



## 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”, “composto”, “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

Grado 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  $\text{cm}^{-1}$ , being the internal C = C reference peak at 1638  $\text{cm}^{-1}$  [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 full-text 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 pilot-tested 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 Calculation                            | Spectra Exposition | 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 <sup>-1</sup> . Carbonyl absorption peak (C=O) at 1730 cm <sup>-1</sup> . Urethane absorption peak (N-H) at 3350 cm <sup>-1</sup>  | 5 | Ratio of the reference absorption peaks   | yes                | A     |
| Shin         | 1993 | FT Raman Scattering and Raman spectroscopy             | Experimental proportion of Bis-GMA, TEGDMA, UDMA, HDDM, EBPADMA. | light-cured (130 mW of a filtered tungsten source) and heat oven at 100°C heat-cured | 3 mm-thick NMR tubes  | FT Raman: InGaAs detector attached on FTIR 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 <sup>-1</sup> | C = C peak 1640 cm <sup>-1</sup> for all monomers. C=O peak 1718 cm <sup>-1</sup> for Bis-GMA, ABPADMA, EGDMA; 1714 cm <sup>-1</sup> for TEGDMA, 1716 cm <sup>-1</sup> for UDMA, 16-HDDM; 1738 cm <sup>-1</sup> for EGDA. | - | Ratio of the reference absorption peaks   | yes                | B     |
| 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 compressed between two glass plates | standard  | C = C band at 1640 cm <sup>-1</sup> , and the aromatic C - C band at 1610 cm <sup>-1</sup>  | 3 | Ratios of polymer peaks to monomers peaks | no                 | B     |
| Peutzfeldt   | 1994 | Light-intensity tester (CL-Tester) and IR spectroscopy | Experimental proportion of BISGMA, TEGDMA                        | Halogen light and LED light  | thin film between two KBr-disks   | standard  | C = C band at 1640 cm <sup>-1</sup> , and the aromatic C - C band at 1610 cm <sup>-1</sup>  | 3 | Ratios of polymer peaks to monomers peaks | no                 | B     |
| Jancar       | 2000 | FTIR   | Experimental proportion of Bis-GMA, TEGDMA                       | vacuum oven at 120° C for 1 h and then post-cured at 250° C for 1 h.                 | specimens with 25 x 10 x 0,5 mm <sup>3</sup>  | standard  | the C = C peak at 1634 cm <sup>-1</sup> and peak at 1716 cm <sup>-1</sup>   | - | -   | yes                | C     |
| Peutzfeldt   | 2000 | IR   | Comercial composites with BisGMA/TEGDMA. Shade A3 and A 3.5.     | Halogen light 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 <sup>-1</sup> and the aromatic ... (C - C)† band at 1608 cm <sup>-1</sup>  | 3 | Ratios of polymer peaks to monomers peaks | no                 | B     |

| First Author | Year | Method                | Material   | Light Intensity/ Curing  | Specimens Description                                     | Laser / Wavelength   | Peak Vibration Reference   | N        | DC Calculation                            | Spectra Exposition | 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 <sup>-1</sup> and a carbonyl group COO at 1730 cm <sup>-1</sup>                       | -        | Ratios of polymer peaks to monomers peaks | yes                | B     |
| Tarte        | 2002 | FTIR                  | Comercial composites. Do not specified the monomers. Shade: A3 | Three different light sources. 1. LED (9 mW cm <sup>-2</sup> ); 2. PLASMA(1370 mW cm <sup>-2</sup> ); 3. HALOGEN lamp (560 mW <sup>-2</sup> )    | sample pressed into KBr pellets with 1 cm of diameter     | standard   | aliphatic C = C peak at 1637 cm <sup>-1</sup> and aromatic C = C at 1608 cm <sup>-1</sup>                                | 20 scans | Ratios of polymer peaks to monomers peaks | yes                | B     |
| Yoon         | 2002 | FTIR                  | Comercial composites. Do not specified the monomers. Shade: A3 | Three different light sources: 1. LED(400 mW cm <sup>-2</sup> ); 2. PLASMA(1472 mW cm <sup>-2</sup> ); 3. HALOGEN lamp(400 mW cm <sup>-2</sup> ) | sample pressed into KBr pellets with 50 - 70 μm thickness | standard   | The aliphatic C = C group has a peak around 1636 cm <sup>-1</sup> . The aromatic C = C peak around 1609 cm <sup>-1</sup> | 3        | Ratios of polymer peaks to monomers peaks | yes                | B     |
| Mendes       | 2005 | FTIR                  | Comercial composites. Do not specified the monomers. Shade: B  | -  | -   | standard   | peak height absorbances at 1637 and 1610 cm <sup>-1</sup>  | -        | Ratios of polymer peaks to monomers peaks | no                 | C     |
| Nomoto       | 2006 | FTIR                  | Comercial composites. Do not specified the monomers. Shade: B  | LED/Halogen lighth   | specimens with 4 mm of diameter and 8 mm of depth         | standard   | C = C absorbance peak at 1637cm <sup>-1</sup> ; aromatic absorbance peak at 1608cm <sup>-1</sup>                         | 3        | Ratios of polymer peaks to monomers peaks | yes                | B     |
| Väikpärta    | 2006 | FTIR spectroscopy     | Experimental proportion of BISGMA, TEGDMA, POLI AMIDE          | Halogen light (wavelength 480 nm, light intensity ca 650 mW/cm <sup>2</sup> ) for 40 s   | powder from specimens form of 2x2x25 mm <sup>3</sup>      | standard   | aliphatic C = C peak at 1637 cm <sup>-1</sup> ; and the aromatic C = C peak at 1609 cm <sup>-1</sup>                     | 3        | Ratios of polymer peaks to monomers peaks | no                 | B     |
| Filho        | 2008 | FTIR                  | Experimental proportion of Bis-GMA, TEGDMA, UDMA               | Halogen lighth (850 mW/cm <sup>2</sup> for 20 s) and (1000 mW/cm <sup>2</sup> for 10 s)  | Films in 3 mm of diameter and 0.1 thick                   | standard   | aliphatic C = C peak at 1638 cm <sup>-1</sup> and C = O peak at 1720cm <sup>-1</sup>                                     | 5        | Ratios of polymer peaks to monomers peaks | yes                | A     |
| Goncalves    | 2009 | FT-Raman spectroscopy | Experimental proportion of BISGMA, TEGDMA, BIS-EMA             | Halogen lighth (420 mW cm <sup>-2</sup> )  | 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 (1640 cm <sup>-1</sup> ) and aromatic (1610 cm <sup>-1</sup> ) carbon double bond absorption                   | -        | Ratios of polymer peaks to monomers peaks | no                 | C     |
| Galvão       | 2010 | FTIR                  | Comercial composites with Bis-GMA, Bis-EMA, TEGDMA             | LED for 40s  | 5 mg ground powder  | 300 to 4000 cm <sup>-1</sup> wavelength                                | aliphatic C = C peak at 1637 cm <sup>-1</sup> ; and the aromatic C = C peak at 1610 cm <sup>-1</sup>                     | -        | Ratios of polymer peaks to monomers peaks | no                 | C     |

**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<br>Defined methods for analysis of degree of conversion<br>The reliability of evaluation methods described<br>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<br>Defined methods for analysis of degree of conversion<br>The reliability of evaluation methods described<br>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<br>Defined methods for analysis of degree of conversion<br>The reliability of evaluation methods described<br>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. Stansbury | 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 homopolymerizations 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 hardness 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                    |

| 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 composition of light-polymerized resin composites on post-gel strains and interface integrity         |
| D Skrtic                        | 2007 | Effect of chemical structure and composition of the resin phase on vinyl conversion of amorphous calcium phosphate-filled composites   |
| Francisco López-Suevos          | 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 nonleakage 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 nonleakage 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)/SiO <sub>2</sub> with Grafting-Type                             |
| Vesna Miletic                   | 2010 | Micro-Raman Assessment of the Ratio of Carbon-Carbon Double Bonds of Two Adhesive Systems Cured with LED or Halogen Light-curing Units |
| Hideaki Tokuyama                | 2010 | Preparation of poly(N-isopropylacrylamide) 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      |
| Leticia Cristina 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)/SiO <sub>2</sub> 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 ( $\sim 109$  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  $\text{cm}^{-1}$  and the absorbance of the aromatic (C - C) band at approximately 1608  $\text{cm}^{-1}$  [5,14,15,18,19,20,23,25,26]. The band peak variations up to  $\sim 5 \text{ cm}^{-1}$ , 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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## ORIGINAL ARTICLE

# Influence of glass fiber reinforcement and resin viscosity on the resistance to fracture of Adhesive Partial Fixed Prosthesis

Influência do reforço de fibra de vidro e viscosidade da resina sobre a resistência a fratura de Prótese Parciais Fixas Adesivas

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## ABSTRACT

**Objective:** The aim of this study was to evaluate the influence of glass fiber reinforcement and resin viscosity on the resistance to fracture of adhesive fixed partial prosthesis (AFPP). **Materials & Methods:** A stainless steel molding was constructed simulating occlusal preparation for AFPP on a maxillary first pre-molar (4 x 4 x 2 mm) and molar (6 x 4 x 2 mm), with a proximal slot (2 x 1 x 4 mm) and with 7 mm of distance between them. These moldings were duplicated in polyurethane (n = 60) and divided into two groups according to the resin viscosity: G - Grandio SO (VOCO) (n = 30) and GHF - Grandio SO Heavy flow (VOCO) (n = 30). These groups were subdivided into three subgroups according to the glass fiber reinforcement used (n = 10): Subgroup N - without glass fiber reinforcement; Subgroup V - GRANDTEC (VOCO); subgroup S - everStick C & B (Stick Tech). Four increments divided into 2 layers, 2 in the lower part and 2 in the upper part of the preparations were light-cured according to the manufacturers' instructions. A vertical load was applied on the center of the pieces at a speed of 1mm/min. Data were obtained in Kgf and submitted to two-way ANOVA and Tukey test ( $\alpha = 0.05$ ). **Results:** ANOVA showed significant differences for glass fiber reinforcement. Subgroup N ( $24.45 \pm 3.60$ )a was significantly different from subgroup S ( $32.54 \pm 6.94$ )b and subgroup V ( $37.18 \pm 5.33$ )c. **Conclusion:** The glass fibers tested were capable of improving the resistance to fracture of AFPP. GRANDTEC fiber exhibited the greatest values of resistance to fracture and for the resins studied the viscosity did not influence on the resistance to fracture of AFPP.

## KEYWORDS

Glass fiber reinforcement; Adhesive partial fixed prostheses; Resin Composite.

## RESUMO

**Objetivo:** O objetivo deste estudo foi avaliar a influência do reforço com fibra de vidro e viscosidade da resina na resistência à fratura de prótese parcial fixa adesiva (PPFA). **Material e Método:** Um molde de aço inoxidável foi confeccionado simulando preparos oclusais para PPFA em primeiro pré-molar superior (4 x 4 x 2 mm) e molar (6 x 4 x 2 mm), com uma caixa proximal (2 x 1 x 4 mm) e 7 mm de distância entre eles. Esses moldes foram duplicados em poliuretano (n = 60) e divididos em dois grupos de acordo com a viscosidade da resina: G - Grandio SO (VOCO) (n = 30) e GHF - Grandio SO Heavy flow (VOCO) (n = 30). Estes grupos foram subdivididos em três subgrupos de acordo com o reforço de fibra de vidro utilizado (n = 10): Subgrupo N - sem reforço de fibra de vidro; Subgrupo V - GRANDTEC (VOCO); subgrupo S - everStick C & B (Stick Tech). Quatro incrementos divididos em 2 camadas, 2 na parte inferior e 2 na parte superior dos preparos foram fotopolimerizados de acordo com as instruções dos fabricantes. Uma força vertical foi aplicada no centro das peças a uma velocidade de 1 mm/min. Os dados foram obtidos em Kgf e submetidos a ANOVA a 2 fatores e teste de Tukey ( $\alpha = 0,05$ ). **Resultado:** A ANOVA mostrou diferenças significativas para reforço de fibra de vidro. Subgrupo N ( $24,45 \pm 3,60$ )a foi significativamente diferente do subgrupo S ( $32,54 \pm 6,94$ )b e subgrupo V ( $37,18 \pm 5,33$ )c. As fibras de vidro testadas foram capazes de melhorar a resistência à fratura de PPFA. **Conclusão:** A Fibra GRANDTEC apresentou os maiores valores de resistência à fratura e, para as resinas estudadas, a viscosidade não teve influência na resistência à fratura de PPFA.

## PALAVRAS-CHAVE

Reforço por fibra de vidro; Prótese parcial fixa adesiva; Resina composta.