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Influence of soft-start curing protocol in the degree of conversion of a nanohybrid resin composite

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ABSTRACT

OBJECTIVE: To analyze the influence of the soft-start curing protocol in the degree of conversion of a nanohybrid composite.

METHODS: Ten specimens were prepared from the polymerization of the composite resin Llis using two protocols: conventional (1000 mW/cm² for 20 seconds) or gradual (250 mW/cm² for 20 seconds + 1000 mW/cm² for 15 seconds). Then, analyzes were performed using a spectrophotometer and data were subjected to Two-way ANOVA and Holm-Sidak test. The significance level was 5%.

RESULTS: There was no statistical difference between the average resin conversion degree of composite values in the base area and top for both curing protocols ($p > 0.05$).

CONCLUSION: Therefore, the use of soft-start curing protocol did not interfere in the degree of conversion of a nanohybrid composite.

Keywords: composite resins; polymerization; physical properties.

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Influência do protocolo de fotoativação gradual no grau de conversão de uma resina composta nanohíbrida

RESUMO

OBJETIVO: Analisar a influência do protocolo de fotoativação gradual no grau de conversão de uma resina composta nanohíbrida.

METODOLOGIA: Dez espécimes foram confeccionados a partir da polimerização da resina composta Llis, utilizando os seguintes protocolos: convencional (1000 mW/cm², durante 20 segundos) ou gradual (250 mW/cm² durante 20 segundos + 1000 mW/cm² durante 15 segundos). Posteriormente, foram realizadas análises com espectrofotômetro e os dados foram submetidos à análise de variância a dois critérios e o teste de Holm-Sidak. O nível de significância adotado foi 5%

RESULTADOS: Não houve diferença estatística entre a média dos valores de grau de conversão da resina composta na superfície de topo e de base, para ambos os protocolos de fotoativação ($p > 0,05$).

CONCLUSÃO: o uso do protocolo de fotoativação gradual não interferiu no grau de conversão de uma resina composta nanohíbrida.

Palavras-chave: resinas compostas; polimerização; propriedades físicas.

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INTRODUCTION

The clinical performance of composite resin restorations has improved significantly in the last decades [1]. However, the occurrence of enamel and dentin cracks, cusp fractures, postoperative sensitivity and formation of gaps between the restoration and the tooth continue to be problems frequently observed in dental practice [2]. Many of these setbacks are related to the polymerization contraction of the composite resins and the stress generated at the tooth/restoration interface [3].

The polymerization reaction begins with the interaction between photons of visible light, at a wavelength between 400 and 500 nm, and the initiators, producing extremely reactive free radicals [4]. Free radicals, in turn, promote the breakdown of the unsaturated carbon bonds (C=C), aliphatic (1636 nm) and aromatic (1608 nm) groups, and the dimethacrylate monomers, initiating a chain reaction [4]. A high degree of conversion of resinous monomers into polymers is fundamental for the composite resins to have satisfactory physical properties [5].

On the other hand, the process of forming the polymer chain of the composite resins leads to the approximation of the monomers, and reduces the intermolecular distance of approximately 4 Å to 1.5 Å. Thus, the polymerization will lead to a significant volumetric reduction, which can vary between 2 and 4% [6]. Such volumetric shrinkage, when occurring in the pre-gel phase (fluid composite resin), can be dissipated through the flow of the molecules of the material, allowing the stress relief [7]. However, when the gel-point is reached (passage from the fluid state to the viscous state), a significant increase occurs in the amount of crosslinking and in the modulus of elasticity of the material, resulting in the transmission of stresses to the dental structure [8]. The polymerization shrinkage stress can damage the marginal sealing of adhesive restorations, form gaps and promote cusp displacement [3, 9].

An alternative for reducing the polymerization shrinkage stress is the use of gradual curing protocol, also known as soft start [10]. In this protocol, a low power density is applied during the first phase of the polymerization period and a higher power density is used at the end of the irradiation [10]. This method of photoactivation allows the pre-gel phase to be prolonged, resulting in a greater relief of tensions generated during the polymerization shrinkage [11]. Studies have shown that the soft-startcuring protocol gives the composite resins satisfactory mechanical characteristics, similar to those obtained with the conventional protocol [12, 13].

The degree of conversion of the composite resins is influenced by several factors, among them the composition of the material [14]. Nanohybrid composite resins were marketed in order to be used on anterior and posterior teeth due totheir excellent initial polishing, gloss maintenance over time, and satisfactory mechanical properties [15]. These materials have inorganic particles of nanometric size (0.02 µm) combined with particles of conventional size (1 µm) [15]. Although composite nanohybrid resins are widely used, there are few studies that evaluate the use of the soft-startcuring protocol associated with this type of composite resin.

The aim of this study was to analyze the influence of the soft-start curing protocol on the degree of conversion of a nanohybrid composite resin. The null hypothesis tested was that there will be no significant difference in the degree of conversion of a nanohybrid composite resin independent of the curing protocol used.

METHODS

Ten specimens were performed with a nanohybrid composite resin – Llis®; FGM, Joinville, SC, Brazil – (**Table 1**) using a metal matrix with a hole in the center with dimensions of 5 mm in diameter × 2 mm in height.

Each specimen was obtained from the insertion of a single increment of the composite resin in the center of the matrix, with the aid of a SuprafillDuflex spatula (SS White, Rio de Janeiro, RJ, Brazil). Subsequently, the composite resin increment was accommodated with a Hollenback #5 metal condenser (Golgran, Sao Caetano do Sul, SP, Brazil), and covered with a polyester matrix and a glass cover, which was manually pressed by 60 seconds, promoting extravasation of excess material.

After removal of the glass cover, the specimens were photoactivated through the polyester tape by using a light curing unit (Poly Wireless; Kavo®, Joinvile, SC, Brazil) following two curing protocols (**Table 2**).

Table 1. Composition of the composite resin.

Composite resin	Composition
Composite resin Llis® (color dA3)	Bis-GMA monomers, Bis EMA, TEGDMA, camphorquinone, co-initiators, silane. Micronized barium-aluminosilicate glass, pigments and nanometric silica.

Bis-GMA: Bis-PhenolAdi-Glycidyl Methacrylate; Bis EMA: Bis-PhenolAdi-Glycidyl Methacrylatoethoxylate; TEGDMA: Triethyleneglycoldimethacrylate.

Table 2. Description and energy density of the curing protocols.

Curing protocol	Description	Energy density
Conventional (n=5)	1000 mWcm^2 for 20 s	20 J/cm^2
Soft-start (n=5)	250 mW/cm^2 for 20 s + 1000 mW/cm^2 for 15 s	20 J/cm^2

Immediately after photoactivation, the top and bottom surfaces of the specimens were identified and stored individually in amber vials at 37°C for 24 hours. To determine the degree of conversion, analyseswere made on top (0 mm) and bottom (2 mm) surfaces of each specimen by a Raman micro spectrometer (Xplora; Horiba Scientifc, Kyota, Japan). The spectrum was excited from the use of a laser with wavelength at 532 nm through an

objective (100 X). The spectrum was obtained, according to the following conditions: irradiation time: 60 s; number of accumulations: 10 and grade: 1200 lines/mm. The degree of conversion was calculated based on the reduction of the peak intensity corresponding to the C=C methacrylate groups at 1,636 cm -1 and 1,608 cm -1 polymerized (P) compared to the unpolymerized (U) specimen, according to the following equation:

$$Degree\ of\ conversion = \left(1 - \frac{P}{U}\right) \times 100$$

From the data obtained, Shapiro-Wilk test was applied. Subsequently, Two-way ANOVA (curing protocol and surface area) and Holm-Sidak test for *post-roc* comparisons were applied. Statistical procedures were performed with Sigmastat 3.5 software for Windows (Systat Software Inc., San Jose, CA, USA) and in all situations, the level of significance was 5%.

RESULTS

The degree of conversion values are shown in **Table 3**. The results were not influenced by the curing protocol ($p=0.921$, $F=0.0103$) and by the surface of the composite resin ($p=0.242$, $F=1.477$). The interactions were not statistically significant ($p=0.550$, $F=0.373$).

There was no statistically significant difference between the degree of conversion values to the bottom and top surface for both curing protocols ($p>0.05$). In each surface (top or bottom), no statistically significant difference was observed between the degree of conversion values obtained from each curing protocol ($p>0.05$).

Table 3. Mean of the degree of conversion (%) and standard deviation, between the surfaces, according to the curing protocol.

Curing protocols	Surfaces	
	Top	Bottom
Conventional ($n=5$)	$81,2 \pm 4,0^{A,a}$	$77,3 \pm 4,0^{A,a}$
Soft-start ($n=5$)	$80,1 \pm 2,4^{A,a}$	$78,8 \pm 7,4^{A,a}$

Notes: Similar capital letters indicate no significant difference in the columns; similar lowercase letters indicate no significant difference on the lines.

DISCUSSION

The degree of conversion of a composite resin corresponds to the percentage of double carbon bonds converted into single bonds to form a polymer chain [16]. This percentage is related to the mechanical properties of the composite resin, where a high degree of conversion is fundamental for its satisfactory clinical performance [17].

Different tests can be used to evaluate the degree of conversion of resin monomers, such as infrared Fourier transform spectroscopy [18], Raman spectroscopy [19,20], and microhardness tests [21]. In the present study, the direct method of evaluating the degree of conversion of the composite resins using Raman spectroscopy was used. Raman spectroscopy quantifies the degree of conversion during polymerization by comparing non-polymerized residual methacrylate vibration bands [9].

The proper polymerization of the composite resins is directly related to their degree of conversion. In the attempt to promote high conversion of monomers into polymers associated with low shrinkage stress, different curing protocols were developed [22]. In the conventional curing protocol, a highpower light is applied, in a constant mode, which should result in higher degree of conversion values [23]. However, some studies have shown that the use of this protocol does not result in a greater degree of conversion when compared to other protocols [13, 24]. These data corroborate with the findings of the present study (**Table 3**).

On the other hand, the possibility of negative influence of the high light intensity on the development and relaxation of the shrinkage stress should be considered. The instantaneous passage from a less viscous (pre-gel) state to a rigid (post-gel) state, rapidly increases the modulus of elasticity and reduces the possibility of relief from the polymerization shrinkage [25]. In attempt to reduce this issue, the soft-startcuring protocol was developed. This protocol uses low initial light intensity, forming a smaller number of free radicals, which limits the amount of methacrylate monomers groups converted into polymers [11]. This way, the polymerization reaction proceeds more slowly, allowing stress relief through the flow of the molecules (pre-gel phase). When the composite resin reaches the gel point, the maximum flow will occur, and then light with high intensity will be applied to complement the polymerization reaction [11].

When polymerization is carried out with low light intensity, the composite resin can be sub-polymerized, resulting in a low degree of conversion of monomers into polymers, with consequent damage to the properties of the material [21]. However, Emami and Solderholm [24] concluded that different protocols using equivalent energy density give similar degree of convertion values, regardless of the low initial energy intensity applied. These findings corroborate with the results of the present study. This way, the null hypothesis failed to be rejected.

The methodology used to evaluate the degree of conversion of the composite resins through Raman spectroscopy does not provide data on the shrinkage stress, being a limitation of the present study. Therefore, new studies should be developed to evaluate the influence of soft-startcuring protocol on the reduction of shrinkage stress of composite resins.

CONCLUSION

According to the applied methodology, it was verified that the use of soft-startcuring protocol did not interfere in the degree of conversion of a composite nanohybrid resin.

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