of specimens to the number of measured scans (= 20) [15], which is used simply to improve the quality of the spectra (or signal-to-noise ratio). In other words, to precisely evaluate the DC, the measurements should be statistically significant, or acquired from different specimens [23,24].

CONCLUSIONS

Based on this review, the authors suggest that, despite the great methodology variations, the studies from Grade A are the most reproducible to directly inspect the degree of conversion of resin composites

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