LEONARDO GONÇALVES CUNHA

Cirurgião Dentista

INFLUÊNCIA DO MÉTODO DE FOTOATIVAÇÃO SOBRE A RESISTÊNCIA DA UNIÃO E PROPRIEDADES FÍSICAS DE UM COMPÓSITO ODONTOLÓGICO EM DIFERENTES CONFIGURAÇÕES CAVITÁRIAS

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do **Título de Doutor em Materiais Dentários.**

Orientador: Prof. Dr. Mário Alexandre Coelho Sinhoreti

Piracicaba – SP

2007

FICHA CATALOGRÁFICA ELABORADA PELA

BIBLIOTECA DA FACULDADE DE ODONTOLOGIA DE PIRACICABA

Bibliotecário: Marilene Girello – CRB-8ª. / 6159

C914i

Cunha, Leonardo Gonçalves.

Influência do método de fotoativação sobre a resistência da união e propriedades físicas de um compósito odontológico em diferentes configurações cavitárias. / Leonardo Gonçalves Cunha. -- Piracicaba, SP : [s.n.], 2007.

Orientador: Mário Alexandre Coelho Sinhoreti.

Tese (Doutorado) – Universidade Estadual de Campinas, Faculdade de Odontologia de Piracicaba.

1. Resinas compostas. 2. Tensão. 3. Fotopolimerização. 4. Conversão. I. Sinhoreti, Mário Alexandre Coelho. II. Universidade Estadual de Campinas. Faculdade de Odontologia de Piracicaba. III. Título.

(mg/fop)

Título em Inglês: Influence of the photoactivation method on the bond strength and physical properties of a resin composite in different C-factor levels
Palavras-chave em Inglês (Keywords): 1. Composite resins. 2. Stress. 3. Photopolymerization. 4. Conversion

Área de Concentração: Materiais Dentários

Titulação: Doutor em Materiais Dentários
Banca Examinadora: Mário Alexandre Coelho Sinhoreti, Alysson Noriyuki Kajishima Konno, Regina Maria Puppin Rontani, Mario Fernando de Góes, Roberto Ruggiero Braga
Data da Defesa: 23-02-2007
Programa de Pós-Graduação: Materiais Dentários

UNICAMP

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FACULDADE DE ODONTOLOGIA DE PIRACICABA

A Comissão Julgadora dos trabalhos de Defesa de Tese de DOUTORADO, em sessão pública realizada em 23 de Fevereiro de 2007, considerou o candidato LEONARDO GONÇALVES CUNHA aprovado.

PROF. DR. MARIO ALEXANDRE COELHO SINHORETI

PROF. DR. ROBERTO RUGGIERO BRAGA

PROFa. DRa. REGINA MARIA PUPPIN RONTANI

PROF. DR. MARIO FERNANDO DE GOES

PROF. DR. ALYSSON NORIYUKI KAJISHIMA KONNO

DEDICO ESTE TRABALHO

Aos meus pais**, MÁRIO E DULCE,** constantes mestres no aprendizado, exemplos de integridade, honestidade e dedicação, cujo amor e incentivo foram e são indispensáveis nas minhas concretizações e realizações.

Aos meus irmãos, **FABIANO E DANIEL,** pela amizade e companheirismo nas horas difíceis e nas horas de descontração e alegria.

A minha namorada, **CAROL,** pela amizade e o grande amor que sentimos um pelo outro, e pela compreensão nos momentos da minha ausência.

AGRADECIMENTO ESPECIAL

Ao **PROF. DR. MÁRIO ALEXANDRE COELHO SINHORETI**, pela amizade demonstrada em todos os momentos. Agradeço pela solicitude ao orientar, paciência ao ensinar e a dedicação e competência para a realização deste trabalho, sem as quais não seria possível a concretização deste projeto. Esperando ter correspondido, a minha admiração e meus agradecimentos.

Ao **PROF. DR. JACK LIBÓRIO FERRACANE**, pela pronta aceitação de me orientar no meu estágio de doutoramento no exterior e pela preocupação e atenção a mim dispensada desde a minha chegada aos Estados Unidos. Sua competência e genialidade foram fundamentais para a concretização deste projeto. Esperando mais uma vez ter correspondido, a minha admiração e meus agradecimentos.

AGRADECIMENTOS

À Direção da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, na pessoa do seu diretor **PROF. DR. FRANCISCO HAITER NETO** e do Diretor Associado **PROF. DR. MARCELO DE CASTRO MENEGHIM.**

Ao **Prof. Dr. Lourenço Correr Sobrinho**, Titular da Área Materiais Dentários, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, pelo constante estímulo e incentivo ao aprendizado.

Ao **Prof. Dr. Simonides Consani**, Titular da Área Materiais Dentários, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, pelo exemplo de excelente educador, cujo incentivo e conhecimento científico contribuíram para a minha formação.

Ao **Prof. Dr. Mario Fernando de Góes**, Titular da Área Materiais Dentários, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, pela contribuição em minha formação científica, pessoal e pelo estímulo para a busca de novos conhecimentos.

A **Profa. Dra. Regina Maria Puppin-Rontani**, Titular da Área Odontopediatria, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, pela amizade demonstrada desde o primeiro momento e contribuição em minha formação pessoal e profissional.

Ao técnico especializado do laboratório da Área Materiais Dentários, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, engenheiro **Marcos Blanco Cangiani**, pelo auxílio em diversos momentos da realização deste projeto.

À **Selma Aparecida Barbosa de Souza Segalla**, secretária da Área Materiais Dentários, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas, pela amizade e atenção prestada em vários momentos.

Aos colegas de Pós-Graduação **Alysson, Américo, Ana Flávia, Cíntia, Daniela, Eduardo, Eliane, Giovane, Jacy, Juliana, Luciana, Marcelo, Mônica, Murilo, Osvaldo, Piva, Ricardo (artista), Ricardo ("carinha"), Rogerinho, Rubã, Tango e Vinicius,** pela amizade e companheirismo dentro e principalmente fora do curso, pelos momentos de descontração vivenciados nesses anos, tornando inesquecíveis os diversos eventos realizados.

A grande amiga **Mirela**, pelo apoio e ajuda em diversos momentos do nosso doutorado. Apoio, afeto e sinceridade sempre foram suas qualidades.

À grande amiga **Gisele**, pela amizade e companheirismo constante durante o curso, sendo uma das pessoas com importância significante no meu estagio de doutoramento, e pela maravilhosa convivência.

À enorme amiga **Roberta,** presente na maioria dos momentos da minha formação, sempre oferecendo auxílio nos momentos difíceis, bom humor nos momentos de alegria e uma amizade verdadeira e constante. Mais do que amiga, uma irmã.

Aos grandes amigos **Carlos, Álvaro, Fabinho e Leo Bixo**, com os quais tive o prazer de morar durante o Doutorado, pela especial amizade em todos os momentos. Por me proporcionarem, através do verdadeiro companheirismo, excelentes momentos de convivência, que serão inesquecíveis e dos quais sentirei muita falta.

Aos amigos (as) das outras Áreas, hoje uma parte já de respeitados docentes: **Alessandra, Alex, Andréa, Carol Calil (aliás, minha cunhada), Ciça, Daniel, Denise, Eduardo, Larissa, Laura, Lu Machion, Magge, Marcelo Goiano, Marlise, Paty, Paula**, **Rogério e Santista,** pela amizade e companheirismo constantes, pela ajuda nos momentos difíceis e pela maravilhosa convivência.

A todos que indiretamente contribuíram para a realização deste trabalho.

Meus sinceros agradecimentos

EPÍGRAFE

“O que quer que você possa fazer,
ou sonhe que o possa,
faça-o.
Coragem contém genialidade,
poder e magia.”

Goethe

RESUMO

O objetivo deste estudo foi avaliar o efeito de diferentes métodos de fotoativação e níveis de Fator-C sobre a tensão da contração de polimerização, taxa de geração de tensões e grau de conversão de um compósito odontológico, além de avaliar o efeito do método de fotoativação sobre a resistência da união de restaurações em compósito. No Capitulo 1 foi verificado o efeito de métodos de fotoativação modulados sobre a resistência da união de restaurações em compósito. Foi possível concluir que a modulação da intensidade luminosa através dos métodos de fotoativação *Pulse Delay* e *Soft-Start* aumentou significativamente a resistência da união da interface adesiva. Nos Capítulos 2 e 5, a influência de quatro métodos de fotoativação utilizando luz de lâmpada halógena foi avaliada sobre a tensão da contração de polimerização, taxa de geração de tensões e grau de conversão de um compósito odontológico em níveis de Fator-C 3,0 (Capítulo 2) e 1,5 e 3,0 (Capitulo 5), além de avaliar o efeito dos mesmos métodos de fotoativação sobre a resistência da união de restaurações em compósito em Fator-C 3,0 (Capítulo 2). Embora os métodos de fotoativação modulados, de maneira geral, não tenham apresentado diferenças significativas em relação ao método convencional por luz contínua na tensão máxima gerada, estes se mostraram efetivos em reduzir a taxa de geração de tensões, proporcionando aumento significativo da resistência da união de restaurações em compósito, sem redução significativa do grau de conversão. Os Capítulos 3 e 4 avaliaram a influência do tipo da fonte luminosa (Halógena ou LED), da intensidade de luz e da modulação do método de fotoativação sobre a tensão da contração de polimerização, taxa de geração de tensões e grau de conversão de um compósito odontológico em níveis de Fator-C 3,0 (Capítulo 4) e 1,5 e 3,0 (Capitulo 3), além de avaliar o efeito dos mesmos métodos de fotoativação sobre a resistência da união de restaurações em compósito em Fator-C 3,0 (Capítulo 4). A utilização de métodos de fotoativação modulados ou métodos com exposição luminosa em intensidade reduzida mostrou-se efetiva na redução da taxa de geração de tensões, proporcionado aumento significativo da resistência da união de restaurações em compósito, sem redução significativa do grau de conversão. O tipo de

fonte luminosa não apresentou efeito significativo no desenvolvimento das propriedades físicas do compósito quando métodos com a mesma dose energética e mesma intensidade de luz foram comparados. A avaliação da influência do nível de Fator-C nos Capítulos 3 e 5 mostrou, em ambos os estudos, que o nível de Fator-C apresentou relação diretamente proporcional com a taxa de geração de tensões e com a tensão total gerada pela contração de polimerização, ou seja, quanto maior o nível de Fator-C, maior a taxa de geração de tensões e maior a tensão final gerada. O nível de Fator-C, entretanto, não apresentou influência sobre o grau de conversão do compósito.

ABSTRACT

The objective of this study was to verify the influence of curing methods and C-factor levels on contraction stress, stress rate, and degree of conversion of a restorative composite. Besides, the effect of the curing method on the bond strength of composite restoratives was also evaluated. Chapter 1 tested the influence of modulated curing methods on the bond strength of composite restoratives. It was shown that the modulation of the irradiance using Pulse Delay and Soft-Start curing methods was effective to improve the bond strength of the adhesive interface. In Chapters 2 and 5 the influence of four curing methods using halogen light was evaluated on contraction stress, stress rate, and degree of conversion of a restorative composite at C-factor levels 3.0 (Chapter 2) and 1.5 and 3.0 (Chapter 5). Besides, the influence of the same curing methods on the bond strength of composite restoratives at C-factor 3.0 was also evaluated (Chapter 2). In spite of the lack of statistical difference among some of the modulated curing methods tested when compared to the conventional continuous light method as to the maximum stress generation, the reduction in the stress rate observed for the modulated curing methods proved to be effective to improve the strength of the bonded interface, with no adverse effect on the degree of conversion of the restorative composite. Chapter 3 and 4 evaluated the effect of the light source (Halogen or LED), of the irradiance, and of modulation of the curing method on contraction stress, stress rate, and degree of conversion of a restorative composite at C-factor levels 3.0 (Chapter 4) and 1.5 and 3.0 (Chapter 3). Besides, the influence of the same curing methods on the bond strength of composite restoratives at C-factor 3.0 was also evaluated (Chapter 4). Modulated curing methods or curing methods using reduced irradiance was shown to be effective in reducing contraction stress rate and improving the strength of the bonded interface, and without compromising the degree of conversion of the restorative composite. The light source showed no influence on the development of physical properties of the restorative composite when curing methods using the same energy dose and the same irradiance were compared. The evaluation of the C-factor level in Chapters 3 and 5 showed, in both works, a negative influence of the C-factor on the stress rate and on the amount of stress generated. Therefore, higher the C-factor, higher the stress rate and higher the amount of stress generated. However, the C-factor level showed no influence on the degree of conversion of the restorative composite.

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INTRODUÇÃO

Os primeiros compósitos resinosos foram introduzidos no início dos anos 60 para serem utilizados em dentes anteriores, em substituição ao cimento de silicato e à resina acrílica (BOWEN, 1963). Posteriormente, aprimoramentos relacionados à modificação na composição, tipo, formato e quantidade de carga inorgânica promoveram melhor desempenho destes materiais e expandiram suas indicações, sendo possível sua utilização também em região de dentes posteriores.

O método de polimerização dos compósitos resinosos mais empregado é a utilização de fontes de luz visível, dentro da zona azul do espectro luminoso, entre 410 a 550 nm (IRIE *et al.*, 2002). A exposição do compósito à luz azul promove a ativação do fotoiniciador presente na formulação deste material, sendo a canforoquinona um dos fotoiniciadores mais freqüentemente utilizados (ANUSAVICE, 1998).

Entretanto, durante a reação de polimerização do compósito é observado, como conseqüência, significante percentual de contração volumétrica (DAVIDSON & DE GEE, 1984), devido à redução da distância intermolecular dos monômeros durante a formação da cadeia polimérica (BRAGA ET AL., 2005). Estudos *in vitro* concluíram que o percentual de contração dos compósitos restauradores se situa entre 1,9% e 6% (LABELLA ET AL., 1999; KLEVERLAAN & FEILZER, 2005).

Quando a reação de polimerização acontece em confinamento, a união do material restaurador às paredes cavitárias faz com que a inerente contração seja relacionada ao desenvolvimento de tensões (BRAGA ET AL., 2005). O desenvolvimento significativo de tensões pode acarretar no rompimento da interface dente-restauração, caso a tensão supere a resistência da união estrutura dental-compósito restaurador. Com o rompimento da união, verifica-se a formação de fendas, as quais permitem a passagem de fluidos orais e bactérias, podendo levar a ocorrência de sensibilidade, pigmentação da interface e recorrência da lesão cariosa (GORDAN ET AL., 2006).

A intensidade da tensão gerada está relacionada à técnica restauradora empregada, assim como à composição do material restaurador utilizado, e a configuração da cavidade a ser restaurada (Fator-C). Para o cálculo do Fator-C, toma-se como referência a relação entre a área total de paredes unidas pela área total de paredes livres (FEILZER ET AL., 1987). Dessa forma, quanto maior o Fator-C, menor será a área de paredes livres presentes nessa configuração cavitária, reduzindo a possibilidade de escoamento e de acomodamento do compósito nos momentos inicias da reação de polimerização.

Entretanto, devido ao fato de que a configuração final da cavidade dificilmente pode ser modificada, algumas técnicas restauradoras alternativas têm sido sugeridas com o objetivo de diminuir os efeitos deletérios da contração de polimerização. Entre essas técnicas, destacam-se os métodos de fotoativação modulados. O método de fotoativação *Soft-Start* representa um desses métodos, no qual faz-se uso inicialmente de intensidade de luz reduzida (SILIKAS ET AL, 2000), promovendo menor grau de conversão nos estágios iniciais da reação de polimerização. Com o retardamento do aumento do módulo de elasticidade do compósito, ocorre a possibilidade do material escoar e liberar parcialmente a tensão gerada, reduzindo a magnitude final de tensão (FEILZER ET AL., 1990). Por outro lado, resultados menos encorajadores foram apresentados em alguns estudos, nos quais diferenças quanto à adaptação marginal de restaurações em compósito entre os métodos *Soft-Start* e Luz Contínua não foram verificadas, tanto *in vivo* (LINDBERG ET AL., 2005) quanto *in vitro* (ALONSO ET AL., 2004).

O método de fotoativação *Pulse Delay* representa uma variação do método *Soft-Start*, no qual um intervalo de 3 a 5 minutos é realizado entre os dois períodos de exposição à luz, objetivando uma lenta continuidade da reação de polimerização durante o intervalo na ausência da luz (KANCA & SUH, 1999), e posteriormente, ao final do intervalo, promovendo-se uma segunda exposição luminosa em alta intensidade para assegurar satisfatório desenvolvimento das propriedades físico-mecânicas do compósito.

Embora existam evidências de que o método de fotoativação *Pulse Delay* promova melhor integridade marginal (LUO ET AL, 2002), reduzida incidência de fendas cavo-superficiais e fraturas de esmalte (KANCA & SUH, 1999; SAHAFI ET AL, 2001), além de redução nos níveis de tensão residual do compósito (SUH ET AL, 1999), ainda não existe um protocolo definido sobre qual seria a intensidade inicial adequada para se utilizar este método. Diferentes trabalhos apresentam uma variabilidade de intensidades de 60 a 425 mW/cm^2 (YAP ET AL. 2002; HACKMAN ET AL., 2002; LIM ET AL., 2002).

Outro aspecto está relacionado à influência que diferentes fontes de luz podem exercer sobre o desenvolvimento das propriedades físico-mecânicas. Comparado com a fonte luminosa mais tradicionalmente utilizada (quartzo-tungstênio-halógeno - QTH), a fonte de luz emitida por diodo (LED) possui a característica de apresentar estreito espectro luminoso, na faixa de 438 a 501 nm, e pico de intensidade em 465 nm, coincidente com o pico de absorção da canforoquinona, o fotoiniciador mais utilizado em compósitos comerciais (FUJIBAYASHI ET AL, 1998; JANDT ET AL., 2000; NOMURA ET AL., 2002). Devido a esta vantagem, fabricantes têm associado à fonte de luz LED, maior eficiência na polimerização de compósitos resinosos, possibilitando a redução do tempo de exposição à luz, sem que haja comprometimento do grau de conversão do compósito (DENTALMAN INTERN.; DENMED DIRECT SERVICES). Além disso, os aparelhos fotoativadores LED não necessitam de filtros para seleção do comprimento de onda, artefato indispensável nos aparelhos de lâmpada halógena. Entretanto, não existem evidências na literatura de que grau de conversão superior seja alcançado pelo compósito quando fotoativado por uma fonte LED comparativamente a uma fonte de lâmpada halógena, ao se fazer uso da mesma densidade energética (MILLS ET AL., 2002; UHL ET AL., 2005).

Dessa forma, a preservação da interface substrato dental–compósito restaurador é um dos principais fatores envolvidos na longevidade da restauração. Por sua vez, a preservação desta interface está relacionada à concentração total de tensão gerada pela contração dos compósitos, fato associado ao método de fotoativação e material utilizados, configuração final da cavidade a ser restaurada, ou pela junção destes mecanismos.

Proposição

Considerando a inerente ocorrência da contração de polimerização dos compósitos restauradores, com conseqüente geração de tensões, e possível comprometimento da interface dente-restauração, este estudo verificou, em cinco capítulos [1]:

1. O efeito de sete métodos de fotoativação na resistência da união de restaurações em compósito.
2. A influência de diferentes métodos de fotoativação sobre a tensão de contração, taxa de geração de tensões e grau de conversão de um compósito restaurador, e ainda sobre a resistência da união de restaurações em compósito.
3. O efeito de diferentes métodos de fotoativação e fontes de luz sobre a tensão de contração, taxa de geração de tensões e grau de conversão de um compósito restaurador em dois diferentes níveis de Fator-C.
4. O efeito de diferentes métodos de fotoativação e fontes de luz sobre a tensão de contração, taxa de geração de tensões e grau de conversão de um compósito restaurador, e ainda sobre a resistência da união de restaurações em compósito.
5. O efeito de diferentes métodos de fotoativação sobre a tensão de contração, taxa de geração de tensões e grau de conversão de um compósito restaurador em dois diferentes níveis de Fator-C.

[1] Este trabalho foi realizado no formato alternativo, com base na deliberação da Comissão Central de Pós-Gradução (CCPG) da Universidade Estadual de Campinas (UNICAMP), número 001/98.

CAPÍTULO 1

EFFECT OF DIFFERENT PHOTOACTIVATION METHODS ON THE BOND STRENGTH OF COMPOSITE RESTORATIONS BY PUSH-OUT TEST

Manuscrito aceito para publicação no periódico

Quintessence Internacional

Effect of the light-curing method on the bond strength of composite restorations by push-out test

ABSTRACT

Objectives: Modulated curing methods could lead to higher probability of bond preservation. Therefore, the aim of this study was to evaluate the effect of seven curing methods on bond strength of composite restorations. **Method and Materials:** Seventy bovine incisors were selected. A conical cavity was prepared in the buccal surface. Single Bond adhesive system was applied following the manufacturer's instructions and the cavities were filled with a single increment of Esthet X. The specimens were randomly assigned into 7 groups (n=10) according to the photoactivation method: 1) Control Continuous light 700 (700mW/cm^2); 2) Continuous Light 150 (150mW/cm^2); 3) Continuous light 250 (250mW/cm^2); 4) Soft-Start 75 (75mW/cm^2 + 700mW/cm^2); 5) Soft-Start 150 (150mW/cm^2 + 700mW/cm^2); 6) Pulse delay (150 mW/cm^2 + 3min + 700 mW/cm^2); 7) Intermittent light (cycles at 600 mW/cm^2). The energy density for all groups was 14 J/cm^2. The bond strength of the composite restorations was measured performing push-out test in a universal testing machine (Instron). The results were submitted to ANOVA and Tukey's test ($p<0.05$). **Results:** Pulse Delay, Soft-Start 150, and Soft-Start 75 methods showed a significant increase on bond strength when compared with the Control Continuous Light 700 method. Low power density and intermittent light groups showed intermediate results. **Conclusion:** Modulation of the energy density during light curing of resin composites using pulse delay or soft-start methods increased the bond strength of composite restorations.

Key words: push-out test, bond strength, resin composite restorations, photoactivation methods, power density, pulse-delay, soft-start polymerization.

INTRODUCTION

Light cured resin composites are commonly used in daily clinical practice to restore anterior and posterior teeth for their many advantages: esthetic, bonding to tooth structure, and mechanical properties. However, these materials undergo significant volumetric shrinkage when polymerized.[1] *In vitro* measurements of polymerization shrinkage of resin composites range from 1.9% to 6%.[2]

When under confinement, due to bonding to cavity walls, the shrinkage is associated with the development of stresses.[3] Such stress may disrupt the bonding between the composite and the cavity walls or may even cause cohesive failure of the restorative material or the surrounding tooth tissue.[1]

The rate of monomer conversion depends on the power density. The higher the power density, the faster the monomer conversion, and the higher the stress generation.[4] The polymerization using low power density could reduce the stress. However, the light exposure time must be prolonged in order to maintain the energy density similar to that used in the conventional methods.[5]

Studies on alternative curing methods have shown the beneficial effects of a modulated polymerization.[6,7] In Soft-Start method, the curing starts using a reduced power density. This could increase the material flow during the earlier stages of polymerization, leading to better marginal adaptation.[8,9] Previous studies[10,11] have shown similar mechanical properties such as shrinkage, surface hardness, and residual monomer concentration when Soft-Start was compared to conventional curing, as long as the total energy density is the same.

A variation of Soft-start polymerization, known as the Pulse Delay technique, was introduced as an attempt to reduce contraction stress by prolonging the period in which the composite remains in a viscous state, which in turn would allow for plastic deformation before the more rigid state was reached. This is achieved by an initial short pulse of light using low energy, followed by a lag period from 1 to 5 minutes before the final light exposure is performed.[12-13] It has been suggested that this technique allows for an enhanced flow or deformation of the composite, which is reported to reduce the incidence of cavosurface marginal gaps and enamel fractures[13-14], theoretically by reducing residual stress in the composite[12].

Intermittent light curing method was introduced by Obici *et al*[15]. The curing of the composite occurs in cycles of light on and light off. The light-off period could modify the polymerization kinetics of the composite, with reduction or modification in the distribution of the stress generated.[16] It was demonstrated that the Intermittent light could effectively reduce the polymerization shrinkage[15] and enhance marginal adaptation of composite restorations.[16]

Therefore, the purpose of this study was to evaluate the effect of different curing methods on the bond strength of composite restorations using a new methodology, the push-out test in teeth cavities. The tested hypothesis was that the modulated curing methods would significantly increase the bond strength values when compared to conventional continuous light.

METHOD AND MATERIALS

Selection and Teeth Preparation

Seventy bovine incisors free from cracks or any other kind of structural defect were selected under x20 magnification. The teeth were disinfected in 0.5% aqueous solution of Chloramine T at 4^{o}C for no more than one week. The crowns were cut off in the cement-enamel junction (Figure 1A) using a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil).

Conical preparations (top diameter of 5.0 mm, bottom diameter of 4.0 mm, and 2.0 mm in height - Figure 1C) were prepared in the buccal surface of each tooth using a diamond tipped bur (#3131; KG Sorensen, São Paulo, SP, Brazil), mounted in a high-speed hand piece (Kavo, Joinville, SC, Brazil), under constant air-water cooling in a standard cavity preparation appliance (Figure 1B). The diamond burs were replaced after every 5th preparation. The C-factor of the preparation was calculated to be 2.2.

Restorative procedures

Preparations were etched using 35% phosphoric acid (Scotchbond Etchant, 3M-ESPE, St. Paul, MN, USA, batch number 5EN) for 15 seconds on dentin and 30 seconds on enamel. Single bond adhesive system (3M-ESPE, St. Paul, MN, USA, batch number 4KB) was applied according to manufacturer's instructions and cured for 10 seconds, at 700 mW/cm^2 (XL 2500 - 3M-ESPE, St Paul, MN, USA). The composite was placed in bulk (Esthet-X, shade A2, Dentsply/Caulk – Mildford, DE - 19963-0359, batch number 0110161). A mylar strip and a microscope slide were placed over the cavity, and used to force the composite to adapt to the preparation walls and to extrude the excess material. The specimens were randomly assigned into seven groups (n=10), according to the curing method (Table 1). The power density was frequently checked by a radiometer (Demetron Research Corp., Danbury, USA)

After applying the light curing procedures, the samples were stored in distilled water at 37^{o}C for 24 hours.

A diamond tipped bur (#3017HL, Fava Metalúrgica, São Paulo, SP, Brazil) was used to partially grind the lingual face of the crown, with the goal to expose the bottom (axial) surface of the restoration. The mesial and distal crown segments on the lingual surface were preserved to reinforce the specimen (Figure 1D). After that, restorations were polished using Sof-Lex (3M-ESPE, St Paul, MN, USA) on the buccal and lingual surface.

The bond strength test was conducted using a push-out test. The sample was positioned on top of a metallic device that had an aperture that allowed the smaller diameter of the restoration to be in contact with an aspheric device, connected to the load cell of a universal testing machine (Instron, model 4411, Buckinghamshire, England). This aspheric device applied a compressive force on the smaller diameter surface of the restoration, until rupture of the tooth-composite bond was achieved. (Figure 1E). The push-out test was carried out at a cross head speed of 0.5 mm/min. Maximum load was divided by the bonded surface area of each sample.

After the test, the fractured specimens were examined under a stereomicroscope at 40X (Carl Zeiss, Manaus, AM, Brazil) and classified as to the characteristic of failure:

cohesive failure in composite, cohesive failure in dentin, adhesive failure, or mixed (adhesive and cohesive in composite).

Bond strength values were subjected to ANOVA and Tukey's post-hoc test at a pre-determined significance level of 0.05.

RESULTS

Bond strength values and standard deviations for all curing methods are listed in Table 2. Pulse Delay, Soft-Start 75, and Soft-Start 150 curing methods showed the highest mean values. There was no statistical difference among these methods, and they presented statistically higher mean values when compared to Continuous Light 700 curing method ($p<0.05$). Continuous Light 150, Continuous Light 250, and Intermittent Light curing methods presented intermediate results, neither differing statistically among themselves nor to the other evaluated groups ($p>0.05$).

Failure mode classification is showed on Figure 2. Continuous Light 700, Intermittent Light, and Continuous Light 250 groups showed adhesive failure as the most frequent failure mode. Continuous Light 150, Soft-Start 150, and Soft-Start 75 showed equal percentages of adhesive failure and mixed failure. Pulse Delay showed the lower percentage of adhesive failure and was the only group that presented cohesive failure in composite.

DISCUSSION

Different methodologies have been used to measure bond strength, such as shear bond strength, tensile bond strength, and microshear bond strength. One disadvantage of these methodologies is that the test is generally performed in flat surfaces. In such situation, the C-factor is very low and the development of shrinkage stress is not directed to the bonding interface. The present study was developed using a push-out test. Usually, push-out test is used to evaluate bond strength of endodontic cements in the radicular conduct.[17,18] To the present study, the push-out test was adapted to evaluate bond strength of composite restorations in simulated Class I cavities. The advantage of using push-out test was that the bond strength could be evaluated in a high C-factor cavity (2.2), with high stress generation directed to the bonding area.[19] All bonding area was submitted to the compressive force at the same time, allowing shear bond strength to be evaluated in a cavity. In addition, the confidence of this methodology could be confirmed by low variability of the data, once the results showed low standard deviation.

The analysis of the results revealed that there was no significant difference among the continuous light groups (Continuous Light 700, Continuous Light 150, and Continuous Light 250). However, a tendency of higher bond strength was observed when reduced power density was used. This tendency was also confirmed by the analysis of the failure mode of the specimens. Continuous Light 700 curing method predominantly presented adhesive failure (80%). For this method, the reaction might have evolved too fast, virtually eliminating the opportunity for viscous flow, leading to a dramatic increase in stiffness after a relatively low degree of conversion[3,20]. As a result, stress develops almost immediately after polymerization is triggered[21] and nearly all of the conversion occurs after the polymer matrix has reached a significant level of rigidity[3]. The orientation of this stress to the adhesive interface in a fast mode reduces the probability of bond preservation.

However, to the curing method Continuous Light 150, a percentage of 50% of adhesive failure and 50% of mixed failure was observed. The reduction on the frequency of adhesive failure when compared to the Continuous Light 700 curing method could be associated with partial preservation of the adhesive interface. The use of a reduced power density during the curing of the composite could be related to a reduction on polymerization rate, with consequent slower generation of stress. Neves *et al* [22] stated that lower power densities were able to reduce the maximum polymerization rate and delay the formation of a rigid network. However, the reduction on the stress rate reached by the curing methods Continuous Light 150 and 250 were not enough to significantly increase the bond strength.

To the Intermittent light curing method, the halogen light is exposed in cycles, inserting intervals of light on and light off.[6,15,16,23] Theoretically, the light-off periods would increase the viscous-elastic stage of the composite, promoting partial stress release.

Some studies [6,15,16,23] have pointed out the benefits of intermittent light on the curing of composite. However, in these studies, the energy dose of the intermittent light method was lower than that of the other curing methods. The reduction on the energy dose leads to reduction on the shrinkage level, and consequently, reduction on the shrinkage stress. In this study, the energy dose was standardized around 14 J/cm^2 for all curing methods, in order to ensure similar degree of conversion and similar final volumetric shrinkage of the composite. The Intermittent light showed results of bond strength statistically similar to those obtained for Continuous light 700 curing method and the adhesive failure was the most frequent failure mode observed, likewise in Continuous light 700 method. However, Intermittent light method showed an intermediate mean value of bond strength. There was no significant difference of this group from the other modulated curing methods. Therefore, it can be hypothesized that the light off period was not long enough to cause a significant stress release and better results. Other types of cycles must be evaluated in order to find better results of composite restoration cure using this method.

To the Soft-start methods, two different values of initial power density were tested (150 and 75 mW/cm^2). The mean values of bond strength for these groups were statistically higher than the ones observed for Continuous Light 700 method. The use of initial reduced power density could be related to a slower development of stress, due to the lower polymerization rate in the initial period of curing compared to continuous curing in high power density. [4,8,24] In a previous study [25], the marginal and internal gap of composite restoration photoactivated using Soft-start technique were evaluated. The use of Soft-start polymerization showed a significant reduction of internal gap formation. This could confirm that the higher bond strength of these groups is related to the preservation of the bonding interface. Other studies have also revealed bond preservation and reduction of marginal gap formation using Soft-start technique. [9, 24]

The Pulse Delay photoactivation method is a variation of the Soft-start technique. The initial cycle of light exposure in reduced power density and the 3-minute light-off interval limited the polymerization rate, while maximized the degree of conversion that occurred as a result of the low irradiance first step. [26] At this point, the conversion, and consequent shrinkage, of the composite occurred while the material maintained a low elastic modulus, before the composite became predominantly rigid, elastic solid. Shrinkage prior to the acquisition of substantial modulus can be compensated by molecular rearrangement of the polymeric chains. The introduction of a light-off period between the two cycles of light exposure may prolong the low modulus phase, allowing the stress development to be partial relieved by polymer flow and deformation.[27] This situation was confirmed in this work. The Pulse Delay curing method was responsible for a significant increase in the bond strength compared to Continuous Light 700 method. The mean value of bond strength of the Pulse Delay curing method was 7.27 MPa, statistically higher than the of Continuous light 700 method. Besides, the predominant failure mode of this group was mixed, and this method was the only one in which cohesive failure in composite was observed. Uno *et al* [28] demonstrated reduction of gap formation using Pulse delay technique.

Based on the results of this study, the tested hypothesis was partially validated. The modulation of the power density in the curing process using Soft-start and Pulse Delay techniques was associated with higher bond strength values when compared to the conventional curing by Continuous light in high power density. Therefore, the clinical use of modulated photoactivation methods may be beneficial, since they could be associated with better preservation of the interface tooth-restoration, a factor highly significant for the clinical longevity of composite restorations.

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Table 1 – Description of the light curing methods.

Curing Method *	Power density and Time exposure	Light Curing Unit
Continuous Light 700	700mW/cm^2 during 20s	XL 2500 (3M/ESPE)
Continuous Light 150	150mW/cm^2 during 94 s	XL 2500 (3M/ESPE) †
Continuous Light 250	250mW/cm^2 during 56 s	XL 2500 (3M/ESPE) †
Soft-Start 75	10s at 75mW/cm^2 +19s at 700mW/cm^2	XL 2500 (3M/ESPE) †
Soft-Start 150	10s at 150mW/cm^2 + 18s at 700mW/cm^2	XL 2500 (3M/ESPE) †
Pulse Delay	5s at 150mW/cm^2 + 3min + 19s at 700mW/cm^2	XL 2500 (3M/ESPE) †
Intermittent Light	56s at 600mW/cm^2 (2s light on + 2s light off)	Optilux 150 Demetron adapted ‡

* The energy dose applied for all groups was 14 J/cm^2.

† The reduction of the power density in these groups was obtained using a standard separator.

‡ The curing unit used in this curing method was an experimental curing unit developed in Dental Materials Department, Piracicaba Dental School, UNICAMP. The experimental curing unit was assembled from a commercial curing unit (Optilux 150 – Demetron) that uses halogen light. This unit was adapted to an electric circuit that allows a cyclic irradiation (2 s light on and 2 s light off).

Table 2. Mean values of bond strength (MPa) for all curing methods.

Curing method	Bond strength (MPa)
Pulse Delay	7.27 a (1.26)
Soft-Start 75	6.45 a (1.10)
Soft-Start 150	6.30 a (1.06)
Continuous Light 150	6.23 ab (0.99)
Continuous Light 250	5.91 ab (0.73)
Intermittent Light	5.66 ab (1.55)
Continuous Light 700	4.64 b (1.50)

Mean values followed by different letters differ statistically among themselves for the Tukey test at the level of 5%.

( ) – Standard Deviation

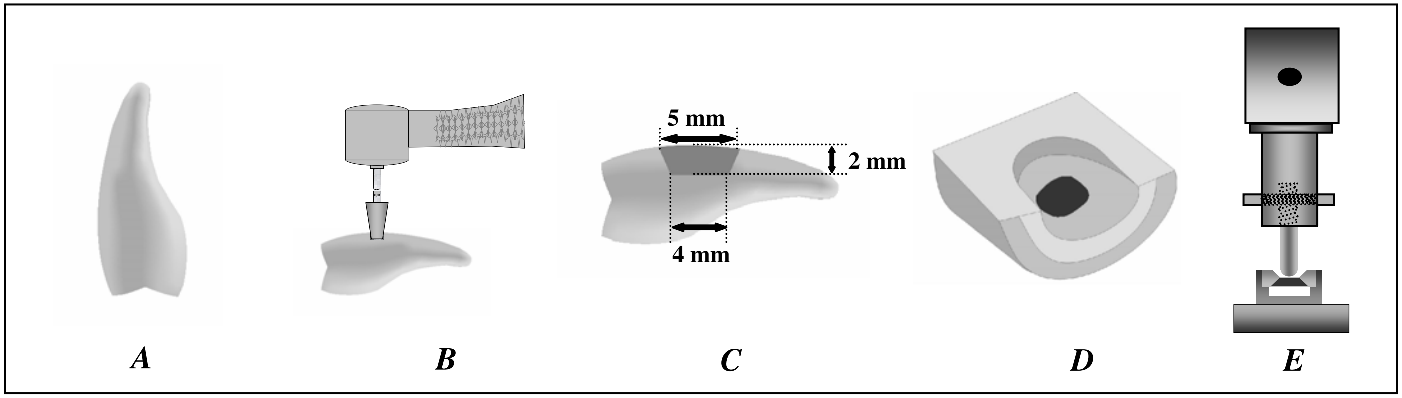
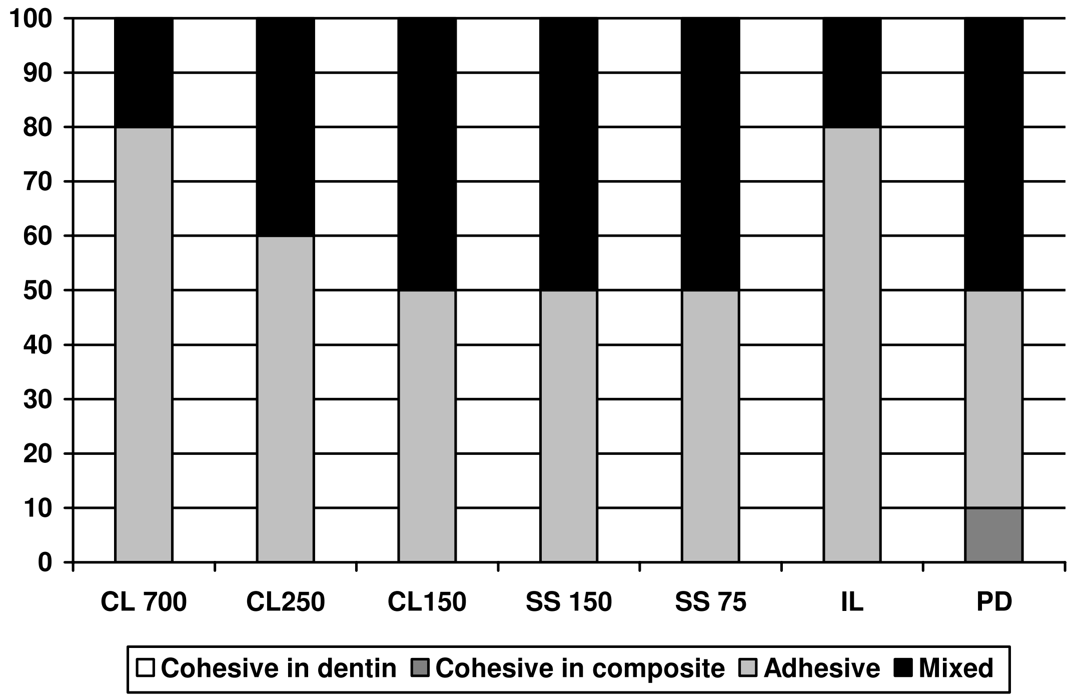


Figure 1 – Schematic representation of the push-out test: **A** - Dental fragment; **B** - Cavity preparation in the standard cavity preparation appliance; **C** - Lateral view of the restored sample (2.0 mm in height, top diameter of 5.0 mm, and bottom diameter 4.0 mm); **D -** Selective wear of the lingual surface and exposure of the bottom region of the restoration; **E** - Lateral view of the complete test system.

FIGURE 2 - PERCENTAGE OF FAILURE MODE FOR ALL CURING METHODS.



CAPÍTULO 2

MODULATED PHOTOACTIVATION METHODS: INFLUENCE ON CONTRACTION STRESS, DEGREE OF CONVERSION AND PUSH-OUT BOND STRENGTH OF COMPOSITE RESTORATIVES

Manuscrito aceito para publicação no periódico

Journal of Dentistry

Modulated photoactivation methods: influence on contraction stress, degree of conversion and push-out bond strength of composite restoratives

Summary

Objectives: Verify the influence of curing methods on contraction stress, stress rate, and degree of conversion (DC) of a restorative composite and on bond strength of composite restoratives.

Methods: For the stress test, composite (0.84 mm thick) was applied between two 5-mm diameter glass rods, mounted in a servohydraulic machine. Stress rate was taken by the value of stress/time at each second. DC was measured by micro-FTIR. Bond strength testing was performed using a push-out test. The C-factor in all tests was 3.0. Four curing methods were tested: Continuous Light (CL), Soft-Start (SS), and two Pulse Delay methods using different initial irradiances - 150 (PD150) and 80 mW/cm^2 (PD80). Results were analyzed by ANOVA and Tukey's test ($\alpha = 0.05$).

Results: Stress values ranged from 7.9 MPa (PD80) to 10.3 MPa (CL). No statistical difference was verified among CL, SS, and PD150. PD80 presented statistically lower stress values compared to CL and SS. CL presented the highest maximum stress rate, followed by SS, PD150 and PD80. Mean DC values ranged from 54.2% (PD150) to 55.9% (PD80), with no difference observed among the methods. For the bond strength test, values ranged from 26.4 MPa (CL) to 35.5 MPa (PD150). PD150 and PD80 were both statistically superior to SS and CL. SS presented statistically higher bond strength compared to CL.

Conclusions: Modulated curing methods were shown to be effective in reducing contraction stress rate and improving the strength of the bonded interface, and without compromising the DC of the restorative composite.

Keywords: curing methods; restorative composite; FTIR; contraction stress; bond strength; composite restoratives.

Introduction

Contraction stress in composite restorations is the result of polymerization contraction taking place under the confinement, produced by bonding to cavity walls.[1] Resin polymerization contraction,[2] specimen geometry,[3] and light-curing method [4,5] have all been shown to play significant roles in the development of contraction stresses during the cure of composites. The clinician has some control of each of these factors.

Traditionally, quartz-tungsten-halogen (QTH) lights have been used in a continuous output mode with a fairly high irradiance to polymerize dental composites.[6-7] However, radiation from this type source can be applied in different manners. The "soft-start" method employs an initial light activation at low irradiance followed by a second at a higher irradiance that is typically equivalent to that used in the continuous method.[7] The "soft start" method has been associated with better marginal integrity of composite restorations *in vitro*.[8-9] However, less encouraging results have been presented by others, showing no difference between Soft-start and Continuous light methods in the *in vitro* marginal adaptation [10] and *in vivo* microleakage [11], and in the contraction strain.[12]

A variation of soft-start polymerization, known as the Pulse Delay technique, was introduced as an attempt to reduce contraction stress in dental composites.[13] This is achieved by an initial short pulse of light, followed by a waiting time from 3 to 5 minutes before the final light exposure is performed.[13-14] It has been suggested that this technique allows for an enhanced flow or deformation of the composite, which is reported to reduce the incidence of cavosurface marginal gaps and enamel fractures,[13,15-16] thereby improving marginal integrity,[17] theoretically by reducing residual stress or strain in the composite.[16] However, studies on the pulse delay method do not present a standard protocol for its use. Authors suggest irradiance levels from 60 to 425 mW/cm^2 for the first pulse,[15, 17-21] without consensus.

The aim of the present study was to evaluate the effect of four light-curing methods on the degree of conversion, contraction stress, and contraction stress rate developed by a resin-based composite, and on the bond strength of the composite to dentin. The tested hypothesis was that modulated curing methods provide a significant reduction in contraction stress, leading to improved bond strength, with no reduction in the degree of conversion of the restorative composite.

Materials and Methods

The VIP light-curing unit (Variable Intensity Polymerizer, Bisco, Schaumburg, IL, USA), which has the capability to provide different levels of irradiance, was used in all experiments. Filtek Z250 restorative composite (shade A2, 3M-ESPE, St. Paul, MN, USA, batch number 5JH) was used for all experiments. The curing methods evaluated in the following three tests are described in Table 1.

Polymerization Contraction Stress and Stress Rate

Polymerization Contraction Stress Test was performed with a closed loop servohydraulic testing instrument (MTS 858, MTS Systems, Eden Prairie, MN, USA). Two borosilicate glass rods 5 mm in diameter were sandblasted (250 µm Al_2O_3) and treated with a silane (Silane ceramic primer, 3M-ESPE, St. Paul, MN, USA, batch number 5WJ) and light-cured adhesive resin (Scotchbond MP, 3M-ESPE, St. Paul, MN, USA, batch number 0MA). One of the glass rods (12 mm in height) was attached to a metallic fixture connected to the actuator, which had a slot through which the light curing guide was kept in contact with the opposing side of the rod. The other rod was 10 mm in height and was bonded to a fixture attached to the load cell. Contraction stress was measured by placing a 0.84 mm layer of composite between the two rods. The ratio of bonded-to-unbonded surface area was 3.0 in this configuration. A near zero compliance system was set up by using an eddy current feedback system (Kaman Instruments, Colorado Springs, CO, USA) that kept the distance between the rods constant. A light-curing guide was directed down through the upper rod and the contraction forces recorded during 10 min from the initiation of light activation. Maximum force was divided by cross-sectional area to calculate average axial stress. Five samples of each curing method were tested. Stress rates were calculated as the change in stress vs. time at each second during the measurement period.

Prior to testing, the light intensity at the top of the specimen was measured using the power meter (Power Maximum 5200, Molectron, Portland, OR, USA). A turbo light guide and neutral density filters were used to control the irradiance reaching the specimen in this experiment. When the irradiance at the end of the Turbo light guide was 770, 220, and 120 mW/cm^2, the irradiance at the surface of the composite was 550, 150, and 80 mW/cm^2, respectively, considering a reduction of 30% in the light intensity when the light passed through the glass rod.

Degree of Conversion

The degree of conversion of the resin composite in the four curing methods tested was determined using a micro-Fourier Transform Infrared Spectroscopy (FTIR) analyser (DS20/XAD, Analect Instruments, Irvine, CA, USA). Glass rings of 4 mm-diameter and 2mm-height and glass slides were sandblasted and silanated (Silane ceramic primer, batch number 5WJ). The rings were bonded to the slide using a thin coat of adhesive (Scotchbond MP, batch number 2MT), resulting in a glass cavity with a bonded-to-unbonded area ratio of 3.0. The glass cavity was bulk filled with the restorative composite. A mylar strip and glass slide were pressed over the glass cavity to force the composite to adapt to the cavity walls and to extrude the excess material. The composite was then light-activated through the glass slide at the bottom of the cavity. A turbo light guide and neutral density filters were used to control the curing parameters. When the irradiance at the end of the Turbo light guide was 610, 170, and 90 mW/cm^2, the irradiance at the bottom surface of the composite was 550, 150, and 80 mW/cm^2, respectively, considering a reduction of 10% in the light intensity when the light passed through the glass slide. Three samples for each experimental condition were prepared and stored dry for 24h at room temperature. Chips of composite approximately 50x100 µm in size were removed with a scalpel from the specimen's top surface (opposite surface from the light exposure) under safe yellow light and subsequently analyzed in transmission at 8 cm^{-1} resolution. Three spectra were analyzed per specimen. The ratio between the intensities of aliphatic C=C (at 1637.3 cm^{-1}) and aromatic C=C (at 1608.3 cm^{-1}) peaks for uncured and cured samples was used to calculate the degree of conversion, according to the following equation:

$$DC = \left[1 - \left(\frac{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{C arom)] cured resin}}{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{C arom)] uncured resin}} \right) \right] \times 100$$

Where: *DC* is the degree of conversion, *Abs (C=C) arom* is the height of the benzene ring peak and *Abs (C=C) aliph* is the height of the aliphatic C=C bonds peak, for both cured and uncured composites.

Push-out Bond Strength

Selection and Teeth Preparation

Forty bovine incisors free from cracks or any other kind of structural defect were selected under x20 magnification. The teeth were disinfected in 0.5% chloramine for 15 days and stored for less than 1 month in 0.9% saline solution. The crowns were cut off at the cement-enamel junction (Figure 1A) using a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil). All buccal surfaces were ground and flattened under water cooling with a 180 grit SiC paper, to standardize the thickness of the enamel to approximately 0.5 mm. A diamond bur (ref. 3017HL, Fava, São Paulo, SP, Brazil) was used to partially grind the lingual face of the crown, resulting in 2.5 mm tooth substrate in height, preserving the mesial and distal crown segments in the lingual surface to reinforce the specimens (Figure 1C).

Conical cavities were prepared in the buccal surface of each tooth with a diamond bur (ref. 3131, KG Soresen) with a high-speed water-cooled handpiece (Kavo SA, Joenville, SC, Brazil) in a standard cavity preparation appliance (Figure 1B). The diamond bur was replaced after every 5^{th} preparation. The cavity presented a conical form 2.5 mm in height, with a diameter of 2.1 mm at the top and diameter of 1.5 mm at the bottom (Figure 1D), resulting in a C-factor of 3.0.

Restorative procedures

Preparations were etched using 35% phosphoric acid (Scotchbond Etchant, 3M-ESPE, St. Paul, MN, USA, batch number 5EN). Single bond adhesive system (3M-ESPE, St. Paul, MN, USA, batch number 4KB) was applied according to manufacturer's instructions and photoactivated for 10 seconds, at 700 mW/cm^2 (XL 2500 - 3M-ESPE, St Paul, MN, USA). The composite was placed in bulk (Filtek Z250, shade A2, batch number 5JK). A mylar strip and a microscope slide were placed over the cavity, and used to force the composite to adapt to the preparation walls and to extrude the excess material. The slide was then removed, and the light curing tip was positioned against the mylar, followed by photoactivation. The four light curing methods evaluated are described in Table 1. The irradiances of 550, 150 and 80 mW/cm^2 were reached by placing neutral density filters between the light source and the guide. There was no need for a turbo light guide with VIP in this experiment.

The samples were stored in distilled water at 37^{o}C for 24 hours. Restorations were polished with Sof-Lex disks (3M-ESPE, St Paul, MN, USA) on the buccal and lingual surface. The bond strength was evaluated using a push-out test. The sample was positioned on top of a metallic device that had an aperture that allowed the smaller diameter of the restoration to be in contact with an aspheric device, connected to the load cell of a universal testing machine (Instron, model 4411, Buckinghamshire, England). This aspheric device applied a compressive force on the smaller diameter surface of the restoration, until rupture of the tooth-composite bond was achieved. (Figure 1E). The push-out test was carried out at a cross head speed of 0.5 mm/min. Values in MPa were obtained by dividing maximum load by the bonded surface area of each specimen.

After the test, the fractured specimens were examined under a stereomicroscope at 40x (Carl Zeiss, Manaus, AM, Brazil) and classified as to the characteristic of failure: cohesive failure in composite, cohesive failure in dentin, adhesive failure, or mixed (adhesive and cohesive in composite).

Statistical analysis

Maximum contraction stress, degree of conversion, and bond strength values were analyzed by one-way ANOVA and Tukey's test at a significance level of 5%. Correlation analysis of initial irradiance vs. contraction stress (Figure 5A) and initial irradiance vs. stress rate (Figure 5B) were also performed.

Results

Influence on Polymerization Contraction Stress and Stress rate

As shown in Figure 2, a sharp increase in contraction stress was observed for all curing methods immediately following light activation. A continuous increase in the stress values was observed for all methods when the light was turned off, but at different rates. The stress rate in the first 20 seconds of light exposure is shown in Figure 3 and the maximum stress rate is listed in Table 2. CL presented the highest stress rate (0.32 MPa/s), reached at 3.3s, followed by SS, with a maximum stress rate of 0.21 MPa/s, reached at 3.3s. The PD methods presented the lowest maximum stress rates: 0.15 MPa/s for PD150 at 3.3s and 0.04 MPa/s for PD80 at 4.3s. Figure 4 shows stress rates for the PD methods during the second cycle of light exposure. PD80 presented a maximum stress rate of 0.21 MPa/s, while PD150 had a rate of 0.15 MPa/s. Table 2 lists the mean contraction stress values and standard deviations for all curing methods. The CL (10.3 MPa) and SS (10.2 MPa) methods presented the highest mean values, and were statistically different from PD80 (7.9 MPa). The curing method PD150 (9.3 MPa) presented an intermediate value, equivalent to all other methods evaluated ($p > 0.05$).

Correlation curves of initial irradiance vs. maximum contraction stress and initial irradiance vs. stress rate showed a reasonable fit with a logarithmic function (Figure 5). The contraction stress and stress rate demonstrated an increase with increasing initial irradiance values, but both tended to level off at the higher energy levels.

Influence on Degree of Conversion

The degree of conversion of the restorative composite ranged from 54.2 % (PD150) to 55.9 % (PD80) at 24 hours (Table 2). No statistical difference was observed among the curing methods.

Influence on Bond Strength

Bond strength results are shown in Table 2. PD150 and PD80 curing methods presented the highest mean values, 35.5 MPa and 34.4 MPa respectively, statistically higher than CL and SS methods. SS curing method presented a mean bond strength value of 30.6 MPa, statistically higher than the ones presented by CL, which in turn had the lowest mean value of bond strength, 26.4 MPa. The CL method showed adhesive failure to be the most frequent failure mode (80%). SS showed equal percentages of adhesive failure (50%) and mixed failure (50%). The PD methods presented the lower percentage of adhesive failure: 30% for both. Mixed failure was the most frequent failure mode for the PD methods: 60% for PD150 and 70% for PD80. PD150 was the only method that presented cohesive failure in composite (10%).

Discussion

In this study, the highest stress mean value was reached by the CL method, which can be partially explained by its respectively higher maximum stress rate (Table 2). For this method, the reaction might have evolved too fast, virtually eliminating the opportunity for viscous flow, leading to a dramatic increase in stiffness after a relatively low degree of conversion.[1,22] As a result, stress develops almost immediately after polymerization is triggered,[23] and the vast majority of the conversion occurs after the polymer matrix has reached a significant level of rigidity.[1]

From the correlation between stress and irradiance ($r^2 = 0.73$), it was shown that stress development seems to be directly proportional to the increase in irradiance. However, the maximum stress was not significantly different among CL (10.3 ± 1.08 MPa), SS (10.2 ± 0.84 MPa), and PD150 (9.3 ± 1.34 MPa) curing methods, in contrast to the differences in the maximum stress rate values. The maximum stress rate reached by SS (0.21 MPa/s) and PD150 (0.15 MPa/s) methods was respectively 34 and 53 % lower than that presented by CL (0.32 MPa/s). It is possible that, for SS and PD150, the initial low irradiance led to a decreased initial polymerization, reflected as a reduction in the stress rate, thereby modifying the generation and distribution of stress, as also verified by Hofmann et al. [24] Thus, it appears that although the net stress might be similar for different curing methods, the path to arrive at the maximum stress differs.[5]

The reduction in the stress rate observed for SS and PD150 could be related to improved bond preservation, as verified in this study. PD150 presented a mean value of bond strength statistically superior (35.5 MPa) to SS (30.6 MPa) and to CL (26.4 MPa). The same situation was shown for SS, which presented a mean value of bond strength statistically superior to CL. Besides, from the analysis of failure mode, CL curing method predominantly presented adhesive failure (80%), in contrast to SS, in which half of the samples presented mixed failure, symbolizing a higher probability of bond preservation for the SS method. Therefore, in contrast to the lack of statistical difference among SS, PD150, and CL in the maximum mean stress values, differences in the initial stress rates related to the lower initial irradiance of SS and PD150 resulted in a significant increase of the mean bond strength value for these methods.

The correlation analysis between stress rate and irradiance ($r^2 = 0.82$) showed a directly proportional relation between these factors. Indeed, the method using the highest initial irradiance (CL) was associated with the highest stress rate, when compared to lower initial irradiance curing methods, such as PD80. In fact, PD80 led to the lowest stress rate (0.04 MPa/s – first cycle of light exposure) in this study, being 85% lower than the highest rate for CL (0.32 MPa/s)

In addition, PD80 was the only curing method which showed a statistically lower mean value of contraction stress (7.9 ± 1.04 MPa) when compared to CL. However, differences were observed for PD methods after the second cycle of light exposure. For PD150, the maximum stress rate reached after the 3-minute interval was 0.15 MPa/s, which was the same value reached in the first irradiation. The initial pulse limited the contraction force produced when the second, higher irradiance exposure was applied. Therefore, the

PD150 method resulted in lower stress rate during the second cycle of light exposure, even using the same irradiance as the CL method. However, the same was not observed for PD80, in which the second cycle of light exposure produced a maximum stress rate of 0.21 MPa/s, an increase of 320% in relation to the first irradiation. Also, the stress rate for the second pulse in this case was almost 30% higher than that produced by PD150 in the same cycle. This could be explained by the fact that, at this very low irradiance in the first pulse, there is insufficient photon-density to initiate a significant part of the reaction.[5,25] Therefore, it can be speculated that a significant proportion of the conversion took place only after the second irradiation. This was expected to have lead to a higher stress rate in the second cycle, since more remaining double bonds would still be present in this case.[5,25-26] A significant influence of the delayed stress generation was observed in the bond strength results, in which no statistical difference was verified between PD150 (35.5 MPa) and PD80 (34.4 MPa), despite the lower irradiance (80 mW/cm^2) and the lowest initial stress rate (0.04 MPa/s) related to PD80 in this study. However, the mean values of bond strength observed for both PD methods were statistically superior to CL and SS methods. Besides, the predominant failure mode of the PD methods was mixed, and PD150 curing method was the only one in which cohesive failure in composite was observed.

Stress rate is dependent on irradiance and not dependent on total light energy, i.e., radiant exposure.[5] The stress rate calculated here is an instantaneous stress rate, as opposed to a final stress/total time, as reported by others.[27] Therefore, it was possible to determine that the maximum stress rate occurred early in the curing cycle. Moreover, after the stress rate peak was reached (the largest being at 4.3s for PD80 in this study), it declined regardless of the irradiance and duration of exposure. The highest stress rate was observed for the CL method (0.32 MPa/s, at 3.3s). SS and PD150 presented intermediate values of maximum stress rate, respectively 0.21 and 0.15 MPa/s, reached at 3.3s for both methods. The lowest maximum stress rate was related to PD80, 0.04 MPa/s, at 4.3s. Thus, a lower irradiance can always be expected to produce a lower contraction stress rate when compared to higher irradiance irrespective of the duration of light exposure.[5]

It has to be pointed out, however, that reducing contraction stress by changing polymerization rate has limitations. Some studies [28-30] in the literature concluded that modulated curing methods may lead to the formation of a more linear polymer network, which could be more prone to leachability in organic solvents Even if they produce equivalent degrees of conversion, non-continuous curing methods may lead to significantly more ethanol degradation compared to continuous high-intensity irradiation. In contrast to these studies, Lovell et al. [31] observed that an experimental composite based on Bis-GMA/TEGDMA exhibited similar network structure, regardless of the method or rate of cure. Therefore, more studies should evaluate this fact, to certify what is the real influence of modulated curing methods on the polymer network.

The degree of conversion results showed a similar conversion of the composite when the specimens were exposed to equivalent light dose (16 J/cm^2), providing evidence for a reciprocal relationship between irradiance and exposure time.[25] The absence of statistical differences among CL (55.1 ± 0.5%), SS (54.8 ± 1.3%), PD150 (54.2 ± 2.6%) and PD80 (55.9 ± 0.6%), confirm the findings of different studies, [5, 25] in which statistical equivalence was verified for the combinations of exposure time and irradiance within the same total dose. Therefore, the advantages of the modulated methods, when compared to CL, such as the lower stress generation for PD80 and the superior bond strength values for SS, PD150, and PD80, were reached with no reduction of the degree of conversion.

In conclusion, the tested hypothesis that modulated curing methods would provide a significant reduction of contraction stress leading to improved bond strength was partially validated by the results. In spite of the lack of statistical difference among CL, SS, and PD150 as to the maximum stress generation, the reduction in the stress rate observed for SS, PD150 and PD80 proved to be effective in significantly increasing the bond strength, with no adverse effect on the degree of conversion of the restorative composite. It remains to be seen if these advantages could associate the modulated curing methods with significantly higher clinical longevity and better performance of composite restorations.

Acknowledgements

This study is part of a thesis, for partial fulfillment of the PhD degree in Dental Materials. Supported by CAPES (PDEE grants BEX3428/04-4).

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Table 1. Light-curing methods evaluated with their outputs and the respective energy density

Method	Mode of Irradiation	Radiant Exposure
Continuous Light (CL)	30s at 550 mW/cm^2	16 J/cm^2
Soft-Start (SS)	10s at 150 mW/cm^2 + 27s at 550 mW/cm^2	16 J/cm^2
Pulse Delay 150 (PD150)	5s at 150 mW/cm^2 + 3 min + 28s at 550 mW/cm^2	16 J/cm^2
Pulse Delay 80 (PD80)	5s at 80 mW/cm^2 + 3 min + 29s at 550 mW/cm^2	16 J/cm^2

Table 2. Contraction stress, maximum stress rate, degree of conversion, and bond strength generated by the light-curing methods.

	Contraction Stress (MPa)	Stress rate (MPa/s)	DC (%)	Bond Strength (MPa)
Continuous Light	10.3 (1.1) a	0.32	55.1 (0.5) a	26.4 (2.4) c
Soft-Start	10.2 (0.8) a	0.21/0.19 *	54.8 (1.3) a	30.6 (2.2) b
Pulse Delay 150	9.3 (1.3) ab	0.15/0.15 #	54.2 (2.6) a	35.5 (3.4) a
Pulse Delay 80	7.9 (1.0) b	0.04/0.21 #	55.9 (0.6) a	34.4 (1.7) a

* First value is maximum stress rate during first ten seconds of light exposure in reduced light intensity while second value corresponds to maximum stress rate during cure after the ten initial seconds

First value is maximum stress rate during primary step of pulse-delay cure while second value corresponds to maximum stress rate during cure after the delay.

Mean values followed by different small letters in the same column differ statistically among themselves for the Tukey test at the level of 5%. ( ) – Standard Deviation

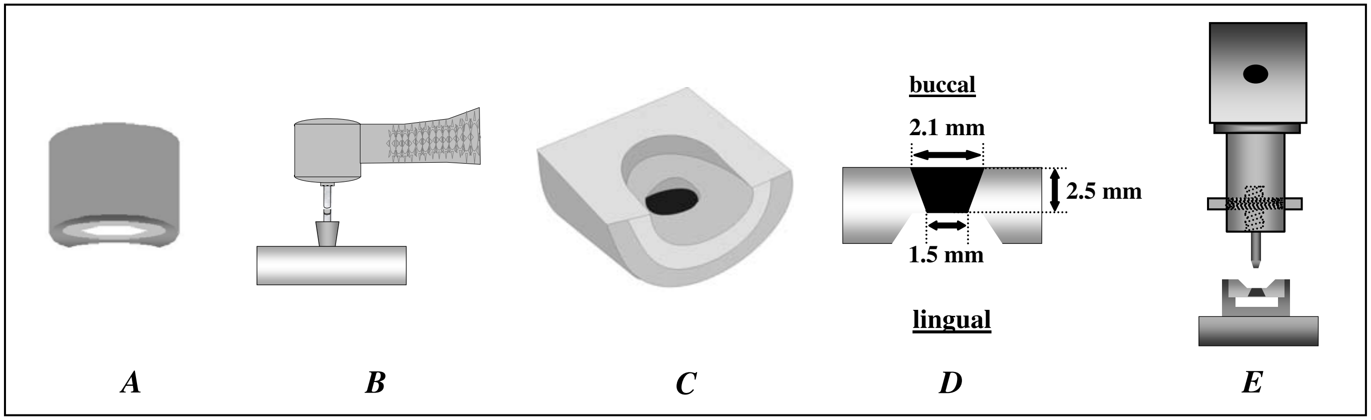

Figure 1. Schematic representation of the "push-out" test: ***A.*** Dental fragment after flattening of the buccal surface and selective grinding of the lingual surface; ***B.*** Cavity preparation in the standard cavity preparation appliance; ***C.*** Lingual view of the dental sample with the cavity done; ***D.*** Lateral view of the restored sample (2.5 mm in height, top diameter of 2.1 mm, and bottom diameter 1.5 mm); ***E.*** Lateral view of the complete system for the accomplishment of the test.

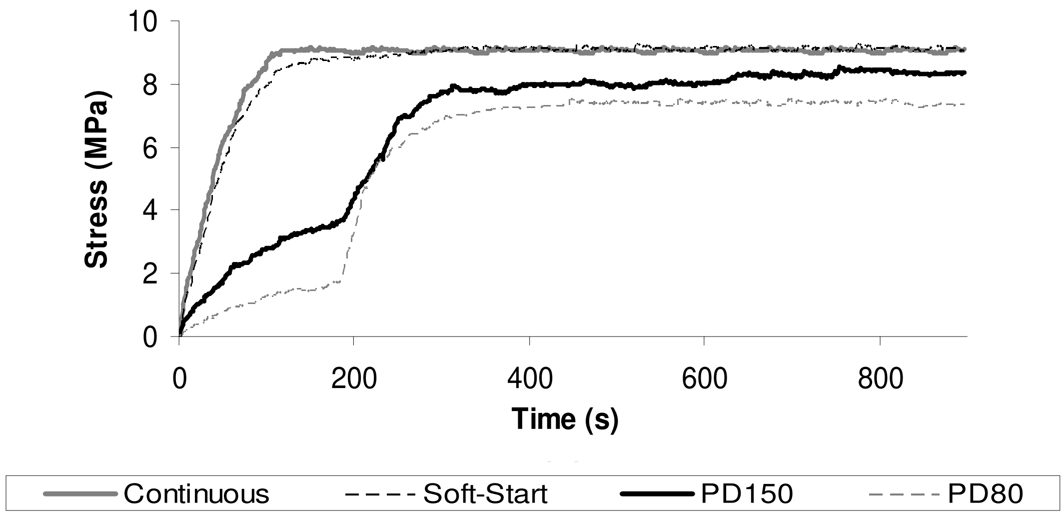

Figure 2. Stress values (MPa) for each light-curing method as a function of tim

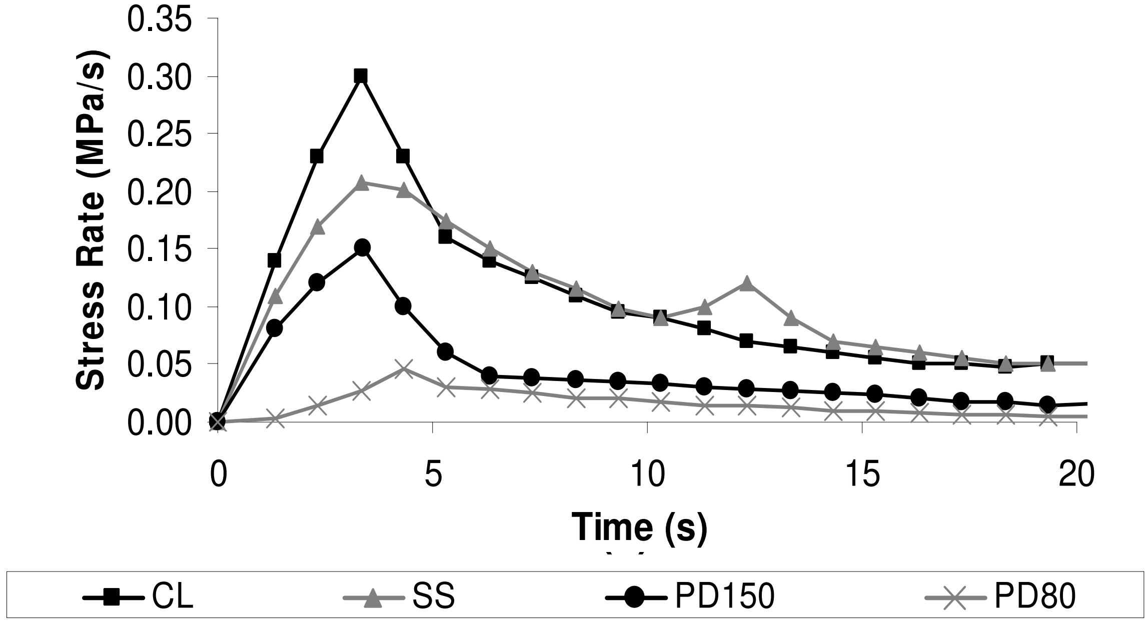

Figure 3. Stress rate (MPa/s) during the first 20 seconds of curing for each light-curing method as a function of time

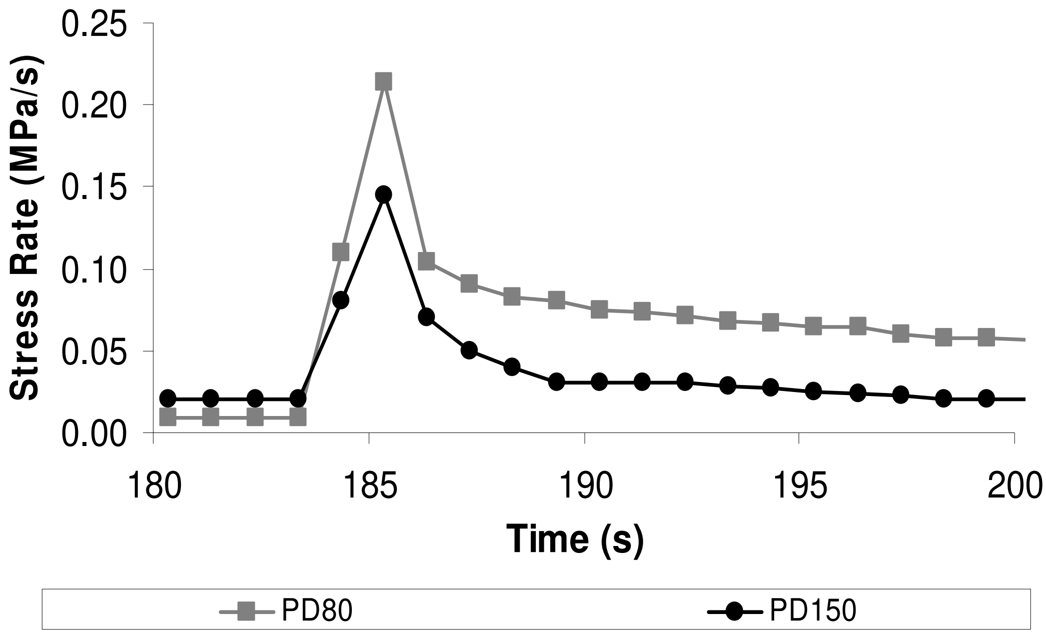

Figure 4. Stress rate (MPa/s) during the second cycle of light exposure to PD80 and PD150 curing methods as a function of time

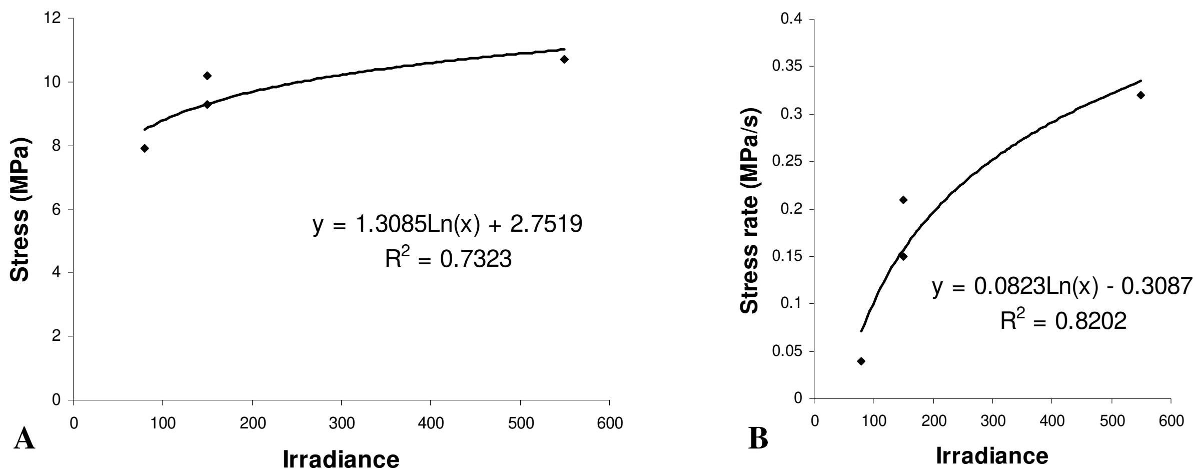

Figure 5. Correlation curves of irradiance vs. stress (**A**) and irradiance vs. stress rate (**B**)

CAPÍTULO 3

PHYSICAL PROPERTIES AND CONTRACTION STRESS OF A RESIN COMPOSITE IRRADIATED USING HALOGEN AND LED LIGHT CURING WITH DIFFERENT IRRADIANCES AT TWO C-FACTOR LEVELS

Manuscrito submetido para publicação no periódico

Journal of Dentistry

Physical properties and contraction stress of a resin composite irradiated using halogen and LED light curing with different irradiances in two C-factor levels

Summary

Objectives: Verify the influence of five curing methods and two light sources on contraction stress, stress rate, and degree of conversion (DC) of a restorative composite at two C-factor levels.

Methods: For the stress test, composite (0.84 mm thick) was applied between two 5-mm diameter glass rods, mounted in a servohydraulic machine. Stress rate was taken by the value of stress/time at each second. DC was measured by FTIR. Five curing methods were tested at two C-factor levels (1.5 and 3.0): High Intensity LED (LED HI), Continuous Light (QTH CL), Medium Intensity LED (LED MI), Low Intensity LED (LED LI), and Pulse Delay (QTH PD). Results were analyzed by ANOVA and Tukey's test.

Results: To the stress test, at CF 1.5, statistical differences were only observed for QTH PD when compared to LED HI, QTH CL, and LED LI. At CF 3.0, no difference was observed for the curing methods. Stress values at CF 3.0 were statistically higher than the ones at CF 1.5. To stress rate, in both C-factor levels, LED HI presented the highest maximum stress rate, followed by QTH CL, LED MI, LED LI, and QTH PD. In the DC test, no difference was observed among the methods, in both CF levels.

Conclusions: Curing methods using lower irradiance levels were shown to be effective in reducing stress rate, with no damage to the degree of conversion of the restorative composite. C-factor was shown to influence negatively the stress rate and the amount of stress generated.

Keywords: curing methods; restorative composite; FTIR; contraction stress, stress rate.

Introduction

Clinical failure of composite restorations is often the result of an incomplete sealing of the tooth/restoration interface.[1] The role of composite polymerization stress as one of the main causes of marginal integrity loss and consequent post-operative occurrences such as hypersensitivity, microleakage and secondary caries, has been described by some studies.[2-3]

Stress magnitude is related to the restorative technique employed, as well as to composite composition and degree of conversion. One of the factors associated with the restorative procedure is the restoration's bonded-to-unbonded ratio (cavity configuration factor, or C-factor).[4] The confinement of the composite would hinder the volume reduction compensation by viscous flow from the free surface that may take place before the gelation of the material.[5]

It has been proposed that the method by which light energy is delivered to the composite is capable of delaying the acquisition of elastic modulus, allowing polymeric chains to re-arrange and microscopically and macroscopically accommodate the reduction in volume by plastic deformation.[6] The use of low irradiances has become widespread in clinical practice, as several studies have shown that the use of continuous low intensity curing routines, as well as those characterized by reduced irradiance at the initial seconds, may lead to significant reductions in microleakage and gap formation in composite restorations.[7-8]

Different light sources may also influence the development of physical properties. Blue light emitting diode (LED) has the advantage of narrower spectral range than the QTH light, and the better match of the light emitted by LED LCUs with the absorption spectrum of the photoinitiator camphorquinone.[9-11]

Therefore, the purpose of this study was to evaluate the effect of different irradiance levels and light sources (QTH and LED) on the stress generated, stress rate, and degree of conversion of a resin composite, in two C-factor levels. The light-curing methods using reduced irradiance levels were hypothesized to promote a reduction in the contraction stress and stress rate, with no reduction in the degree of conversion of the restorative composite. A higher C-factor level was hypothesized to generate an increase in the stress level and stress rate, with no influence on the degree of conversion values.

Materials and Methods

Light-curing was performed with two light-curing units: VIP (Variable Intensity Polymerizer, Bisco, Schaumburg, IL, USA), a halogen light unit, which provides different levels of irradiance, and Free-Light II (3M-ESPE, St. Paul, MN, 55144, USA), a LED light source of high irradiance. In order to accomplish the irradiances used in the protocols evaluated (Table 1) in all of the following three tests, for both light-curing units neutral density filters were placed between the light source and the guide. For VIP, a Turbo tip was also used, except on the bond strength test. Filtek Z250 (shade A2, 3M-ESPE, St. Paul, MN, USA, batch number 5JH) was used for all experiments.

Polymerization Contraction Stress and Stress Rate

Polymerization Contraction Stress Test was performed with a closed loop servohydraulic testing instrument (MTS 858, MTS Systems, Eden Prairie, MN, USA). Two borosilicate glass rods 5 mm in diameter were sandblasted (250 µm Al_2O_3) and treated with a silane (Silane ceramic primer, 3M-ESPE, USA) and light-cured adhesive resin (Scotchbond MP, 3M-ESPE, USA, batch number 0MA). One of the glass rods (12 mm in height) was attached to a metallic fixture connected to the actuator, which had a slot through which the light curing guide was kept in contact with the opposing side of the rod. The other rod was 10 mm in height and was bonded to a fixture attached to the load cell. Contraction stress was measured by placing two different thickness of composite between the two rods: 1.66 mm (ratio of bonded-to-unbonded surface area - 1.5) and 0.84 mm (ratio of bonded-to-unbonded surface area - 3.0). A near zero compliance system was set up by using an eddy current feedback system (Kaman Instruments, Colorado Springs, CO, USA) that kept the distance between the rods constant. A light-curing guide was directed down through the upper rod and the contraction forces recorded during 10 min from the initiation of light activation. Maximum force was divided by cross-sectional area to calculate average axial stress. Five samples of each curing method were tested. Stress rates were calculated as the change in stress vs. time at each second during the measurement period.

Prior to testing, the light intensity at the top of the specimen was measured using the power meter (Power Maximum 5200, Molectron, Portland, OR, USA). For the VIP unit, when the irradiance at the end of the Turbo light guide was 780 and 220 mW/cm^2, the irradiance at the surface of the composite was approximately 550 and 150 mW/cm^2, respectively, considering a reduction of 30% in the light intensity when the light passed through the glass rod. Considering the same reduction for the Free-Light unit, irradiances of 1200, 780, and 290 mW/cm^2 at the end of the light guide corresponded approximately to 850, 550, and 200 mW/cm^2 at the surface of the composite.

Degree of Conversion

The degree of conversion of the resin composite in the five curing methods tested was determined using a micro-Fourier Transform Infrared Spectroscopy (FTIR) analyser (DS20/XAD, Analect Instruments, Irvine, CA, USA). Glass rings of two different thickness (0.5 and 2.0 mm) and 4 mm diameter and glass slides were sandblasted and silanated (Silane ceramic primer, batch number 5WJ). The rings were bonded to the slide using a thin coat of adhesive (Scotchbond MP, batch number 2MT), resulting in two different glass cavities of bonded-to-unbonded area ratio of 1.5 (4 mm diameter and 0.5 mm depth) and 3.0 (4 mm diameter and 2 mm depth). The glass cavity was bulk filled with the restorative composite. A mylar strip and glass slide were pressed over the glass cavity to force the composite to adapt to the cavity walls and to extrude the excess material. The composite was then light-activated through the glass slide at the bottom of the cavity. For the VIP unit, when the irradiance at the end of the Turbo light guide was 610 and 170 mW/cm^2, the irradiance at the bottom surface of the composite was approximately 550 and 150 mW/cm^2, respectively, considering a reduction of 10% in the light intensity when the light passed through the glass slide. Considering the same reduction to the Free-Light unit, irradiances of 950, 610, and 220 mW/cm^2 at the end of the light guide corresponded approximately to 850, 550, and 200 mW/cm^2 at the surface of the composite. Three samples for each experimental condition were prepared and stored dry for 24h at room temperature. Chips of composite approximately 50x100 µm in size were removed with a scalpel from the specimen's top surface (opposite surface from the light exposure) under safe yellow light and subsequently analyzed in transmission at 8 cm^{-1} resolution. Three spectra were analyzed per specimen. The ratio between the intensities of aliphatic C=C (at 1637.3 cm^{-1}) and aromatic C=C (at 1608.3 cm^{-1}) peaks for uncured and cured samples was used to calculate the degree of conversion, according to the following equation:

$$DC = \left[1 - \left(\frac{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{C arom)] cured resin}}{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{C arom)] uncured resin}} \right) \right] \text{ x } 100$$

Where: *DC* is the degree of conversion, *Abs (C=C) arom* is the height of the benzene ring peak and *Abs (C=C) aliph* is the height of the aliphatic C=C bonds peak, for both cured and uncured composites.

Statistical analysis

Maximum contraction stress and degree of conversion values were analyzed by one-way ANOVA and Tukey's test at a significance level of 5%. Correlation analysis of initial irradiance vs. stress rate was also performed for C-factor 1.5 (Figure 3A) and for C-factor 3.0 (Figure 3B).

Results

Effect on Polymerization Contraction Stress and Stress rate

As shown in Figure 1, a sharp increase in contraction stress was observed immediately following light activation, for all curing methods and both C-factors. A continuous increase in the stress values was observed when light was turned off, for all methods and both C-factors. The stress rate in the first 20 seconds of light exposure for the curing methods in both C-factors, and the influence of the C-factor on the stress rate is shown in Figure 2. Maximum stress rate is listed in Table 2. At C-factor 1.5, LED HI presented the highest stress rate (0.26 MPa/s), reached at 3.3s, followed by QTH CL, with a maximum stress rate of 0.24 MPa/s, reached at 4.3s. LED MI presented an intermediate value of maximum stress rate, 0.19 MPa/s at 4.3s. The LED LI and QTH PD methods presented the lowest maximum stress rates: 0.14 MPa/s for LED LI at 7.3s and 0.07 MPa/s for QTH PD at 4.3s. At C-factor 3.0, the same order was observed: LED HI showed the highest maximum stress rate (0.46 MPa/s at 2.3s), followed by QTH CL and LED MI (0.32 MPa/s reached at 3.3 for both methods), LED LI (0.21 MPa/s at 4.3s) and QTH PD (0.15 MPa/s at 3.3s). Table 2 lists the mean maximum contraction stress values and standard deviations for all curing methods at the different C-factor levels. At C-factor 1.5, HI LED (8.6 MPa), QTH CL (8.6 MPa), and LI LED (8.7 MPa) methods were responsible for the highest mean values, not differing statistically among themselves and presenting statistical superior mean values ($p < 0.05$) to QTH PD method (7.3 MPa). The curing method MI LED (8.0 MPa) presented an intermediate mean value, equivalent to all other methods evaluated ($p > 0.05$). At C-factor 3.0, no statistical difference ($p > 0.05$) was observed among the curing methods. For all curing methods, the mean maximum stress values reached at C-factor 3.0 were statistically superior ($p < 0.05$) to the ones reached at C-factor 1.5.

Correlation curve of initial irradiance vs. stress rate at C-factor 1.5 showed a reasonable fit with a logarithmic function (Figure 3A). The stress rate demonstrated an increase with increasing initial irradiance values, but tended to level off at the higher energy levels. At C-factor 3.0, the correlation curve of initial irradiance vs. stress rate showed a linear function (Figure 3B).

Effect on degree of conversion

As can be seen in Table 2, no statistical difference ($p>0.05$) was observed for the curing methods tested, in which of the C-factors evaluated. At C-factor 1.5, mean values ranged from 52.6 % (MI LED) to 54.6 % (QTH CL). To the C-factor 3.0, mean values ranged between 52.5 % (HI LED) and 54.6 % (LI LED). To each curing method, no influence of the C-factor levels was observed on the mean values of degree of conversion.

Discussion

From the results of this study, no statistical difference was observed for the curing methods to the maximum contraction stress values at C-factor 3.0, and at C-factor 1.5, QTH PD was the only method in which a statistical inferior mean contraction stress value was observed when compared to LED HI and QTH CL. However, it was possible to observe a significant influence of the curing method on the contraction stress rate. The correlation analysis between irradiance and stress rate at C-factor 1.5 ($r^2 = 0.91$) and at C-factor 3.0 ($r^2 = 0.98$) showed a directly proportional relation between these factors. Indeed, the method using the highest initial irradiance (LED HI) was associated with the highest stress rate, when compared to lower initial irradiance curing methods, such as LED LI and QTH PD. In fact, QTH PD led to the lowest stress rates (0.07 and 0.15 MPa/s – at C-factors 1.5 and 3.0 respectively) in this study, being 73% lower at C-factor 1.5 and 67% lower at C-factor 3.0 than the highest stress rates found in this study (LED HI - 0.26 and 0.46 MPa/s at C-factors 1.5 and 3.0 respectively). At C-factor 1.5, the slower polymerization reaction increased the probability of partial stress release. Indeed, the reduction of 73% in the stress rate observed for QTH PD could be responsible for a significant reduction of the final stress generated, resulting in a statistical inferior mean value of contraction stress to QTH PD when compared to LED HI.

However, the same situation was not observed at C-factor 3.0. Though the curing rate of QTH PD was significantly reduced (67% lower), it did not result in a significant decreasing in final contraction stress. Findings of previous studies [12-14] suggest that polymerization rate must be reduced under a certain threshold in order to significantly reduce final contraction stress, maybe because the gelation of metacrylates takes place at very low degrees of conversion [15] and once the reaction has started, an autoaccelaration fenomena is observed and stresses are expected to increase drastically because of the consequent increase in stiffness given by crosslinking.[16] Therefore, the period allowed for the material to flow is very restricted, and a substantial decrease in curing rate is required to significantly affect contraction stress development. This situation is even worse in a high C-factor level, in which the faster polymerization reaction decreases the probability of partial stress release. However, it is also important to notice that materials with distinct compositions may show different behaviors when submitted to the same curing methods tested. A study evaluating the effect of low curing rates on contraction stress development of three commercial materials found stress reductions between 19 and 30%.[17]

One possible explanation for the similar final contraction stress values found for all experimental groups at C-factor 3.0 is that the final radiant exposure employed for all of them was similar (16 J/cm^2). According to previous findings[18], this would lead to the same degree of conversion, as also observed in the present study. Silikas et al. [19] found high correlation (r^2>0.99) between degree of conversion and stress generation. Therefore, if the reduction in the stress rate caused by the modulated curing methods was not enough to significantly decrease the final stress of the composite used, as observed for the high C-factor level groups, the degree of conversion turns to the most important factor affecting the development of final contraction stress in dental composites.[20] Thus, the statistical equivalence in the degree of conversion to the curing methods could explain the results found in this study for the maximum stress mean values at C-factor 3.0.

For all curing methods, the final stress reached at C-factor 1.5 were statistically inferior to the ones reached at C-factor 3.0. Since composite flow is more likely to occur from the free surfaces of the specimen, a lower level of C-factor will indicate a higher proportion of free composite surface, causing a smaller restriction to shrinkage, thereby reducing stress. [21-22] In this study, a mean reduction of 20% in the maximum stress was found at C-factor 1.5, when compared to the values reached at C-factor 3.0. Besides, the influence of the C-factor level was not just related to the amount of stress generated, but it also had a significant influence on the stress rate. As it can be seen in the mean values of stress rate on Table 2 and on Figure 2, the maximum stress rate found at C-factor 1.5, independent of the curing method, was around 35% lower than the one related to C-factor 3.0.

Stress rate is dependent on irradiance and not dependent on radiant exposure.[23] The stress rate values reported here were estimated in real time, as opposed to final stress/total time, as reported by others,[24] making it possible to determine the time in the curing cycle at which the maximum stress rate occurred. It could be observed that maximum stress rate developed early in the curing cycle and after the stress rate peak was reached, it declined regardless of the irradiance and duration of exposure. Interestingly, the method that presented the highest stress rates (LED HI) also showed the most premature maximum stress rates development, 3.3s and 2.3s, at C-factors 1.5 and 3.0 respectively. Besides, the C-factor level showed significant influence in the stress rate development. For all curing methods, the maximum stress rate was reached faster at C-factor 3.0, when compared to the values within the same method at C-factor 1.5.

Comparatively, the methods LED LI and QTH PD used similar low irradiances (200 and 150 mW/cm^2, respectively), but the first in continuous mode and the latter in a 5 sec exposure, followed by a 3 minute interval and a second exposure at 550 mW/cm^2. If we take into consideration only the stress rate values, it can be said that QTH PD was more efficient to reduce the stress rates maybe because the first exposure used not only low irradiance but was performed for a short time. Since conversion was expected to keep on increasing in the dark period, when the second, higher irradiance exposure was applied, there were less carbon double bonds left to be used in the growing chains. Therefore, QTH PD method resulted in lower stress rates during the second cycle of light exposure (0.12 and 0.15 MPa/s at C-factors 1.5 and 3.0 respectively), even using the same irradiance of the QTH CL or LED MI methods.

Another interesting comparison that can be made from the results of the present study is between QHT CL and LED MI methods themselves. Some manufactures claim that LED units can reach higher degree of conversion compared to halogen units, only in a short exposure, which would be clinically advantageous.[25-26] The rationale is the narrow light spectrum of LED units, with wavelengths between 438 and 501 nm, and peak intensity at 465 nm,[9-11] which is coincident with the absorption peak of the camphorquinone, between 465 and 470 nm,[27] making its conversion more efficient. In the present study, comparing the curing methods LED MI and QTH CL, both using the same irradiance of 550 mW/cm^2, and the same radiant exposure (16 J/cm^2), no statistical difference was observed for the groups in the degree of conversion and maximum contraction stress at both C-factor levels. At least for the degree of conversion, this fact corroborates the results of other studies,[28-29] in which no difference was observed in the development of physical properties when LED and QTH units were compared.

In conclusion, the tested hypothesis that curing methods using reduced irradiance or modulation of the light exposure would provide a significant reduction on contraction stress was partially validated by the results. In spite of no statistical difference among LED methods and QTH CL at C-factor 1.5, and also for all methods at C-factor 3.0 as to the maximum stress generation, a significant reduction in the stress rate values was observed for the curing methods using reduced irradiance levels. This was reached with no expense for the degree of conversion. The hypothesis concerning the C-factor was validated by the results. Higher the C-factor level, higher the amount of stress generated, and faster the generation of stresses. C-factor was proven to have no effect on the degree of conversion of the restorative composite.

Acknowledgements

This study is part of a thesis, for partial fulfillment of the PhD degree in Dental Materials. Supported by CAPES (PDEE grants BEX3428/04-4).

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Table 1. Light-curing methods evaluated with their outputs and the respective energy density

Method	Irradiation protocol	Light Source	Radiant exposure
High Intensity LED (HI LED)	19s at 850 mW/cm^2	LED	16 J/cm^2
Continuous light (QTH CL)	30s at 550 mW/cm^2	Halogen	16 J/cm^2
Medium Intensity LED (MI LED)	30s at 550 mW/cm^2	LED	16 J/cm^2
Low Intensity LED (LI LED)	80s at 200 mW/cm^2	LED	16 J/cm^2
Pulse Delay (QTH PD)	5s at 150 mW/cm^2 + 3 min + 28s at 550 mW/cm^2	Halogen	16 J/cm^2

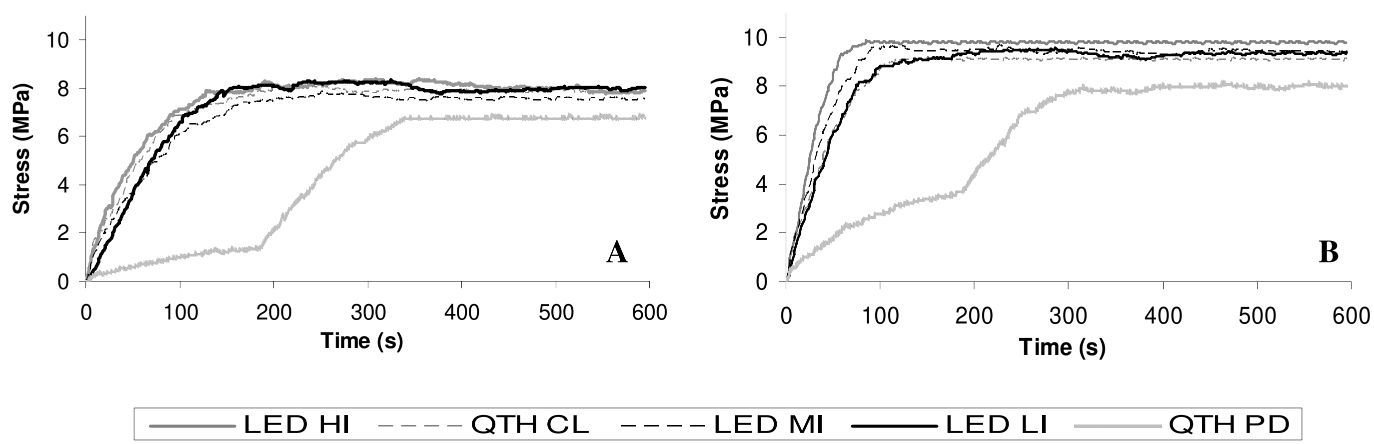
Table 2. Contraction Stress, maximum stress rate, and degree of conversion generated by the light-curing methods at each C-factor level

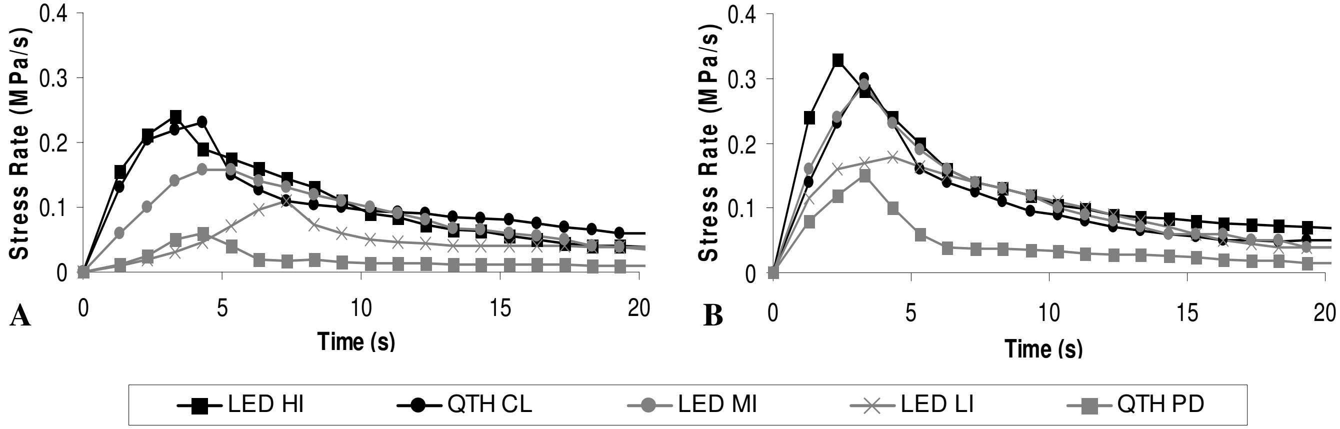
	Contraction Stress (MPa)		Stress Rate (MPa/s)		Degree of Conversion (%)	
	CF 1.5	**CF 3.0**	**CF 1.5**	**CF 3.0**	**CF 1.5**	**CF 3.0**
LED HI	8.6 (0.6) a, B	10.5 (1.2) a, A	0.26	0.46	53.0 (2.2) a, A	52.5 (1.2) a, A
QTH CL	8.6 (0.5) a, B	10.3 (1.1) a, A	0.24	0.32	54.6 (0.9) a, A	54.5 (0.5) a, A
LED MI	8.0 (1.2) ab,B	10.4 (1.2) a, A	0.19	0.32	52.6 (2.1) a, A	53.1 (1.8) a, A
LED LI	8.7 (0.8) a, B	10.3 (0.6) a, A	0.14	0.21	54.1 (2.5) a, A	54.6 (1.9) a, A
QTH PD	7.3 (0.6) b, B	9.3 (1.3) a, A	0.07/0.12 [1]	0.15/0.15 [1]	53.6 (1.3) a, A	54.2 (2.6) a, A

[1] First value is maximum stress rate during primary step of pulse-delay cure while second value corresponds to maximum stress rate during cure after the delay.

Mean values followed by different small letters in the column and capital letters in the row differ statistically among themselves for the Tukey test at the level of 5%. ( ) – Standard Deviation

Figure 1. Stress values (MPa) for each light-curing as a function of time at C-factor 1.5 (**A**) and 3.0 (**B**)





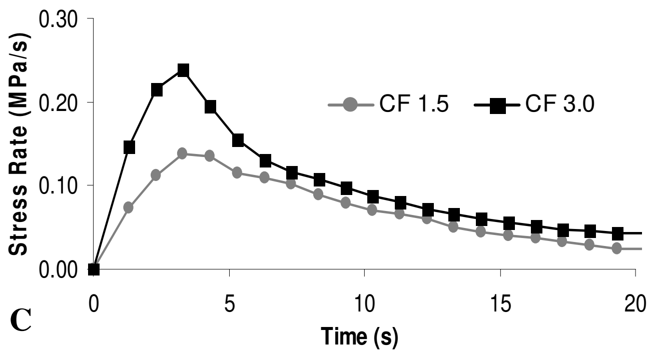
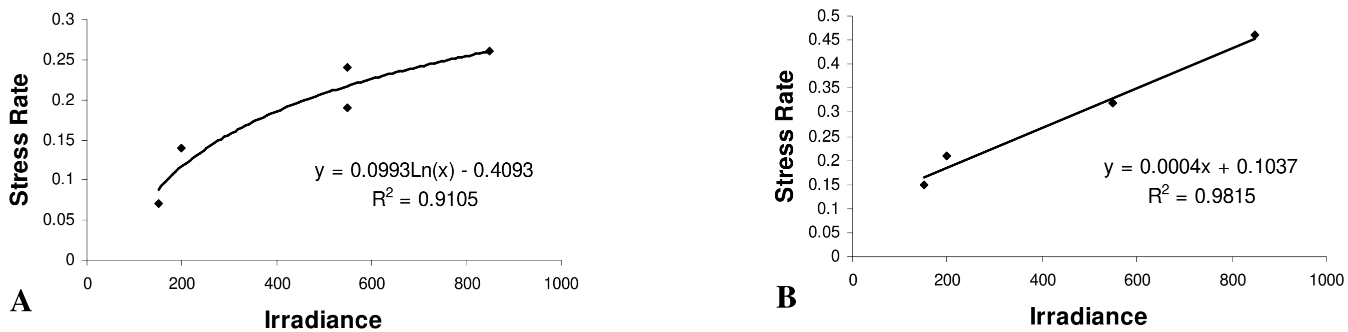


Figure 2. Stress rate (MPa/s) during the first 20 seconds of curing for each light-curing method. (**A**) at C-factor 1.5; (**B**) at C-factor 3.0; **(C)** influence of the C-factor, independent of the light-curing method

Figure 3. Correlation curves of irradiance vs. stress rate at C-factor 1.5 (**A**) and at C-factor 3.0 (**B**).



CAPÍTULO 4

EFFECT OF IRRADIANCE ON BOND STRENGTH AND PHYSICAL PROPERTIES OF A RESIN-BASED COMPOSITE IRRADIATED USING HALOGEN AND LED LIGHT-CURING

Manuscrito submetido para publicação no periódico

American Journal of Dentistry

Effect of irradiance on bond strength and physical properties of a resin-based composite irradiated using halogen and LED light-curing

ABSTRACT

Purpose: The objective of this study was to evaluate the influence of five curing methods on contraction stress, stress rate, and degree of conversion (DC) of a composite and on bond strength of composite restoratives to dentin. **Methods**: For the stress test, composite was applied between two 5-mm diameter glass rods, mounted in a servohydraulic machine. Stress rates were calculated as the change in stress vs. time. DC was measured by FTIR. Bond strength testing was performed using a push-out test in bovine incisors. The C-factor (ratio of bonded to non-bonded area) was 3.0 for all tests. Five methods were evaluated: High Intensity LED (LED HI), Continuous Halogen Light (QTH CL), Medium Intensity LED (LED MI), Low Intensity LED (LED LI), and Pulse Delay Halogen Light (QTH PD). Results were analyzed by ANOVA and Tukey's test ($\alpha = 0.05$). **Results**: Stress values ranged from 9.25 MPa (QTH PD) to 10.46 MPa (LED MI). No statistical difference was observed among the methods. LED HI presented the highest maximum stress rate, followed by LED MI, QTH CL, LED LI, and QTH PD. No difference in the DC was observed among the methods. Bond strength values ranged from 24.6 MPa (LED HI) to 35.4 MPa (QTH PD), with the QTH PD presenting a statistically higher value compared to the other methods. LED LI and LED MI were both statistically superior to LED HI, which presented the lowest mean value.

CLINICAL SIGNIFICANCE

Curing methods using lower irradiance levels were shown to be effective in reducing stress rate and improving the strength of the bonded interface, and without compromising the DC of the restorative composite.

INTRODUCTION

While composite restorations have become popular because of their aesthetic appeal, some drawbacks intrinsic to the polymerization reaction still need to be overcome. As the material cures, an increase in stiffness accompanying volumetric changes that are confined by the cavity walls results in stresses that challenge the integrity of the bond between the composite restoration and the tooth.[1] Even in cases where bonding integrity is maintained, contraction stress is a potential source for problems like cuspal deflection [2] or enamel cracks.[3]

The magnitude of such stresses is dependent upon several factors related to the geometry of the cavity (C-Factor), material characteristics such as the monomer composition, catalyst concentration and filler type and content, and restorative technique (placement technique and light delivering method, for the photoactivated systems).[4] The clinician only has control over some of these factors, such as the irradiance and exposure time.[5]

It has been proposed that the modulation of the light energy, using low irradiances, during curing of the composite could delay the acquisition of elastic modulus. The rationale is that polymer growth centers would be formed at a slower rate, allowing the polymeric chains opportunity to re-arrange by viscous flow, and macroscopically accommodate the reduction in volume.[6] In that way, the stress development is expected to be lower. Indeed, some studies[7-8] have shown that the use low intensity curing routines, either in continuous or in stepped modes, lead to significant reductions in microleakage and gap formation in composite restorations.

Another aspect is the influence different light sources may have on the development of physical properties. Compared to the more traditional QTH (Quartz tungsten halogen) light, blue light emitting diode (LED) has the advantage of presenting narrower spectral range, which more appropriately targets the absorption wavelength of camphoroquinone, with a peak value of 470 nm.[9-11] It was claimed that this could increase conversion efficiency and reduce the required exposure time.[12-13] In addition, LED units have no need for filters that are required with halogen units for wavelength selection. From this point of view, LED units represent an improvement over halogen lamps. However, there is no evidence that, for a given radiant exposure, LED units provide better results compared to QTH units.[14-15]

Therefore, the purpose of this study was to evaluate the effect of different irradiance levels and light sources (QTH and LED) on the maximum stress generated, stress rate, bond strength to dentin, and on the degree of conversion of a resin composite. The tested hypothesis was that curing methods using reduced irradiance could provide a significant reduction in stress rate, thus reducing the contraction stress, leading to improved bond strength, and without compromising the degree of conversion of the composite.

MATERIALS AND METHODS

Light-curing was performed with two light-curing units: VIP (Variable Intensity Polymerizer, Bisco, Schaumburg, IL, USA), a halogen light unit, that provides different levels of irradiance, and Free-Light II (3M-ESPE, St. Paul, MN, USA), a LED light source of high irradiance. In order to accomplish the irradiances used in the protocols evaluated (Table 1) in all of the following three tests, neutral density filters were placed between the light source and the guide for both light-curing units. For VIP, a Turbo tip was also used, except on the bond strength test. Filtek Z250 (shade A2, 3M-ESPE, St. Paul, MN, USA) was used for all experiments.

Polymerization Contraction Stress and Stress Rate

The polymerization contraction stress test was performed with a closed loop servohydraulic testing instrument (MTS 858, MTS Systems, Eden Prairie, MN, USA). Two borosilicate glass rods 5 mm in diameter were sandblasted (250 µm Al_2O_3) and treated with a silane (Silane ceramic primer, 3M-ESPE, St. Paul, MN, USA) and light-cured adhesive resin (Scotchbond MP, 3M-ESPE, St. Paul, MN, USA). One of the glass rods (upper rod; 12 mm in height) was wrapped with eight layers of adhesive cellophane tape and attached with four set screws to a metallic fixture connected to the actuator, with a slot through which the light curing guide was kept in contact with the opposing side of the rod. The other rod (lower rod, 10 mm in height) was bonded with dental composite to a steel fixture attached to the load cell. Contraction stress was measured by placing a 0.84 mm layer of composite between the two rods, cured through the upper rod. The ratio of bonded-to-unbonded surface area was 3.0 in this configuration. A near zero compliance system was set up by using an eddy current feedback system (Kaman Instruments, Colorado Springs, CO, USA) that kept the distance between the rods constant during the experiment. Load was recorded for 10 min, time period chosen based on the results of a previous study. Maximum force was divided by cross-sectional area to calculate axial stress. Five samples of each curing method were tested. Stress rates were calculated as the change in stress vs. time during each second of the measurement period.

Prior to testing, the light intensity at the top of the specimen (i.e. bottom of the upper glass rod) was measured using a power meter (Power Maximum 5200, Molectron, Portland, OR, USA). For the VIP unit, when the irradiance at the end of the Turbo light guide was 780 and 220 mW/cm^2, the irradiance at the surface of the composite was approximately 550 and 150 mW/cm^2, respectively, demonstrating a reduction of 30% in the light intensity when the light passed through the glass rod. Considering the same reduction for the Free-Light unit, irradiances of 1200, 780, and 290 mW/cm^2 at the end of the light guide corresponded approximately to 850, 550, and 200 mW/cm^2 at the top surface of the composite.

Degree of conversion

The degree of conversion of the resin composite in the five curing methods tested was determined using a micro-Fourier Transform Infrared Spectroscopy (FTIR) analyser (DS20/XAD, Analect Instruments, Irvine, CA, USA). Glass rings of 4 mm-diameter and 2mm-height and glass slides were air-abraded sandblasted (50 µm alumina) and silanated (Silane ceramic primer). The rings were bonded to the slide using a thin coat of adhesive (Scotchbond MP), resulting in a glass cavity with a bonded-to-unbonded surface area ratio of 3.0. The glass cavity was bulk filled with the restorative composite, and then a mylar strip and glass slide were pressed over the glass cavity to force the composite to adapt to the cavity walls and to extrude the excess material. The composite was then light-activated through the glass slide at the bottom of the cavity. For the VIP unit, when the irradiance at the end of the Turbo light guide was 610 and 170 mW/cm^2, the irradiance at the bottom surface of the composite was approximately 550 and 150 mW/cm^2, respectively, based on a measured reduction of 10% in the light intensity when the light passed through the glass slide. Considering the same reduction to the Free-Light unit, irradiances of 950, 610, and 220 mW/cm^2 at the end of the light guide corresponded approximately to 850, 550, and 200 mW/cm^2 at the surface of the composite. Three samples for each experimental condition were prepared and stored dry for 24h at room temperature. Chips of composite approximately 50x100 µm in size were removed with a scalpel from the specimen’s top surface (opposite surface from the light exposure) under safe yellow light and subsequently analyzed in transmission at 8 cm^{-1} resolution in the FTIR microscope. Three spectra were analyzed per specimen. The ratio between the intensities of aliphatic C=C (at 1637.3 cm^{-1}) and aromatic C=C (at 1608.3 cm^{-1}) peaks for uncured and cured samples was used to calculate the degree of conversion, according to the following equation:

$$DC = \left[1 - \left(\frac{[\text{Abs (C=C aliph)/Abs (C} \cdots \text{ C arom)}] \text{ cured resin}}{[\text{Abs (C=C aliph)/Abs (C} \cdots \text{ C arom)}] \text{ uncured resin}} \right) \right] \times 100$$

where *DC* is the degree of conversion, *Abs (C=C) arom* is the height of the benzene ring peak and *Abs (C=C) aliph* is the height of the aliphatic C=C bonds peak, for both cured and uncured composites.

Bond Strength

Fifty fresh bovine incisors free from cracks or any other kind of structural defect were selected under x20 magnification. The teeth were disinfected in 0.5% chloramine for 15 days and stored for less than 1 month in 0.9% saline solution. The crowns were cut off at the cement-enamel junction (Figure 1A) using a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil). All buccal surfaces were ground and flattened under water cooling with a 180 grit SiC paper, to standardize the thickness of the enamel around 0.5 mm. A diamond bur (ref. 3017HL, Fava, São Paulo, SP, Brazil) was used to partially grind the lingual face of the crown, resulting in 2.5 mm tooth substrate in height (Figure 1C). The mesial and distal crown segments of the lingual surface were preserved to reinforce the specimens (Figure 1C).

Conical preparations were prepared in the buccal surface of each tooth with a diamond tipped bur (ref. 3131, KG Sorensen) mounted in a high-speed water-cooled hand piece (Kavo SA, Joenville, SC, Brazil) in a standard cavity preparation appliance (Figure 1B). The diamond tipped burs were replaced every five preparations. The resulting cavity was conical, 2.5 mm in height, with a 2.1 mm top diameter and 1.5 mm bottom diameter (Figure 1D), resulting in a C-factor of 3.0.

Restorative procedures

Preparations were etched using 35% phosphoric acid (Scotchbond Etchant, 3M-ESPE, St. Paul, MN, USA). Single bond adhesive system (3M-ESPE, St. Paul, MN, USA) was applied according to manufacturer's instructions and cured for 10 seconds, at 700 mW/cm^2 (XL 2500 - 3M-ESPE, St Paul, MN, USA). The composite was placed in bulk. A mylar strip and a microscope slide were placed over the cavity, and used to force the composite to adapt to the preparation walls and to extrude the excess material. The slide was then removed, and the light curing tip was positioned against the mylar, followed by photoactivation, according to the protocols described in Table 1. Ten samples of each curing method were tested. There was no need for a turbo light guide with the VIP in this experiment.

The samples were stored in distilled water at 37^{o}C for 24 hours. Restorations were polished with Sof-Lex disks (3M-ESPE, St Paul, MN, USA) on the buccal and lingual surface. The bond strength test was conducted using a push-out test. The sample was positioned on top of a metallic holder with an aperture that allowed the smaller diameter of the restoration to be in contact with an aspheric device connected to the load cell of a universal testing machine (Instron, model 4411, Buckinghamshire, England). A compressive force was applied through the aspheric device to the smaller diameter surface of the restoration at a cross head speed of 0.5 mm/min until rupture of the tooth-composite bond (Figure 1E). Stress in MPa was obtained by dividing maximum load by the bonded surface area of each specimen.

After the test, the fractured specimens were examined with a stereomicroscope at 40x (Carl Zeiss, Manaus, AM, Brazil) and classified as to the mode of failure: cohesive failure in composite, cohesive failure in dentin, adhesive failure, or mixed (adhesive and cohesive in composite or dentin).

Statistical analysis

Maximum contraction stress, degree of conversion, and bond strength values were analyzed by one-way ANOVA and Tukey's test at a significance level of 5%. The correlation between contraction stress rate and bond strength (Figure 5A) and initial irradiance and contraction stress rate (Figure 5B) were calculated as well.

RESULTS

Effect on Polymerization Contraction Stress and Stress rate

Contraction stress results are listed in Table 2. All curing protocols resulted in statistically equivalent total contraction stress ($p>0.05$). As shown in Figure 2, a continuous increase in the stress values was observed for all methods even after the light was turned off. The maximum stress rate is listed in Table 2 and the stress rate in the first 20 seconds of light exposure is shown in Figure 3. LED HI presented the highest stress rate (0.46 MPa/s), reached at 2.3s. QTH CL and LED MI presented equivalent values of maximum stress rate (0.32 MPa/s), reached at 3.3s for both methods. LED LI and QTH PD methods presented the lowest maximum stress rates: 0.21 MPa/s for LED LI at 4.3s and 0.15 MPa/s for QTH PD at 3.3s. In addition, for the QTH PD during the second cycle of light exposure, the same maximum stress rate mean values was observed (0.15 MP/s) and was reached at 5.3 seconds.

Contraction stress rate and bond strength presented an inverse linear correlation, with $r^2 = 0.79$ (Figure 5A), i.e., bond strength decreased with increasing maximum contraction stress rate. The interaction between irradiance and contraction stress rate showed a reasonable fit with a logarithmic function, with $r^2 = 0.93$ (Figure 5B). The contraction stress rate demonstrated an increase with increasing irradiance values, but both tended to level off at the higher irradiance levels.

Effect on Degree of Conversion

The degree of conversion of the composite ranged from 52.5 ± 1.2% (LED HI) to 55.1 ± 0.5% (QTH CL) at 24 hours. Mean values are listed in Table 2. No statistical difference was observed among the curing methods ($p>0.05$).

Effect on Bond Strength

The QTH PD curing method presented the highest mean bond strength, 35.5 ± 3.4 MPa (Table 2), statistically higher than the others. LED MI and LED LI showed similar mean bond strengths, 29.2 ± 2.6 and 29.3 ± 2.6 MPa, respectively, both being statistically higher than that of LED HI (24.6 ± 2.0 MPa). No statistical difference was observed for QTH CL (26.4 ± 2.4 MPa) when compared to LED LI, LED MI and LED HI. Adhesive failure was the most frequent failure mode for LED HI, QTH CL, and LED MI (80%, 80%, and 70%, respectively). LED LI showed equal percentages of adhesive failure (50%) and mixed failure (50%). Mixed failure was the most frequent failure mode for the QTH PD method (60%) and this method was the only one that presented any cohesive failure in the composite (10%).

DISCUSSION

In this study, no statistical difference was observed for the curing methods in relation to the maximum contraction stress values. However, from the correlation between stress rate and irradiance ($r^2 = 0.93$), it was shown that stress rate development seems to be directly proportional to the increase in irradiance. Indeed, the groups photoactivated with LED at high irradiance (850 mW/cm^2) showed the highest stress rate (0.46 MPa/sec), followed by QTH CL (0.32 MPa/sec) and LED MI (0.32 MPa/sec). The lowest stress rates were observed for QTH PD (0.15 MPa/s) and LED LI (0.21 MPa/s), respectively 67 and 54% lower than the LED HI. Thus, it appears that although the net stress might be similar for different curing methods, the path to achieve the maximum stress differs.[5] It is possible that, for QTH PD and LED LI, the low irradiance led to a decreased polymerization rate, modifying the generation and distribution of stress, as also verified by Hofmann and others.[16] However, though the curing rate can be significantly reduced, it does not mean that the decrease in contraction stress will also be significant. Findings of previous studies [17-19] suggest that polymerization rate must be reduced below a certain threshold in order to significantly reduce contraction stress, perhaps because the gelation of methacrylates takes place at very low degrees of conversion [20]. Once the reaction begins, an auto-acceleration phenomena is observed and stresses are expected to increase dramatically because of the consequent increase in stiffness produced by crosslinking.[21] Therefore, the time available for the material to flow is very restricted, and a substantial decrease in curing rate is required to significantly affect contraction stress development. Thus, even a reduction of 67% in the stress rate observed for QTH PD when compared to the highest stress rate of this study (LED HI) was not enough to lead to a significant reduction of the maximum stress value for the QTH PD method. However, it is also important to note that materials with distinct compositions may show different behaviors when submitted to the same curing methods tested. A study evaluating the effect of low curing rates on contraction stress development for three other commercial materials found stress reductions between 19 and 30%.[22]

One possible explanation for the similar final contraction stress values found for all experimental groups is that the final radiant exposure employed for all of them was similar (16 J/cm^2). According to previous findings [23], which are supported by the results of this study, this would lead to the same degree of conversion. Silikas and others [24] and Vandewalle and others [25] found a high correlation (r^2>0.99) between degree of conversion and stress generation. Therefore, if the reduction in the stress rate caused by the modulated curing methods was not sufficient to significantly decrease the final stress of the composite used, the degree of conversion becomes the most important factor affecting the development of final contraction stress in dental composites.[26] Thus, the statistical equivalence in the degree of conversion for the five curing methods may explain the results found in this study for the maximum stress mean values.

Stress rate is dependent on irradiance and not on radiant exposure.[5] The stress rate values reported here were estimated in real time, as opposed to final stress/total time, as reported by others [27], making it possible to determine the time in the curing cycle at which the maximum stress rate occurred. It could be observed that maximum stress rate developed early in the curing cycle and after the stress rate peak was reached, it declined regardless of the irradiance and duration of exposure. Interestingly, the method that presented the highest stress rate (LED HI) also showed the most premature maximum stress rate development, in this case, at 2.3s. QTH CL, LED MI, and QTH PD reached maximum stress rate at 3.3s. The LED LI method reached maximum stress rate (0.21 MPa/s) at 4.3s.

A high correlation was found between stress rate and bond strength (r^2=0.79). The QTH PD method produced both the lowest maximum stress rate (0.15 MPa/s) and the highest bond strength (35.5±3.4 MPa), statistically superior to all the other methods. The analysis of failure mode also showed that the QTH PD curing method predominantly presented mixed failure (60%), symbolizing a higher probability of bond preservation, in contrast to LED HI, QTH CL, and LED MI, in which most of the samples failed adhesively. The same trend could be observed for LED using low and intermediate irradiance (bond strengths of 29.3±2.6 and 29.2±2.6 MPa, respectively), both of which were statistically superior to LED using high irradiance (24.6±2.4 MPa). Therefore, it can be speculated that although no statistical difference was observed in final contraction stress for all methods, the stress rate for QTH PD and LED LI was significantly reduced, and this was reflected in the increase of the mean bond strength values reached with these methods. Comparatively, the methods LED LI and QTH PD used similar low irradiances (200 and 150 mW/cm^2, respectively), but the first in continuous mode and the latter in a 5 sec exposure, followed by a 3 minute interval and a second exposure at 550 mW/cm^2. If we take into consideration the stress rate values only, it can be said that QTH PD was more efficient in reducing rate, perhaps because the first exposure used not only low irradiance but was performed for a short time (i.e. low radiant exposure). Since conversion was expected to continue in the dark period between pulses, when the second, higher irradiance exposure was applied, there were less carbon double bonds left to be used in the growing chains. Therefore, QTH PD method resulted in lower stress rate during the second cycle of light exposure (0.15 MPa/s), though it used an equivalent irradiance to that of the QTH CL or LED MI methods. Although it did not seem to have an influence on contraction stress or degree of conversion, this protocol was correlated with better bond strength results.

Another interesting comparison that can be made from the results of the present study is between QTH CL and LED methods themselves. Some manufacturers claim that LED units can reach higher degree of conversion compared to halogen units using shorter exposure times, which would be clinically advantageous.[12-13] The rationale is the narrow light spectrum of LED units, with wavelengths between 438 and 501 nm, and peak intensity at 465 nm [9-11], which is coincident with the absorption peak of the camphorquinone, between 465 and 470 nm [28], theoretically making it more efficient in activating the camphorquinone. In the present study, comparing the curing methods LED MI and QTH CL, both using the same irradiance of 550 mW/cm^2 and the same radiant exposure (16 J/cm^2), no statistical difference was observed in the degree of conversion, maximum contraction stress, maximum stress rate or bond strength for the groups. At least for the degree of conversion, this fact corroborates the results of other studies [14-15], in which no difference was observed in the development of physical properties when LED and QTH units were compared.

In conclusion, the tested hypothesis that curing methods using reduced irradiance or modulation of the light exposure would provide a significant reduction on contraction stress, leading to improved bond strength was partially validated by the results. The reduction in the stress rate observed for QTH PD group significantly increased the bond strength when compared to the other curing methods tested. LED LI and LED MI were also effective in increasing the bond strength when compared to a curing method using continuous output in high light intensity (LED HI). Such advantage of QTH PD, LED LI, and LED MI was reached with no adverse effect on the degree of conversion of the restorative composite. It remains to be seen if these advantages could be associated with significantly higher clinical longevity and better performance of composite restorations.

Acknowledgements

This study is part of a thesis, for partial fulfillment of the PhD degree in Dental Materials. Supported by CAPES (PDEE grants BEX3428/04-4).

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Table 1. Light-curing methods evaluated with their outputs and the respective energy density

Method	Irradiation protocol	Light Source	Radiant exposure
High Intensity LED (LED HI)	19s at 850 mW/cm^2	LED	16 J/cm^2
Continuous light (QTH CL)	30s at 550 mW/cm^2	Halogen	16 J/cm^2
Medium Intensity LED (LED MI)	30s at 550 mW/cm^2	LED	16 J/cm^2
Low Intensity LED (LED LI)	80s at 200 mW/cm^2	LED	16 J/cm^2
Pulse Delay (QTH PD)	5s at 150 mW/cm^2 + 3 min + 28s at 550 mW/cm^2	Halogen	16 J/cm^2

Table 2. Contraction stress, maximum stress rate, degree of conversion, and bond strength generated by the light-curing methods.

	Contraction Stress (MPa)	Stress rate (MPa/s)	DC (%)	Bond Strength (MPa)
LED HI	10.5 (1.2) a	0.46	52.5 (1.2) a	24.6 (2.0) c
QTH CL	10.3 (1.1) a	0.32	55.1 (0.5) a	26.4 (2.4) bc
LED MI	10.4 (1.2) a	0.32	53.1 (1.8) a	29.2 (2.6) b
LED LI	10.3 (0.6) a	0.21	54.6 (1.9) a	29.3 (2.6) b
QTH PD	9.3 (1.3) a	0.15/0.15 [1]	54.2 (2.6) a	35.5 (3.4) a

[1] First value is maximum stress rate during primary step of pulse-delay cure while second value corresponds to maximum stress rate during cure after the delay.

Mean values followed by the same lower case letter in the same column are statistically equivalent (with $p<0.05$).

( ) – Standard Deviation

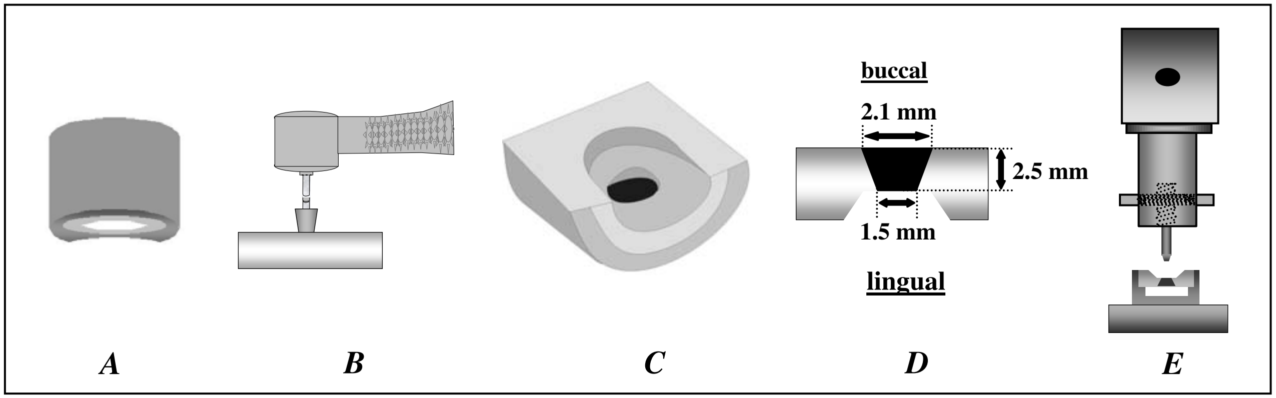

Figure 1. Schematic representation of the "push-out" test: ***A.*** Tooth fragment after flattening of the buccal surface and selective grinding of the lingual surface; ***B.*** Cavity preparation in the standard cavity preparation appliance; ***C.*** Lingual view of the tooth fragment with the cavity completed; ***D.*** Lateral view of the restored sample (2.5 mm in height, top diameter of 2.1 mm, and bottom diameter 1.5 mm); ***E.*** Lateral view of the complete system with inverted specimen for the accomplishment of the push-out test

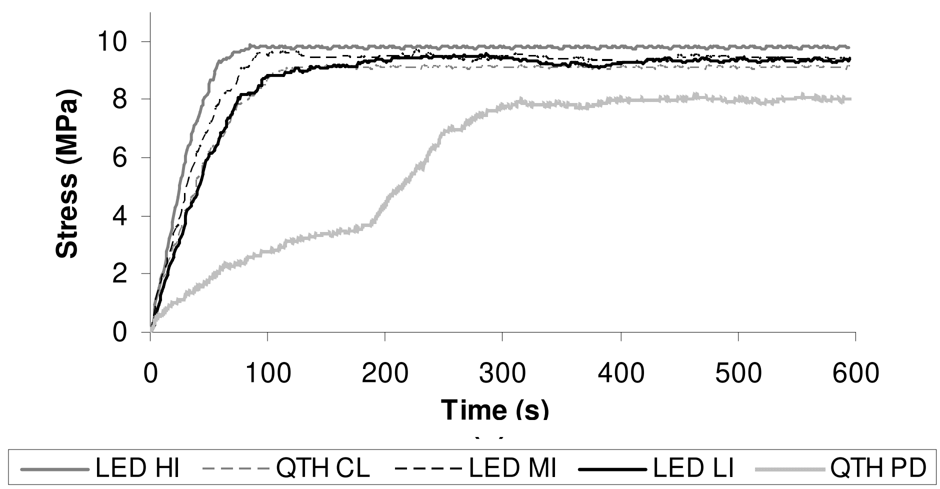

Figure 2. Stress values (MPa) for each light-curing as a function of time

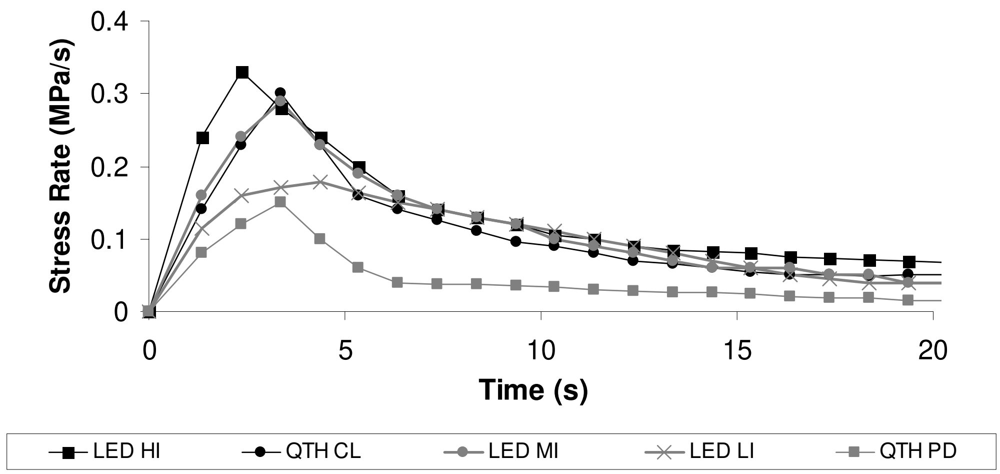

Figure 3. Representative curves of stress rate (MPa/s) during the first 20 seconds of curing for each light-curing method as a function of time

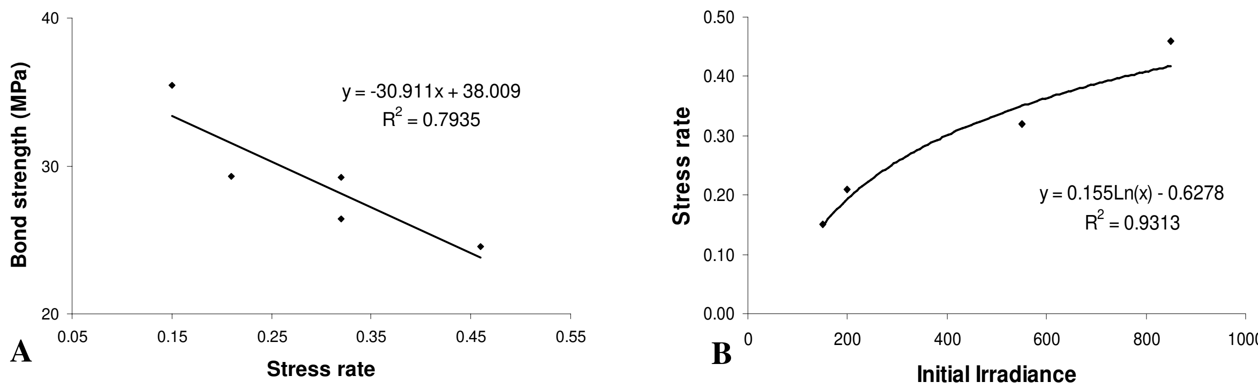

Figure 4. Correlation curves of stress rate vs. bond strength (**A**) and irradiance vs. stress rate (**B**)

CAPÍTULO 5

CONTRACTION STRESS AND PHYSICAL PROPERTIES DEVELOPMENT OF A RESIN-BASED COMPOSITE IRRADIATED USING MODULATED CURING METHODS AT TWO C-FACTOR LEVELS

Manuscrito submetido para publicação no periódico

Dental Materials

Summary

Objectives: Verify the influence of curing methods on contraction stress, stress rate, and degree of conversion (DC) of a restorative composite at two C-factor levels.
Methods: For the stress test, composite was applied between two 5-mm diameter glass rods, mounted in a servohydraulic machine. Stress rates were calculated as the change in stress vs. time at each second. DC was measured by micro-FTIR. Four curing methods were tested at two C-factor levels (1.5 and 3.0): Continuous Light (CL), Soft-Start (SS), and two Pulse Delay methods using different initial irradiances - 150 (PD150) and 80 mW/cm^2 (PD80). Results were analyzed by ANOVA and Tukey's test ($\alpha = 0.05$).
Results: For the stress test, at CF 1.5, PD80 presented the lowest mean value, statistically different from the others. PD150 also showed a mean value statistically inferior to CL. At CF 3.0, no statistical difference was observed among CL, SS, and PD150. PD80 presented statistically inferior stress values compared to CL and SS. Stress values at CF 3.0 were statistically higher than the ones at CF 1.5, for all curing methods. CL presented the highest maximum stress rate, followed by SS, PD150 and PD80, in both C-factors. In the DC test, no difference was observed among the methods and between the C-factor levels.
Significance: Modulated curing methods were shown to be effective in reducing contraction stress rate, without compromising the DC of the restorative composite. C-factor was shown to influence negatively the stress rate and the amount of stress generated.

Keywords: composite resin, curing method, contraction stress, degree of conversion

Introduction

Although extremely popular for its aesthetic appeal, an inherent disadvantage of light-activated resin composites is the shrinkage consequent to polymerization [1-3]. When this shrinkage takes place under confinement, due to bonding to cavity walls, stresses on the bond interface may develop [4], leading to failure at the restoration-tooth boundary. The final outcome is microleakage of oral and dentinal fluids as well as accumulation of microorganisms and debris that are largely responsible for the major clinical problems with dental composite: sensitivity, staining, and secondary caries [5].

The magnitude of such stresses can be related to the restorative technique, as well as to material composition and degree of conversion [6]. One of the factors associated with the restorative procedure that have been shown to be directly related to stress is the restoration's bonded-to-unbonded ratio (cavity configuration factor, or C-factor) [4,6]. The higher the C-factor, the less free surfaces there will be, leading to a situation where there is little opportunity for the composite to flow and accommodate changes in volume [6]. Since it is hardly ever possible to change the cavity configuration, some techniques have been suggested to minimize the potential stress generation. For example, a photoactivation with an initial low irradiance followed by an exposure with higher intensity has been proposed, in methods known as "soft-start" [7]. The rationale is that the polymerization reaction would occur in a slower rate, delaying the gelation (or elastic modulus development), giving the material opportunity to accommodate by viscous flow and ultimately reduce stress [8]. One variation of this technique is to introduce an interval between the two pulses (pulse-delay technique) [9], aiming to allow the polymerization to continue in the dark at slower rates and complete the irradiation with higher irradiance to ensure good degree of conversion and mechanical properties.

Although there has been evidence that such methods provide better marginal integrity [10-12], reduced incidence of cavosurface marginal gap and enamel fracture [9,13], besides reducing residual stress in the composite [14], studies on pulse delay method do not present a standard protocol for the use of this method. Different authors suggest a range of irradiances for the first pulse, from 60 to 425 mW/cm^2 [13-17] and still there is no consensus on which initial low-irradiance step should be the ideal one.

Thus, the aim of the present study was to evaluate the effect of four light-curing methods on the degree of conversion, contraction stress and stress rate developed by a resin-based composite in two C-factor levels. The tested hypothesis was that modulated curing methods provide a significant reduction on contraction stress, without compromising the degree of conversion of the restorative composite. A higher C-factor level was hypothesized to generate an increase in the stress level and stress rate, with no influence on the degree of conversion values.

Materials and Methods

The VIP light-curing unit (Variable Intensity Polymerizer, Bisco, Schaumburg, IL, USA), which has the capability to provide different levels of irradiance, was used in this study. Filtek Z250 restorative composite (shade A2, 3M-ESPE, St. Paul, MN, USA, batch number 5JH) was used for all experiments. The curing methods evaluated in the following tests are described in Table 1.

Polymerization Contraction Stress and Stress Rate

Polymerization Contraction Stress Test was performed with a closed loop servohydraulic testing instrument (MTS 858, MTS Systems, Eden Prairie, MN, USA). Two borosilicate glass rods 5 mm in diameter were sandblasted (250 µm Al_2O_3) and treated with a silane (Silane ceramic primer, 3M-ESPE, USA) and light-cured adhesive resin (Scotchbond MP, 3M-ESPE, USA, batch number 0MA). One of the glass rods (12 mm in height) was attached to a metallic fixture connected to the actuator, which had a slot through which the light curing guide was kept in contact with the opposing side of the rod. The other rod was 10 mm in height and was bonded to a fixture attached to the load cell. Contraction stress was measured by placing two different thickness of composite between the two rods: 1.66 mm (ratio of bonded-to-unbonded surface area - 1.5) and 0.84 mm (ratio of bonded-to-unbonded surface area - 3.0). A near zero compliance system was set up by using an eddy current feedback system (Kaman Instruments, Colorado Springs, CO, USA) that kept the distance between the rods constant. A light-curing guide was directed down through the upper rod and the contraction forces recorded during 15 min from the initiation of light activation. Maximum force was divided by cross-sectional area to calculate average axial stress. Five samples of each curing method were tested. Stress rates were calculated as the change in stress vs. time at each second during the measurement period.

Prior to testing, the light intensity at the top of the specimen was measured using the power meter (Power Maximum 5200, Molectron, Portland, OR, USA). A turbo light guide and neutral density filters were used to control the irradiance reaching the specimen in this experiment. When the irradiance at the end of the Turbo light guide was 780, 220, and 120 mW/cm^2, the irradiance at the surface of the composite was approximately 550, 150, and 80 mW/cm^2, respectively, considering a reduction of 30% in the light intensity when the light passed through the glass rod.

Degree of conversion Test

The degree of conversion of the resin composite in the four curing methods tested was determined using a micro-Fourier Transform Infrared Spectroscopy (FTIR) analyser (DS20/XAD, Analect Instruments, Irvine, CA, USA). Glass rings of two different thickness (0.5 and 2.0 mm) and 4 mm diameter and glass slides were sandblasted and silanated (Silane ceramic primer, batch number 5WJ). The rings were bonded to the slide using a thin coat of adhesive (Scotchbond MP, batch number 2MT), resulting in two different glass cavities of bonded-to-unbonded area ratio of 1.5 (4 mm diameter and 0.5 mm depth) and 3.0 (4 mm diameter and 2 mm depth). The glass cavity was bulk filled with the restorative composite. A mylar strip and glass slide were pressed over the glass cavity to force the composite to adapt to the cavity walls and to extrude the excess material. The composite was then light-activated through the glass slide at the bottom of the cavity. A turbo light guide and neutral density filters were used to control the curing parameters. When the irradiance at the end of the Turbo light guide was 610, 170, and 90 mW/cm^2, the irradiance at the bottom surface of the composite was 550, 150, and 80 mW/cm^2, respectively, considering a reduction of 10% in the light intensity when the light passed through the glass slide. Three samples for each experimental condition were prepared and stored dry for 24h at room temperature. Chips of composite approximately 50x100 µm in size were removed with a scalpel from the specimen's top surface (opposite surface from the light exposure) under safe yellow light and subsequently analyzed in transmission at 8 cm^{-1} resolution. Three spectra were analyzed per specimen. The ratio between the intensities of aliphatic C=C (at 1637.3 cm^{-1}) and aromatic C=C (at 1608.3 cm^{-1}) peaks for uncured and cured samples was used to calculate the degree of conversion, according to the following equation:

$$DC = \left[1 - \left(\frac{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{C arom)] cured resin}}{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{C arom)] uncured resin}} \right) \right] \times 100$$

where *DC* is the degree of conversion, *Abs (C=C) arom* is the height of the benzene ring peak and *Abs (C=C) aliph* is the height of the aliphatic C=C bonds peak, for both cured and uncured composites.

Statistical analysis

Maximum contraction stress and degree of conversion values were analyzed by one-way ANOVA and Tukey's test at a significance level of 5%. Correlation analysis of irradiance vs. stress rate was also performed for C-factor 1.5 (Figure 4A) and for C-factor 3.0 (Figure 4B).

Results

Polymerization Contraction Stress and Stress rate

As shown in Figure 1, a sharp increase in contraction stress was observed immediately following light activation, for all curing methods and both C-factors. A continuous increase in the stress values was observed when light was turned off, for all methods and both C-factors. The stress rate in the first 20 seconds of light exposure for the curing methods in both C-factors, and the influence of the C-factor on the stress rate is shown in Figure 2, and the maximum Stress rate is listed in Table 2. At C-factor 1.5, CL presented the highest stress rate (0.24 MPa/s), reached at 4.3s, followed by SS, with a maximum stress rate of 0.14 MPa/s, reached at 5.3s. The PD methods presented the lowest maximum stress rates: 0.07 MPa/s for PD150 at 3.3s and 0.01 MPa/s for PD80 at 5.3s. At C-factor 3.0, the same order was observed. CL showed the highest maximum stress rate (0.32 MPa/s at 3.3s), followed by SS (0.21 MPa/s at 3.3s), PD 150 (0.15 MPa/s at 3.3s), and PD80 (0.05 MPa/s at 4.3s). Table 2 lists the mean contraction stress values and standard deviations for all curing methods. At C-factor 1.5, CL presented the highest mean stress value (8.6 MPa), statistically different from PD150 (7.4 MPa) and PD80 (5.3 MPa). PD80 presented also a statistically inferior mean value when compared to PD150 and SS (8.6 MPa). No statistical difference ($p > 0.05$) was observed between SS and PD150. At C-factor 3.0, CL (10.3 MPa) and SS (10.2 MPa) methods presented the highest mean values, statistically different from PD80 (7.9 MPa). The curing method PD150 (9.3 MPa) presented an intermediate value, equivalent to all methods evaluated ($p > 0.05$). For all curing methods, the mean maximum stress values reached at C-factor 3.0 were statistically superior ($p < 0.05$) to the ones reached at C-factor 1.5.

Correlation curve of initial irradiance vs. stress rate at C-factor 1.5 (Figure 4A) and at C-factor 3.0 (Figure 4B) showed reasonable fits with logarithmic function. The stress rate in both cases demonstrated an increase with increasing initial irradiance values, but tended to level off at the higher energy levels.

Degree of conversion

As can be seen in Table 2, no statistical difference ($p > 0.05$) was observed for the curing methods tested in this study, in each of the C-factors evaluated. At C-factor 1.5, the values ranged from 53.6 % (PD150) to 55.5 % (PD80). For C-factor 3.0, the mean values ranged between 54.2 % (PD150) and 55.9 % (PD80). For each curing method, no statistical difference ($p > 0.05$) was observed between 1.5 and 3.0 C-factor levels.

Discussion

The results of this study supported the hypothesis that the stress generated by the contraction of the composite is related to the curing method, the C-factor level, or both. For all curing methods, the maximum stress mean values reached at C-factor 1.5 were statistically inferior to the ones reached at C-factor 3.0. Since composite flow is most likely to occur from the free surfaces of the specimen, a lower C-factor will indicate a higher proportion of free composite surface, reducing the restriction to shrinkage, thereby reducing stress [18-19]. In this study, a mean reduction of 21% in the maximum stress was found at C-factor 1.5, when compared to the values reached at C-factor 3.0. Besides, the influence of the C-factor level was not just related to the amount of stress generated, but it also had a significant influence on the stress rate. As it can be seen in the mean values of stress rate in Table 2 and on Figure 2, the maximum stress rate found at C-factor 1.5, independent of the curing method, was around 22% lower than the one related to C-factor 3.0. For all curing methods, at C-factor 3.0, the maximum stress rate was higher and reached faster, when compared to the values at C-factor 1.5.

The influence of the curing method was also clear. In both C-factor levels, the highest stress mean value was reached by the CL method, 8.6 and 10.3 MPa, respectively for C-factor 1.5 and 3.0. These results can be partially explained by their respectively higher maximum stress rates (Table 2). For this method, the reaction might have evolved too fast, virtually eliminating the opportunity for viscous flow, leading to a dramatic increase in stiffness after a relatively low degree of conversion [4,20]. As a result, stress develops almost immediately after polymerization is triggered [21], and the vast majority of the conversion occurs after the polymer matrix has reached a significant level of rigidity [4]. However, at C-factor 1.5, no statistical difference was observed for maximum stress between CL (8.6 MPa) and SS (8.6 MPa), and the same was observed among CL (10.3 MPa), SS (10.2 MPa), and PD150 (9.3 MPa) at C-factor 3.0, instead of differences in the maximum stress rate values. The correlation analysis between irradiance and stress rate at C-factor 1.5 ($r^2 = 0.84$) and at C-factor 3.0 ($r^2 = 0.93$) showed a directly proportional relation between these factors. Indeed, the method using the highest initial irradiance (CL) was associated with the highest stress rate, in both C-factors, when compared to lower initial irradiance curing methods, such as PD150. At C-factor 1.5, the maximum stress rate reached by SS (0.16 MPa/s) was almost 34 % lower than the one presented by CL (0.24 MPa/s). In addition, the maximum stress rate reached by SS (0.21 MPa/s) and PD150 (0.15 MPa/s) at C-factor 3.0, was respectively 35 and 53 % lower than the one presented by CL (0.32 MPa/s). It is possible that, for SS and PD150, the initial low irradiance had led to a decreased initial polymerization, reflected as a reduction in the stress rate, modifying the generation and distribution of stress, as also verified by Hofmann et al. [22]. Thus, it appears that although the net stress might be similar for different curing methods, the path to get to the maximum stress differs [23]. However, though the curing rate can be significantly reduced, it does not mean that the decrease in contraction stress will also be significant. Findings of previous studies suggest that polymerization rate must be reduced under a certain threshold in order to significantly reduce contraction stress [24-26]. According to some authors, the gelation of metacrylates happens at very low degrees of conversion [20]. Therefore, the period allowed for the material to flow is very restricted, and a substantial decrease in curing rate is needed to significantly affect contraction stress development. Thus, even a reduction of 53% in the stress rate observed for PD150 when compared to the maximum stress rate of CL curing method was not enough to promote a significant reduction of the maximum stress mean value to the PD150 method. However, it is also important to notice that composites from different manufactures may show different behaviors when submitted to the same curing methods tested. A study evaluating the effect of low curing rates on contraction stress development of three commercial materials found stress reductions between 19 and 30% [17].

PD80 led, at both C-factor levels, to the lowest contraction stress mean values and to the lowest maximum stress rates. This method was related to statistically inferior mean values of contraction stress when compared to CL and SS at both C-factor levels, and also to PD150 at C-factor 1.5. However, when comparing the mean values of maximum stress rate, differences were observed for PD methods after the second cycle of light exposure. At C-factor 3.0, for PD150, the maximum stress rate reached after the 3-minute interval (0.15 MPa/s) was the same value reached with the first irradiation. The initial pulse limited the contraction force when the second, higher irradiance exposure was applied. Therefore, PD150 method resulted in lower stress rate during the second cycle of light exposure, even using the same irradiance of the CL method. However, the same was not observed for PD80, in which after the second cycle of light exposure a maximum stress rate of 0.21 MPa/s was observed, an increase of 320% in relation to the first irradiation. Also, the stress rate for the second pulse in this case was almost 30% higher than the one produced by PD150 in the same cycle. At C-factor 1.5, the same relationship was found. The stress rate for the second pulse of PD80 in this case was 34% higher than the one produced by PD150 in the same cycle. This could be explained by the fact that, at this very low irradiance, there is insufficient photon-density to initiate a significant part of the reaction [23,27]. Therefore, it can be speculated that a significant amount of the conversion took place only after the second irradiation. This was expected to have led to a higher rate in the second cycle, since more remaining double bounds would still be present in this case [23, 27-28].

Stress rate is dependent on irradiance and not dependent on light energy density [23]. The stress rate calculated here is instantaneous stress rate, as opposed to final stress/total time, as reported by others [29]. Therefore, it was possible to determine that maximum stress rate occurred early in the curing cycle. Moreover, after the stress rate peak was reached (the largest being at 5.3s for SS and PD80 at C-factor 1.5, in this study), it declined regardless of the irradiance and duration of exposure.

An interesting finding of this study was that modulated curing methods tend to have higher efficacy at lower levels of C-factor. At a high level of C-factor, the faster polymerization reaction decreased the probability of partial stress release. At C-factor 3.0, the maximum stress rate for SS curing method (0.21 MPa/s) was reached during the first 10 seconds of curing, even using a low irradiance, and no statistical difference was found between PD150 and CL. However, at C-factor 1.5, in which a slow polymerization reaction was observed, the modulated curing methods were more effective. PD150 were responsible for a statistically inferior mean value of stress compared to CL, and a lower stress rate was observed for SS during the first 10 seconds, when compared to the second cycle of light exposure at high irradiance.

The degree of conversion data shows a similar conversion of the composite when the specimens were exposed to equivalent radiant exposure (16 J/cm^2), providing evidence for a reciprocal relationship between irradiance and exposure time [27]. The absence of statistical differences among the methods in both C-factor levels confirm the findings of different studies [23,27], in which statistical equivalence was verified for the combinations of exposure time and irradiance within the same total radiant exposure. Therefore, the advantages of the modulated methods, when compared to CL, such as stress generation statistically inferior for PD80 and also to PD150 at C-factor 1.5 were reached with no reduction of the degree of conversion. In addition, no influence of the C-factor was observed in the degree of conversion, for all curing methods.

In conclusion, the tested hypothesis that modulated curing methods would provide a significant reduction on contraction stress was partially validated by the results. In spite of no statistical difference between CL and SS at C-factor 1.5, and also among both methods and PD150 at C-factor 3.0 as to the maximum stress generation, a significant reduction in the stress rate values was observed for the modulated curing methods when compared to CL, with no expense for the degree of conversion. The hypothesis concerning the C-factor was validated by the results. The higher the C-factor level, the higher the amount of stress generated, and the faster the stress development. C-factor was proven to have no effect on the degree of conversion of the restorative composite.

Acknowledgements

This study is part of a thesis, for partial fulfillment of the PhD degree in Dental Materials. Supported by CAPES (PDEE grants BEX3428/04-4).

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Table 1. Light-curing methods evaluated with their outputs and the respective energy density

Method	Mode of Irradiation	Energy Density
Continuous Light (CL)	30s at 550 mW/cm^2	16 J/cm^2
Soft-Start (SS)	10s at 150 mW/cm^2 + 27s at 550 mW/cm^2	16 J/cm^2
Pulse Delay 150 (PD150)	5s at 150 mW/cm^2 + 3 minutes + 28s at 550 mW/cm^2	16 J/cm^2
Pulse Delay 80 (PD80)	5s at 80 mW/cm^2 + 3 minutes + 29s at 550 mW/cm^2	16 J/cm^2

Table 2. Contraction Stress (MPa), maximum stress rate (MPa/s), and degree of conversion generated by the light-curing methods at each C-factor level

	Contraction Stress		Stress Rate		Degree of Conversion	
	CF 1.5	**CF 3.0**	**CF 1.5**	**CF 3.0**	**CF 1.5**	**CF 3.0**
CL	8.6 (0.5) a, B	10.3 (1.1) a, A	0.24	0.32	54.6 (0.9) a, A	55.1 (0.5) a, A
SS	8.6 (0.6) a, B	10.2 (0.8) a, A	0.14/0.16 *	0.21/0.19 *	55.3 (2.0) a, A	54.8 (1.3) a, A
PD150	7.4 (0.6) b, B	9.3 (1.3) ab, A	0.07/0.12 #	0.15/0.15 #	53.6 (1.3) a, A	54.2 (2.6) a, A
PD80	5.3 (0.6) c, B	7.9 (1.0) b, A	0.01/0.18 #	0.05/0.21 #	55.5 (2.2) a, A	55.9 (0.6) a, A

* First value is maximum stress rate during first ten seconds of light exposure in reduced light intensity while second value corresponds to maximum stress rate during cure after the ten initial seconds

First value is maximum stress rate during primary step of pulse-delay cure while second value corresponds to maximum stress rate during cure after the delay.

Mean values followed by different small letters in the same column differ statistically among themselves for the Tukey test at the level of 5%. ( ) – Standard Deviation

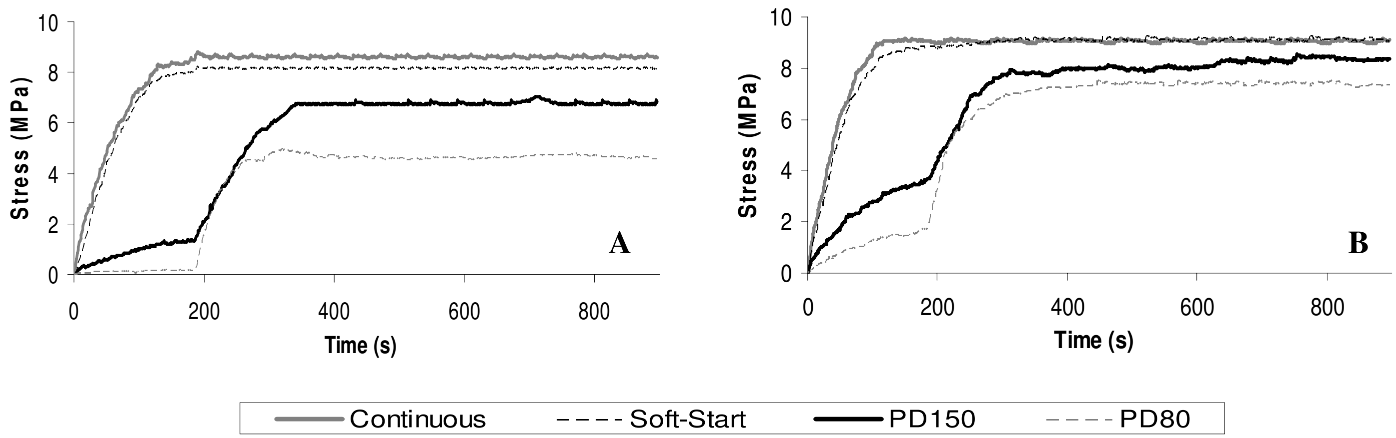
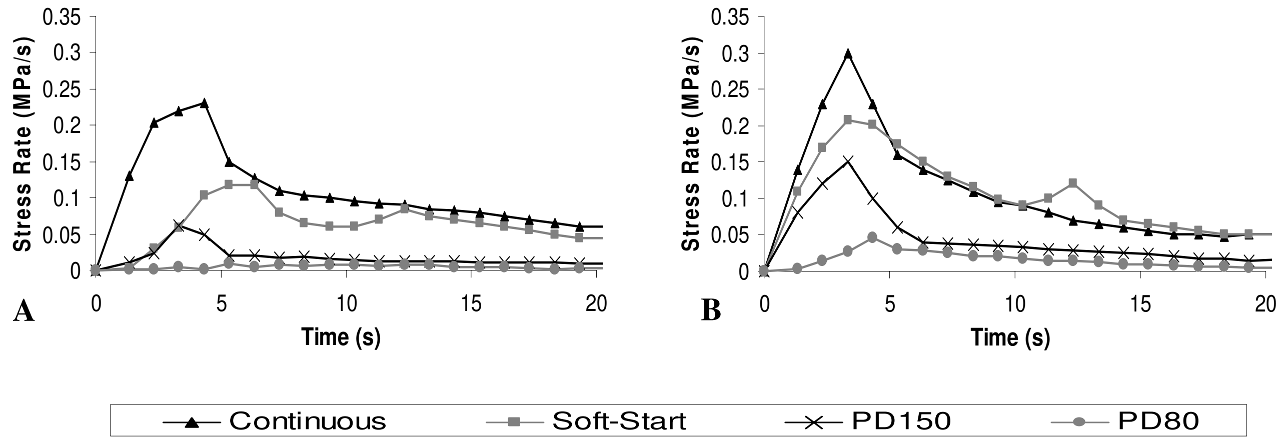


Figure 1. Stress values (MPa) for each light-curing as a function of time at C-factor 1.5 (**A**) and 3.0 (**B**)



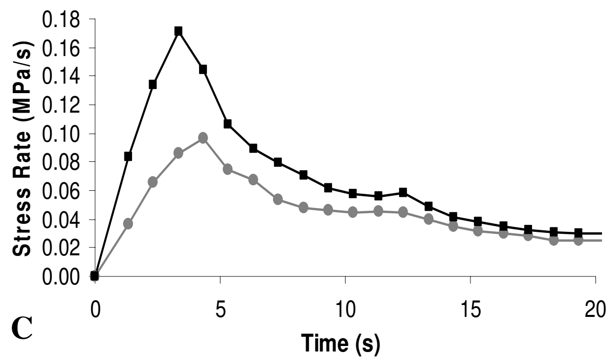


Figure 2. Stress rate (MPa/s) during the first 20 seconds of ccuring for each light-curing method. (**A**) at C-factor 1.5; (**B**) at C-factor 3.0; (**C**) influence of the C-factor, independent of the light-curing method

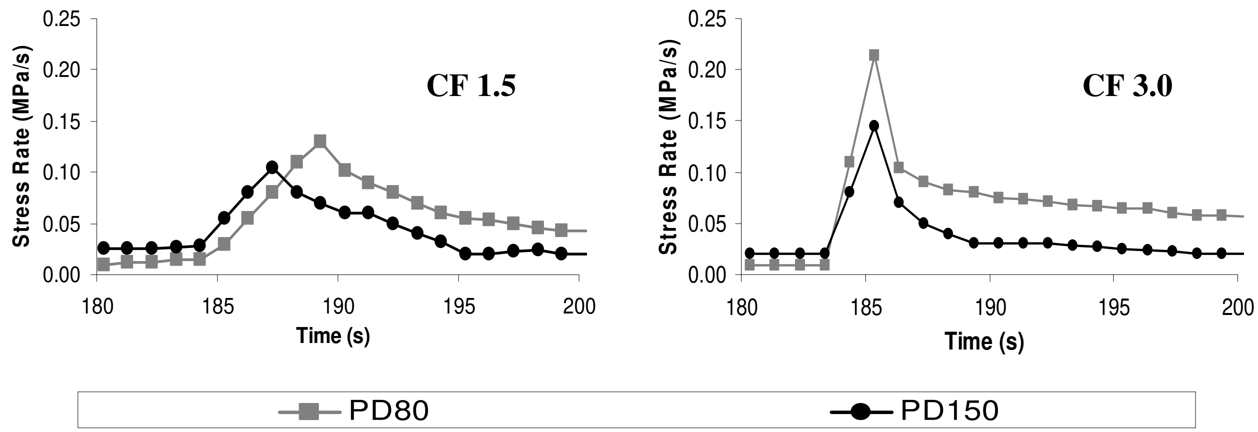


Figure 3. Stress rate (MPa/s) during the second cycle of light exposure to PD80 and PD150 curing methods as a function of time at each C-factor level

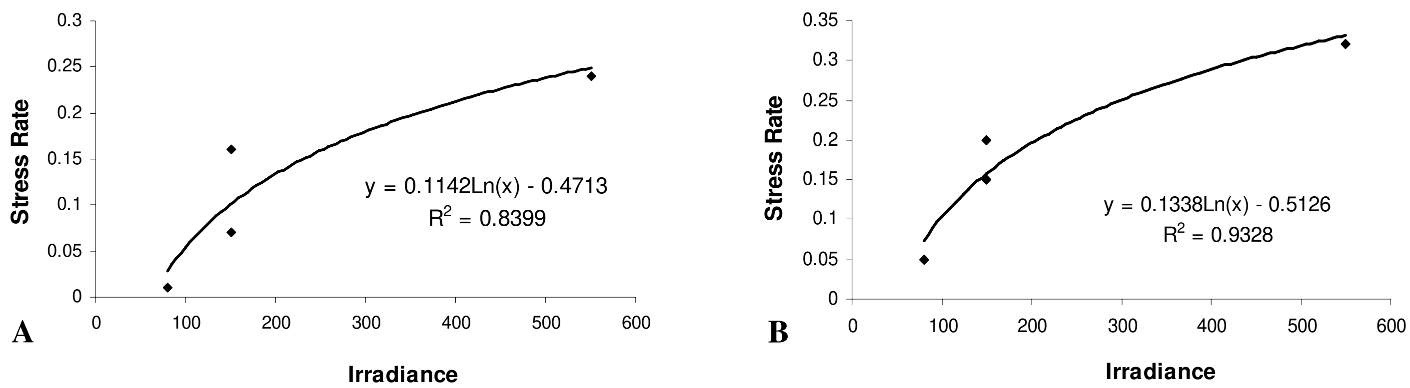


Figure 4. Correlation curves of irradiance vs. stress rate at C-factor 1.5 (**A**) and at C-factor 3.0 (**B**)

CONSIDERAÇÕES GERAIS

Os resultados encontrados nos estudos apresentados comprovaram a hipótese de que, tanto o método de fotoativação, quanto o nível de Fator-C, são fatores significativos na geração de tensão pela contração de polimerização. Para todos os métodos de fotoativação, a tensão máxima alcançada em nível de Fator-C 1,5 foi estatisticamente inferior à observada para o nível de Fator-C 3,0. Tais resultados podem ser associados ao fato de que a capacidade de escoamento do compósito nos momentos iniciais da polimerização está relacionada à área total de superfícies livres presente em determinada configuração cavitária. Assim, em menores níveis de Fator-C onde há maior área de superfície livre, possivelmente ocorrerá maior probabilidade de escoamento, e, portanto, redução da tensão gerada (LAUGHLIN ET AL., 2002; FEILZER ET AL., 1989). Nos estudos realizados, a redução média de 20,5% foi associada aos valores máximos de tensão encontrados para os métodos de fotoativação em nível de Fator-C 1,5, quando comparado com os resultados obtidos em nível de Fator-C 3,0. Além disso, a influência do nível de Fator-C não foi relacionada somente à quantidade de tensão gerada, mas também apresentou significante influência na taxa de geração de tensões. A partir dos resultados obtidos nos estudos, foi possível verificar que, a taxa de geração de tensões, independente do método de fotoativação, foi cerca de 22% menor nos métodos de fotoativação testados em nível de Fator-C 1,5 quando comparados aos resultados em nível de Fator-C 3,0. Assim, para todos os métodos fotoativadores, em nível de Fator-C 3,0, a tensão máxima gerada foi maior e alcançada mais rapidamente, quando comparado aos valores obtidos em Fator-C 1,5.

A influência do método de fotoativação também foi evidente. Os altos valores de correlação obtidos nos diferentes estudos comprovaram o fato de que a taxa de geração de tensão é diretamente proporcional à intensidade inicial de luz. Métodos que fizeram uso de altos valores de intensidade inicialmente, como o de Luz Contínua (550 mW/cm^2 nos capítulos 1 e 4) e LED em Alta Intensidade (850 mW/cm^2 nos capítulos 2 e 3), quando comparados aos métodos modulados de fotoativação, foram sempre associados a maiores taxas de geração de tensão. Para estes métodos, a reação de polimerização deve ter acontecido em alta velocidade, eliminando a oportunidade de escoamento no início da polimerização, e acarretando aumento significativo da rigidez do material logo após a obtenção de baixa conversão (BRAGA ET AL., 2005; KANNURPATTI ET AL., 1997). Como conseqüência, o início do desenvolvimento da tensão é observado imediatamente após o inicio da polimerização (CALHEIROS ET AL., 2004), e a maior parte da conversão do compósito ocorre após a matriz polimérica ter alcançado significante nível de rigidez (BRAGA ET AL., 2005).

Entretanto, embora intensidades luminosas com grandes diferenças tenham sido avaliadas nos diferentes métodos de fotoativação, em muitos dos casos não foi observada diferença estatística nos valores máximos de tensão alcançados pelos métodos, como, por exemplo, entre o método LED Baixa Intensidade e o método LED Alta Intensidade, nos quais não foi encontrada diferença significativa na tensão máxima gerada, tanto em Fator-C 1,5 como em Fator-C 3,0 (capítulo 2), apesar de as intensidades luminosas testadas terem sido significativamente diferentes, 200 e 850 mW/cm^2, respectivamente, em exposição contínua. Por outro lado, apesar da ausência de diferença estatística quanto à tensão máxima gerada, diferenças significativas foram verificadas na taxa máxima de geração de tensão entre os métodos fotoativadores nos diferentes estudos. No Capítulo 1, apesar da não diferença estatística na tensão máxima gerada entre os métodos *Pulse Delay* 150 (9,3 MPa) e Luz Contínua (10,3 MPa), uma redução de 53% foi observada na taxa de geração de tensão para o método *Pulse Delay* (0,15 MPa/s) em comparação ao método Luz Contínua (0,32 MPa/s). O mesmo quadro foi observado no capítulo 3, no qual não foi observada diferença estatística na tensão máxima gerada entre os métodos *Pulse Delay* (9,3 MPa) e LED Alta Intensidade (10,5 MPa), embora uma redução de 67% tenha sido observada na taxa de geração de tensão para o método *Pulse Delay* (0,15 MPa/s) em comparação ao método LED Alta Intensidade (0,46 MPa/s). A redução da taxa de geração de tensões está possivelmente associada à baixa intensidade utilizada inicialmente para os métodos modulados de fotoativação, promovendo redução da taxa de polimerização, e, conseqüentemente, modificando o processo de geração de tensões (HOFMANN ET AL., 2003). Dessa forma, parece que embora a tensão final gerada possa ser similar entre os métodos avaliados, o meio pelo qual ela é alcançada difere (SAKAGUCHI ET AL., 2004). Entretanto, embora a taxa de geração de tensões tenha sido reduzida significativamente, não significa que a redução da tensão máxima também será significante. Estudos anteriores (WITZEL ET AL., 2005; BOUSCHLICHER & RUEGGEBERG, 2000; BRAGA & FERRACANE, 2002) concluíram que a taxa de polimerização precisa ser reduzida de maneira altamente significativa para que redução da tensão máxima gerada seja verificada, porque o ponto gel dos metacrilatos é alcançado em baixos níveis de grau de conversão (KANNURPATTI ET AL., 1997), e, uma vez que a reação foi iniciada, é esperado que a tensão aumente rapidamente com conseqüente aumento da rigidez pela ocorrência de ligação cruzada entre as cadeias poliméricas recém-formadas (STANSBURY ET AL., 2005). Portanto, o período permitido para o escoamento do material é muito restrito, e uma redução substancial da taxa de polimerização é requerida para significativamente afetar o desenvolvimento de tensões. Entretanto, é importante o fato de que materiais com diferentes composições podem apresentar diferentes comportamentos quando submetidos aos mesmos métodos de fotoativação testados nestes estudos. Um estudo avaliando o efeito de métodos fotoativadores modulados sobre o desenvolvimento da tensão de três compósitos comerciais encontrou percentagens de redução de tensão entre 19 e 30% (LIM ET AL., 2002).

Por outro lado, a redução da taxa de geração de tensões observada pelos métodos fotoativadores modulados promoveu, de uma maneira geral, aumento da resistência da união de restaurações em compósito. Como exemplo, os métodos *Pulse Delay* foram associados aos maiores valores de resistência da união, 35,5 MPa para o método *Pulse Delay* com intensidade inicial de 150 mW/cm^2 e 34,4 MPa para o mesmo método com intensidade inicial de 80 mW/cm^2 (Capítulo 1). Este método de fotoativação apresentou resultados de resistência da união estatisticamente superiores aos alcançados pelo método Luz Halógena Contínua (capítulos 1 e 3) e LED Alta Intensidade (Capítulo 3). Além disso, o padrão de fratura apresentado pelos métodos modulados foi predominantemente mista, simbolizando preservação parcial da interface adesiva, em oposição aos métodos não modulados Luz Contínua e LED Alta e Média Intensidade, que apresentaram o padrão de fratura adesiva como predominante. Portanto, apesar da ausência de diferença estatística quanto à tensão máxima gerada entre os diferentes métodos de fotoativação, a redução da taxa de geração de tensões associou os métodos modulados a maior resistência da união, permitindo a conclusão de que o meio de geração da tensão é mais importante do que a tensão máxima alcançada.

Alguns estudos (DENTALMAN INTERN.; DENMED DIRECT SERVICES) observaram que as fontes de luz LED poderiam promover maior grau de conversão ao compósito comparada às fontes de lâmpada halógena, o que seria vantajoso clinicamente. Entretanto, quando as fontes de lâmpada halógena e LED foram comparadas nos capítulos 2 e 3, através dos métodos Luz Halógena Contínua e LED Média Intensidade (ambos utilizando intensidade luminosa de 550 mW/cm^2 e mesma densidade energética de 16 J/cm^2), não foram observadas diferenças estatísticas entre os métodos para os valores de grau de conversão e tensão máxima, para ambos níveis de Fator-C. A ausência de diferença estatística para os valores de grau de conversão também foi observada por estudos prévios (MILLS ET AL., 2002; UHL ET AL., 2005), nos quais não houve diferença no desenvolvimento de propriedades físico-mecânicas quando as fontes luminosas LED e halógena foram comparadas.

Os métodos LED Baixa Intensidade e *Pulse Delay* 150 foram testados com intensidades luminosas similares (200 e 150 mW/cm^2 respectivamente), porém o primeiro em exposição contínua e o segundo com exposição inicial de 5 segundos seguido por intervalo de 3 minutos e segunda exposição a 550 mW/cm^2. Considerando os resultados obtidos, foi possível concluir que o método *Pulse Delay* foi mais efetivo na redução da taxa de geração de tensões pelo possível fato de que a primeira exposição não utilizou somente intensidade de luz reduzida, mas também foi feita por curto período de tempo (5 segundos). Considerando que a conversão continuou a acontecer no período no qual não houve exposição à luz, quando a segunda exposição foi efetuada, significativo percentual de conversão já tinha ocorrido. Dessa forma, durante o segundo ciclo de exposição à luz, foi observado para o método *Pulse Delay* menores taxas de geração de tensão (0,12 e 0,15 MPa/s nos níveis de Fator-C 1,5 e 3,0 respectivamente), mesmo utilizando para a segunda exposição a mesma intensidade de luz dos métodos Luz Halógena Contínua e LED Média Intensidade (Capítulos 2 e 3).

Quanto aos resultados de grau de conversão, para todos os métodos e níveis de Fator-C comparados nos diferentes capítulos, diferenças estatísticas não foram observadas.

Tais resultados podem estar associados ao fato de que a mesma densidade energética (16 J/cm^2) tenha sido utilizada para os diferentes métodos, comprovando a relação entre intensidade e tempo de exposição (HALVORSON ET AL., 2002), possibilitando a combinação de diferentes intensidades e tempos de exposição, promovendo a mesma conversão se a dose energética total utilizada for a mesma. Portanto, as vantagens associadas aos métodos fotoativadores modulados previamente citadas foram alcançadas sem que houvesse redução do grau de conversão do compósito.

Concluindo, apesar de não terem sido verificadas, de uma maneira geral, diferenças estatísticas entre os métodos comparados quanto à tensão máxima, a redução observada na taxa de geração de tensões para os diferentes métodos modulados de fotoativação ou aqueles nos quais a exposição luminosa foi feita em intensidade de luz reduzida, provou ser efetiva para aumentar significativamente a resistência da união da interface adesiva, sem que houvesse redução do grau de conversão do compósito. O nível de fator-C apresentou influência significativa. Quanto maior o nível de Fator-C, maior a quantidade de tensão gerada, e maior a taxa com que esta se desenvolve. Entretanto, o nível de Fator-C não apresentou efeito no grau de conversão do compósito. Agora se faz necessário verificar se tais vantagens podem ser associadas também *in vivo* com maior longevidade clínica de restaurações em compósito.

Conclusões Gerais

1. A modulação do método de exposição à luz durante a fotoativação do compósito resinoso foi associada a aumento da resistência da união da interface adesiva em restaurações de compósito.

2. Métodos de fotoativação modulados mostraram-se efetivos em reduzir a taxa com que a tensão é gerada, promovendo aumento da resistência da união da interface adesiva, sem que, no entanto, houvesse redução do grau de conversão do compósito restaurador.

3. O tipo de fonte de luz utilizada não influenciou o desenvolvimento das propriedades físicas do compósito quando a mesma energia era utilizada para ambas fontes luminosas.

4. O nível de Fator-C apresentou influência significativa sobre a taxa de geração de tensão e tensão total gerada pela contração de polimerização do compósito. Entretanto, o nível de Fator-C não apresentou influência sobre o grau de conversão desenvolvido pelo compósito.

5. Os métodos de fotoativação modulados apresentaram aumento da efetividade com diminuição do nível de Fator-C. Dentre os métodos modulados, *Pulse Delay* apresentou-se o mais efetivo, com significativa redução do *stress rate* e aumento significativo da resistência da união de restaurações adesivas.

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* De acordo com a norma utilizada na FOP/UNICAMP, baseada no modelo Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

* Referências pertinente à Introdução Geral do trabalho e as Considerações Gerais.

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APÊNDICES

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Progress report 727

Title: Effect of different photoactivation methods on the bond strength of composite restorations by push-out test

Type: Original Article

Author: Leonardo Goncalves Cunha

Manuscript file: Download manuscript(pdf) 111kB	**Resubmitted Manuscript:** Download manuscript(pdf) 100kB

First Submission

Date received:2005-08-25		Review Time: 33.1 weeks
Number of requests sent to external reviewers:	1	
Number of external reviewers that accepted to review:	1	
Number of external reviews that have been completed:	1	
Decision: **Reject But Resubmit**	Decision date: 2006-04-16	Print reviews as a pdf file >>

Resubmission

Date resubmitted:2006-05-23		Re-review Time: 5.1 weeks
Number of external re-reviews in progress:	0	
Number of external re-reviews in returned:	1	
Decision: **Accept**	Decision date: 2006-06-29	Print reviews as a pdf file >>

J Dent. 2006 Nov 17; [Epub ahead of print]

Modulated photoactivation methods: Influence on contraction stress, degree of conversion and push-out bond strength of composite restoratives.

- **Cunha LG**,
- **Alonso RC**,
- **Pfeifer CS**,
- **Correr-Sobrinho L**,
- **Ferracane JL**,
- **Sinhoreti MA**.

Department of Restorative Dentistry, Dental Materials Area, Piracicaba, School of Dentistry, UNICAMP Av. Limeira, 901, Bairro Areiao, CEP 13414-018, Piracicaba, Sao Paulo, Brazil.

OBJECTIVES: Verify the influence of curing methods on contraction stress, stress rate, and degree of conversion (DC) of a restorative composite and on bond strength of composite restoratives. METHODS: For the stress test, composite (0.84mm thick) was applied between two 5-mm diameter glass rods, mounted in a servohydraulic machine. Stress rate was taken by the value of stress/time at each second. DC was measured by micro-FTIR. Bond strength testing was performed using a push-out test. The C-factor in all tests was 3.0. Four curing methods were tested: continuous light (CL), soft-start (SS), and two pulse delay methods using different initial irradiances-150mW/cm(2) (PD150) and 80mW/cm(2) (PD80). Results were analyzed by ANOVA and Tukey's test (alpha=0.05). RESULTS: Stress values ranged from 7.9MPa (PD80) to 10.3MPa (CL). No statistical difference was verified among CL, SS, and PD150. PD80 presented statistically lower stress values compared to CL and SS. CL presented the highest maximum stress rate, followed by SS, PD150 and PD80. Mean DC values ranged from 54.2% (PD150) to 55.9% (PD80), with no difference observed among the methods. For the bond strength test, values ranged from 26.4MPa (CL) to 35.5MPa (PD150). PD150 and PD80 were both statistically superior to SS and CL. SS presented statistically higher bond strength compared to CL. CONCLUSIONS: Modulated curing methods were shown to be effective in reducing contraction stress rate and improving the strength of the bonded interface, and without compromising the DC of the restorative composite.

PMID: 17113700 [PubMed - as supplied by publisher

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Piracicaba, 27 de fevereiro de 2007.

Leonardo Gonçalves Cunha
RG: 30.517.504-X
AUTOR

Prof. Dr. Mário Alexandre Coelho Sinhoreti
RG: 18.897.368
ORIENTADOR