EDUARDO DALL'MAGRO

Cirurgião – Dentista

AVALIAÇÃO DA DUREZA KNOOP E DA RESISTÊNCIA DE UNIÃO DE RESTAURAÇÕES EM COMPÓSITO APÓS DIFERENTES PROTOCOLOS DE FOTOATIVAÇÃO

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

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Prof. Dr. Mário Alexandre Coelho Sinhoreti

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Prof. Dr. Tarcísio José de Arruda Paes Júnior
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SUMÁRIO

RESUMO

Este estudo apresenta três experimentos na mesma linha de pesquisa. No primeiro estudo, foi avaliada a resistência de união através do método *push out* e a dureza Knoop do compósito dental Z250 (3M/ESPE) fotoativado utilizando o aparelho de lâmpada halógena XL2500 (3M/ESPE) sob diferentes protocolos de fotoativação: modo contínuo (CH) (700 mW/cm^2) por 20 segundos; modo contínuo de baixa intensidade (CL) (150 mW/cm^2) por 20 segundos; e, *pulse-delay* com ativação na intensidade de 150 mW/cm^2 por 2s(P2), 3s(P3), 5s(P5), 10s(P10) ou 15 segundos (P15), seguindo-se 1 minuto de espera, e complementado por 700 mW/cm^2 por mais 20 segundos. Para o teste de *push out*, após a fotoativação, os corpos-de-prova foram armazenados a 37ºC ± 1 por 24h ± 1 até serem desgastados e polidos, e, os valores de resistência à união foram observados numa máquina de ensaio universal (Instron) com célula de carga de 500 N e velocidade de 0,5 mm/min. Para o ensaio de dureza, a fotoativação seguiu o mesmo protocolo. Após a confecção, as amostras foram embutidas em resina de poliestireno e submetidas ao acabamento e polimento com lixas d'água, a fim de obter as medidas de dureza, na superfície, a 1mm, 2, 3, 4, e 5 mm de profundidade, num durômetro (HMV 2, Shimadzu), utilizando carga de 50g, por 15 segundos. Os dados foram submetidos à Análise de Variância e as médias ao teste de Tukey (5%). Os resultados de resistência à união mostraram que o grupo P5, apresentou valor de resistência de união significantemente maior que os demais grupos. O grupo CL apresentou resistência de união menor que o grupo anteriormente citado, mas superior aos demais grupos. Não houve diferença estatística significativa entre os grupos P2, P3, P10, P15 e CH. A dureza Knoop mostrou que os grupos CH e P15 apresentaram as maiores médias de dureza na superfície e até 4 mm de profundidade. Os corpos-de-prova do grupo CL apresentaram as menores médias de dureza. Na profundidade de 3 mm , a dureza Knoop de todos os grupos foi estatisticamente inferior a da superfície. O segundo experimento avaliou a resistência de união através do método de *push out* e a dureza Knoop do compósito odontológico Z250 (3M/ESPE), fotoativado com o aparelho de lâmpada halógena XL2500 (3M/ESPE), utilizando diferentes protocolos de fotoativação: modo contínuo (700 mW/cm^2 por 20s) (CO); *soft-start*

(50 mW/cm^2 por 5s, seguido por 700 mW/cm^2 for 15s) (SS1); *soft-start* (100 mW/cm^2 por 5s, seguido por 700 mW/cm^2 por 15s) (SS2); *soft-start* (150 mW/cm^2 por 5s, seguido por 700 mW/cm^2 por 15s) (SS3); *soft-start* (200 mW/cm^2 por 5s, seguido por 700 mW/cm^2 por 15s) (SS4); *soft-start* (250 mW/cm^2 por 5s, seguido por 700 mW/cm^2 por 15s) (SS5); *soft-start* (300 mW/cm^2 por 5s, seguido por 700 mW/cm^2 por 15s) (SS6). Os testes de *push out* e de dureza Knoop foram realizados como no primeiro experimento. Os dados foram submetidos à Análise de Variância e as médias ao teste de Tukey (5%). Os resultados mostraram que o grupo SS3, obteve a maior resistência de união quando comparado ao grupo CO. Não houve diferença estatística entre os outros modos *soft-start* em relação aos demais grupos. Os outros resultados de dureza, não apresentaram diferença estatística entre os grupos na região de superfície e até 4 mm de profundidade. Também foi observado que para cada grupo, não houve diferença estatística entre a região de superfície até a profundidade de 2 mm. O terceiro experimento avaliou a resistência de união através do método de *push out* do compósito odontológico Z250 (3M/ESPE), fotoativado com o aparelho de lâmpada halógena XL2500 (3M/ESPE), utilizando diferentes protocolos de fotoativação: *soft-start* (ativação na intensidade de 150 mW/cm^2 por 2s(SS2), 3s(SS3), 5s(SS5), 10s(SS10) ou 15 segundos (SS15), e complementado por 700 mW/cm^2 por mais 15 segundos); *pulse-delay* (ativação na intensidade de 150 mW/cm^2 por 2s(P2), 3s(P3), 5s(P5), 10s(P10) ou 15 segundos (P15), seguindo-se 1 minuto de espera, e complementado por 700 mW/cm^2 por mais 15 segundos). Os testes de *push out* foram realizados como no primeiro experimento. Os dados foram submetidos à Análise de Variância e as médias ao teste de Tukey (5%). Os resultados mostraram que não houve diferença estatística entre os modos *soft-start*. Para o modo *pulse-delay*, o grupo P5 apresentou resistência de união estatisticamente superior aos demais grupos. Os grupos fotoativados pelo modo *pulse-delay* apresentaram resistência de união superior comparados aos grupos fotoativados pelo modo *soft-start*.

Palavras-chave: aparelhos fotoativadores; compósito; dureza Knoop; resistência de união.

ABSTRACT

This study showed three experiments in the same research line. The first study has assessed the bond resistance through the *push out* method and the Knoop hardness of the dental composite Z250 (3M/ESPE), photoactivated with the equipment of halogen light XL2500 (3M/ESPE), using different protocols of photoactivation: the continuous mode of high intensity (CH) (700mW/cm^2) for 20 seconds; the continuos mode of low intensity (CL) (150mW/cm^2) for 20 seconds; and the *pulse-delay* with 150mW/cm^2 for 2s(P2), 3s(P3), 5s(P5), 10s(P10) or 15 seconds (P15), with a 1-minute delay, followed by 700mW/cm^2 for 20 seconds. For the *push out* test, the test specimens were stored at 37^0 C ± 1 for 24h ± 1 until they were ground and polished, and, the bond strength values were observed through a universal test machine (Instron) with a charge cell of 500N and the speed of 0.5 mm/min. For the Knoop hardness, the specimens were made by using the same photoactivation protocols. After, the specimens were embedded in polyestiren resin and sanded and polished in order to obtain the hardness measures on top, 1 mm, 2, 3, 4, and 5 mm deep in a hardness measure machine (HMV 2, Shimadzu), using a charge of 50g, for 15 seconds. The data were submitted to ANOVA and Tukey's test (5%). The results of bond strength showed that the P5 group presented significantly higher resistance values than the other groups.The CL group presented a lower bond resistance than the group previously mentioned, though higher than the other groups. There was no statistic difference between the groups P2, P3, P10, P15 and CH. The results of Knoop hardness showed that the groups CH and P15 presented higher values of Knoop hardness in surfaces up to 4 mm deep. The specimens of the CL group presented the lowest averages of hardness. At 3 mm deep, the Knoop hardness of all groups was statistically inferior to the surface hardness. The second study evaluated the bond strength through the *push out* method and the Knoop hardness of the dental composite Z250 (3M/ESPE), photoactivated with halogen lamp unit XL2500 (3M/ESPE), using different protocols of photoactivation: continuous mode (700mW/cm^2 for 20s) (CO); *soft-start* (50 mW/cm^2 for 5s, followed by 700 mW/cm^2

for 15s) (SS1); *soft-start* (100 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS2); *soft-start* (150 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS3); *soft-start* (200 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS4); *soft-start* (250 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS5); *soft-start* (300 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS6). For the *push out* test and Knoop hardness test the specimens were made how the first experiment. The data were submitted to ANOVA and Tukey test (5%). The results showed that the SS3 group obtained the higher bond strength when compared to the CO group. There were no differences among the other modes of *soft-start* in relation to the other groups. The other results in hardness, showed no statistic differences among the groups in the surface region and up to 4 mm depth. It was also observed that within each group there were no statistic differences between the surface region up to the 2 mm depth. The third study evaluated the bond strength through the *push out* method of the dental composite Z250 (3M/ESPE), photo activated with halogen lamp unit XL2500 (3M/ESPE), using different protocols of photo activation: *soft-start* (150 mW/cm^2 for 2s(SS2), 3s(SS3), 5s(SS5), 10s(SS10) or 15 seconds (SS15), followed by 700 mW/cm^2 for 15 seconds); *pulse-delay* (150 mW/cm^2 for 2s(P2), 3s(P3), 5s(P5), 10s(P10) or 15 seconds (P15), with a 1-minute delay, followed by 700 mW/cm^2 for 15 seconds). For the *push out* test the specimens were made how the first experiment. The data were submitted to ANOVA and Tukey test (5%). The results showed there were no statistic differences among groups photo activated using *soft-start* mode. For *pulse delay* mode P5 obtained the highest bond strength, statistically higher than other groups. The groups photoactivated with *pulse-delay* mode showed bond strength statistically higher than *soft-start* mode.

Key words: curing units; composite; Knoop hardness; bond strength.

INTRODUÇÃO GERAL

Nos anos 60, as resinas compostas começaram a substituir gradativamente materiais restauradores como a resina acrílica e cimento de silicato em restaurações de dentes anteriores, porém, com algumas limitações em suas aplicações (Ferracane, 1992, Peutzfeldt, 1997) . Um dos principais obstáculos encontrados foi a infiltração marginal, causada pela perda do selamento marginal devido à contração existente durante o processo de polimerização (Rees & Jacobsen, 1989, Uno & Shimokobe, 1994).

Desde a introdução do sistema de ativação por luz visível para essas resinas, surgiram algumas vantagens como tempo de trabalho mais adequado, maior resistência à compressão e lisura superficial, entre outras. Porém, independente do compósito utilizado, ainda se verificava contração de polimerização devido a formação de ligações covalentes entre as moléculas do monômero, contraindo a cadeia polimérica (Peutzfeldt, 1997).

Dentre os fatores que influenciam a contração de polimerização desses compósitos pode-se citar: tamanho das moléculas dos monômeros, quantidade das partículas de carga, grau de conversão e natureza do monômero (Walls *et al.*, 1988). A contração de polimerização pode formar fendas nas margens das restaurações, devido às tensões geradas que rompem a união entre compósito e dente, originando com isso infiltrações marginais e, conseqüentemente, irritação pulpar, sensibilidade pós–operatória, recidiva de cárie e manchamento marginal (Uno & Shimokobe, 1994).

Dentre as alternativas para se melhorar a integridade marginal das restaurações com compósitos são citados: uso de materiais de base como resina *flow* e ionômero de vidro que apresentam menor módulo de elasticidade que o compósito para absorver parcialmente as tensões geradas pela contração; sistemas adesivos que contemplem efetivamente a união com a estrutura dental; uso de técnica incremental e controle do escoamento do compósito durante a fotoativação (Asmussen & Peutzfeldt, 2001, Feilzer *et al.*, 1995, Ikemi & Nemoto, 1994, Lutz *et al.*, 1986, Venhoven *et al.*, 1996, Versluis *et al.*, 1998).

Em relação ao escoamento durante a fotoativação, várias técnicas têm sido propostas com o objetivo de controlar a velocidade durante o processo de polimerização (Sakaguchi & Berge, 1998, Uno & Asmussen, 1991). Este controle para reduzir a tensão gerada pela contração pode ser conseguido, através de uma polimerização inicial com intensidade de luz baixa seguida de polimerização final com elevada intensidade, distanciando a ponta do aparelho fotopolimerizador da superfície do compósito, ou, usando aparelhos emissores de luz com intensidade variável (Feilzer *et al.*, 1995, Koran & Kurschner, 1998, Pires *et al.*, 1993, Unterbrink & Muessner, 1995). Assim, a menor taxa de conversão inicial permite o escoamento do material, gerando menor tensão interna decorrente da contração e posteriormente, a alta intensidade na polimerização promove adequado grau de conversão para obtenção de propriedades físicas e mecânicas satisfatórias (Davidson *et al.*, 1984, Feilzer *et al.*, 1995, Watts & al Hindi, 1999).

Como esses fatores parecem contribuir para manutenção da união do compósito restaurador ao dente, este estudo propõe avaliar o efeito de se propor várias alternativas de se modular a luz emitida pelo aparelho fotoativador durante o processo de fotoativação, na resistência de união e também avaliar, de maneira indireta, o grau de conversão do compósito através do ensaio de dureza Knoop.

PROPOSIÇÃO

Este estudo tem como proposição:

1 – Avaliar a influência da variação do tempo de exposição inicial usando o método de fotoativação *pulse-delay* na resistência de união (*push-out*) e dureza Knoop do compósito Z250.

2 – Avaliar a irradiância inicial ideal no método de fotoativação *soft-start* para promover maior resistência de união verificada pelo teste (*push-out*) e dureza Knoop do compósito Z250.

3 - Avaliar a influência da variação do tempo de exposição inicial usando os métodos de fotoativação *pulse-delay* e *soft-start* na resistência de união (*push-out*) do compósito Z250, e comparar os dois modos de fotoativação.

Este trabalho foi realizado no formato alternativo com base na deliberação da Comissão Central de Pós-Graduação (CCPG) da Universidade Estadual de Campinas (UNICAMP) nº001/98.

CAPÍTULO 1

Effect of different modes of the light modulation on the bond strength and Knoop hardness of dental composite

(a ser enviado - formatado para o Journal of Dentistry)

Summary

Objective: This study verified the bond strength through the push-out method and the Knoop hardness of the composite Z250, photoactivated with the curing unit XL2500 (3M/ESPE), using different photoactivation protocols: continuous mode - high intensity (CH) (700mW/cm^2) for 20 s.; continuos mode - low intensity (CL) (150mW/cm^2) for 20 s.; and pulse-delay with 150mW/cm^2 for 2(P2), 3(P3), 5(P5), 10(P10) or 15 seconds (P15), with a 1-minute delay, followed by 700mW/cm^2 for 20 seconds.

Methods: For push-out test (n=10), the bond strength values were obtained using a universal test machine (Instron) with a cross-head speed of 0.5mm/min. For Knoop hardness (n=5), the specimens were made using the same photoactivation protocols. After, the hardness measures were made in a hardness machine (Shimadzu), using a load of 50g for 15 s.

Results: The data were subjected to ANOVA and Tukey's test (alpha=0.05). The results of bond strength showed that the P5 group presented significantly higher values. The CL group presented a lower value than the CL group, though higher than the other groups. There was no difference between the groups P2, P3, P10, P15 and CH. The results of Knoop hardness showed that the CH and P15 groups presented the highest values in surface up to 4mm deep. The specimens of the CL group presented the lowest mean. From 3mm deep, the means of all groups were statistically inferior to the surface.

Conclusion: The initial time exposition for pulse-delay method can influence on the bond strength and Knoop hardness of Z250 composite resin.

Keywords: dental composite, bond strength, Knoop hardness, pulse-delay.

Introduction

Resin composites are the direct restorative materials more used in dentistry [1]. Current dental composites are expected to have optical and mechanical properties comparable to tooth enamel and dentin and provide a service life of 10 years or more. However, the polymerization shrinkage still remains the primary problem of dental composites. Shrinkage is inherent in current commercially available system and will persist until non-shrinking or low-shrinkage polymers [2] or expanding fillers are available in commercial composites. This creates contraction stress and marginal gap, leading to invasion of oral fluids and bacteria [3,4]. However, several factors limit the performance of this material, especially depth of cure and degree of conversion. Light of an appropriate wavelength initiates photo-polymerization of methacrylate groups producing a highly cross-linked polymer matrix. Light from the curing source must be able to adequately polymerize deeper composite regions than just the top, irradiated surface. However, as light passes through the composite, it is absorbed and scattered, reducing its effectiveness to initiate polymerization, and consequently resulting in variation of cure with depth [5]. Although high light intensity provides higher DC values, it also produces greater polymerization shrinkage, which may cause gap formation and further micro-leakage, compromising the longevity of the restoration [6-8]. Another problem is that the insufficient polymerization with high residual monomers may be released within the first days in oral conditions [9,10]. The unreacted monomers result in inferior physical properties. Several methods of light modulation are used for the photo-activation of the composites with the objective of minimizing the stress generated by the polymerization shrinkage. These photo-activation methods have been suggested to initiate the composite cure reactions, maintaining DC values similar to that found using high light intensity, whilst reducing the polymerization shrinkage. Soft-start polymerization and pulse-delay polymerization adopt an initially low light intensity followed by a final cure with high light intensity. A slower rate of

conversion allows for better flow of the material, which in turn decreases contraction stresses, leading to better marginal adaptation. The complement of photo-activation with high irradiance is intended to allow the composite to achieve the maximum degree of conversion [7,11,12]. Irradiance diminishes as the curing tip is moved away from resin composite restorative material [13], and can be used for two-steps photo-activation methods. Furthermore, the viscoelastic nature of the polymerizing composite must also be considered. During polymerization, there is an irreversible increase in the elastic modulus of the composite. The moment that the loss of the flowing capacity of the material occurs is called gel-point. Prior to the gel-point, polymerization contraction will not create stress at the restoration margins or within the material, as it is compensated by flow. However, rigid contraction after the gel-point has received much attention and has been responsible for the induction of stress in the bonding interface [14,15,16] demonstrated that the speed of the polymerization reaction has a great effect in the generation of stress.

The testing design for this study was a push-out model. Extrusion testing in dentistry was first described in 1970 by Roydhouse [17]. He proposed pushing out dental material cylinders from tooth discs. Previous studies making use of a conical version of the push-out design demonstrating the different bonding properties of direct and indirect restorations with highly reproducible measurements [18]. Besides that, the cavity-like configuration of the bonding area allows the determination of both composite bond strength and marginal adaptation on the same sample [18].

Independently of the photo-activation method applied, the DC must be as high as possible, ensuring that the material possesses the best properties. Studies in the literature demonstrating a good correlation between DC and hardness of composites and, hence, hardness is utilized as an indicator of DC [1,19,20].

The ideal exposure time with low irradiance in the initial light exposition for two-steps polymerization methods is primordial to optimal marginal adaptation and mechanical properties of resin composites. The objective of this study was to evaluate the influence of initial light exposure time variations using pulse-delay

photo-activation methods on bond strength (push-out test) and Knoop hardness of a resin composite.

Materials and Methods

For this study the composite resin Filtek Z250 (3M/ESPE, St.Paul, MN, USA), shade A3, was used.

Push-out test

It was used 70 round metallic matrices with 3mm height containing a truncated-cone cavity with 6mm in the lower diameter and 9mm in the upper diameter.

These matrices were internally sandblasted with aluminum oxide 40 µm using a micro-etcher (Danville Eng. Inc., USA). After sandblasting, the matrices were etched with phosphoric acid 37% for 15 seconds, water rinsed and dried. The bonding agent silane (Ceramic Primer, 3M/ESPE) was applied and dried for 5 seconds. The adhesive system (Adper Single Bond, 3M/ESPE) was applied, air-dried for 20 seconds and photoactivated for 10 seconds using light-cured with halogen lamp XL2500 (3M/ESPE).

During the application of resin composite the matrices were set on a Mylar strip positioned on a glass slab. The resin composite was bulk inserted and then covered with a polyester strip and pressed with a microscope sheet to accommodate the material into the matrix and remove composite excesses.

Composites were light-cured with halogen lamp XL2500 (3M/ESPE). The maximum irradiance checked with the radiometer (Demetron Research Corp., Danbury, USA), was 700 mW/cm^2. The specimens were photo-activated according to the following methods: continuous high irradiance mode (700 mW/cm^2 for 20s) (CH); continuous low irradiance mode (150 mW/cm^2 for 20s.) (CL); pulse-delay (150 mW/cm^2 for 2s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P2); pulse-delay (150 mW/cm^2 for 3s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P3);

pulse-delay (150 mW/cm^2 for 5s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P5); pulse-delay (150 mW/cm^2 for 10s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P10); pulse-delay (150 mW/cm^2 for 15s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P15). For reduced irradiances, the tip of curing units was moved away from composite surface. To standardize the photo-activation distance, spacers of acrylic resin (JET, Artigos Odontológicos Clássico, São Paulo, SP, Brazil) were interposed between the surface of the composite and the tip of the light curing units.

After photo-activation, the specimens were stored in incubator at 37ºC ± 1 for 24h ± 1, in a dark and dry container, before push-out test. The top and bottom surfaces of restorations were grounded using 400 grit sandpapers (Carborundum, Saint-Gobain Abrasivos Ltda, PE, Brazil) on an automated polisher under water cooling to remove the composite excess, promoting a correct positioning of specimen while testing.

For push-out test an acrylic resin apparatus containing a central hole was attached in an universal testing machine (Instron model 4411, England). An sphere-shaped rod attached to a compression load cell (500N) was used to load the composite restorations until failure at a crosshead speed of 0.5 mm/min. The push-out bond strength was determined by computing the quotient of maximum load (N) and adhesion area (equation for calculation of truncated cones; mm^2). The data were submitted to one-way ANOVA, followed by Tukey's test at the 5% significance level.

Knoop hardness test

For the Knoop hardness test, one metallic mold (5 mm height X 5 mm diameter) were used to obtain 70 specimens. The resin composite was bulk inserted. A polyester strip was seated on surface of the specimen and pressed manually to remove composite excesses. Composites were light-cured with halogen lamp, according to methods proposed to push-out test: continuous high irradiance mode (700 mW/cm^2 for 20s) (CH); continuous low irradiance mode (150

mW/cm^2 for 20s.) (CL); pulse-delay (150 mW/cm^2 for 2s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P2); pulse-delay (150 mW/cm^2 for 3s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P3); pulse-delay (150 mW/cm^2 for 5s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P5); pulse-delay (150 mW/cm^2 for 10s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P10); pulse-delay (150 mW/cm^2 for 15s, delay of 1 min., followed by 700 mW/cm^2 for 20s) (P15).

After photo-activation, the specimens were stored in incubator at 37ºC ± 1 for 24h ± 1, in a dark and dry container. Elapsed 24 hours, the specimens were embedded in polyestiren resin for grounding and polishing. After curing of polyestiren resin, the specimens were ground and polished using 320, 400, 600 and 1200 grit sandpapers (Carborundum) on an automated polisher under water-cooling. The specimens were dried and submitted to Knoop hardness measurements in a hardness tester (HMV 2, Shimadzu, Tokyo, Japan) with load of 50 g for 15 s. For each specimen, five readings were taken and an average was calculated.

The data was submitted to two-way ANOVA (photo-activation mode x depth), followed by Tukey's test at the 5% significance level.

Results

Push-out bond strength

Table 1 displays the means and standard deviations for bond strength results. The P5 group revealed significant higher bond strength than other groups. Specimens photo-activated using continuous mode with low irradiance (CL) presented bond strength statistically lower than group P5, but higher strength than other groups. The push-out test did not reveal statistical differences among P2, P3, P10, P15 and CH groups.

Knoop hardness test

Table 2 displays the means and standard deviations for Knoop hardness. From the top surface up to 4mm depths, groups CH and P15 presented the highest Knoop hardness means, which were statistically higher than groups P2 and CL ($p<0.05$). Specimens photo-activated using continuous mode with low irradiance (CL) presented the lowest Knoop hardness means ($p<0.05$). There were no statistical differences among groups P3, P5, P10 and CH at any depth. Specimens of P2 presented Knoop hardness means statistically lower than group CH at top, 1, 2, 3, and 4 mm depths. At 5 mm, composites of group CL presented too soft for Knoop hardness testing (mean 0.00). There were no statistical differences among other groups.

The comparisons in the row (Table 2) show that at 2 mm depth the Knoop hardness of groups P2, P5, P10, and CL, was statistically lower than top surface. The Knoop hardness for all groups at 3 mm depth was statistically lower than top surface.

Discussion

The composite polymerization is a complex phenomenon that involves innumerous factors. The magnitude of stress development depends on the volume fraction of fillers, filler geometry, ratio of modulus between polymer and particle, adhesion between polymer and particle, inherent shrinkage of the particle, and degree of cure of polymer. The shrinkage stress is essential to longevity and success composites, and depends on the polymerization rate, composition of polymeric matrix, specimen geometry, filler volume fraction, photo-initiators concentration, and curing method. The rate of polymerization is influenced by photo-initiators concentration, monomer reactivity, molecular weight of monomer, leading to different viscosities and mobility [21]. Moreover, characteristics related to light source, such as energy density and spectral flux, also alter final material properties [5,22-26]. Energy density is the product of irradiance and the duration of

light exposure [5,23,24], whilst spectral flux represents the optical power output from the light-curing unit, in milliwatts, at each given wavelength [22,25]. The DC in a photo-activated composite is dependent on the total energy density, at the correct wavelength, supplied to the photo-initiator [27]. Reduction of this initial speed can be obtained through the use of low intensity units that produce few free radicals [28]. This lower speed gives the composite more time for molecular rearrangement, reducing the stress caused by polymerization shrinkage [29].

The pulse-delay method described by Kanca (1999) [30], uses the premise that a short pulse of light followed by a relaxation period in dark, allows the relief of stresses prior to inducing additional stresses during the continuing polymerization [31]. In this work, pulse-delay using 5 seconds of initial exposure time produced bond strength statistically higher as compared to other groups. Besides, the presence of the highest bond strength means the Knoop hardness was similar to CH groups. In those specimens induced less stress at bond interface, however the degree of conversion was maintained. According to Sakaguchi et al. [31], the maximum contraction force rate occurs within the first 10 s of photo-activation. The contraction force can lead to rupture in bond interface, promoting lower bond strength. We observed that beyond irradiance, the exposure time has a great importance for bond strength when the pulse-delay method was used. When short exposure time was used, P2 and P3 groups, showed that the bond strength was similar to CH group. Yap et al. [32], use the pulse-delay method in which the initial activation was carried out for 3 s at an intensity of 100 mW/cm^2, like in this study, and did not detect any shrinkage of the composite during photo-activation and the waiting period. The authors believe that the energy density of the initial photo-activation was not enough to effectively initiate a polymerization reaction and all the shrinkage the composite underwent, was compensated for by the flow of the material. The reaction only became effective when the second high irradiance exposure occurred. Thus, the reaction occurred under high intensity with a similar reaction speed to that of the CH. However, the Knoop hardness of P2 group was affected by short exposure time. For longer initial exposure times, P10 and P15 groups, bond strength were similar to CH group. The high energy density during

the first curing cycle for P10 (15.5 J/cm^2) and P15 (16.25 J/cm^2) produced high conversion of double bonds, leading to high shrinkage polymerization and contraction force. This can be seen in the similar Knoop hardness results of P10, P15 and CH groups. When specimens were photo-activated using continuous low irradiance (CL), bond strength was statistically lower than P5, but higher than other groups. The low irradiance during the photo-activation cycle, the polymerization reaction occurs slowly, allows the relief of shrinkage stresses, decreasing the stress in the bond interface. However, CL group presented the lowest Knoop hardness means. Those specimens were photo-activated using the lowest energy density (3 J/cm^2). Regardless of light modulation method, it has been recommended that energy densities similar to those supplied by conventional photo-activation (continuous mode) are used. This is recommended because previous studies related that the degree of conversion depends on energy density that is supplied to the composite than photo-activation method [33,34]. Koran and Kurschner [12] found that the increase in density led to an increase in the hardness values until photo-activation with 17 J/cm^2, after which there was no significant alteration in hardness values. In this study the maximum energy was given using P15 (16.25 J/cm^2). Groups that presented the lowest energy density (P2 and CL) showed Knoop hardness means statistically lower than CH. The bond strength of CL was statistically lower than P5. According to the results of this work, the relaxation period in dark is of great importance for stress relief in composite.

There were great differences between bond strength values. Push-out tests enable measurements of bond strength under more cavity-like conditions, demonstrating the properties of the restorative system better than in the shear or tensile tests. Confined spaces are less favorable from a mechanical perspective as a result of a high C-factor [35]. Therefore, more stresses occur during polymerization that pulls the bonded restoration away from the dentin walls, creating stress at the tooth-restorative interface [36]. If shrinkage stress overcomes the bond strength, a gap may be formed at the tooth-restorative interface. It was observed that specimens without gap formation presented high bond strength. However, when gap was present, there was a pronounced decrease in the bond strength. The

gaps are sites of stress concentration. During the push-out test, the stresses are focused in these sites, leading to rupture of bond. In those specimens without or less gap formation, the stresses are distributed for all bond interface, producing high bond strength values.

Conclusion

Within the limitations of this study, it can be concluded that the initial light exposure time variations for pulse-delay photo-activation method influences on bond strength and Knoop hardness of the resin composite Z250.

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Table 1: Push-out bond strength means and standard deviation ( ) for resin composite Z250 photo-activated using different photo-activation methods and exposure times.

Groups	Exposure time (s)	Bond strength (MPa)
P2	2+20	05.40 (1.89) c
P3	3+20	06.38 (1.16) c
P5	5+20	34.20 (8.47) a
P10	10+20	07.80 (2.33) c
P15	15+20	06.12 (1.73) c
CL	20	18.09 (3.35) b
CH	20	04.87 (0.87) c

Means followed by different small letter represent statistical difference (5%) by Tukey´s test.

Table 2: Knoop hardness means and standard deviation ( ) for resin composite Z250 photo-activated using different methods and exposure times.

Group	Exposure time	Depth (mm)					
		Top	1	2	3	4	5
P2	2+20	55.07 (2.57) b, A	51.75 (2.73) b, AB	48.60 (3.42) bc, BC	44.71 (2.70) b, CD	40.05 (3.10) b, D	26.40 (4.57) a, E
P3	3+20	56.90 (2.20) ab, A	55.66 (1.97) ab, A	52.85 (2.32) ab, AB	49.62 (2.93) ab, B	44.03 (2.27) ab, C	26.26 (1.56) a, D
P5	5+20	57.52 (1.62) ab, A	55.30 (1.69) ab, AB	51.02 (1.60) abc, BC	48.91 (1.75) ab, CD	44.53 (1.03) ab, D	25.20 (4.46) a, E
P10	10+20	59.77 (1.42) ab, A	55.00 (1.41) ab, AB	51.03 (2.19) abc, BC	47.57 (2.28) ab, CD	44.04 (0.87) ab, D	27.92 (1.94) a, E
P15	15+20	60.36 (0.54) ab, A	57.77 (1.30) ab, A	54.70 (2.31) a, AB	51.05 (2.06) a, BC	46.22 (2.43) a, C	24.80 (3.09) a, D
CL	20	55.24 (1.61) b, A	51.82 (0.42) b, A	45.33 (1.95) c, B	35.90 (2.25) c, C	10.96 (3.90) c, D	00.00 (0.00) b, E
CH	20	62.10 (5.54) a, A	59.45 (4.60) a, A	56.46 (3.66) a, AB	53.00 (2.76) a, BC	49.38 (2.40) a, C	28.27 (3.47) a, D

Means followed by different lowercase letter in the column and capital letter in the row represent statistical difference (5%) by Tukey´s test.

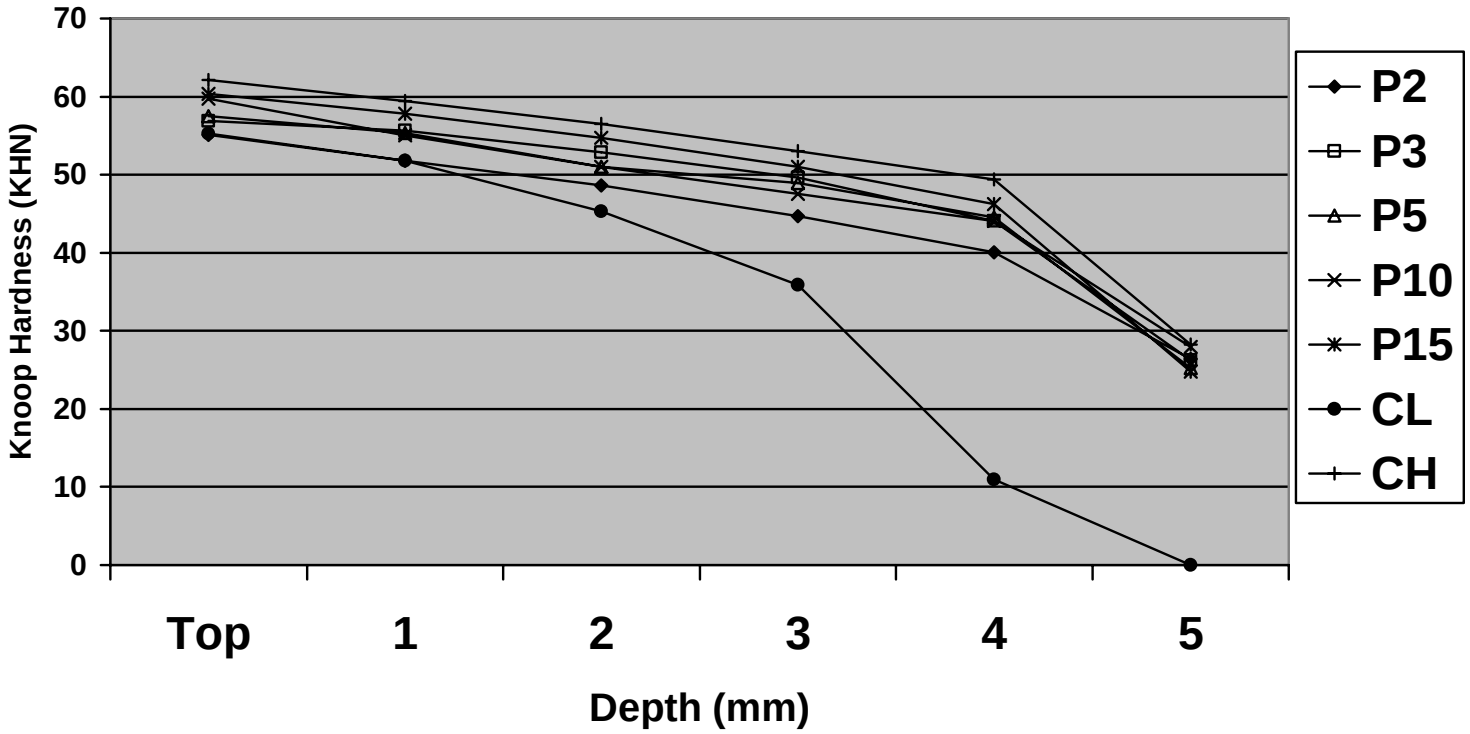

Figure 1: - Knoop hardenss as a function of depth from the surface, considering samples cured using different photo-activation protocols: Pulse-delay 2 seconds (P2), Pulse-delay 3 seconds (P3), Pulse-delay 5 seconds (P5), Pulse-delay 10 seconds (P10), Pulse-delay 15 seconds (P15), continuos mode with low intensity (CL), continuos mode with high intensity (CH).

CAPÍTULO 2

Effect of different initial light intensity by the soft-start photo-activation technique on the bond strength and Knoop hardness of dental composite

(enviado para o Brazilian Dental Journal)

Abstract

This study evaluated the bond strength through the push-out method and the Knoop hardness of the dental composite Z250 (3M/ESPE), photo activated with halogen lamps XL 2500 (3M/ESPE), using different protocols of photo activation: continuous mode (700mW / cm^2 for 20s) (CO); soft-start (50 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS1); soft-start (100 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS2); soft-start (150 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS3); soft-start (200 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS4); soft-start (250 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS5); soft-start (300 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS6). For the push-out test, the specimens were stored at 37°C ± 1 for 24h ± 1 until they are sanded and polished and the bond strength values were obtained through a universal testing machine (INSTRON) with a load cell of 500N and the speed of 0.5 mm/min. The data was submitted to the variance analysis ANOVA and the average to the Tukey test at a 5% significance level. For the Knoop hardness test the specimens were made using the same protocols of photo activation. After that, they were embedded in polyestiren resin and afterwards sandblasted and polished in order to obtain the hardness measurements in hardness tester (HMV 2, Shimadzu), using a 50g load, for 15 seconds. For each specimen five readings were taken. The averages were submitted to the variance analysis and the averages to the Tukey test at a 5% significance level. The results of bond resistance showed that the SS3 group obtained the higher bond resistance when compared to the CO group. There were no difference among the other modes of soft-start in relation to the other groups. Regarding the other results in hardness, there have been no statistic differences among the groups in the surface region and up to 4 mm depth. It

was also observed that within each group there were no statistic difference between the surface region up to the 2 mm depth.

Key words: dental composite, bond strength, Knoop hardness, soft-start.

Introduction

Resin composites are the direct restorative materials most used in dentistry. Current dental composites are expected to have optical and mechanical properties comparable to tooth enamel and dentin and provide a service life of 10 years or more. However, the polymerization shrinkage still remains the primary problem of dental composites. Regardless resin composite formulation, contraction polymerization occurs due to covalent bond between monomer molecules, leading to polymeric chain shortening (1). Walls *et al.* (2) related that contraction polymerization is influenced by the molecular weight of monomer, the filler content, the degree of conversion and the monomer formulation.

This creates contraction stress, leading to marginal gap, invasion of oral fluids and bacteria, determining the composite longevity (1). However, several factors limit the performance of this material, especially depth of cure and degree of conversion (DC). Light of an appropriate wavelength initiates photo-polymerization of methacrylate groups producing a highly cross-linked polymer matrix. Light from the curing source must be able to adequately polymerize deeper composite regions than just the top, irradiated surface. However, as light passes through the composite, it is absorbed and scattered, reducing its effectiveness to initiate polymerization, and consequently resulting in variation of the cure with depth (3). Although high irradiance provides higher DC values,

it also produces greater polymerization shrinkage, which may cause gap formation and further micro-leakage, compromising the longevity of the restoration. Another problem is that the insufficient polymerization with high residual monomers. The amount of residual monomers depends on the light source and the photoatctivation method (4). This monomer may be released within the first days in oral conditions. The unreacted monomers result in inferior physical properties, and can be leached of polymeric matrix (5). Marginal integrity can be increased by absorbing stress materials under restorative materials; adhesive systems with high bond strength to tooth structure, incremental technique filling, and flowing control by light modulation during photo-activation (6).

Several methods of light modulation have been used for the photo-activation of the composites with the objective of minimizing the stress generated by the polymerization shrinkage. These photo-activation methods have been suggested to initiate the composite cure reactions, maintaining DC values similar to that found using high light intensity, whilst reducing the polymerization shrinkage (7). Soft-start polymerization adopts an initially low irradiance followed by a final cure with high irradiance. A slower rate of conversion allows for better flow of the material, which in turn decreases contraction stresses, leading to better marginal adaptation. The complement of photo-activation with high irradiance is used in order to make the composite achieve the maximum degree of conversion (6). Irradiance diminishes as the curing tip is moved away from resin composite restorative material, and can be used for two-steps photo-activation methods. Furthermore, the viscoelastic nature of the polymerizing composite must also be considered. During

polymerization there is an irreversible increase in the elastic modulus of the composite. The moment when the loss of the flowing capacity of the material occurs is called gel-point. Prior to the gel-point, polymerization contraction will not create stress at the restoration margins or within the material, as it is compensated by flow. However, rigid contraction after the gel-point has received much attention and has been responsible for the induction of stress in the bonding interface (8). Kinomoto et al. (9) demonstrated that the speed of the polymerization reaction has a great effect in the generation of stress. The ideal photoactivation method is primordial to optimal marginal adaptation, mechanical properties and depth of cure of resin composites (10). By using a lower irradiance, free radicals formation will be lower than when a higher irradiance is used. A lower free radicals formation means that linear polymerization will initially occur to a greater extent, thus the propagating chain comes close to an adjacent free radical or initiated chain as cross-linkage can occur. The aim of this study was to evaluate the ideal initial irradiance using soft-start photo-activation methods to produce the highest bond strength (push-out test) and Knoop hardness for a resin composite.

Materials and methods

For this study the composite resin Z250, shade A3 was used (3M, St. Paul, MN, USA)

Push-out test

Seventy round metallic matrices with 3mm height containing a round cavity with 6mm in the lower diameter and 9mm in the upper diameter were used.

These matrices were internally sandblasted with aluminum oxide 40 µm using a micro-etcher (Danville Eng. Inc., USA). After sandblasting, the matrices were etched with phosphoric acid 37% for 15 seconds, water rinsed and dried. The bonding agent silane (Ceramic Primer) was applied and dried for 5 seconds. The adhesive system (Single Bond, 3M, St.Paul, MN, USA) was applied, air-dried for 20 seconds and photo-activated for 10 seconds.

During the application of resin composite the matrices were set on a Mylar strip positioned on a glass slab. The resin composite Z250 was bulk inserted and a Mylar strip was seated on the specimen surface and manually pressed using a microscope sheet to remove composite excesses.

Composites were light-cured with halogen lamp XL2500 (3M/ESPE, St Paul, MN, USA). The maximum irradiance checked with the radiometer (Demetron Research Corp., Danbury, USA), was 700 mW/cm^2. Composite resin were photo-activated according to the following methods: continuous mode (700 mW/cm^2 for 20s) (CO); soft-start (50 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS1); soft-start (100 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS2); soft-start (150 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS3); soft-start (200 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS4); soft-start (250 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS5); soft-start (300 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS6). For reduced

irradiances, the tip of curing units was moved away from composite surface. To standardize the photo-activation distance, spacers of acrylic resin (JET, Artigos Odontológicos Clássico, São Paulo, SP, Brazil) were interposed between the surface of the composite and the tip of the light curing units.

After light curing, the specimens were stored in an incubator at 37ºC ± 1 for 24h ± 1, in a dark and dry container, before the push-out test. The top and bottom surfaces of restorations were ground using 400 grit sandpapers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE, Brazil) on an automated polisher under water cooling to remove the composite excess, promoting the correct positioning of specimen while testing.

For push-out test an acrylic resin apparatus containing a central hole was attached in an universal testing machine (Instron model 4411, England). A sphere-shaped rod attached to a compression load cell (500N) was used to load the composite restorations until failure at a crosshead speed of 0.5 mm/min. The push-out bond strength was determined by computing the quotient of maximum load (N) and adhesion area (equation for calculation of truncated cones; mm^2). The data was submitted to one-way ANOVA, followed by Tukey's test at the 5% significance level.

Knoop hardness test

For the Knoop hardness test 70 specimens were made with a metallic mold (5 mm height X 5 mm diameter). The resin composite was bulk inserted. A polyester strip was seated on surface of the specimen and pressed manually to remove composite excesses. Composites were light-cured with halogen

lamp, according to the methods suggested for the push-out test: continuous mode (700 mW/cm^2 for 20s) (CO); soft-start (50 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS1); soft-start (100 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS2); soft-start (150 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS3); soft-start (200 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS4); soft-start (250 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS5); soft-start (300 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS6). For reduced irradiances, the tip of curing units was moved away from the composite surface. To standardize the photo-activation distance, spacers of acrylic resin (JET, Artigos Odontológicos Clássico, São Paulo, SP, Brazil) were interposed between the surface of the composite and the tip of the light curing units.

After light curing, the specimens were stored in incubator at 37ºC ± 1 for 24h ± 1, in a dark and dry container. Elapsed 24 hours, the specimens were embedded in polyestiren resin for grinding and polishing. After curing the polyestiren resin, the specimens were ground and polished using 320, 400, 600 and 1200 grit sandpapers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE, Brazil) on an automated polisher under water cooling. The specimens were dried and submitted to Knoop hardness measurements in a hardness tester (HMV 2, Shimadzu, Tokyo, Japan) with load of 50 g for 15 s. For each specimen, five readings were taken and an average was calculated.

The data was submitted to two-way ANOVA (photo-activation mode x depth), followed by Tukey's test at the 5% significance level.

Results

Push-out bond strength

Table 1 displays the means and standard deviations for bond strength results. The SS3 group revealed significant higher bond strength than CO groups. There were no statistical differences among different soft-start methods. There were no statistical differences among groups SS2, SS4, SS5, SS6 and CO photo-activation methods.

Knoop hardness test

Table 2 displays the means and standard deviations for Knoop hardness. There were no statically differences for Knoop hardness from top up to 4mm depth between soft-start method and control group. At 5mm, group SS4 presented the highest Knoop hardness means, which were statistically higher than groups SS2 and SS3 ($p<0.05$).

The comparisons in the row (Table 2) show that at 2 mm depth, the Knoop hardness of groups SS1, SS2, SS5, and SS6 was statistically lower than top surface. The Knoop hardness for all groups at 3mm depth was statistically lower than top surface.

Discussion

The testing design for this study was a push-out model. Extrusion testing in dentistry was first described by Roydhouse in 1970 (11). He proposed

pushing out dental material cylinders from tooth discs. Previous studies using a conical version of the push-out design demonstrated the different bonding properties of direct and indirect restorations with highly reproducible measurements (12). Besides that, the cavity-like configuration of the bonding area allows the determination of both composite bond strength and marginal adaptation on the same sample (12). Independently of the photo-activation method applied, the DC must be as high as possible, ensuring that the material achieves the best properties. Studies in the literature demonstrate a good correlation between DC and hardness of composites and, hence, hardness is useful as an indicator of DC (5).

The composite polymerization is a complex phenomenon that involves innumerous factors. The magnitude of stress development depends on the volume fraction of fillers, filler geometry, ratio of modulus between polymer and particle, adhesion between polymer and particle, inherent shrinkage of the particle, and degree of polymer cure. The shrinkage stress has great importance to longevity and success composites, and depends on the polymerization rate, composition of polymeric matrix, specimen geometry, filler volume fraction, photo-initiators concentration, and curing method. The rate of polymerization is influenced by the photo-initiators concentration, the monomer reactivity, the molecular weight of monomer, leading to different viscosities and mobility (13). Moreover, characteristics related to light curing unit, such as energy density and spectral flux, also alter final material properties (3). Energy density is the product of irradiance and the duration of light exposure, whilst spectral flux represents the optical power output from the light-curing unit, in milliwatts, at each given wavelength (14). The DC in a photo-activated

composite is dependent on the total energy density, at the correct wavelength, supplied to the photo-initiator. Reduction of this initial speed can be obtained through the use of low irradiance units that produce few free radicals (15). This lower speed gives the composite more time for molecular rearrangement, reducing the stress caused by polymerization shrinkage (16).

The soft-start method described by Uno and Asmussen (17) uses the premise that a short pulse of light allows the relief of stresses prior to inducing additional stresses during the continuing polymerization (18). In this work, the amount of photons supplied to composite within the same exposure time was changed. If the polymerization reaction depends on the generation of free radicals, the speed reaction would be changed for different irradiances during photo-activation. The results showed the soft-start method using 150 mW/cm^2 initial irradiance (SS3) produced bond strength statistically higher than control group (CO). The low irradiance during the photo-activation cycle makes the polymerization reaction to proceed slowly, allowing the relief of shrinkage stresses and decreasing the stress at tooth-restorative material interface. Besides that, SS3 presented Knoop hardness means similar to CO groups ($p>0.05$). That means that, in those specimens, less stress occurred at bond interface, whilst the degree of conversion was maintained. According to Sakaguchi et al. (18), the maximum contraction force rate occurs within the first 10 s of photo-activation. The contraction force can lead to rupture in bond interface, promoting lower bond strength. The initial irradiance when soft-start method is used has a great importance for bond strength. When low irradiance was used, SS1 and SS2 groups, bond strength was similar to CO group. We expected that by using low irradiance in the photo-activation, the bond strength

would be increased. However, we believe that in those specimens the energy density of the initial photo-activation was not enough to effectively initiate the polymerization reaction. The reaction only became effective when the second high irradiance exposure occurred. Thus, the reaction occurred under high intensity similar to CO, leading to gap formation and lower bond strength (19). This may be seen in the Knoop hardness test, in which SS1 and SS2 groups presented Knoop hardness means similar to CO.

There were no statistical differences among SS4, SS5, SS6 and CO for bond strength. The speed reaction has been decreased to give the composite more time for molecular rearrangement, reducing the stress caused by polymerization shrinkage (16). Maybe, the high energy density during the first curing cycle for SS4, SS5, and SS6 produced high conversion of double bonds, leading to high shrinkage polymerization and contraction force. This can be seen in the similar bond strength results to CO. Regardless of the light modulation method, it has been recommended that energy densities similar those supplied by conventional photo-activation (continuous mode) be used. This is recommended because previous studies reported that the degree of conversion depends on the energy density that is supplied to the composite and the photo-activation method (20). Koran and Kurschner (7) found that the increase in energy density led to an increase in the hardness values until photo-activation with 17 J/cm^2, after which there was no significant alteration in hardness values. However, the ideal energy density for photo-activation is composite dependent. In this study the energy density variations (10.95-14.00 J/cm^2) have no effect on DC, seen on Knoop hardness values.

Conclusion

Within the limitations of this study, it can be concluded that the initial irradiance variations for the soft-start photo-activation method influences on bond strength, but do not have any effects on Knoop hardness values of the resin composite Z250. The best alternative for the traditional mode (continuous mode) is the soft-start mode with 150mW/cm^2 initial pulse.

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Table 1: Push-out bond strength means and standard deviation ( ) for resin composite Z250 photo-activated using different methods.

Groups	Irradiance (mW/cm^2)	Bond strength (MPa)
SS1	50+700	6.07 (0.83) ab
SS2	100+700	5.97 (0.73) ab
SS3	150+700	7.08 (1.34) a
SS4	200+700	5.91 (0.54) ab
SS5	250+700	6.22 (1.12) ab
SS6	300+700	6.17 (1.51) ab
CO	700	5.22 (0.78) b

Means followed by different lower case letters represent statistical difference (5%) by Tukey´s test.

Table 2: Knoop hardness means and standard deviation ( ) for resin composite Z250 photo-activated using different methods.

Group	Irradiance (mW/cm^2)	Knoop Hardness Means (KHN)					
		Top	1mm	2mm	3mm	4mm	5mm
SS1	50 + 700	58.32 (4.85) a, A	54.52 (4.97) a, AB	51.35 (3.69) a, B	49.46 (3.80) a, B	43.54 (3.54) a, C	29.07 (5.75) abc, D
SS2	100 + 700	59.40 (3.77) a, A	56.62 (2.04) a, AB	53.45 (2.01) a, B	51.40 (1.25) a, B	43.84 (2.20) a, C	23.20 (3.32) c, D
SS3	150 + 700	60.36 (5.60) a, A	58.82 (1.64) a, A	56.08 (1.42) a, AB	51.64 (0.47) a, BC	45.92 (2.41) a, C	26.60 (5.64) bc, D
SS4	200 + 700	58.37 (3.92) a, A	55.55 (4.94) a, AB	55.52 (2.69) a, AB	52.36 (1.98) a, BC	48.68 (3.04) a, C	33.07 (2.74) a, D
SS5	250 + 700	63.15 (2.36) a, A	60.17 (1.36) a, AB	56.67 (1.82) a, BC	53.55 (1.77) a, CD	49.61 (1.92) a, D	28.47 (2.03) abc, E
SS6	300 + 700	61.14 (3.14) a, A	57.54 (2.61) a, AB	55.02 (1.29) a, BC	49.49 (1.01) a, CD	44.33 (1.88) a, D	31.60 (2.26) ab, E
CO	700	62.10 (5.54) a, A	59.45 (4.60) a, A	56.46 (3.66) a, AB	53.00 (2.76) a, BC	49.38 (2.40) a, C	28.27 (3.47) abc, D

Means followed by different small letter in the column and capital letter in the row represent statistical difference (5%) by Tukey´s test.

Capítulo 3

Effect of different photo-activation techniques on the bond strength of dental composite

Abstract

This study evaluated the bond strength through the *push out* method of the dental composite Z250 (3M/ESPE), photo activated with halogen lamp unit XL2500 (3M/ESPE), using different protocols of photo activation: *soft-start* (150 mW/cm^2 for 2s(SS2), 3s(SS3), 5s(SS5), 10s(SS10) or 15 seconds (SS15), followed by 700 mW/cm^2 for 15 seconds; *pulse-delay* (150 mW/cm^2 for 2s(P2), 3s(P3), 5s(P5), 10s(P10) or 15 seconds (P15), with a 1-minute delay, followed by 700 mW/cm^2 for 15 seconds). The test specimens were stored at 37^{0}C ± 1 for 24h ± 1 until they were ground and polished, and, the bond strength values were observed through a universal test machine (Instron) with a charge cell of 500N and the speed of 0.5 mm/min. The data were submitted to ANOVA and Tukey test (5%). The results showed there were no statistic differences among groups photo activated using *soft-start* mode. For *pulse-delay* mode, P5 get the highest bond strength, statistically higher than other groups. The groups photo-activated with *pulse-delay* mode showed bond strength statistically higher than *soft-start* mode.

Introduction

The resin composites are the direct restorative materials most used in dentistry[14]. This is due to the possibility of bonding to dental structure, through adhesive systems, as well as its mechanical properties, that permit this material to be used both anterior and posterior restorations, and due to esthetic characteristics. However, composites present drawbacks like limited degree of conversion, due to the creation of a highly cross-linked network that limits the mobility of the reaction system [10]. However, the primary problem of dental composites is still the polymerization shrinkage.

The polymerization shrinkage depends on filler content, type of monomer used in resin matrix, i.e., monomer with high molecular weight present lower contraction than monomer with low molecular weight[1]. Another alternative is the use of non-shrinking or low-shrinkage monomers[2]. The polymerization shrinkage produces stresses. If these stresses are not relief internally, they can be concentrated at the bonding interface and produce gap formation[3]. Shrinkage stress at the bonding interface tends to destroy the bond between the resin composite and the tooth structure[5, 8].

The magnitude of shrinkage stress is decurrently of polymerization reaction speed[7]. The operator can minimize the shrinkage stress and gap formation at interface during photo activation using different photo activation methods[12] or incremental techniques[18]. To decrease speedy of polymerization reaction, and consequently the shrinkage stress, photo activation techniques that suggest low irradiance during the initial period of photo activation were proposed[7, 20]. Soft-start polymerization technique adopts an initially low light intensity followed by a final cure with high light intensity. A slower rate of conversion allows for better flow of the material, which in turn decreases contraction stresses, leading to better marginal adaptation[19, 20]. The complement of photo-activation with high irradiance is to composite achieves the maximum degree of conversion[5, 7, 20]. In the pulse-delay photoactivation technique, there is a short time period (about 1-3 minutes) between initial exposition and final cure. This procedure can reduce the incidence of cavosurface marginal gap and enamel fracture[6]. Another advantage of this photoactivation methods is that the degree of cure is similar to conventional method[4, 13].

However there are doubts as the best photo activation method and light exposure time to promote adequate bond strength between resin composite and dental structure. The aim of this study was to evaluate the bond strength of a resin composite photo activated using soft-start or pulse-delay technique, with different initial light exposure time during the first photo activation cycle.

Materials and Methods

It was used 100 round metallic molds with 3mm height containing a round cavity with 6mm in the lower diameter and 9mm in the upper diameter (Figure 1).

These molds were internally sandblasted with aluminum oxide 40 µm using a micro-etcher (Danville Eng. Inc., USA). After sandblasting, the molds were etched with phosphoric acid 37% for 15 seconds, water rinsed and dried. The bonding agent silane (Ceramic Primer, 3M/Espe, St. Paul, MN, USA) was applied and dried for 5 seconds. The adhesive system (Single Bond, 3M/Espe, St.Paul, MN, USA) was applied, air-dried for 20 seconds and photo-activated for 10 seconds.

During set, a Mylar strip was positioned on a glass slab. The resin composite Z250 (3M/Espe, St.Paul, MN, USA) shade A3, was bulk inserted and a Mylar strip was seated on surface of the specimen and pressed manually using a microscope sheet to remove composite excesses.

Composites were light-cured with halogen lamp (XL2500, 3M/ESPE, St Paul, MN, USA). The maximum irradiance checked with the radiometer (Demetron Research Corp., Danbury, USA), was 700 mW/cm^2. Resin composites were photo-activated according to follow methods: *soft-start* (150 mW/cm^2 for 2s, followed by 700 mW/cm^2 for 15s) (SS2); *soft-start* (150 mW/cm^2 for 3s, followed by 700 mW/cm^2 for 15s) (SS3); *soft-start* (150 mW/cm^2 for 5s, followed by 700 mW/cm^2 for 15s) (SS5); *soft-start* (150 mW/cm^2 for 10s, followed by 700 mW/cm^2 for 15s) (SS10); *soft-start* (150 mW/cm^2 for 15s, followed by 700 mW/cm^2 for 15s) (SS15); *pulse-delay* (150 mW/cm^2 for 2s, delay for 1 min, followed by 700 mW/cm^2 for 15s) (P2); *pulse-delay* (150 mW/cm^2 for 3s, delay for 1 min, followed by 700 mW/cm^2 for 15s) (P3); *pulse-delay* (150 mW/cm^2 for 5s, delay for 1 min, followed by 700 mW/cm^2 for 15s) (P5); *pulse-delay* (150 mW/cm^2 for 10s, delay for 1 min, followed by 700 mW/cm^2 for 15s) (P10); *pulse-delay* (150 mW/cm^2 for 15s, delay for 1 min, followed by 700 mW/cm^2 for 15s) (P15). For reduced irradiances, the tip of curing units was moved away from composite surface and to standardize the photoactivation distance, spacers of acrylic resin (JET, Artigos Odontológicos

Clássico, São Paulo, SP, Brazil) interposed between the surface of the composite and the tip of the light curing units.

After light-curing, the specimens were stored in incubator at 37ºC ± 1 for 24 h ± 1, in a dark and dry container, before push-out test. The top and botom surfaces of restorations were ground using 400 grit sandpapers (Carborundum, Saint-Gobain Abrasivos Ltda, Cruz de Rebouças/Igaraçu, PE, Brazil) on an automated polisher under water cooling to remove the composite excess, promoting a correct positioning of specimen while testing.

For push-out test an acrylic resin apparatus containing a central hole was attached in a universal testing machine (Instron model 4411, England). A sfere-shaped rod attached to a compression load cell (500N) was used to load the composite restorations until failure at a crosshead speed of 0.5 mm/min. The push-out bond strength (PBS) was determined by computing the quotient of maximum load (N) and adhesion area (equation for calculation of truncated cones; mm^2). The data were submitted to two-way ANOVA (photoactivation mode x exposure time), followed by Tukey's test at the 5% significance level.

Results

The Table 1 displays the means and standard deviations for bond strength results. For *pulse-delay* photoactivation method group, P5 presented mean statistically higher than other groups. Group P2 presented the lowest *push-out* bond strength mean, statistically different of P5 and P10 groups. *Push-out* bond strength test didn´t show statistical differences among groups photoactivated using *soft-start* methods. The comparisons between photoactivation methods showed that P3 presented higher bond strength than SS3 ($p<0.05$); P5 presented higher bond strength than SS5 ($p<0.05$), and P10 presented higher bond strength than SS10 ($p<0.05$). There were no statistical differences neither specimens of group P2 and SS2, nor specimens of groups P15 and SS15 ($p>0.05$).

Discussion

During photoactivation of the resin composites occur generation of free radicals that initiate polymerization reaction. The higher irradiance emitted from light source, the higher free radicals generation and faster the speed of polymerization reaction[10]. To decrease speedy of polymerization reaction, and consequently the shrinkage stress, photo activation techniques that commend low irradiance during the initial period of photo activation were proposed[6, 7, 20]. In this work, soft-start and pulse-delay technique of light modulation were used. The results showed that pulse-delay produced higher bond strength than soft-start, depending on initial light exposure time. Soft-start and pulse-delay techniques adopt an initially low light intensity followed by a final cure with high irradiance. The difference between soft-start and pulse-delay is that for pulse-delay, after the first polymerization cycle, there is a period in “dark” (1 minute) before the second polymerization cycle. Thus the composite has more time to molecular rearrangement and stress relief. In this dark period still occurs specimen conversion when samples are exposed to low irradiance. This is due to the free radicals that persist in the network after irradiation has ceased. The lower initial conversion produces greater mobility and allows more dark-cure and stress relief[11]. For soft-start technique the activation of some free radicals during the first photo activation cycle occurs and initiates the polymerization reaction. Immediately after the first polymerization cycle the samples are photo activated using the maximum irradiance emitted by light source. The high irradiance after the first cycle doesn’t allow that the stress be relief internally[16]. Thus the stress produced by fast polymerization reaction by second polymerization cycle added to stresses produced in the first cycle were directed to bond interface, leading to gap formation and reducing the bond strength as compared to pulse-delay technique[15].

When soft-start was used, the initial light exposure time had not influence on the bond strength values. However, for pulse-delay technique intermediate light exposure time during the first photo activation cycle produced bond strength statistically higher than other groups. When the light exposure time is reduced, a little amount of free radicals and double bonds conversion are produced9. Thus the great amount of reaction occurs in the second

polymerization cycle, with a fast speed of polymerization. As the polymerization in the second cycle occurs at high speed, there is more shrinkage stress, leading to lower bond strength values[17].

The groups that used longer initial light exposure times during the first photo activation cycle (10 and 15s) also presented bond strength lower than intermediate group. When there is a longer light exposure time during the first photo activation cycle, a great amount of polymerization reaction occurs. Therefore there is no time to stress relief and the shrinkage stress is concentrate at bond interface, decrease the bond strength.

Conclusion

Within the limitations of this study, it can be concluded that the initial light exposure time variations for pulse-delay photo-activation method influences on bond strength of the resin composite Z250. However for soft-start the initial light exposure time variations had no influence. Pulse-delay presented bond strength statistically higher than soft-start photo activation method.

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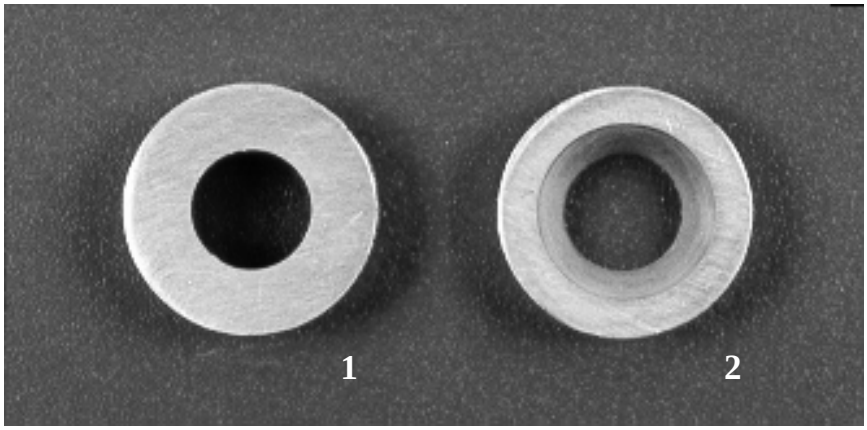
Table 1. Push-out bond strength means and standard deviation ( ) for resin composite Z250 photoactivated using different methods and exposure times.

Exposure time (s)	Bond strength (MPa)	
	Pulse-delay (P)	Soft-start (SS)
2 + 15	5.40 (1.89) c,A	4.16 (0.78) a,A
3 + 15	6.38 (1.16) bc,A	4.21 (1.16) a,B
5 + 15	34.20 (8.47) a,A	5.24 (0.95) a,B
10 + 15	7.80 (2.33) b,A	4.06 (0.63) a,B
15 + 15	6.12 (1.73) bc,A	5.26 (1.19) a,A

Means followed by different small letter in the column and capital letter in the row represent statistical difference (5%) by Tukey´s test.

A)



B)

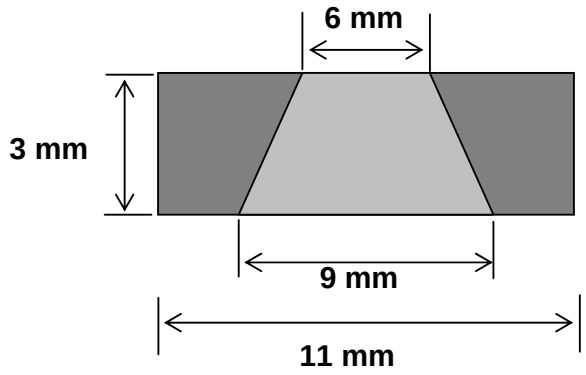


Figure 1. A – Metallic mold used: 1) view of the lower diameter region; 2) upper diameter region; B – schematic illustration of the mold.

CONSIDERAÇÕES FINAIS

Apesar da evolução do compósito odontológico, a contração de polimerização ainda é o principal obstáculo desse material. Durante a contração, tensões provenientes da aproximação das moléculas dos monômeros ocorrem e se não forem liberadas internamente, podem se concentrar na interface dente-material restaurador, levando à falha quando do procedimento restaurador. Para minimizar este problema, surgiram técnicas de fotoativação que visam modular a velocidade da reação, possibilitando um escoamento do compósito durante os períodos iniciais da polimerização, diminuindo a concentração de tensões entre as moléculas de polímero na interface. Dentre estas técnicas se destacam os modos *soft-start* e *pulse-delay*. Entretanto, não existe um protocolo para utilização destas técnicas, pois o tempo de exposição e a irradiância ideal para fotoativação ainda não foram estabelecidos. Portanto, procurou-se neste estudo verificar a influência do tempo de exposição e da irradiância durante o primeiro ciclo de fotoativação utilizando os modos *soft-start* e *pulse-delay.* Para isto, utilizou-se o compósito Z250 submetido aos ensaios mecânicos de resistência de união (*push-out*) e de dureza Knoop.

O primeiro estudo mostrou que o tempo de exposição inicial para o modo *pulse-delay* teve influência significativa sobre a resistência de união e sobre a dureza Knoop do compósito Z250. Durante o primeiro ciclo de foto-ativação pelo modo *pulse-delay*, o tempo de exposição de 5s com 150 mW/cm^2 inicial e 700 mW/cm^2 de irradiância final apresentou os maiores valores de resistência de união, enquanto manteve os valores de dureza Knoop semelhante ao grupo controle. Prolongando-se o tempo de exposição à luz, mais fótons atingem o compósito, ativando maior quantidade de foto-iniciadores, ocorrendo desta forma, maior grau de conversão. Durante os períodos iniciais de fotoativação é que ocorre a maior geração de tensão de contração. A fotoativação com baixa irradiância propicia maior escoamento das cadeias monoméricas, diminuindo a geração de tensão na interface, produzindo maiores valores de resistência de união. O grupo fotoativado pelo modo contínuo com baixa irradiância apresentou resistência de união inferior

ao grupo foto-ativado pelo modo *pulse-delay* com tempo de exposição de 5s, mas superior aos demais grupos. Entretanto, este grupo apresentou valores de dureza Knoop inferiores ao grupo controle. Portanto, os menores valores de dureza podem ser devidos ao menor grau de conversão obtido pelo compósito quando fotoativado por este modo, inviabilizando sua utilização. Os demais grupos não apresentaram valores de resistência diferentes do grupo controle, o que mostra que não foram capazes de diminuir a tensão de contração.

O segundo estudo mostrou que a irradiância durante o primeiro ciclo de foto-ativação quando o modo *soft-start* é utilizado tem influência nos valores de resistência de união, mas não na dureza Knoop do compósito Z250 até 4mm de profundidade. O grupo fotoativado com irradiância de 150 mW/cm^2 apresentou média de resistência de união superior ao grupo controle (CO), mas semelhante aos demais grupos. A irradiância está relacionada com a velocidade em que a reação ocorre. Quanto maior for a irradiância, maior será a velocidade de reação, levando a maior concentração de tensões na interface. Nos grupos fotoativados com baixa irradiância pouca reação deve ter ocorrido nos períodos iniciais de fotoativação. Portanto, a maior parte da reação pode ter ocorrido durante o segundo ciclo de fotoativação, que é realizado com alta irradiância, semelhante ao grupo controle. Já, para os grupos fotoativados com irradiância maior que 150 mW/cm^2, a maior parte da reação deve ter ocorrido durante o primeiro ciclo de fotoativação, numa grande velocidade. Desta forma, não foi possível a liberação das tensões internas, proporcionando valores de resistência de união semelhante ao grupo controle. Entretanto, os valores de dureza Knoop foram semelhantes aos do grupo controle, mostrando que o grau de conversão monomérica foi semelhante para todos os grupos.

O terceiro estudo mostrou que o tempo de exposição para o modo *pulse-delay* teve influência significativa sobre a resistência de união do compósito Z250, mas não teve influência para o modo *soft-start*. Além disso, os espécimes fotoativados pelo modo *pulse-delay* mostraram resistência de união superior comparados ao modo *soft-start*. Quando os espécimes são fotoativados pelo modo *pulse-delay*, logo após o primeiro ciclo de fotoativação com baixa irradiância, existe um tempo de espera antes do segundo ciclo com alta irradiância. Durante este tempo de espera ocorre o relaxamento das cadeias poliméricas e a liberação das tensões de contração internamente no

compósito. Já os espécimes fotoativados pelo modo *soft-start*, imediatamente após o primeiro ciclo de fotoativação os espécimes são fotoativados com alta irradiância. Deste modo não há tempo para a “acomodação” das cadeias poliméricas, e as tensões são concentradas na interface dente-material restaurador, diminuindo os valores de resistência de união. Para o modo *soft-start* o tempo de exposição não influenciou nos valores de resistência de união, porque não há tempo para a “acomodação” das cadeias poliméricas, diferentemente do modo *pulse-delay*.

Assim, através dos resultados obtidos neste estudo, pode-se concluir que a modulação da luz emitida pelos aparelhos de lâmpada halógena de quartzo-tungstênio é um método simples e eficaz para o clínico minimizar a formação de tensões durante a fotoativação dos compósitos. A maneira como se faz essa modulação pode fornecer resultados superiores ou inferiores ao método convencional.

CONCLUSÃO GERAL

Dentro das limitações deste estudo pode-se concluir que:

1 - A variação no tempo inicial de exposição da luz para o método de fotoativação *pulse-delay* com aparelho de lâmpada halógena influenciou na resistência de união e na dureza Knoop do compósito Z250.

2 – A melhor alternativa de tempo inicial de fotoativação para essa técnica foi o de 5 segundos.

3 - A variação no tempo inicial de exposição da luz para o método de fotoativação *soft-start* com aparelho de lâmpada halógena não influenciou na resistência de união do compósito Z250.

4 – A variação na irradiância inicial para o método de fotoativação *soft-start* influenciou na resistência de união, entretanto não afetou os valores de dureza Knoop do compósito Z250.

5 – A melhor irradiância para o modo *soft-start* foi com pulso inicial de 150 mW/cm^2 e final de 700 mW/cm^2.

6 – O modo *pulse-delay* mostrou valores de resistência de união superiores comparados ao modo *soft-start*.

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Prezado Doutor,

Acusamos o recebimento do artigo **EFFECT OF DIFFERENT INITIAL LIGHT INTENSITY BY THE SOFT-START PHOTO-ACTIVATION TECHNIQUE ON THE BOND STRENGTH AND KNOOP HARDNESS OF DENTAL COMPOSITE.** Informamos que seu trabalho será enviado para avaliação do corpo editorial. Para esclarecimento posterior o número de registro do seu trabalho é BDJ 845.

Atenciosamente,

Prof. Dr. Manoel D. Sousa Neto
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