

UNIVERSIDADE ESTADUAL DE CAMPINAS  
FACULDADE DE ODONTOLOGIA DE PIRACICABA

KARLA MYCHELLYNE COSTA OLIVEIRA

Análise fotoelástica da tensão de contração da polimerização  
de compósitos resinosos sob influência de técnicas  
restauradoras

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Banca examinadora:

Simonides Consani [Orientador]

Alexandre Luiz Souto Borges

Luciano de Souza Gonçalves

Mario Alexandre Coelho Sinhoreti

Luis Roberto Marcondes Martins

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Prof. Dr. SIMONIDES CONSANI

Prof. Dr. ALEXANDRE LUIZ SOUTO BORGES

Prof. Dr. LUCIANO DE SOUZA GONÇALVES

Prof. Dr. MARIO ALEXANDRE GOELHO SINHORETI

Prof. Dr. LUIS ROBERTO MARCONDES MARTINS

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## RESUMO

A tensão de contração da polimerização é uma característica indesejável e inevitável das restaurações adesivas. É importante entender a distribuição da tensão para melhorar a efetividade clínica das restaurações de resina composta.

Capítulo 1: o objetivo foi avaliar a tensão de contração da polimerização e o grau de conversão de um compósito resinoso com diferentes modos de ativação e densidades de energia. O compósito Z350 foi inserido no orifício central do disco de resina fotoelástica e fotoativado com LED (Bluephase-2) com densidade de energia de 12, 24 ou 36 J/cm<sup>2</sup> usando os seguintes protocolos de fotoativação: contínuo de alta intensidade (HI: 1200 mW/cm<sup>2</sup> por 10, 20 ou 30 s), contínuo de baixa intensidade (LI: 650 mW/cm<sup>2</sup> por 18, 36 ou 54 s) e soft-start (SS: 150 mW/cm<sup>2</sup> por 5 s + 1200 mW/cm<sup>2</sup> por 9, 19 ou 29 s) (n=5). A tensão de contração de polimerização foi avaliada por meio de análise fotoelástica e o FTIR foi usado para análise do grau de conversão imediatamente e 24 horas após a ativação. ANOVA 3-fatores foi usado para avaliar os principais efeitos e as interações correspondentes, depois ANOVA 2-fatores foi realizado para cada tempo (p<0,05). As imagens mostraram que a tensão aumentou quando maior densidade de energia foi usada. A tensão não foi estatisticamente significativa entre os métodos de ativação de alta e baixa intensidade. O método soft-start gerou tensões menores do que os modos contínuos quando 12 J/cm<sup>2</sup> usado. O grau de conversão do modo soft-start foi estatisticamente menor que os modos de alta e baixa intensidade. Não foram encontradas diferenças no grau de conversão entre as doses de energia testadas. Pode-se concluir que a tensão de contração foi influenciada pela dose de energia e pelos protocolos de polimerização. O grau de conversão não foi influenciado pelo aumento das densidades de energia.

Capítulo 2: O objetivo foi avaliar a tensão de contração criada em modelos fotoelásticos usando diferentes técnicas de inserção do compósito. Vinte réplicas de dentes em resina fotoelástica com cavidade classe II foram obtidos e distribuídos em quatro



grupos (n=5) de acordo com as seguintes técnicas restauradoras: inserção horizontal (H), inserção oblíqua (O), compósito fluido + inserção horizontal (F+H), compósito fluido + inserção oblíqua (F+O). Os modelos restaurados foram analisados por meio de polariscópio plano. A tensão ao longo de 7 áreas da interface adesiva foi analisada a partir das imagens obtidas após inserção de cada incremento e 24h após a fotoativação. Os valores de tensão máxima foram submetidos a ANOVA 2-fatores, seguido pelo teste de Tukey ( $p < 0.05$ ). As técnicas de inserção horizontal e oblíqua apresentaram diferenças no padrão de tensão. Apesar disso, nenhuma diferença no valor de tensão entre as técnicas horizontal e oblíqua foi encontrada. Os grupos com resina fluida mostraram maior tensão que aqueles restaurados apenas com compósito convencional.

Palavras chaves: Compósitos, tensão de contração de polimerização, técnicas de inserção, modos de ativação, análise fotoelástica.

## ABSTRACT

Polymerization shrinkage stress is an undesirable and inevitable characteristic of adhesive restorations. It is important to understand the stress distribution to improve the clinical effectiveness of resin composite restoration. Chapter 1: The aim was to evaluate the polymerization shrinkage stress and degree of conversion of a resin composite material submitted to different photoactivation protocols. A bulk increment of composite Filtek Z350 was placed in the central perforation of photoelastic disc and photoactivated using LED-based curing unit (BluePhase II) with energy density of 12, 24 or 36 J/cm<sup>2</sup> using the following photopolymerization protocols: continuous high intensity (HI: 1200 mW/cm<sup>2</sup> during 10, 20 or 30 s), continuous low intensity (LI: 650 mW/cm<sup>2</sup> during 18, 36 or 54 s) and soft-start (SS: 150 mW/cm<sup>2</sup> during 5 s + 1200 mW/cm<sup>2</sup> during 9, 19 or 29 s) (n=5). Photoelastic analysis was used to evaluate polymerization shrinkage stress of composite and FTIR was performed to access the degree of conversion of composite according to the curing modes. Statistical test ANOVA 3-way was used to determine the main effects and the corresponding interactions, then ANOVA 2 way was performed for each time (p<0.05). The images showed that polymerization shrinkage stress increases with higher values of power density. No significant differences should be noticed on polymerization shrinkage stress between high and low intensity modes of activation. Soft-start method generates stresses that were statistically lower than continuous modes except when 12 J/cm<sup>2</sup> was applied. Similar degree of conversion was observed for photoactivation modes used, except for soft-start mode with 12, 24 and 36 J/cm<sup>2</sup> that showed lowest levels of conversion. It could be concluded that polymerization shrinkage stress was significantly influenced by energy density and the curing protocol. No benefits on degree of conversion were observed by increasing the energy density. Chapter 2: The aim was to evaluate the polymerization shrinkage stress created in tooth photoelastic models using different filling techniques. Twenty class II tooth models were obtained. They were distributed into four experimental groups (n=5) according to the following

restorative techniques: resin composite with horizontal insertion (H), resin composite with oblique insertion (O), flowable composite + resin composite with horizontal insertion (F + H) and flowable composite + resin composite with oblique insertion (F + O). Restored photoelastic models were analyzed using a plane polariscope. The stress along seven points of adhesive interface was analyzed from images of each insertion, at the baseline (immediately) and 24 h after polymerization. Maximum shear stress data was obtained and submitted to ANOVA 2-way, followed by Tukey's post hoc test at  $p = 0.05$ . Horizontal and oblique techniques have shown differences in stress patterns. No difference between horizontal and oblique techniques was found. Groups restored with flow composite showed significant higher stress levels than those restored only with regular composite. In conclusion, the use of flow composite created higher polymerization stress effects on the class II restoration than does the conventional restoration technique.

Keywords: Composites, polymerization shrinkage stress, filling techniques, photopolymerization modes, photoelastic analysis.

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## INTRODUÇÃO

A contração da polimerização em materiais resinosos ocorre devido à conversão de ligações Van Der Waals em ligações covalentes C-C (Atai et al., 2005). Quando existe adesão entre o material resinoso e o dente, tensões podem ser criadas nas paredes da cavidade (Davidson, 2000). A tensão de contração da polimerização é clinicamente significativa considerando que pode ser capaz de causar propagação de trincas no esmalte (Yamamoto et al., 2009) e de romper a camada adesiva na interface dente-restauração (Cavalcante et al., 2003; Irie et al., 2002; Peutzfeld & Asmusen, 2004) e conseqüentemente causar a formação de fendas marginais e subsequente infiltração de bactérias, fungos e toxinas que podem levar à hipersensibilidade dentinária, inflamação pulpar (Opdam et al., 1998). A Odontologia Restauradora busca alternativas para diminuir esses efeitos e aumentar a longevidade das restaurações diretas.

Vários fatores exercem influência sobre a tensão de contração da polimerização, dentre eles pode-se identificar desde aspectos inerentes à composição e propriedades do material; até fatores técnicos envolvendo energia fornecida para a ativação, tipo de cavidade e modos de inserção do compósito na cavidade (Bouschlicher et al., 1997; Charton et al., 2007; Dewaele et al., 2009).

A contração volumétrica não deve ser o único aspecto a ser considerado para prever as tensões geradas pela contração de polimerização (Braga et al., 2005). A tensão gerada pela contração de polimerização está diretamente relacionada com a velocidade de polimerização e o grau de conversão; o menor valor do grau de conversão está associado à redução da contração e da tensão (Braga & Ferracane, 2002). A conversão dos monômeros resinosos nunca atinge 100% e monômeros não reagidos que permanecem entre a cadeia polimérica podem agir como plastificadores, prejudicando as propriedades mecânicas da restauração e aumentando o risco de toxicidade aos tecidos adjacentes (Arikawa et al., 2004). O maior grau de conversão pode levar a melhores propriedades mecânicas, estabilidade química e longevidade do material (da Silva et al., 2008).

Tem sido mostrado que diferentes modos de fotoativação podem influenciar a tensão de contração de polimerização e propriedades dos compósitos (Bouschlicher et al., 1997; Braga & Ferracane, 2002; Ilie et al., 2005; Lim et al., 2002; Lu et al., 2005; Visvanathan et al., 2007). O controle da velocidade da reação de polimerização pode aumentar a capacidade de escoamento do compósito, permitindo o alívio das tensões causadas pela contração de polimerização (Tarle et al., 1998). Técnicas de fotoativação têm sido introduzidas com o objetivo de diminuir a tensão criada durante a reação de polimerização (Stansbury et al., 2005). Essas técnicas sugerem que fornecer baixa energia inicial para a polimerização do compósito poderia de certa forma facilitar o relaxamento da cadeia polimérica enquanto o material não atingiu o estágio de rigidez (Braga et al., 2005). Desta forma, a tensão gerada poderia ser diminuída (Lim et al., 2002; Obici et al., 2002). Entretanto, se essas técnicas verdadeiramente diminuem a tensão de contração e não comprometem a conversão das duplas ligações, conversão final dos monômeros e propriedades mecânicas dos compósitos, ainda são questões que estão sob constante debate na literatura e o real benefício dessas técnicas não foi clinicamente comprovado (Asmussen & Peutzfeldt, 2003; Soh & Yap, 2004; Visvanathan et al., 2007; Lopes et al., 2008).

Além disso, fatores técnicos como o método de inserção da resina na cavidade, também devem ser considerados. A distribuição da tensão tanto no material quanto nos tecidos dentários é dependente da forma dos incrementos de resina usados para restaurar a cavidade (Kowalczyk, 2009). Para reduzir a contração de polimerização é recomendado que pequenos incrementos de resina sejam polimerizados sequencialmente, desta forma a tensão criada nas paredes da cavidade também seria diminuída e maior polimerização do compósito na camada mais profunda seria alcançada (Carvalho et al., 1996). A distribuição da tensão em cavidades que possuem alto fator C é considerada desfavorável para a adesão à dentina profunda (Yoshikawa et al., 1999).

Compósitos fluidos tem sido recomendados como base de restaurações de resinas devido a baixa viscosidade e maior umedecimento (Neme et al., 2002).

Seu uso pode diminuir a formação de fendas na interface de união (Kwon et al., 2010) e reduzir a infiltração cervical (Kwon et al., 2010; Sabatini et al., 2010). Entretanto, a viscosidade dos monômeros também pode estar relacionada com a tensão de contração de polimerização dos compósitos resinosos (Charton et al., 2007). Materiais fluidos podem demonstrar maior contração de polimerização e propriedades mecânicas inferiores (Baroudi et al., 2008; Kleverlaan & Feilzer, 2005; Tjandrawinata et al., 2005). Alguns estudos mostraram que compósitos com baixa quantidade de carga apresentam maiores níveis de contração e conseqüentemente, maior tensão (Miyazaki et al., 1991; Baroudi et al., 2007; Gonçalves et al., 2010b, Takahashi et al., 2010). Compósitos que apresentam maior fluidez possuem maior escoamento e podem apresentar diferentes níveis de contração durante o processo de polimerização (Feilzer & Dauvillier, 2003).

A análise fotoelástica tem sido aplicada na Odontologia para visualização das tensões geradas sobre uma determinada estrutura, simulando o que ocorreria clinicamente quando essa estrutura está sob tensão. A técnica é baseada na propriedade óptica de certos materiais plásticos transparentes que apresentam diferentes índices de refração (ou anisotropia óptica) quando submetido à tensão/deformação (Dally & Rille, 1978). Essa metodologia permite a visualização dos eixos de bi-refringência do material fotoelástico que coincidem com os eixos das principais tensões geradas na estrutura (Ernst et al., 2004). Um modelo fotoelástico, quando submetido à carga, mostra-se caracterizado por zonas coloridas em função da passagem de luz branca polarizada. Essas zonas representam o quadro visível da distribuição das pressões por toda a área padrão convergindo para as regiões de concentração de pressão que são denominadas franjas. As franjas criadas possuem relações matemáticas precisas e irão determinar as deformações e tensões geradas no material (Ainola & Aben, 2005), fornecendo evidências sobre a localização e a intensidade da concentração de tensões (Post, 1979). Portanto, a análise fotoelástica tem se mostrado adequada para o bom entendimento da distribuição das tensões o que pode ser favorável

para prever a efetividade clínica de restaurações resinosas (Kinomoto & Torii, 1998).

O objetivo nesta Tese<sup>1</sup> apresentada em dois capítulos foi: 1. Avaliar a influência da densidade de energia e modo de ativação na tensão de contração de polimerização e grau de conversão de compósito resinoso. 2. Analisar a tensão de contração de polimerização gerada em cavidades classe II restauradas com diferentes técnicas de inserção do compósito restaurador.

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<sup>1</sup> Este trabalho foi realizado no formato alternativo, com base na deliberação n° 1985/96 da Comissão Central de Pós-Graduação (CCPG) da Universidade Estadual de Campinas (UNICAMP).



## CAPÍTULO 1 - Shrinkage stress and degree of conversion of a dental composite submitted to different photoactivation protocols

### ABSTRACT

The aim of this study was to evaluate the polymerization shrinkage stress and degree of conversion of a composite submitted to different photoactivation protocols. A bulk increment of composite Filtek Z350 was placed in the central perforation of a photoelastic disc and polymerized using LED-based curing unit (BluePhase II - IvoclarVivadent) with energy density of 12, 24 or 36 J/cm<sup>2</sup> using the following photopolymerization protocols: continuous high intensity (HI: 1200 mW/cm<sup>2</sup> during 10, 20 or 30 s), continuous low intensity (LI: 650 mW/cm<sup>2</sup> during 18, 36 or 54 s) and soft-start (SS: 150 mW/cm<sup>2</sup> during 5 s + 1200 mW/cm<sup>2</sup> during 9, 19 or 29 s) (n=5). Photoelastic analysis was used to evaluate polymerization shrinkage stress of composite and FTIR was performed to access the degree of conversion of composite according to the curing modes. Statistical test ANOVA 3-way was used to determine the main effects and the corresponding interactions, then ANOVA 2-way was performed for each time (p<0.05). The images showed that polymerization shrinkage stress increases with higher values of power density. No statistically significant differences on polymerization shrinkage stress should be noticed between high and low intensity modes of activation. Soft-start method generates stresses that were statistically lower than continuous modes except when 12 J/cm<sup>2</sup> was applied. Similar degree of conversion was observed for photoactivation modes, except for soft-start mode with 12, 24 and 36 J/cm<sup>2</sup> that showed lowest levels of conversion. It could be concluded that polymerization shrinkage stress was significantly influenced by energy density and the photoactivation modes. No benefits on degree of conversion were observed by increasing the energy density.

Keywords: Composites, polymerization shrinkage stress, photoactivation modes, photoelastic analysis.

### INTRODUCTION

The use of resin composites has significantly increased in restorative dentistry due to the importance of their aesthetic, mechanical and physical properties. However, polymerization shrinkage is still an inevitable problem. It happens because of the conversion of Van Der Waals bonds into shorter covalent C-C bonds<sup>1</sup>. When the composite is adhered to the cavity walls, this shrinkage creates stresses that lead to problems on the interface of restoration<sup>2</sup>. Polymerization shrinkage stress is directly related to the degree and rate of conversion<sup>3</sup>. In addition, the clinical performance of composites depends on degree of conversion of the monomers in the material. Monomers conversion never achieve 100%, and many monomers which have not reacted remain within the polymer network, damaging the mechanical qualities of the restoration and being potentially toxic to surrounding tissues<sup>4</sup>. Better mechanical properties, chemical stability and longevity of the composite are associated to higher degrees of conversion<sup>5</sup>.

The rate of polymerization reaction control may increase the resin flow index, allowing release of stress, which were caused by polymerization shrinkage<sup>6</sup>. Different activation modes can influence the polymerization shrinkage stress and final properties of composites<sup>3, 7, 8, 9, 10</sup>. Photoactivation techniques have been introduced with the aim of reducing the stress created during polymerization process. In soft-start technique, the activation begins with low intensity followed by a high intensity irradiation<sup>11</sup>. One hypothesis for the use of this technique is that the initial low irradiance could allow the polymer chains to relieve stresses easier while the material did not achieve its rigidity yet<sup>12</sup>, which could reduce stress levels<sup>8</sup>. However, the literature still discusses if these techniques can reduce the stress without compromise the monomers final conversion and their clinical benefits have not been proved<sup>10, 13, 14</sup>.

Photoelastic analysis allows visualization of birefringent axis in photoelastic materials that coincides with main stress axis in the material<sup>15</sup>. The fringes created

have precise mathematic bases that determine stresses and strains in the material<sup>16</sup>.

Faced with these findings, the aim of this study was to evaluate the polymerization shrinkage stress and degree of conversion of a resin composite material submitted to different photoactivation protocols, involving different activation modes and energy densities.

## MATERIAL AND METHODS

The composite Filtek Z350 (3M-ESPE, St. Paul, MN, USA) was the resinous material elected for this study and the adhesive system SingleBond 2 (3M-ESPE, St. Paul, MN, USA) was used to improve the union of the composite to the photoelastic resin.

The light source tested was the LED BluePhase II (IvoclarVivadent - Schaan, Principality of Liechtenstein) which presents three LED light sources and one that emits ultraviolet radiation.

### 1.1. Samples Preparation

Discs with 20 mm in diameter and 2 mm in thick, with a central orifice 6 mm in diameter were made with Araldite GY 279/ Aradur 2963 resin (Araltec Chemical Products - SP, Brazil). Inner preparation surfaces of photoelastic resin discs were roughened by wear with a drill to improve the adhesion between photoelastic resin composite and composite. Discs were stored at 37°C for 24 hours to allow stress relief in photoelastic resin. Before specimen preparation, the discs were placed in the photoelastic device to verify residual stress, and if detected, it was stored in an oven at 37°C for more 24 hours until stress was completely released.

The spot size of the light source was measured with a digital caliper (Mitutoyo, Japan) for the area measurement (cm<sup>2</sup>). The power (mW) of the light was measured with Ophir 10A-V2-SH (Ophir Optronics, Har – Hotzvim, P.O.B. 45021, Jerusalem 91450, Israel). Then, the irradiance (mW/cm<sup>2</sup>) of light source

was obtained and the density energy or power density was standardized through different exposition times. The energy density ( $\text{J}/\text{cm}^2$ ) is obtained from the irradiance multiplied by the time (seconds) and divided by 1000.

Araldite disc inner surfaces were treated with the adhesive system (SingleBond 2, 3M-ESPE, St. Paul, MN, USA) following the manufacturer's instructions. A bulk increment of composite was placed in the central orifice of photoelastic disc and polymerized using the LED-based curing unit (BluePhase II - IvoclarVivadent) using three different curing modes and three energy densities.

Five specimens were prepared for each group ( $n=5$ ) following photopolymerization protocols were used: continuous high intensity (HI:  $1200 \text{ mW}/\text{cm}^2$  during 10, 20 or 30 s), continuous low intensity (LI:  $650 \text{ mW}/\text{cm}^2$  during 18, 36 or 54 s) and soft-start (SS:  $150 \text{ mW}/\text{cm}^2$  during 5 s +  $1200 \text{ mW}/\text{cm}^2$  during 9, 19, 29 s). The power density was standardized in 12, 24 or  $36 \text{ J}/\text{cm}^2$ .

## 1.2. Optical Calibration

To obtain the photoelastic constant ( $K_\sigma$ ) of Araldite resin, optical calibration was performed<sup>17</sup>. Briefly, a beam of photoelastic resin was loaded using the four-points bending method; this method produces stress patterns of pure bending, generating horizontal isochromatic fringes from a neutral axis on the center of the beam. According to the plot of  $3Pa$  vs.  $h^2N$  the mean coefficient value was  $K_\sigma = 11271 \text{ N}/\text{m}$ . Where  $P$  is the load applied to the beam (N),  $a$  is length between beam supports (m),  $h$  is the beam cross-section height (m) and  $N$  the fringe order.

## 1.3. Photoelastic Analysis

Photoelastic images were recorded with a 2.1 megapixel digital camera (FD MAVICA, SONY, model MVC-FD97, Japan) immediately after light activation and 24 hours post-polymerization in a plane photoelastic device with the exact filter angulations (90 degrees) to obtain constant colored fringes. Fringes formed on photoelastic resin were qualitatively analyzed based on Stress-Optical Law.

Black and white images were taken to obtain constant light and dark fields on images. These images were used to analyze stresses quantitatively based on certain fundamental physical principles. To obtain shrinkage stress values ( $\sigma_0$ ) from the isochromatic-diameter rings the equation used was:  $\sigma_0 = (N_x D_x^2 K_\sigma)/(D_i^2 2d)$ , where:  $N_x$  is the isochromatic ring order;  $D_x^2$  the isochromatic ring diameter (mm);  $D_i^2$  the disc preparation diameter (mm);  $K_\sigma$  the photoelastic constant (N/mm) and  $d$  the Araldite disc thickness (mm).

Polymerization shrinkage stress data (MPa) were calculated based on the location and diameter of the first order isochromatic ring obtained from Araldite discs, on monochromatic images. Three measurements of photoelastic fringe were made in standard directions (vertical, horizontal and diagonal) of each specimen image using the ImageJ software (National Institutes of Health - NIH, Maryland - USA). The mean fringe diameter was obtained and used in the above-mentioned photoelastic equation to calculate stress values of each specimen.

#### 1.4. Degree of Conversion Measurements

Degree of conversion (DC) was measured by Fourier Transformation Infrared - FTIR (Prestige21; Shimadzu, Columbia, ML, USA). Samples with 4.5 mm in diameter and 1mm in thickness were prepared from a metallic mold following each photoactivation protocol. The uncured and cured samples of the composite were placed above a horizontal zinc crystal and were photopolymerized according to respective photoactivation protocol. Measurements were made immediately and 24 hours after photoactivation ( $n=5$ ). Absorption spectra were recorded in a transmission mode using 16 scans at a resolution of  $4 \text{ cm}^{-1}$ . The ratio of absorption maximum was determined using the baseline method considering the peaks of  $1638 \text{ cm}^{-1}$  for aliphatic and  $1608 \text{ cm}^{-1}$  for aromatic chains. The degree of conversion was calculated from the equivalent aliphatic/aromatic molar ratio from polymerized (P) and unpolymerized (U) samples using the formula:  
$$\text{DC (\%)} = (1-P/U) \times 100.$$

### 1.5. Statistic Analysis

The assumption of normality distribution and equality of variance were checked in the variables. In the first step, polymerization shrinkage stress and degree of conversion data were analyzed by ANOVA 3-way considering 3 power density, 3 activation modes and 2 times to determine the main effects and the corresponding interactions. In a second step, separate analysis each time was performed by ANOVA 2 way. The level of significance was 5%.

## RESULTS

Figures 1, 2 and 3 show representative photoelastic images taken immediately and at 24 hours after the different photo-activation modes with 12, 24 and 36 J/cm<sup>2</sup> respectively.

The images show that polymerization shrinkage stress increases with higher values of power density. Slight differences should be noticed between high and low intensity modes of activation. Soft-start method generates less stress considering all power density tested.

Considering polymerization shrinkage stress, three-way ANOVA revealed significant differences ( $p < 0.05$ ) between all factors. Regarding to the interactions only photoactivation mode\*energy density was statistically significant ( $p < 0.05$ ).

The distribution and median of polymerization shrinkage stress values immediately and in 24 hours are shown in Figure 4.

Statistical tests showed that the polymerization shrinkage stress was similar when 12 and 24 J/cm<sup>2</sup> were applied on composite immediately after the photo-activation. The power density of 36 J/cm<sup>2</sup> created the highest stress values in all times evaluated. When 12 J/cm<sup>2</sup> was used, there was no statistical differences between the three activation modes immediately after polymerization, but in 24 hours soft-start and low intensity modes showed values of stress lower than high intensity mode. Immediately after photoactivation, the composites that received 24 J/cm<sup>2</sup> presented statistical significant differences on stress values between low intensity and soft-start modes, lowest values were presented by soft-start mode.

After 24 hours, the stress was comparable for all activation modes. Soft-start mode had the lowest stress values when 36 J/cm<sup>2</sup> of energy was applied on the photoactivation. In 24 hours, the stress created by high intensity mode was higher than low intensity and soft-start modes.

Table 1 shows average values and standard deviation (SD) of polymerization shrinkage stress immediately after photoactivation and after 24 hours for all activation modes tested. No statistical differences between the time were observed for continuous high intensity mode and high energy density groups.

Figure 5 shows the distribution and median of composite degree of conversion immediately and 24 hours after photoactivation. No statistical differences were found among the three energy dose tested ( $p > 0.05$ ). Soft-start photoactivation mode showed lowest values of conversion for all energy doses tested ( $p < 0.05$ ) and no significant differences were found between high and low continuous activation modes.

## DISCUSSION

As consequence of the methacrylate matrix polymerization, the composite material undergoes a fast temperature and shrinkage increase<sup>18</sup>. Curing contraction is a complex process involving many interdependent factors: the number of covalent bonds formed<sup>3</sup>, the nature of the initial monomers, the ratio, size, and type of fillers<sup>19</sup>; the concentration of photoinitiators<sup>20</sup>, and the light energy supplied<sup>5, 21, 22</sup>. Polymerization shrinkage can create destructive stresses when the material is bonded to cavity walls<sup>12</sup>. Degree of conversion measurements helped to better understand and explain polymerization shrinkage stress results.

Polymerization stresses of light-curing polymer-based material are influenced by the amount of energy delivered to the material during irradiation<sup>23</sup>. The control of polymerization rates seems to allow the flow of resin composite during curing and consequently minimize polymerization shrinkage stress<sup>24</sup>. This can be done by

polymerization at low power density followed by final cure at high power density<sup>12</sup>. Besides, when a reduction of the shrinkage rate of the composite is obtained, the effect is a reduction of stress developed into the restorative material locally<sup>25</sup>. Our results showed that soft-start curing mode showed lower stress rates of composite tested when high light energies densities (24 and 36 J/cm<sup>2</sup>) were applied. It has been claimed that slower polymerization causes an improved flow of molecules in the material, decreasing the polymerization shrinkage stress in a restoration<sup>8</sup>. In addition, at a constant energy density, pulse-delay curing reduced the bonding strength of resin composite to dentin compared to continuous curing<sup>26</sup>. Although, light modulation at the beginning of the photoactivation provides low shrinkage stress, low rates of degree of conversion were also observed. This is in accordance with other study<sup>13</sup> that also reported reduction on hardness of composites after ethanol storage<sup>13, 27</sup>.

Considering polymerization shrinkage stress, soft-start curing technique did not show differences from continuous techniques tested when 12 J/cm<sup>2</sup> was applied. During polymerization process, thermal variation can be observed into the composite, and the temperature developed may increase stresses into the material if there is no time to dissipate thermal energy<sup>28</sup>. The low amount of energy may have created less alteration on the composite temperature and consequently stress differences between the curing techniques could not be noticed. Moreover, according to Dewaele et al.,<sup>29</sup> (2009) comparisons of the continuous and the stepped mode of photo-activation are ambiguous. Those authors attribute this to the fact that, in the step-cure mode, power density of the first step has effects on degree of conversion and on density of cross-linking in the opposite direction. When high power density is used, two phenomena can occur: higher concentrations of radicals that rapidly react with each other are produced, thus yielding premature termination and resulting in lower degree of conversion; or a multitude of growth centers are created, increasing the tendency to form a branched polymer (higher crosslink density). On the other side, a lower power density in the first step proportionally yields higher efficiency of the radicals initiated



and this result in a delayed termination of polymerization and in higher degree of conversion. But lower power density would lead to relatively few growth centers, and a relatively linear polymer will be formed. Also, soft curing polymerization can result in reduced shrinkage stress levels and keep the degree of conversion and mechanical properties constant<sup>7</sup>.

At a constant energy density, high and low intensity continuous modes showed similar degree of conversion and polymerization stress by photoelastic analysis. The LED unit used in this study provided power energy of 650 mW/cm<sup>2</sup> when low intensity mode was used, which literature considers medium-high power intensity<sup>23, 30, 31</sup> and this amount of energy was sufficient to satisfactory polymerize the composites and achieve similar levels of conversion of the group that receive the high (1200 mW/cm<sup>2</sup>) light energy, this is in accordance with Dewaele et al., 2009<sup>29</sup>. Once polymerization shrinkage stress is directly related to degree of conversion<sup>3</sup>, this could be an explanation for the comparable stresses found.

The energy density is not only related with degree of conversion, but also with the elastic modulus and the glass transition temperature (T<sub>g</sub>); the higher energy density applied to the material, the higher are elastic modulus and T<sub>g</sub><sup>29</sup>. The visco-elastic properties, such as polymerization shrinkage, polymerization reaction rate and modulus of elasticity are very important in the pattern of shrinkage stress<sup>32</sup>. The results of this study showed that polymerization with 36 J/cm<sup>2</sup> energy presented higher stress than with 12 and 24 J/cm<sup>2</sup>. Despite the same composite has been used for all curing modes in this study, the higher energy density can make the material achieve higher degree of conversion and T<sub>g</sub> values<sup>29</sup>, and consequently should present higher stress levels. In addition, new generations of the LED units emit high-power blue light, which may cause higher temperature rise due to the light irradiation<sup>33</sup>. To achieve 36 J/cm<sup>2</sup> the exposition time of the light source was increased and this may have caused greater increase in temperature than the samples that received less exposition time and energy. Given the amount and the thickness of resin to be cured, temperature and shrinkage rate during the light curing polymerization can raise by increasing the power output depending on

the quality of the emitted light spectra<sup>28</sup>, this can help to explain the higher stresses observed when 36 J/cm<sup>2</sup> of energy was applied to start the composite cure. The use of high energy densities results in an unfavorable relationship between degree of conversion and contraction stress<sup>34</sup>.

In this study, no difference on degree of conversion was observed among the different energy densities applied, probably a satisfactory conversion of the composite was achieved with 12 J/cm<sup>2</sup>. Also, as polymerization reaction occurs, the conversion rate decreases drastically as the stiffness of the material increases and restrict the mobility of monomers or growing polymer chains to continue the reaction; and it becomes difficult to increase conversion significantly, even using high energy densities<sup>35</sup>.

There was no difference in polymerization shrinkage stress when composite was photo-activated with 12 and 24 J/cm<sup>2</sup>. Both power energies seem to be appropriate to polymerize and create comparable conversion of the composite, and consequently no differences on polymerization shrinkage stress was found. The composite can be successfully polymerized at reduced light intensity, while the final conversion value remains high<sup>28</sup>. Despite that, after 24 hours for the high intensity continuous activation, when 12 J/cm<sup>2</sup> of energy was applied the stress was higher than the other modes. Although this fact could not be observed with 24 J/cm<sup>2</sup> of energy density, it is known that degree of conversion of composites is directly related to polymerization shrinkage stress created on the wall that composite is bonded<sup>3</sup>. In addition, thermal stress can reduce the effectiveness of dentin adhesives<sup>36</sup>. Probably the energy provided to composite when 24 J/cm<sup>2</sup> was applied created high stresses after 24 hours that were able to disrupt the bond between composite and photoelastic discs walls, but the integrity of this interface was not a goal on this study.

Within 24 hours, differences between the curing modes were observed when 36 J/cm<sup>2</sup> of energy density was applied. Continuous high intensity mode created higher stresses than continuous low intensity mode, and soft-start mode showed lower stress rates. One explanation can be that huge thermal contraction force is

evident using high power curing unit and shows a steep stress contraction as the light is switched off <sup>34</sup> and this may become more evident after 24 hours on this study. Also, the polymerization contraction is only totally completed after 24 hours of composite activation<sup>37</sup>, and this can help to explain the reason of differences between polymerization modes were more evident after 24 hours. According to Li et al. <sup>38</sup> (2008), polymerization shrinkage followed by Young's modulus of the composite are the factors of most importance in the development of residual stresses. Also, decreasing the viscosity and polymerization rate could reduce the residual stresses in the top section of the restoration, but not those in deeper locations.

Considering the same energy and activation mode, polymerization shrinkage stress of the composite increased after 24 hours only for the continuous low intensity ( $24 \text{ J/cm}^2$ ) and soft-start curing modes ( $12$  and  $24 \text{ J/cm}^2$ ). Properties of light-cured composite are strongly dependent on both the energy dose and the duration of the dark reaction process<sup>25</sup>. Even though, the stress created during the others curing modes have not showed statistical differences between stress values immediately and at 24 hours. The degree of conversion achieved by the composites cured with high power and energy density minutes after the activation did not significantly change within 24 hours and no changes in stress levels could be noticed. Also, no differences on degree of conversion were observed with the time, this can be explained because the monomer conversion achieve a maximum value and after that point no significant changes can be observed <sup>34, 35</sup>.

Within the limitations of this study, it can be concluded that polymerization shrinkage stress is significantly influenced by energy density increase. Soft-start curing mode can promote lower stress levels if the energy density of  $24$  and  $36 \text{ J/cm}^2$  were applied. Stress levels of the composite tested are similar minutes after activation and within 24 hours when the higher power energy and irradiance were applied. No benefits on degree of conversion were observed by increasing the energy density.

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## FIGURES

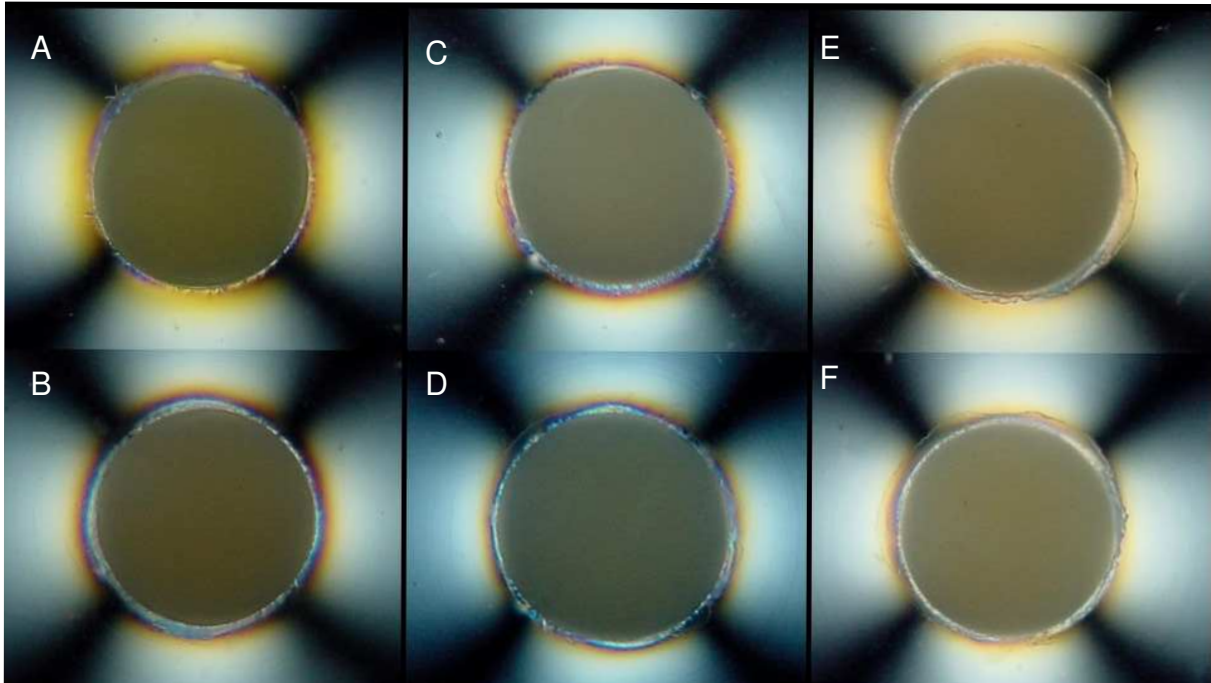


Figure 1 - Stress fringes promoted by polymerization shrinkage stress of composite photo-activated with  $12\text{J}/\text{cm}^2$ : (A and B) High intensity immediate and 24 hours respectively; (C and D) Low intensity immediate and 24 hours; and (E and F) Soft-start immediate and 24 hours.



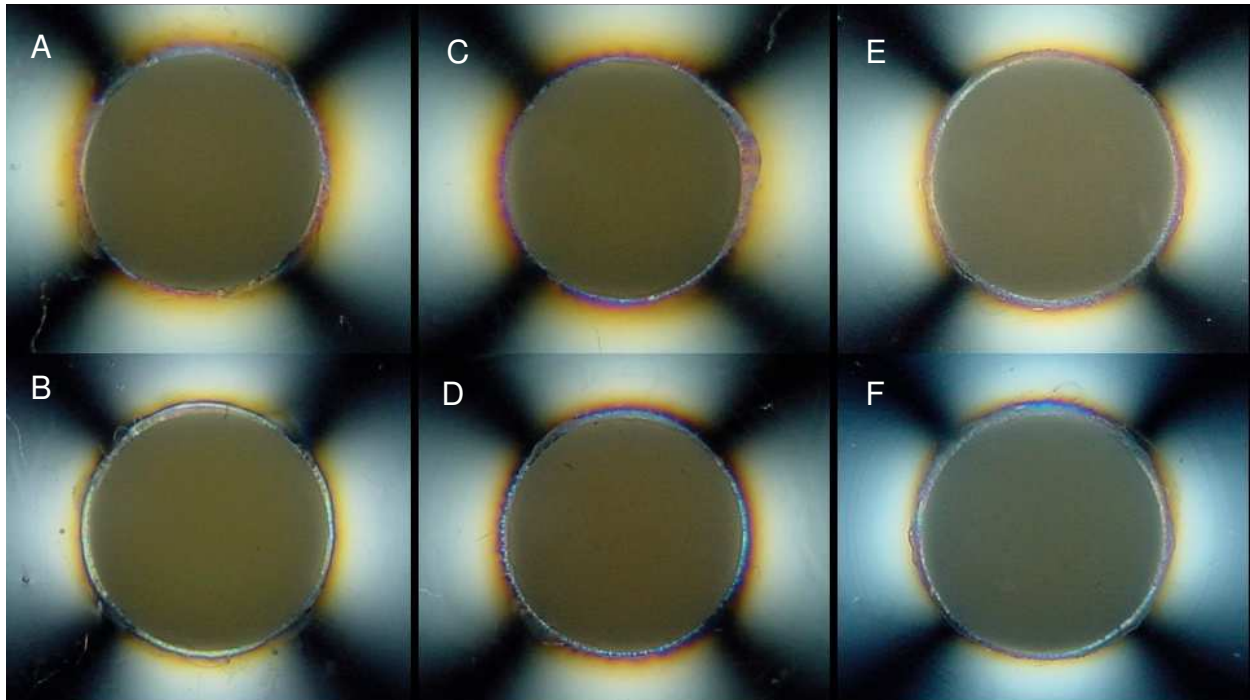


Figure 2 - Stress fringes promoted by polymerization shrinkage stress of composite photo-activated with  $24\text{J}/\text{cm}^2$ : (A and B) High intensity immediate and 24 hours respectively; (C and D) Low intensity immediate and 24 hours; and (E and F) Soft-start immediate and 24 hours

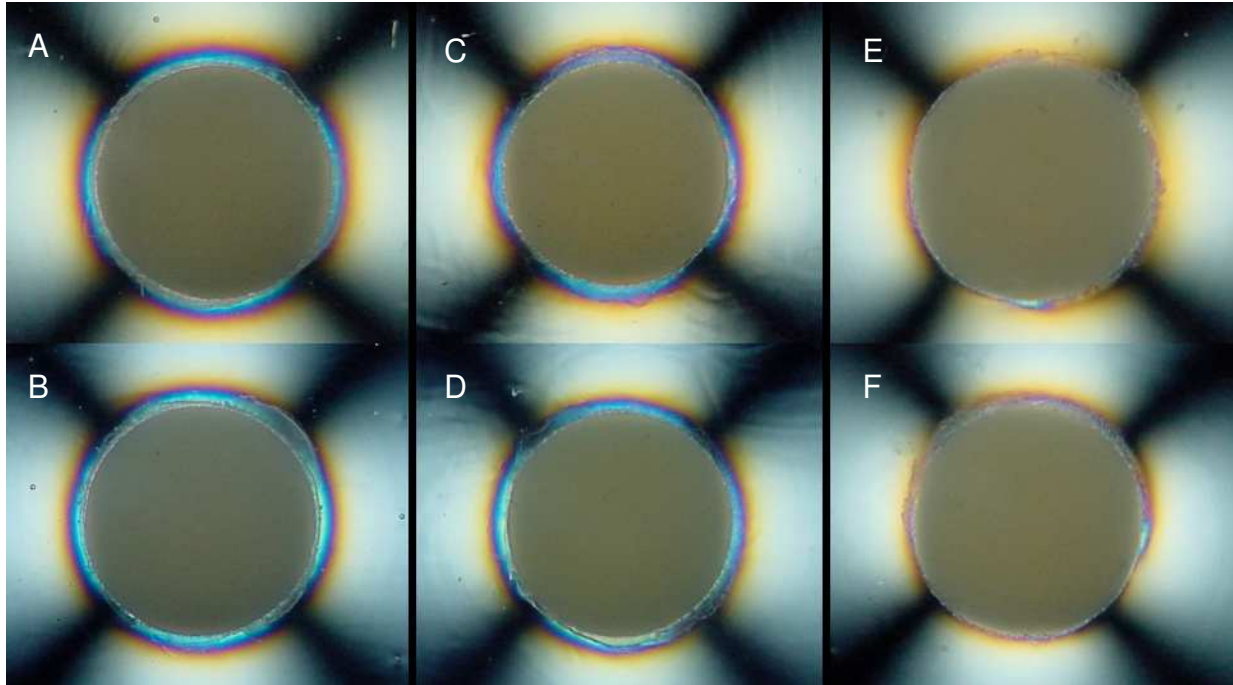


Figure 3 - Stress fringes promoted by polymerization shrinkage stress of composite photo-activated with  $36\text{J}/\text{cm}^2$ : (A and B) High intensity immediate and 24 hours respectively; (C and D) Low intensity immediate and 24 hours; and (E and F) Soft-start immediate and 24 hours.

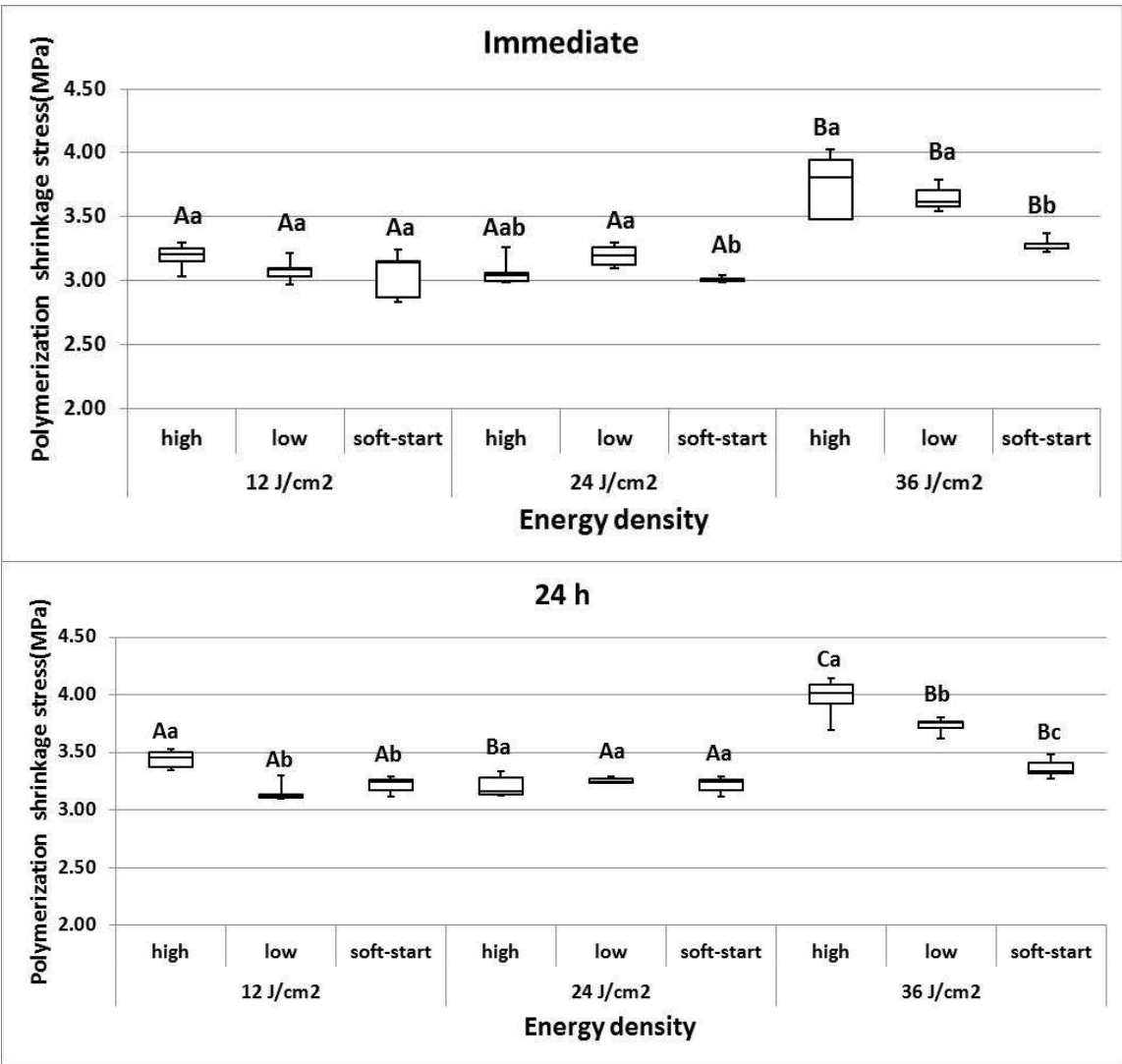


Figure 4 - Charts box plot of polymerization shrinkage stress immediately and 24 hours after different power density and photoactivation modes. \* Different capital letters compare different energy density. Different lowercase letters compare different curing modes ( $p < 0.05$ ).

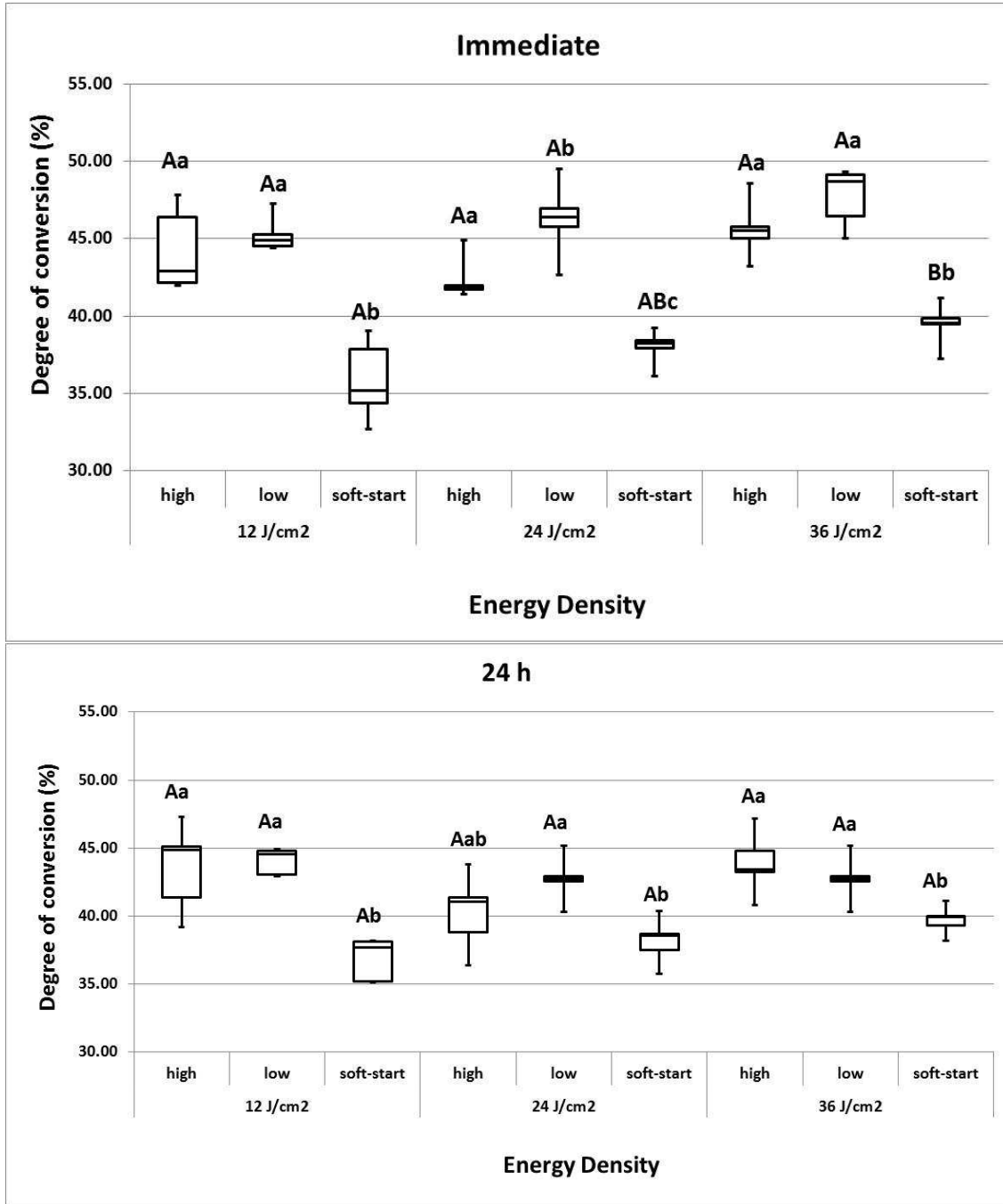


Figure 5 - Charts box plot of degree of conversion immediately and 24 hours after different power density and photoactivation modes. \* Different capital letters compare different energy density. Different lowercase letters compare different curing modes ( $p < 0.05$ ).

TABLES

Table 1 - Average and SD of polymerization shrinkage stress values immediately after photoactivation and after 24 hours for all activation modes tested (MPa).

Energy density	Continuous High Intensity		Continuous Low intensity		Soft-Start	
	Immediate	24 hours	Immediate	24 hours	Immediate	24 hours
<b>12 J/cm<sup>2</sup></b>	3.19 (0.16) A	3.44 (0.17) A	3.08 (0.09) A	3.15 (0.09) A	3.05 (0.18) A	3.13 (0.12) B
<b>24 J/cm<sup>2</sup></b>	3.07 (0.11) A	3.21 (0.10) A	3.19 (0.09) A	3.26 (0.03) B	3.01 (0.02) A	3.22 (0.07) B
<b>36 J/cm<sup>2</sup></b>	3.75 (0.26) A	3.97 (0.24) A	3.65 (0.10) A	3.73 (0.07) A	3.28 (0.05) A	3.36 (0.08) A

\* Different letters mean statistical differences between the time in the same activation mode and energy density (p<0.05).

## CAPÍTULO 2 - Influence of filling techniques on shrinkage stress in class II dental composite restorations<sup>2</sup>

### Summary

Polymerization contraction stress is an undesirable and inevitable characteristic of adhesive restorations. It is important to understand the stress distribution to improve the clinical effectiveness of resin composite restoration. Objective: The aim of this study was to evaluate the polymerization shrinkage stress created in tooth photoelastic models using different filling techniques. Methods: Twenty class II tooth models were obtained. They were distributed into four experimental groups (n = 5) according to the following restorative techniques: resin composite with horizontal insertion (H), resin composite with oblique insertion (O), flowable composite + resin composite with horizontal insertion (F + H) and flowable composite + resin composite with oblique insertion (F + O) restoration. Restored photoelastic models were analyzed using a plane polariscope. The stress along seven points of adhesive interface was analyzed from images of each insertion, at the baseline (immediately) and 24h after polymerization. Maximum shear stress data was obtained and submitted to ANOVA 2-way followed by Tukey's post hoc test at p = 0.05. Results: Horizontal and oblique techniques have shown differences in stress patterns. No difference between stress values of horizontal and oblique techniques was found. Groups restored with flow composite showed significant higher stress levels than those restored only with universal composite. Conclusion: The use of flow composite created higher polymerization stress effects on the class II restoration than does the conventional restoration technique.

Keywords: Composites, polymerization shrinkage stress, filling techniques, photelastic analysis.

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<sup>2</sup> Artigo submetido para apreciação do corpo editorial da Journal of Dentistry.

## INTRODUCTION

An important factor that contributes to deterioration of dental resin composite restorations is contraction stress that occurs during polymerization<sup>1</sup>. The shrinkage of the resin composite is clinically significant, because it creates forces that may disrupt the bond to the cavity walls<sup>2, 3, 4</sup>. This competition between contraction forces within composites and bonds to the cavity wall may cause gaps between dentinal wall and the restorative material<sup>5, 6</sup>, and result in stresses inside the restoration<sup>7</sup>. As a consequence, it can cause marginal failure and subsequent microleakage that permits the passage of bacteria, fluids and toxins and could encourage dentinal hypersensitivity, secondary caries and pulpal inflammation<sup>8</sup>. Besides that, contraction stresses generated by curing dental resin composites are sufficient to cause propagation of cracks from flaws existing in a surrounding brittle material<sup>9</sup>.

The insertion method of the resin on the cavity is important regarding to stress created on it. Also, the stress distribution in the restoration and tooth tissues, is strongly dependent on a shape of the filling layers<sup>10</sup>. A method used to reduce contraction is the resin placement in small layers and its polymerization, decreasing the stress on the cavity walls generated during the polymerization shrinkage and increasing the depth of cure<sup>11</sup>. The distribution of the internal stresses in a composite restoration into a box-shaped cavity is considered to be unfavorable for the deep dentin bond<sup>12</sup>.

Flowable composites have been recommended as liners beneath composite resins due to their low viscosity, increased elasticity and wettability<sup>13</sup>. The use of flowable composites and compomers as liners decrease gap formation<sup>14</sup> and reduce gingival microleakage<sup>15,16</sup>. Although, some studies showed that composites with lower filler content have higher shrinkage levels than conventional viscosity composites<sup>17,18,19</sup> and polymerization shrinkage is directly related to polymerization shrinkage stress<sup>20</sup>.

A good understanding of the stress distribution may improve the clinical effectiveness of resin composite restoration<sup>21</sup>. The photoelastic analysis is a

reliable method to evaluate the stress pattern created during the restorative process. Besides, photoelastic analysis is widely used for problems in which stress or strain information is required for extended regions of the structure. It provides quantitative evidence of highly stressed areas and peak stresses at surface and interior points of the structure<sup>22</sup>.

This study proposed to evaluate the polymerization shrinkage stress created in class II cavities using different filling techniques by photoelastic analysis.

## MATERIAL AND METHODS

### 1. Photoelastic model

A mandibular first molar metallic model with a class I cavity (disto-mesial distance: 7 mm, vestibulo-lingual distance: 3 mm, depth: 3 mm) was duplicated with a resinous material. The replica was sectioned in the proximal to obtain a plane surface that allows better visualization. After the section, a class II cavity was obtained. Silicon (HydroXtreme, Vigodent) molds were made from replicas and a troquel of photoelastic resin (Araldite GY 279/ Aradur 2963 resin - Araltec Chemical Products - SP, Brazil) was obtained from each mold. C-Factor was calculated as the ratio of bonded and unbonded surfaces areas ( $C=2,2$ ).

### 2. Restorative procedures

The composites Filtek Z350 and Filtek Z350 Flow were used in this study (Table 1).

The photoelastic models were assigned to four experimental groups ( $n = 5$ ) according to the following restorative techniques: resin composite with horizontal insertion (H), resin composite with oblique insertion (O), flowable composite + resin composite with horizontal insertion (F + H) and flowable composite + resin



composite with oblique insertion (F + O) restoration. All materials were used according to the manufacturer's instructions.

For all photoelastic replicas, the preparations were acid-etched with 35% phosphoric acid for 60s (ScotchBond Etchant; 3M ESPE, St. Paul, MN, USA) before the restorative procedures to remove any contaminant that could interfere with the bonding procedures. The preparations were then rinsed for 10 seconds and blot-dried using an oil free air for 60 seconds. Then, a thin layer of the adhesive Single-Bond 2 was applied into the cavity and photoactivated according to the manufacturer's instructions.

### 2.1. Horizontal Technique

After adhesive application, a standardized increment ( $\approx 28.3 \text{ mm}^3$ ) of the composite (Filtek Z350, 3M-ESPE) was placed horizontally in the bottom of the cavity filling the third part of its extension. Then the composite was photoactivated with the LED light curing unit (BluePhase II - IvoclarVivadent, Schaan, Principality of Liechtenstein) using the high intensity mode ( $1200 \text{ mW/cm}^2$ ) for 20 seconds. Another 2 increments were placed and photoactivated in the same way to fill the cavity.

### 2.2. Oblique Technique

The first composite increment ( $\approx 28 \text{ mm}^3$ ) was placed into the cavity in contact with the pulpar and vestibular walls. The second increment was placed in contact with the first increment and the lingual wall; then the third increment was placed horizontally over the other increments. Each increment was photoactivated in the same conditions of horizontal technique.

### 2.3. Flow + Horizontal Technique

A thin layer ( $\approx 0.5 \text{ mm}$ ) of the flow composite (Filtek Z350 Flow) was applied on the bottom of the cavity and photoactivated for 20 seconds in similar conditions explained above. Then, two standardized increments of composite were placed horizontally as described for the technique.

#### 2.4. Flow + Oblique Technique

First the flow composite was used as described on item 2.3. Then, two increments of composite were applied as described for the oblique technique.

### 3. Photoelastic analysis

The restored photoelastic models were analyzed using a plane polariscope. Images of each insertion of the restored photoelastic models were recorded at the baseline (immediately) and 24 hours after polymerization. The stress along the adhesive interface was evaluated for all experimental groups. The isochromatic fringe patterns of the stressed sites were determined by their respective fringe orders. The photoelastic color patterns were used to compare the distribution of polymerization shrinkage stress within the different experimental groups (Figure 1A).

The quantitative analysis of stress was recorded immediately and 24 hours after photoactivation of each layer and the values of maximum shear stress ( $f_{max}$ ) calculated in MPa. Nine locations along the adhesive interface were selected and standardized for all specimens. Isochromatic fringe order values were calculated from the 9 standardized selected areas (figure 1B). Then, the relationship between principal stress ( $\partial_1$  and  $\partial_2$ ) and maximum shear stress ( $f_{max}$ ) was defined by the Stress Optic Law using the following equation:

$$\partial_1 - \partial_2 = N \cdot K_{\partial} = 2 \cdot f_{max} \cdot t$$

Where  $\partial_1$  and  $\partial_2$  are the principal stress,  $f_{max}$  is the maximum shear stress, N is the fringe order (isochromatic),  $K_{\partial}$  is the photoelastic constant/optical coefficient, and t is the thickness of the model. The optical coefficient of the photoelastic material was defined using the same calibration technique of Vásquez-Rodríguez et al. (2004)<sup>23</sup> and according to the plot of 3Pa vs.  $h^2N$ ; where P is the load applied to the beam (N), a is length between beam supports (m), h is the beam cross-section height (m) and N the fringe order, the mean coefficient value was  $K_{\partial} =$

11271 N/m. Using the values of photoelastic constant, thickness, and fringe order, the  $f_{\max}$  of each area was calculated using the above equation. The sum of  $f_{\max}$  of all areas was calculated to obtain the stress value into the sample.

#### 4. Statistical analysis

Maximum shear stress data of the sum of all points analyzes along the adhesive interface were submitted ANOVA 2-way, followed by Tukey's post hoc test at  $p = 0.05$ .

## RESULTS

Images of the stress created of photoelastic resin by polymerization shrinkage stress on class II cavities restored with horizontal, oblique, flow+horizontal and flow+oblique techniques are showed in figures 2, 3, 4 and 5 respectively. Although horizontal and oblique techniques have shown similar stress levels, differences in the stress profile were noticed. Horizontal technique created tension more linear pattern than oblique technique. ANOVA 2-way analysis showed that the use of the flow composite, as a liner in dental composite class II restorations, increased the stresses created on photoelastic model ( $p < 0.05$ ). The average of total stress calculated for each group after insertion of the increments is shown in Figure 6.

Statistical analysis showed that there was no difference between horizontal and oblique techniques ( $p > 0.05$ ). Except when only the first increment was considered, stress levels of oblique technique were lower than that observed on horizontal technique. Groups restored with flow composite showed significant higher stress levels than those restored only with regular composite ( $p < 0.05$ ). No statistical differences were observed related to time comparison. The stress levels observed after the third increment were similar after 24 hours for all filling techniques tested ( $p = 0.120$ ).

Nine areas were designated to stress analysis, although the areas 1 and 9 were disregarded because the tooth anatomy of photoelastic model and the capture angle of the images did not allow adequate visualization of these regions. Stress values and standard deviation (SD) in each point and the total stress created on the model on the last insertion and within 24 hours are shown in table 2.

The stress distribution along the 7 points of interface in all increments of the groups tested presented a similar profile. The only exception were the areas 4, 5, 6 and 7 in the increment 1 of oblique group that showed lower values than the other groups (Fig. 7).

## DISCUSSION

The phenomenon of contraction stress development in dental composite is highly complex and remains as a significant clinical concern <sup>24</sup>. Stress profile obtained from photoelastic analysis provided a better comprehension of stresses within the tooth photoelastic model cavity. In this study, the internal stress generated in the prepared cavity could be measured and quantitatively evaluated. Although, the differences of the physical properties such as elastic modulus between the natural teeth and photoelastic resin models used in this study should be considered for interpretation of the stress.

Shrinkage stress occurs when the contraction is obstructed and the material is rigid enough to resist plastic flow and compensate the volume alteration <sup>25</sup>. The use of an incremental filling technique can compensate polymerization shrinkage effects and perhaps decrease tensile stress concentrations at the restoration interface<sup>26, 27</sup> and reduce the cuspal strain <sup>28, 29</sup>. Literature have stated that stresses created by composite polymerization shrinkage reflect on cavity walls and as consequence can compromise adhesive bond and/or cause cusp deflection, compromising the longevity of restoration <sup>3, 30</sup>. Deflection seems to be related to the combination of polymerization shrinkage and elastic modulus <sup>31</sup>. However, the

polymerization shrinkage data cannot predict the marginal integrity of direct restorations<sup>32</sup>. Contraction stresses generated by curing dental resin composites are sufficient to cause propagation of cracks from flaws existing in a surrounding brittle material<sup>9</sup>.

According to the results of this study, horizontal and oblique filling techniques create similar stress levels on the photoelastic model. Others studies revealed insignificant differences between various layering build-up techniques<sup>26</sup> and horizontal incremental filling produces cuspal deflexion similar to oblique incremental filling<sup>29</sup>. It is known that for the same material, polymerization shrinkage is dependent on the volume of composite polymerized<sup>33</sup>, and in this study, the amount of composite for each increment was standardized for all groups, this could help to explain the same stress levels. Also, all experimental groups received the same amount of energy in this study, and polymerization time of the composite material is more important than the restorative procedure in preventing stresses<sup>26</sup>. Although no statistical differences between horizontal and oblique filling techniques were found, from photoelastic images and mapping stress along interface points, it could be noticed that oblique first increment revealed less stress levels (Figure 7). The shape of the increment on the oblique technique covers less surfaces of cavity wall than horizontal technique does; and this could have created less stress during this evaluation phase.

Analysis of the fringes created on photoelastic model revealed that stress directions on horizontal technique are more linear than when oblique technique was applied. This can indicate that during the restoration building, stresses created on cavity walls follow the shape of the composite increment. According to Li et al. (2008)<sup>34</sup> the size of the restoration had relatively little influence on the residual stress development. Moreover, observing the stresses created along the interface, it could be noticed that regions in the pulp wall showed higher shear stresses than axial walls for all techniques tested. During photoactivation, the light initiates the polymerization reaction first on the external surface of composite that may pull pulp

wall and creates more stress in this area. Also, axial walls are less restricted and may permit higher stress relief than pulp walls.

Gonçalves et al. (2010)<sup>35</sup> found a strong correlation between stress and shrinkage; and viscosity followed the same trend, showing a strong, exponential correlation with stress too. Groups restored with flow composites showed higher stress levels on the model than groups where only the composite with conventional viscosity was used. It is known that Filtek Z350 Flow present less filler content compared to Filtek Z350 (information provided by the manufacturer). This is in accordance with the results of Oliveira et al. (2010)<sup>36</sup> which showed an increase of maximum shear stress when flowable resin composite and resin-modified glass-ionomers were used as cavity liner and/or base. Volumetric shrinkage and elastic modulus are highly dependent on the material's filler content<sup>37</sup>. Composites with high filler content have low matrix fraction, which determines the volume reduction observed during formation of a dense cross-linked polymeric network<sup>38</sup>. In addition, the decrease in inorganic content lead to an increase in polymerization stress as result of an increase in composite volumetric shrinkage<sup>35</sup>. The use of flow composites have been indicated as liners in composites restorations, considering its better capacity to flow through pulp wall and improve adaptation of the composite restorations<sup>15</sup>. Flowable composites presented higher organic matrix content and polymerization shrinkage values. Regression analyses showed significantly negative linear correlations between gap formation and strain capacity, and between gap formation and flow, and a significantly positive linear correlation between gap formation and shrinkage (Dewaele et al., 2006). Therefore, the use of flowable composites as liners can represent a negative effect regarding to polymerization stress and integrity of the restoration.

Moreover, the content and type of monomers present on material composition can influence polymerization shrinkage stress<sup>35,37</sup>. TEGDMA is known as a diluent monomer with low molecular weight<sup>39</sup>. Filtek Z350 flow presents more TEGDMA content than Filtek Z350 and this could also have caused the higher stresses levels observed on cavities restored with the flow composite. The

systematic increase in TEGDMA increases the degree of conversion, volumetric shrinkage, elastic modulus and polymerization stress for monomers mixtures, which are harmful for the integrity of the bonded interface of composite restorations<sup>35</sup>.

No differences between total stress after the polymerization of the third increment and within 24 hours were found for the techniques tested. Polymerization shrinkage stress is directly related to degree of conversion and rate of polymerization<sup>40</sup>. Despite the composite shrinkage increases after stopping the light irradiation due to post-polymerization process<sup>41</sup>, the photoelastic model analyzed in this study could compensate the composite additional shrinkage and relief the stresses created. In addition, the final stresses in interface are not necessarily the higher values obtained in the sample<sup>42</sup>. Moreover, resin composite and flowable composite polymerization stress achieves stability after 4 hours<sup>36</sup>.

Clinicians must understand the polymerization shrinkage stress and realize that the quality of composite resin restorations depends on successful management of these stresses<sup>7</sup>. Within the limitations of this study, it was concluded that no differences between layering techniques tested were found. Flowable composites created higher polymerization stress than does the conventional composites.

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## Figures

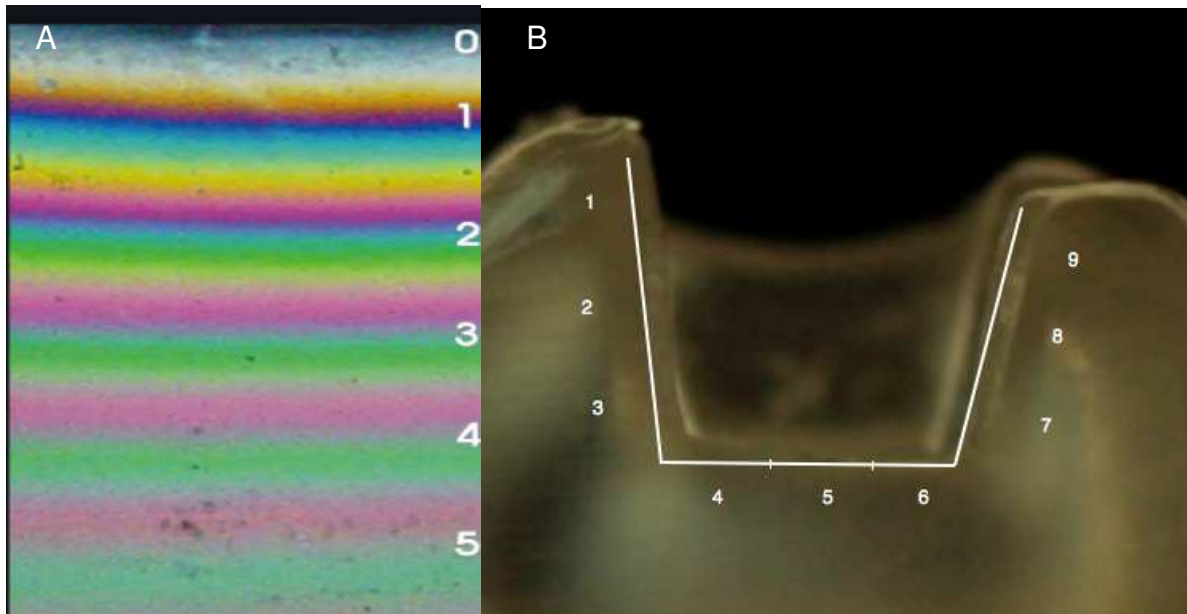


Figure 1 - (A) Schematic demonstration of isochromatic fringe order. (B) Nine points along the cavity interface.

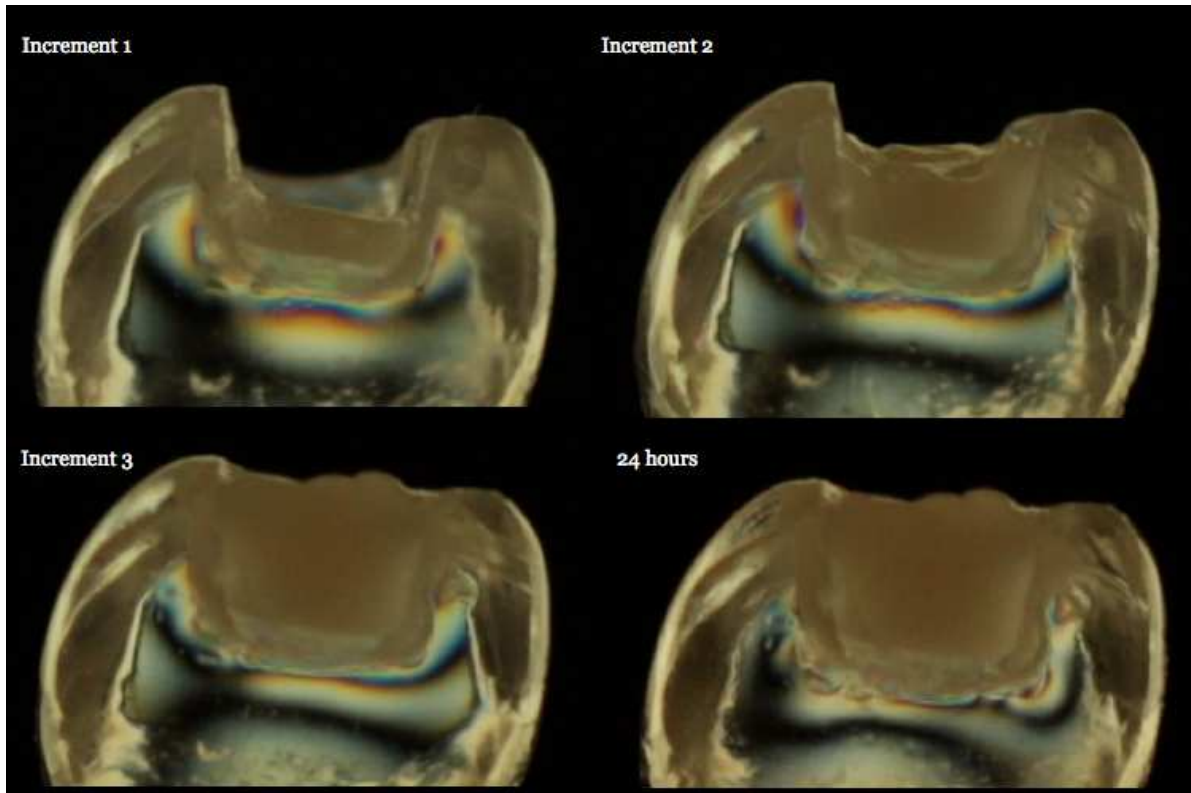


Figure 2 - Horizontal technique

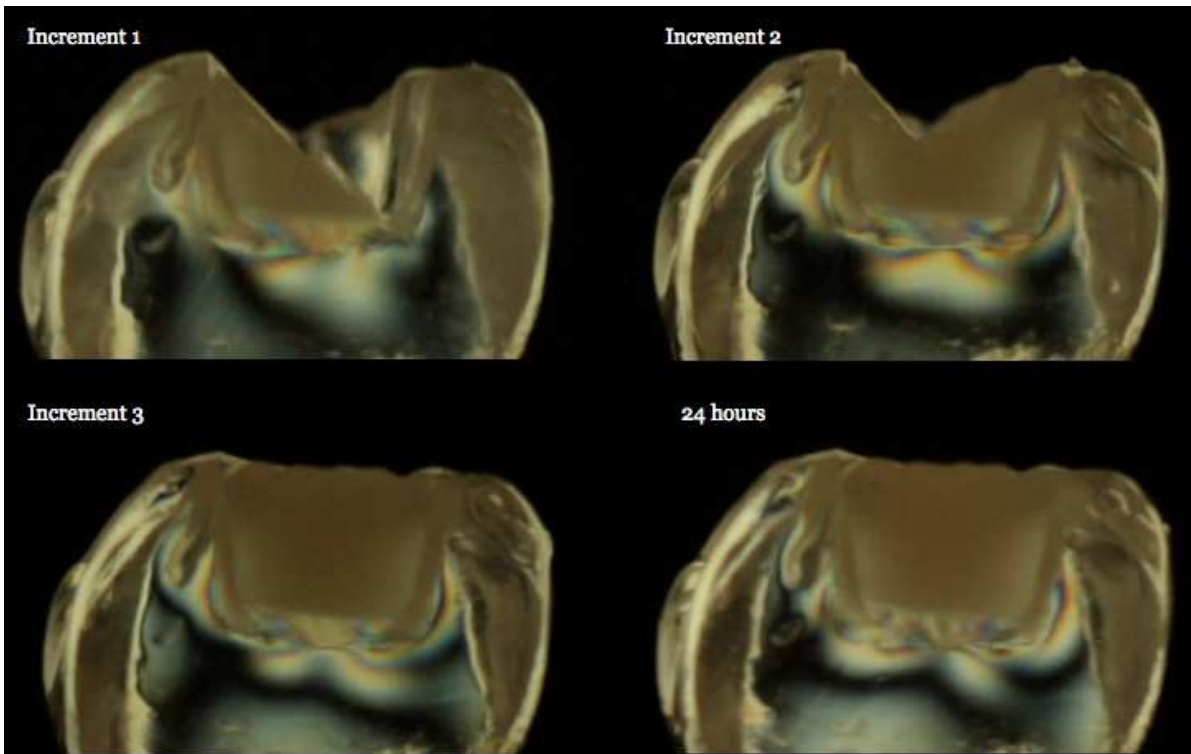
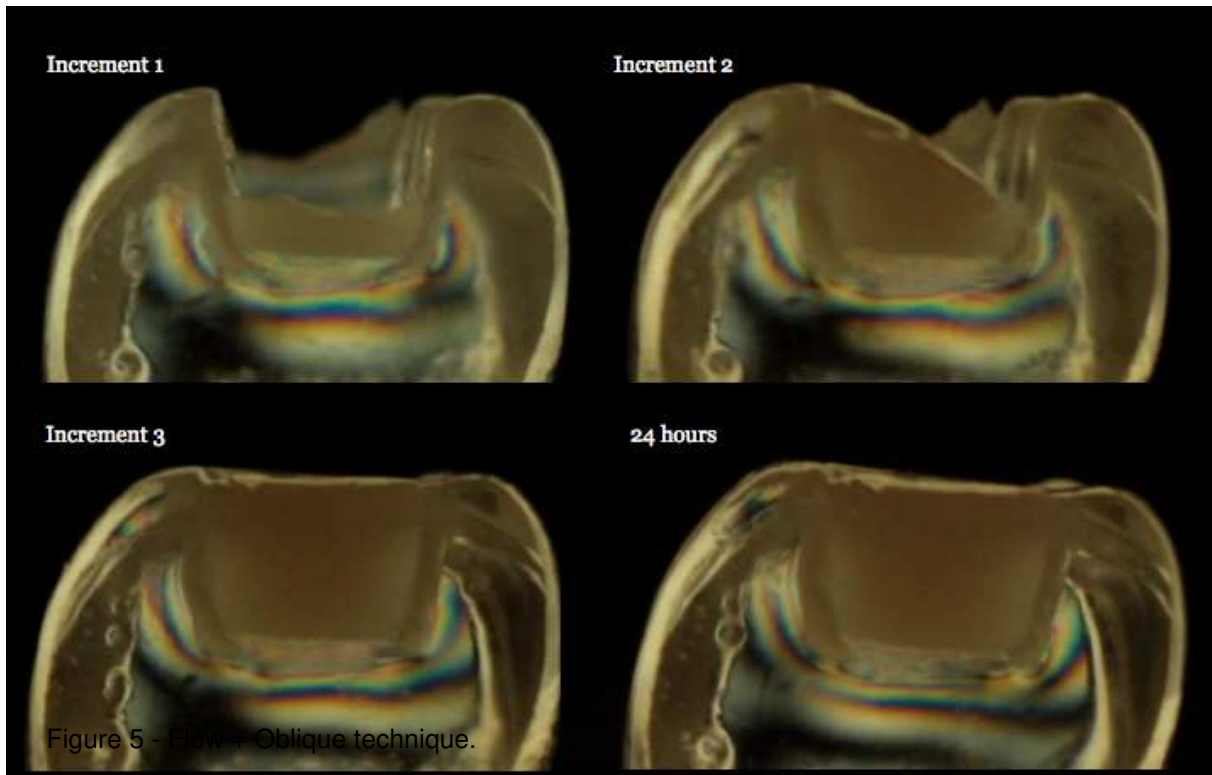
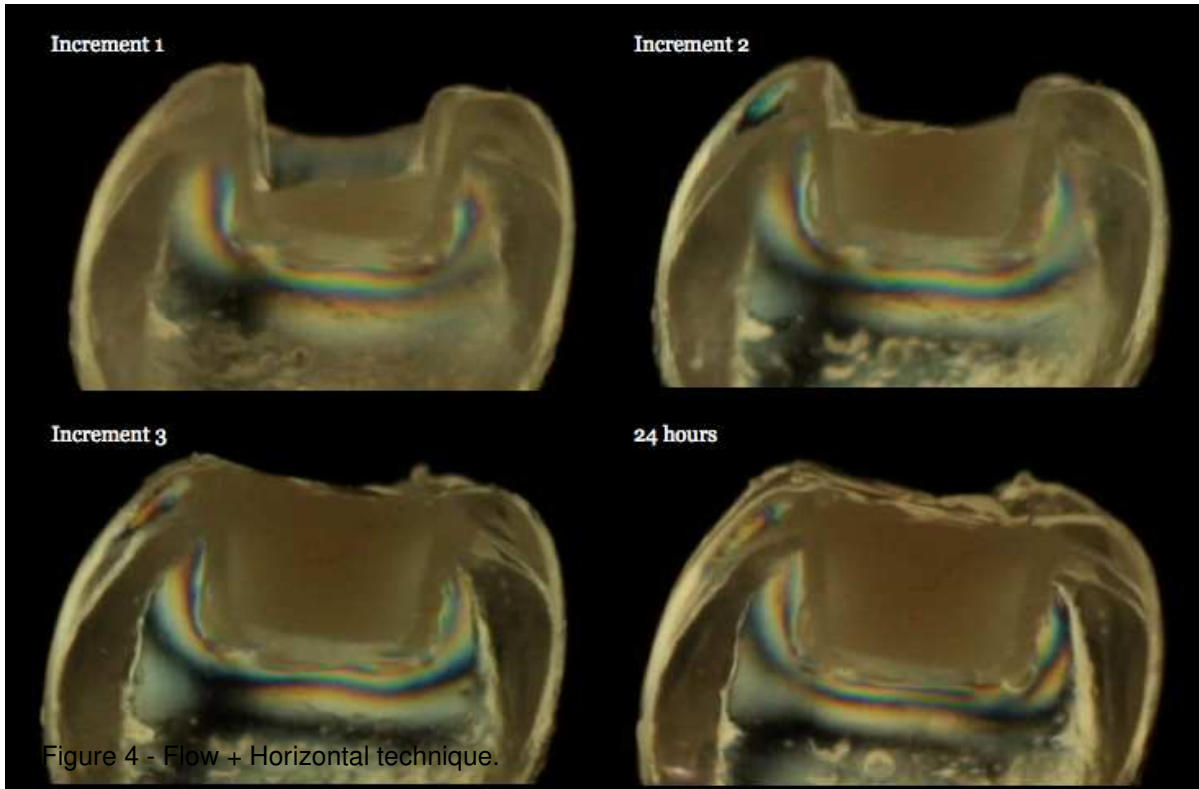


Figure 3 - Oblique technique



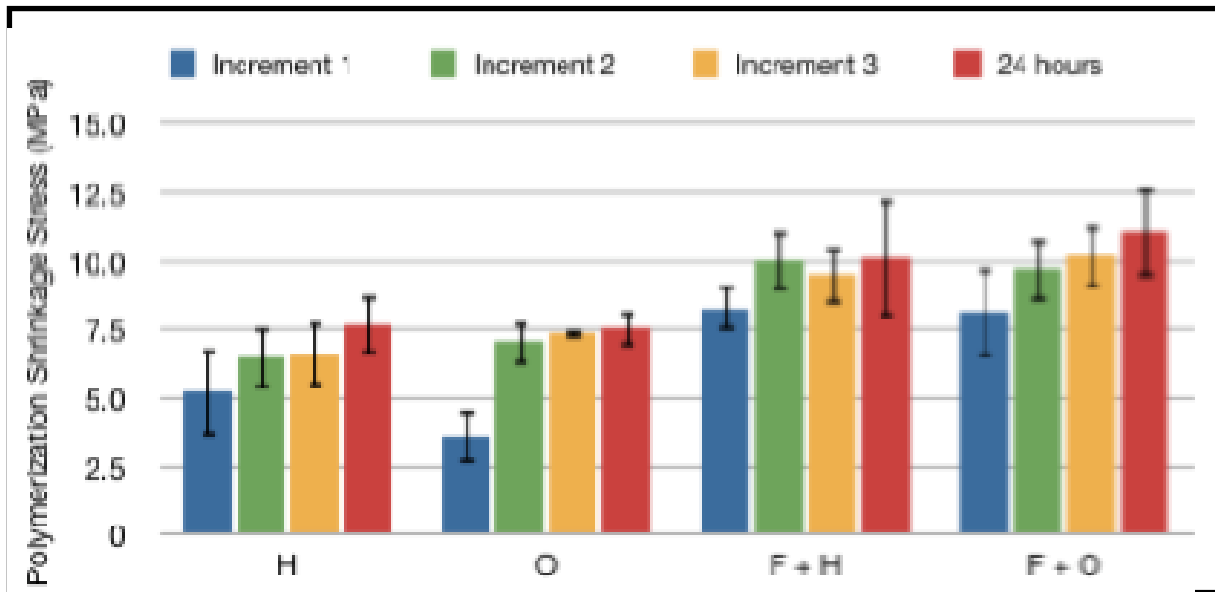


Figure 6 - Chart of the total stress created by polymerization of composite after increments insertion and in 24 hours for all groups tested.

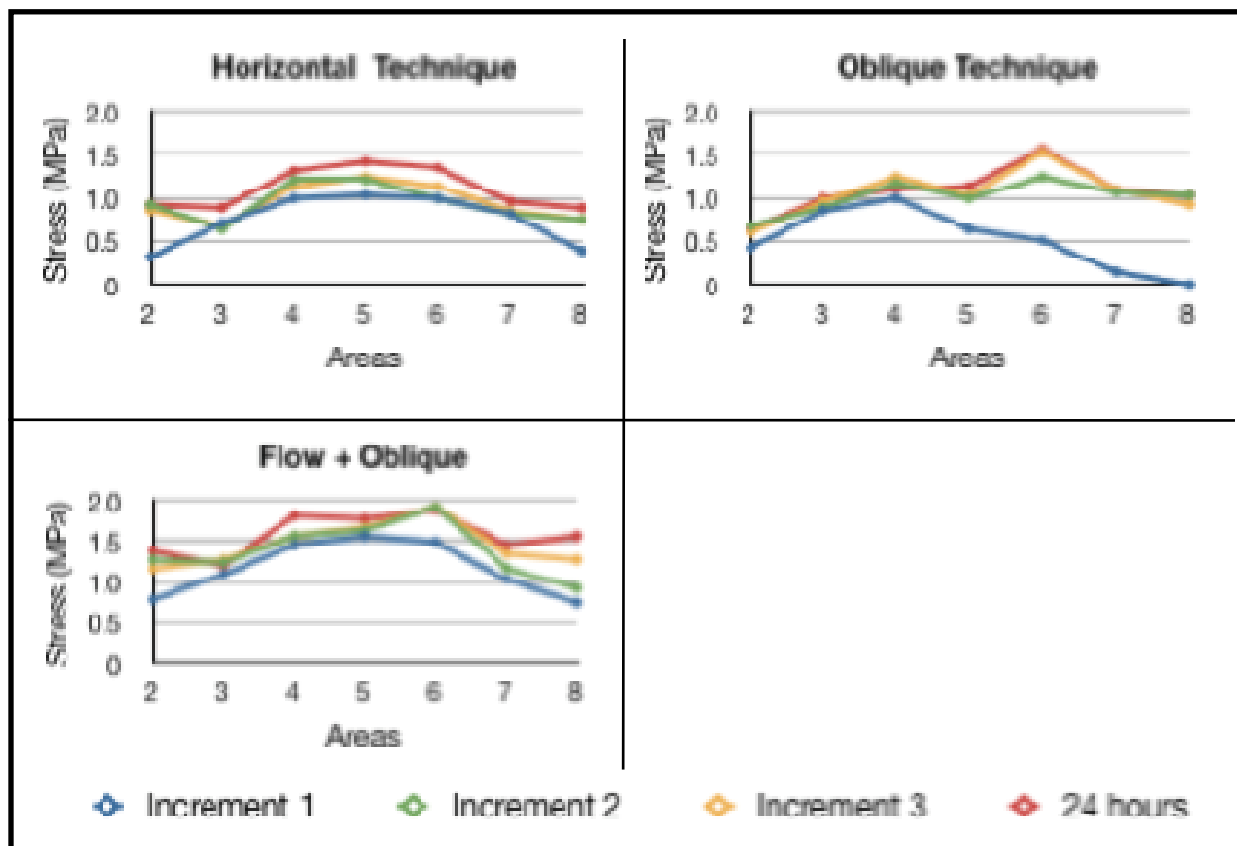


Figure 7 - Charts of stress distribution along restoration interface observed after all increments in all groups tested.

Tables

Table 1 - Composition of composites used in the study.

Composite	Composition	Manufacturer
Filtek Z350	Bis-GMA, Bis-EMA, UDMA with small amounts of TEGDMA. Filler load with 78.5% wt of non-agglomerated/non-aggregated, 20nm nanosilica filler, agglomerated zirconia/silica nanocluster (5-20nm). The cluster particle size range is 0.6 to 1.4 microns.	3M ESPE
Filtek Z350 Flow	Bis-GMA, UDMA, TEGDMA, and Bis-EMA resins. Filler load with 72.5% wt of a combination of non-agglomerated/non-aggregated 20nm silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles).	

Table 2 - Average of stress values (MPa) and SD in all points evaluated and total stress in the photoelastic model for all insertion techniques after insertion of the third increment and within 24 hours.

Areas	Horizontal T.		Oblique T.		Flow+Horizontal T.		Flow+Oblique T.	
	Inc. 3	24 h	Inc. 3	24 h	Inc. 3	24 h	Inc. 3	24 h
2	0.85 (0.30)	0.92 (0.14)	0.60 (0.19)	0.63 (0.26)	0.99 (0.15)	0.88 (0.39)	1.17 (0.23)	1.38 (0.51)
3	0.67 (0.18)	0.89 (0.16)	0.96 (0.15)	0.99 (0.15)	1.06 (0.12)	1.20 (0.28)	1.27 (0.18)	1.20 (0.30)
4	1.13 (0.28)	1.31 (0.30)	1.24 (0.27)	1.10 (0.07)	1.52 (0.15)	1.81 (0.40)	1.56 (0.27)	1.80 (0.32)
5	1.24 (0.17)	1.42 (0.31)	1.03 (0.22)	1.13 (0.25)	1.63 (0.22)	1.84 (0.25)	1.67 (0.19)	1.77 (0.20)
6	1.13 (0.44)	1.35 (0.33)	1.52 (0.30)	1.56 (0.22)	1.84 (0.40)	1.74 (0.54)	1.91 (0.34)	1.88 (0.25)
7	0.85 (0.14)	0.96 (0.09)	1.10 (0.08)	1.10 (0.08)	1.38 (0.18)	1.42 (0.31)	1.35 (0.30)	1.45 (0.34)
8	0.74 (0.27)	0.88 (0.20)	0.92 (0.07)	1.03 (0.07)	1.10 (0.14)	1.24 (0.33)	1.27 (0.28)	1.58 (0.43)
Total	6.62 (1.17) A	7.71 (1.12) A	7.36 (0.19) A	7.53 (0.65) A	9.53 (1.06) B	10.12 (2.15) AB	10.20 (1.13) B	11.04 (1.63) B

\* Different letters mean statistical differences among filling techniques in the same time (p<0.05).



## CONSIDERAÇÕES GERAIS

O fenômeno de desenvolvimento de tensão durante a reação de polimerização do compósito é complexo e segue sendo uma das preocupações clínicas (Ferracane, 2005; Petrovic et al., 2010; Tantbirojn et al., 2011). O perfil da tensão obtido a partir da análise fotoelástica nos capítulos 1 e 2 permitiu a análise quantitativa e qualitativa da tensão de contração de polimerização e contribuiu para melhor entendimento de alguns fatores relacionados com esse problema. Porém, as diferenças entre as propriedades da resina fotoelástica e a estrutura dentária devem ser consideradas durante a interpretação desses resultados.

Como consequência da polimerização dos monômeros, o material resinoso passa por rápido aumento de temperatura e contração (de Santis et al., 2010). A contração de polimerização é um processo complexo que envolve vários fatores interdependentes como o número de ligações covalentes formado (Braga & Ferracane, 2002), a quantidade, forma e tamanho das partículas de carga, a concentração de fotoiniciadores (Pfeifer et al., 2009) e a energia fornecida para a fotoativação (Labella, 1999; Silva et al., 2008; Mortier et al., 2009). A contração de polimerização está diretamente relacionada com as tensões criadas em restaurações de compósitos resinosos (Gonçalves et al., 2010a), ela pode originar a tensões destrutivas que ocorrem quando o material está aderido às paredes da cavidade e se encontra rígido o suficiente para impedir o escoamento e compensar a contração (Braga & Ferracane, 2004; Braga et al., 2005).

As propriedades dos compósitos fotoativados são dependentes tanto da dose de energia quanto do processo de reação após ativação (de Santis, 2009). A tensão de contração de polimerização é influenciada pela quantidade de energia fornecida ao material durante a fotoativação (Takamizawa et al., 2008) estando diretamente relacionado com o grau de conversão e a velocidade de polimerização (Calheiros et al., 2004). A reação mais lenta causa aumento da capacidade de escoamento das moléculas no material, diminuindo a tensão dentro da restauração (Lim et al., 2002). A diminuição da velocidade da reação pode ser

conseguida ativando o compósito usando inicialmente luz em baixa potência seguida pela polimerização final com luz em alta potência (Braga et al., 2005; de Santis, 2009). Essa modulação da luz parece ser uma técnica relevante para alcançar menores valores de tensão (Asmussen & Peutzfeld, 2003). Isso pode ser observado no capítulo 1, quando as irradiâncias de 24 e 36 J/cm<sup>2</sup> foram testadas. Porém, a redução do grau de conversão e da densidade de ligações cruzadas do material também pode ser observada com o uso da técnica soft-start para a ativação (Asmussen & Peutzfeld, 2003; Brandt et al., 2008).

Durante o processo de polimerização, alterações de temperatura também são observadas no compósito, e seu aumento pode elevar as tensões se não houver tempo suficiente para o material dissipar a energia térmica (Tarle et al., 2006). Quando técnicas que modulam a luz são usadas, a baixa energia da ativação inicial tem efeitos no grau de conversão e na densidade de ligações cruzadas (Dewaele et al., 2009; Brandt et al., 2008).

Como observado nos resultados do capítulo 1, o uso de altas doses de energia (36 J/cm<sup>2</sup>) tendem a concentrar tensão nas paredes em que o compósito está aderido. O uso de elevada densidade de energia resulta em desfavorável relação entre o grau de conversão e a tensão de contração do compósito (Calheiros et al., 2004). Apesar disso, o grau de conversão do compósito não sofreu influência da dose de energia aplicada para a fotoativação. Além disso, quando determinada quantidade e espessura de resina é polimerizada, a temperatura e a taxa de contração durante a polimerização pode aumentar com o aumento da potência da luz dependendo do espectro da luz emitida (Tarle et al., 2006). Propriedades visco-elásticas tais como contração de polimerização, velocidade da reação de polimerização e módulo de elasticidade são importantes para determinação da tensão de contração (Sakaguchi et al., 2004).

O compósito pode ser polimerizado com baixa intensidade de energia e o valor de conversão final permanecer alto (Tarle et al., 2006). No capítulo 1, doses de energia de 12 e 24 J/cm<sup>2</sup> parecem ter sido suficientes para polimerizar o compósito adequadamente e promoverem níveis de tensão equivalentes. Além

disso, a medida que a reação de polimerização ocorre, a velocidade da reação diminui e a rigidez do material aumenta, limitando a mobilidade dos monômeros e/ou cadeias em crescimento, e dificultando o aumento da conversão, mesmo quando alta densidade de energia é usada (Halvorson et al., 2002).

A contração térmica é evidente quando alta densidade de energia é usada e apresenta elevada tensão de contração (Atai & Motevasselian, 2009). Além disso, a contração de polimerização só é totalmente finalizada 24 horas após a ativação (Leprince et al., 2010), porém nenhuma diferença no grau de conversão pode ser observada entre os tempos avaliados no capítulo 1, devido à natureza auto-limitante da reação de polimerização (Calheiros et al., 2004). Também no capítulo 1, em 24 horas, diferenças nos níveis de tensão puderam ser observadas entre os modos de ativação quando  $36 \text{ J/cm}^2$  de energia foi aplicado.

Além da energia fornecida para a fotoativação, avaliada no capítulo 1, a influência de técnicas de inserção do compósito sobre a tensão também foram estudadas no capítulo 2. O uso de técnicas incrementais pode minimizar os efeitos da contração, diminuir a tensão na interface da restauração (Kuijs et al., 2003; Petrovic et al., 2010) e reduzir a deformação das cúspides (Lee et al., 2007; Park et al., 2008). Porém, não existe diferença significativa na tensão gerada pelas diferentes formas de inserção do compósito na cavidade (Kuijs et al., 2003). Este resultado também foi encontrado no capítulo 2. Sabe-se que para o mesmo material, a contração de polimerização depende do volume de resina polimerizada (Versluis et al., 1996) e a forma da restauração tem relativamente pouca influência sobre o desenvolvimento de tensão residual (Li et al., 2008).

O uso de compósitos fluidos tem sido indicados como base de restaurações devido a capacidade de escoamento na parede da cavidade e melhorar a adaptação da restauração (Kwon et al., 2010). Porém, existe forte correlação entre a viscosidade do compósito e a tensão gerada pela polimerização (Gonçalves et al., 2010a). Os maiores índices de tensão foram observados quando compósitos de baixa viscosidade foram usados como base nas restaurações (Oliveira et al., 2010), esse fenômeno pode ser confirmado no capítulo 2. A contração volumétrica

e o módulo de elasticidade são dependentes da concentração de partículas inorgânicas na composição do material (Gonçalves et al., 2011). Compósitos com alto conteúdo inorgânico possuem menos componentes orgânicos, que determinam a redução volumétrica durante a reação de polimerização (Takamizawa et al., 2008). A diminuição da quantidade de carga no compósito leva ao aumento da tensão resultante da contração de polimerização (Gonçalves et al., 2010b).

A quantidade e tipo de monômeros presentes na composição da resina também podem influenciar a tensão de contração de polimerização (Gonçalves et al., 2010a; Gonçalves et al. 2011). A resina flow usada no capítulo 2 possui maior quantidade de monômero TEGDMA quando comparado ao compósito de viscosidade convencional. O aumento sistemático desse monômero proporciona aumento do grau de conversão, contração volumétrica, módulo de elasticidade e da tensão de contração de polimerização (Gonçalves et al., 2010a).

As tensões geradas no sistema dente/restauração é ainda é um fator inevitável nas restaurações adesivas que pode comprometer o sucesso da restauração. Clínicos devem entender o conceito de tensão de contração de polimerização e perceber que a qualidade de restaurações de resina composta depende da correta manipulação dessas tensões (Kinomoto et al., 2000). Como verificado nos capítulos 1 e 2, existem muitos fatores envolvidos no processo de polimerização que podem afetar a tensão resultante da contração. Baixos valores de contração podem não ser suficientes para garantir baixos níveis de tensão (Tantbirojn et al., 2011).

## CONCLUSÕES GERAIS

1. Maiores níveis de tensão foram mostrados com  $36 \text{ J/cm}^2$  de energia.
2. O modo de ativação soft-start foi eficiente para reduzir a tensão de contração da polimerização com 24 e  $36 \text{ J/cm}^2$  de energia.
3. O grau de conversão foi semelhante para os modos de ativação testados, exceto para o modo soft-start que apresentou menores níveis de conversão.
4. O grau de conversão não foi influenciado pelo aumento da dose de energia.
5. Não se observou diferenças nas tensões e no grau de conversão entre os tempos imediato e 24 horas quando maiores irradiância e dose de energia foram usadas.
6. Não houve diferença na tensão de contração da polimerização entre as técnicas de inserção horizontal e oblíqua.
7. O uso de material resinoso fluido como forrador e/ou base proporcionou maiores níveis de tensão que o compósito de viscosidade regular.

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<sup>3</sup> De acordo com a norma da UNICAMP/FOP, baseadas na norma do International Committee of Medical Journal Editors - Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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ANEXO 1

Carta de submissão à revista Journal of Dentistry.

20-May-2011

Dear Mrs. Oliveira,

Your submission entitled "Influence of filling techniques on shrinkage stress in class II dental composite restorations" has been assigned the following manuscript number: JJOD-D-11-00332.

You will be able to check on the progress of your paper by logging on to the Elsevier Editorial System as an author.

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Thank you for submitting your work to this journal.

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