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**INFLUÊNCIA DO PRÉ-AQUECIMENTO E DO MODO DE
ATIVÇÃO NA RESISTÊNCIA MÁXIMA À TRAÇÃO E NO GRAU
DE CONVERSÃO DE DOIS CIMENTOS RESINOSOS DE DUPLA
ATIVÇÃO**

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NA RESISTÊNCIA MÁXIMA À TRAÇÃO E NO GRAU DE CONVERSÃO
DE DOIS CIMENTOS RESINOSOS DE DUPLA ATIVAÇÃO**

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Orientador: Prof. Dr. César Augusto Galvão Arrais

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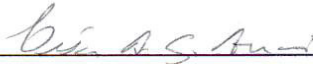
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*“Feliz aquele que transfere o que sabe e aprende o que
ensina”*

Cora Coralina

RESUMO

Esse estudo avaliou o grau de conversão (GC) utilizando análise por infravermelho com transformada de Fourier (FTIR) e teste de resistência máxima à tração (RMT) de cimentos resinosos (CR) de cura dual, pré aquecidos e expostos a diferentes modos de ativação: fotoativação (FA) através de uma lâmina de vidro (LV) ou através de um disco de resina composta cor A2 (A2), ou autoativação (AA). Dois CRs duais foram usados: Rely X ARC (RX) e Variolink II (VII). Corpos-de-prova em forma de disco (1,0 mm de espessura e 5,0 mm de diâmetro) foram obtidos para a análise do GC enquanto espécimes em forma de ampulheta (1,0 mm de espessura e 1,0 mm de largura na região de constrição) foram criados para o teste de RMT, através de um molde vazado de silicone de adição. Os CR foram manipulados a 25° C ou foram aquecidos a 50° C previamente à manipulação. Para os grupos LV e A2, os espécimes foram expostos a luz por 40 segundos. Todos os espécimes (n=10) foram armazenados secos, ao abrigo da luz, a 37° C, por 15 dias. Para a análise de GC, o material foi colocado no molde, sobre o diamante da mesa horizontal de reflectância total atenuada (ATR), conectada na unidade óptica do espectrofotômetro FTIR. Espectros de FTIR (n=5) foram coletados entre as bandas 1680 e 1500 cm^{-1} (resolução de 4- cm^{-1}) e o GC foi calculado pelo método padrão utilizando as alterações nas razões entre as alturas dos picos alifáticos a aromáticos de C=C antes e após polimerização. Para os testes de RMT, os espécimes foram testados em tensão em uma máquina universal de ensaio (velocidade de tração de 1,0 mm/min) até o momento da fratura. Os dados de GC e RMT foram submetidos aos testes ANOVA de dois fatores, seguidos pelo teste de Tukey ($\alpha=5\%$). Ambos os CRs mostraram mais alto GC a 50° C do que a 25° C. Os valores de GC variaram de acordo com o modo de ativação para ambas as temperaturas para RX e apenas a 25° C para VII. Não foram encontradas diferenças significantes nos valores de GC entre os grupos LV e A2 para VII a 50° C. Para o teste de RMT, não foram notadas diferenças significantes entre os grupos A2 a 25° C e A2 a 50° C. Os grupos AA mostraram mais alta RMT a 50° C que a 25° C ($p<0.05$), mas os grupos AA exibiram mais baixos valores de RMT do que os grupos fotoativados, em ambas as temperaturas ($p<0.05$). Concluiu-se que o pré-aquecimento aumentou o GC dos cimentos resinosos em todos os modos de ativação e aumentou a RMT apenas quando os cimentos foram autopolimerizados.

Palavras chaves: cimentos resinosos duais, temperatura, grau de conversão, resistência máxima à tração.

ABSTRACT

This study evaluated the degree of conversion (DC) using Fourier transformed infrared analysis (FTIR) and ultimate tensile strength (UTS) of pre-heated dual-cured resin cements (RCs) exposed to different activating modes: light-activation through a glass slide (GS) or through a pre-cured 2-mm thick resin composite disc (A2 - A2 shade), or specimens were allowed to self-cure (SC). Two dual-cured RCs were used: Rely X ARC (RX) and Variolink II (VII). Disc-shaped specimens (1 mm thick and had 5 mm in diameter) were obtained for DC analysis while hourglass shape specimens (1 mm thick and 1 mm wide at the constriction region) were created for UTS test from addition silicon mold. The RCs were manipulated at 25°C or were pre-heated to 50°C prior to manipulation. For GS and A2 groups, specimens were exposed to light for 40 seconds. All specimens (n=10) were stored dry in the dark at 37°C for 15 days. For FTIR analysis, the material was placed into the mold on the center of a horizontal diamond ATR element in the optical bench of a FTIR spectrometer. FTIR spectra (n=5) were collected between 1680 and 1500 cm⁻¹ (4-cm⁻¹ resolution) and DC was calculated by standard methods using changes in the ratios of aliphatic-to-aromatic C=C absorption peaks from uncured and cured states. For UTS test, specimens were tested in tension on a universal testing machine (crosshead speed of 1 mm/min) until failure. DC and UTS data were submitted to 2-way ANOVA, followed by post-hoc Tukey's test (pre-set alpha of 5%). Both RCs showed higher DC at 50°C than at 25°C. In RX at 25°C and 50°C and in VII at 25°C, DC values depended on the activating mode, while VII did not show significant difference in DC values between GS and A2 groups at 50°C. No significant difference in UTS values was noted between A2 groups at 25°C and A2 groups at 50°C. SC groups showed higher UTS at 50°C than at 25°C (p<0.05), but SC groups exhibited lower UTS values than light-activated groups at both temperatures (p<0.05). The pre-heating increased the DC of RCs in all activating modes and improved UTS only when RCs depended on the self-curing mode.

Keywords: dual-cured resin cements; curing temperature; degree of conversion; ultimate tensile strength; polymerization.

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1. INTRODUÇÃO E JUSTIFICATIVA

Em Odontologia restauradora, cada vez mais, técnicas menos invasivas de preparo dental tem sido preconizadas (BELSER et al.,1997), no sentido de preservar ao máximo a estrutura dental remanescente e reduzir a possibilidade de danos pulpares, proporcionando ao clínico a oportunidade de resultados funcionais e estéticos com maior longevidade.

As técnicas adesivas vem evoluindo continuamente desde que Buonocore em 1955 introduziu o condicionamento ácido do esmalte (BUONOCORE, 1955). Juntamente com a evolução dos procedimentos adesivos, os materiais odontológicos evoluíram consideravelmente, alterando o conceito de macro para microrretenções. Com o desenvolvimento dos cimentos adesivos e dos materiais restauradores, hoje podemos contar com um embricamento mecânico e químico entre dente e restauração. A evolução dos materiais restauradores e dos sistemas de adesão permite-nos maior durabilidade e previsibilidade de resultados (FAHL, 2000), bem como um trabalho restaurador de melhor qualidade técnica e estética, reproduzindo a beleza natural dos dentes.

No mercado odontológico encontra-se grande variedade de materiais usados na cimentação de peças protéticas, tais como os cimentos de fosfato de zinco, cimento de policarboxilato de zinco, cimento de ionômero de vidro, cimento de ionômero de vidro modificado por resina, e os cimentos resinosos. O cimento de fosfato de zinco é o material que vem sendo utilizado por maior tempo entre os agentes cimentantes. Esse cimento não apresenta adesão à estrutura dental, e a retenção da prótese se dá por embricamento mecânico do cimento às paredes cavitárias. O cimento de policarboxilato de zinco foi o primeiro sistema de cimentos desenvolvido com características adesivas às estruturas dentais, unindo-se quimicamente ao dente. Posteriormente, um outro material, o ionômero de vidro, que por meio da quelação entre grupos carboxílicos com os íons cálcio e fosfato da estrutura dental, também apresentou características adesivas, além de potencial bacteriostático e biocompatibilidade, reduzindo a infiltração dos fluidos bucais na interface dente/restauração. Esse cimento foi então modificado com resina, na expectativa de aumentar sua resistência mecânica e diminuir sua solubilidade nos

meios bucais, mas ainda continuava um produto com baixo apelo estético (DIAZ-ARNOLD et al., 1999; WILSON et al., 1983).

Com o desenvolvimento dos sistemas adesivos, as restaurações indiretas de sistemas cerâmicos ou resinas compostas passaram a ser utilizadas com grande frequência pelos clínicos. A segurança oferecida por estes materiais é atribuída às suas ótimas características mecânicas e estéticas, bem como à utilização de cimentos resinosos juntamente com os sistemas adesivos que garantem a adesão. Os cimentos resinosos têm como função promover uma união estável entre dente e restauração, com biocompatibilidade, boa adaptação marginal, livre de infiltração e com baixa solubilidade (MEYER et al., 1998).

Atualmente, os cimentos resinosos são cada vez mais utilizados na clínica odontológica, uma vez que apresentam baixa solubilidade, consistência e espessura de filme adequadas, propriedades mecânicas superiores às dos cimentos anteriormente utilizados e ótima adesividade às estruturas dentais e às restaurações, através de embricamento químico e mecânico, o que garante menor microinfiltração (VAN GROENINGEN et al., 1985; BELSER et al., 1997; BRAGA et al., 2002; DIAZ-ARNOLD et al., 1999).

Os cimentos resinosos tem sua constituição química bastante semelhante às resinas compostas, podendo ser considerados resinas compostas modificadas, diferindo apenas no tipo, tamanho e quantidade das partículas ou conteúdo de carga inorgânica e nas concentrações de monômeros diluentes (DIAZ-ARNOLD et al., 1999). Estes cimentos podem ser classificados de acordo com o modo de ativação que oferecem por meio de variações em sua formulação, podendo ser então classificados como: quimicamente ativados, fotoativados ou duais. Os quimicamente ativados são utilizados para cimentar peças protéticas que não podem ser atravessadas pela luz do fotopolimerizador. Apresentam como desvantagem um tempo de trabalho curto e sem possibilidade de controle do clínico. Os fotoativados por sua vez são amplamente utilizados para a cimentação de facetas e *inlays* estéticos com espessura fina. Possuem amplo tempo de trabalho e melhor estabilidade de cor do que os químicos (PEUTZFELDT, 1995). Os cimentos duais têm sua polimerização iniciada pela luz, porém a mesma pode ser complementada pela ativação química quando a luz fotoativadora é atenuada pela presença da restauração indireta. Desta forma, este tipo de cimento resinoso foi desenvolvido

para garantir uma polimerização adequada em áreas profundas onde a luz fotoativadora não consegue atingir.

Os cimentos duais apresentam em sua composição fotoiniciadores como as cetonas aromáticas (canforoquinona) e iniciadores químicos, como o peróxido de benzoíla. Diversos estudos têm avaliado o seu grau de polimerização, seu grau de conversão, a relação entre esses parâmetros e as características físicas e mecânicas do cimento (BRAGA et al., 2002, CAUGHMAN et al., 2001, EL-MOWAFY & RUBO, 2000, EL-MOWAFY et al., 1999, PEUTZFELDT, 1995). A grande maioria destes estudos tem demonstrado que o mecanismo autopolimerizável é sempre menos efetivo do que o fotopolimerizável (BLACKMAN et al., 1990, CARDASH et al., 1993, CAUGHMAN et al., 2001, EL-MOWAFY & RUBO, 2000, EL-MOWAFY et al., 1999, HASEGAWA et al., 1991, HOFMANN et al., 2001, LEE et al., 2008).

As características físicas e mecânicas dos compósitos resinosos utilizados na Odontologia restauradora são influenciadas pelo seu grau de conversão, que consiste na conversão das ligações duplas alifáticas de carbono dos monômeros em ligações simples covalentes de carbono para a formação da cadeia polimérica (FERRACANE, 1985). Estudos indicam que um aumento da temperatura da resina composta pode resultar em aumento no seu grau de conversão, diminuindo o tempo necessário de exposição à luz ou o tempo necessário para sua polimerização (DARONCH et al., 2005^a, DARONCH et al., 2006^b). Um aumento no grau de conversão pode produzir um aumento na dureza superficial, na resistência à fratura, em maior módulo de flexão, em melhora da resistência à tensão diametral e em mais alta resistência ao desgaste (LOVELL et al., 1999). Desta forma, novos equipamentos têm surgido no mercado visando o aquecimento (de 54° C a 60° C) das resinas compostas antes de sua aplicação clínica. Esse aquecimento aumenta a mobilidade dos radicais melhorando seu grau de polimerização, como resultado da diminuição da viscosidade do material. Além disso, o mesmo processo poderia também melhorar a adaptação dos materiais resinosos às paredes das cavidades, pela diminuição da viscosidade do material resinoso, diminuindo com isso a microinfiltração (DARONCH et al., 2005^a).

Considerando-se que os cimentos resinosos duais dificilmente são expostos à luz fotoativadora com intensidade adequada para iniciar a fotoativação, devido à interposição da peça protética ou da estrutura dental entre a fonte de luz e o

cimento, e que a auto-polimerização destes cimentos não tem a mesma efetividade da polimerização na presença de luz, é possível que o pré-aquecimento de cimentos duais permita maior conversão destes cimentos, resultando na melhora de suas propriedades mecânicas e conseqüentemente maior longevidade clínica. No entanto, os estudos avaliando os efeitos do pré-aquecimento de cimentos resinosos duais em suas propriedades mecânicas são escassos. (CANTORO et al., 2009; CANTORO et al., 2008).

2. PROPOSIÇÃO

Os objetivos desse estudo foram analisar os efeitos do pré-aquecimento de cimentos resinosos duais no seu grau de conversão, e na resistência máxima à tração.

3. METODOLOGIA E RESULTADOS

A presente dissertação está baseada no artigo “Pre-Heated Dual-Cured Resin Cements: Analysis of the Degree of Conversion and Ultimate Tensile Strength in Dual- and Auto- Polymerizing Modes”

PRE-HEATED DUAL-CURED RESIN CEMENTS: ANALYSIS OF THE DEGREE OF CONVERSION AND ULTIMATE TENSILE STRENGTH IN DUAL- AND AUTO-POLYMERIZING MODES

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Running Title: Degree of conversion and ultimate tensile strength of pre-heated resin cements

Abstract

This study evaluated the degree of conversion (DC) using Fourier transformed infrared analysis (FTIR) and ultimate tensile strength (UTS) of pre-heated dual-cured resin cements (RCs) exposed to different activating modes: light-activation through a glass slide (GS) or through a pre-cured 2-mm thick resin composite disc (A2 - A2 shade), or specimens were allowed to self-cure (SC). Two dual-cured RCs were used: Rely X ARC (RX) and Variolink II (VII). Disc-shaped specimens (1 mm thick and had 5 mm in diameter) were obtained for DC analysis while hourglass shape specimens (1 mm thick and 1 mm wide at the constriction region) were created for UTS test from addition silicon mold. The RCs were manipulated at 25°C or were pre-heated to 50°C prior to manipulation. For GS and A2 groups, specimens were exposed to light for 40 seconds. All specimens (n=10) were stored dry in the dark at 37°C for 15 days. For FTIR analysis, the material was placed into the mold on the center of a horizontal diamond ATR element in the optical bench of a FTIR spectrometer. FTIR spectra (n=5) were collected between 1680 and 1500 cm⁻¹ (4-cm⁻¹ resolution) and DC was calculated by standard methods using changes in the ratios of aliphatic-to-aromatic C=C absorption peaks from uncured and cured states. For UTS test, specimens were tested in tension on a universal testing machine (crosshead speed of 1 mm/min) until failure. DC and UTS data were submitted to 2-way ANOVA, followed by post-hoc Tukey's test (pre-set alpha of 5%). Both RCs showed higher DC at 50°C than at 25°C. In RX at 25°C and 50°C and in VII at 25°C, DC values depended on the activating mode, while VII did not show significant difference in DC values between GS and A2 groups at 50°C. No significant difference in UTS values was noted between A2 groups at 25°C and A2 groups at 50°C. SC groups showed higher UTS at 50°C than at 25°C (p<0.05), but SC groups exhibited lower UTS values than light-activated groups at both temperatures (p<0.05). The pre-heating increased the DC of RCs in all activating modes and improved UTS only when RCs depended on the self-curing mode.

Keywords: dual-cured resin cements; curing temperature; degree of conversion; ultimate tensile strength.

INTRODUCTION

Cementing systems (bonding agents/resin cements) are the most used materials to bond indirect restoration and tooth structure. The products were developed to establish a stable and reliable bond between the prepared tooth and restorative material.¹ Resin cements (RCs) are modified resin composites once the organic matrix is similar to that from resin composites, while filler particles differ only regarding type, size, and content in comparison to those from resin composites.¹ In comparison to other cements, RCs have some advantages, such as low solubility, improved mechanical properties, bonding to tooth structure, and reduced microleakage.^{2,3}

RCs can be classified according to their activation mode, so three types of RCs are available: light-, self-, and dual-cured RCs. The self-cured RC is indicated for clinical situations where curing light is totally blocked by the indirect restoration, such as metal crowns. As its cure depends on self-curing components, the clinician cannot control its working time. On the other hand, light-cured RCs allow the clinician to control the working time, but the polymerization effectiveness is dependent upon the intensity of the curing light reaching the RC layer. Therefore, the presence of an indirect restoration between curing unit tip and RC during light-activation may compromise the cement monomer conversion as the indirect restoration severely attenuates the light intensity of the curing light reaching the RC layer.^{4,5} For this reason, another type of RC was developed to associate the benefits from light- and self-curing RCs. The so-called dual-cured RCs are capable of polymerizing even at the bottom of deep cavities or where curing light is attenuated by the presence of the indirect restoration.⁶⁻⁸ However, the self-curing mode of dual-cured RCs is less effective than the dual-curing one,^{6,8} so RCs relying solely on the self-curing components have impaired mechanical properties⁹⁻¹¹ and high solubility,^{12,13} which in turn may compromise the longevity of an indirect restoration.

Some studies have shown that pre-heating of resin composites to temperature ranging from 50°C to 60°C increases their degree of conversion (DC) even when they are exposed to curing light for a shorter period than that recommended by manufacturers.^{14,15} As a consequence, the increase in DC results in higher hardness, higher fracture strength, increased flexural strength and modulus, as well as increased resistance to wear.^{16,17} With this regard, some devices were developed to

pre-heat resin composites before they are applied to the tooth cavity. However, only few studies have evaluated the effects of pre-heating on the DC and mechanical properties of dual-cured RCs in simulated clinical conditions.¹⁸⁻²⁰ Therefore, the aim of the current study was to evaluate the effects of pre-heating on the DC and ultimate tensile strength (UTS) of dual-cured RCs subjected to three activation modes: light-activation through glass slide or through a pre-cured A2-shade resin composite disc, or when the RCs were allowed to self-cure. The research hypothesis was that dual-cured resin cements heated to 50°C during polymerization exhibit higher DC than the same products polymerized at 25°C. It was also anticipated that the increase in DC promoted by high temperature results in higher UTS values of dual-cured RCs regardless of the activating mode.

MATERIALS & METHODS

Specimen preparation

Two dual-cured RCs were used: Rely X ARC (3M ESPE, St. Paul, MN, USA) and Variolink II (Ivoclar Vivadent, Bendererstrasse, Schaan, Liechtenstein). The product compositions are described in Table 1. Disc- (1 mm thick and had 5 mm in diameter) and hourglass- (1 mm thick and 1 mm wide at the constriction region) shaped specimens were created from addition silicon mold (Panasil, Kettenbach GmbH & Co., Eschenburg, Germany). Hourglass shaped-specimens were used for UTS test, while disc shaped-specimens were used for DC analysis.

The products were used at 25±1°C or were heated to 50±1°C prior to and during polymerization. For experimental groups involving pre-heating of RCs, base and catalyst pastes were equally dispensed on a glass plate resting on a heating stirrer surface (Cientec, Piracicaba, SP, Brazil) that was set at 50±1°C. Specimen and glass plate temperatures were constantly measured with a thermocouple (SmartMether, Novus, Porto Alegre, RS, Brazil) to ensure that base and catalyst pastes reached 50°C. For experimental groups without pre-heating, base and catalyst pastes were equally dispensed on a glass plate at 25°C. In specimen preparation for UTS test, a Mylar strip was placed between mold and heated surface. The mold was then filled with the cement at 50°C. The mold allowed direct contact between the heated RC and Mylar strip, so the specimen temperature was maintained during the polymerization. Specimens at room temperature and pre-

heated specimens were subjected to three different activating modes: light activation through a glass slide (GS) or through a pre-cured 2-mm thick resin composite disc (A2 - A2 shade, TPH Spectrum, Dentsply Caulk, Milford, DE, USA) (ANEXO 2) , or specimens were allowed to self-cure (SC). In GS and A2 groups, both glass slide and resin disc were left on the heated plate prior to specimen fabrication, so specimen, glass slide, and indirect restoration had the same temperature during specimen fabrication. The specimen was covered with another Mylar strip to avoid any contact with Oxygen and each side of the specimen was exposed to 20 s of light from light-polymerizing unit (power density: 600 mW/cm², Optilux 501; Demetron Kerr, Danbury, CA, USA), which had the light intensity constantly measured with a radiometer (Cure Rite, Dentsply Caulk). All pre-heated specimens were left on the heated plate for 5 min and were protected from light during this period. All specimens were stored dry in the dark at 37°C for 15 days to ensure maximum polymerization. Ten specimens were prepared for each experimental group (n=10).

Table 1 - Brand, composition and batch number of the dual-cured resin cements used.

| Product (Manufacturer) | Composition (Batch Number) |
|--|--|
| Variolink II (Ivoclar -Vivadent) | Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments Bis-GMA; TEGDMA; UDMA; benzoyl peroxide. (Base: L46354; Catalyst: L 36656) |
| RelyX ARC (3M ESPE, St. Paul, Minn) | Paste A: Silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer Paste B: Silane-treated ceramic, TEGDMA, bis-GMA, silane treated silica, functionalized dimethacrylate polymer (GE9JG) |

TEGDMA: triethylene glycol dimethacrylate; bis-GMA: bisphenol A diglycidyl ether methacrylate; UDMA: urethane dimethacrylate

The specimen was covered with another Mylar strip to avoid any contact with Oxygen and each side of the specimen was exposed to 20 s of light from light-polymerizing unit (power density: 600 mW/cm^2 , Optilux 501; Demetron Kerr, Danbury, CA, USA), which had the light intensity constantly measured with a radiometer (Cure Rite, Dentsply Caulk). All pre-heated specimens were left on the heated plate for 5 min and were protected from light during this period. All specimens were stored dry in the dark at 37°C for 15 days to ensure maximum polymerization. Ten specimens were prepared for each experimental group ($n=10$).

Degree of conversion

For the DC analysis, the mold was placed on the center of a horizontal diamond ATR element (Golden Gate, Specac, Woodstock, GA, USA) in the optical bench of a FTIR spectrometer (Tensor 27, Bruker Optik GmbH; Ettlingen; Germany), so the mold allowed the diamond surface to remain exposed (ANEXO 1). The RCs at 25°C were manipulated according to the manufacturer's instructions and were applied into the mold to create 1-mm thick specimens with the bottom in contact with the diamond surface. For pre-heated groups, the RCs were heated at 50°C as previously described and the diamond surface was also heated with a custom-made heater device. RCs were applied to the diamond surface and were polymerized according to the activating modes described above ($n=5$). For GS and A2 groups, both resin composite disc and glass slide were pre-heated to 50°C . Infrared spectrum from specimen in the uncured state was collected between 1680 and 1500 cm^{-1} at 4 cm^{-1} resolution. For pre-heated SC groups, the RCs were left on the heated diamond surface for 5 min. All specimens were stored in dry condition at 37°C in the dark for 15 days. Afterwards, the cured specimens were placed on the horizontal diamond ATR element and new FTIR spectra were obtained from cured specimens. Monomer conversion was calculated by standard methods using changes in the ratios of aliphatic-to-aromatic C=C absorption peaks in the uncured and cured states obtained from the infrared spectra.^{21,22} The 15-day DC data were submitted to 2-way ANOVA ("temperature", and "activating mode" factors) for each product followed by post-hoc Tukey's test at a pre-set alpha of 5%.

Ultimate tensile strength test

For UTS test, each hourglass-shaped specimen was attached to the grips of a microtensile testing jig with cyanoacrylate (Loctite Super Bonder Gel, Henkel, Düsseldorf, Germany) and was tested in tension on a universal testing machine (EZ Test, Shimadzu Co, Kyoto, Japan) at a crosshead speed of 1 mm/min until failure. After testing, specimens were carefully removed from the fixtures with a scalpel blade and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital micrometer (Series 406; Mitutoyo America Corp., IL, USA). Specimen cross-sectional area was calculated in order to present UTS data in units of stress: MPa. Once the aim of the current study was not to compare products, data were submitted to 2-way ANOVA (“temperature” and “activating mode” factors) for each product followed by post-hoc Tukey’s test at a pre-set alpha of 5% (ANEXO 3).

RESULTS

Degree of conversion

The DC values are displayed on Tables 2 and 3. Significant double interaction was detected between “light-activation” and “temperature” factors for both Rely X ARC ($p=0.0028$) and Variolink II ($p=0.001$). Both pre-heated RCs showed higher DC values than the RCs polymerized at 25°C ($p<0.05$), regardless of the activation mode. However, GS and A2 groups exhibited higher DC values than SC groups at both temperatures ($p<0.05$).

Table 2 - DC means (standard deviation) of Rely X ARC according to curing mode and temperature (%)

| | Light-cured through glass slide | Light-cured through A2-shade RC | Self-cured |
|------|------------------------------------|------------------------------------|---------------|
| 25°C | 68.2 (1.6) Ba | 63.0 (3.5) Bb | 44.5 (5.2) Bc |
| 50°C | 76.4 (2.3) Aa | 71.7 (3.1) Ab | 61.8 (2.0) Ac |

Means followed by different letters (upper case letters within column; lower case letters within row) are significant different at a pre-set alpha of 5%.

Table 3 - DC means (standard deviation) of Variolink II according to curing mode and temperature (%)

| | Light-cured through glass slide | Light-cured through A2- shade RC | Self-cured |
|------|------------------------------------|-------------------------------------|---------------|
| 25°C | 63.5 (2.5) Ba | 56.3 (3.2) Bb | 45.3(2.7) Bc |
| 50°C | 68.8 (1.2) Aa | 67.0 (1.6) Aa | 60.4 (4.3) Ab |

Means followed by different letters (upper case letters within column; lower case letters within row) are significant different at a pre-set alpha of 5%.

GS groups exhibited higher DC values than A2 groups in both Rely X ARC and Variolink II at 25°C and in Rely X ARC at 50°C ($p < 0.05$). No significant difference in DC values was noted between GS and A2 groups when Variolink II was pre-heated at 50°C. The SC groups showed the lowest DC values regardless of product and temperature ($p < 0.001$).

Ultimate Tensile Strength

The UTS values are shown in Table 3 and 4. Significant double interaction was detected between “light-activation” and “temperature” factors for Variolink II, while no significant interaction was detected for Rely X ARC. The change in temperature had no significant effect in UTS values for Rely X ARC. No significant difference in UTS values were noted between groups polymerized at 25°C and those polymerized at 50°C within either GS or A2 groups for both Variolink II and Rely X ARC. On the other hand, the pre-heated SC groups of Variolink II at 50°C exhibited higher UTS values than the SC groups polymerized at 25°C ($p < 0.05$).

The comparison among activating modes demonstrated no significant difference in UTS values between GS and A2 groups for both RCs at either 25°C or 50°C. In both RCs at both temperatures, the SC groups showed lower UTS values than GS and A2 groups ($p < 0.05$).

Table 4 - UTS means (standard deviation) of Rely X ARC according to curing mode and temperature (MPa)

| | | Light-cured through glass slide (GS) | Light-cured through A2-shade RC (A2) | Self-cured (SC) |
|------------|------|---|---|-----------------|
| Rely X ARC | 25°C | 77.3(9.5) Aa | 72.4(7.1) Aa | 35.7(10.7) Ab |
| | 50°C | 68.5(17.3) Aa | 75.4(8.9) Aa | 45.7(15.2) Ab |

Within products, means followed by different letters (Upper case letters within column; lower case letters within row) are significant different at a pre-set alpha of 5%.

Table 5 - UTS means (standard deviation) of Variolink II according to curing mode and temperature (MPa)

| | | Light-cured through glass slide (GS) | Light-cured through A2-shade RC (A2) | Self-cured (SC) |
|--------------|------|---|---|-----------------|
| Variolink II | 25°C | 75.5(9.7) Aa | 72.6(10.3) Aa | 16.8(6.7) Bb |
| | 50°C | 62.4(18.8) Aa | 78.7(13.5) Aa | 35.6(11.2) Ab |

Within products, means followed by different letters (Upper case letters within column; lower case letters within row) are significant different at a pre-set alpha of 5%.

DISCUSSION

In this study, UTS and DC values of two dual-cured RCs were evaluated at different temperatures and activation modes. The increase in temperature of Rely X ARC and Variolink II resulted in higher DC regardless of activation mode, so the research hypothesis was validated for the DC analysis. Such finding is agreement with other results from pre-heated resin composites^{14,15} and might be attributed to the higher radical mobility and collision frequency of unreacted active groups and radicals due to the decrease in cement viscosity promoted by increasing resin temperature.

Despite the overall increase in DC values of Rely X ARC, the values were still dependent on the activating mode. Therefore, the increased temperature did not compensate for the lower light intensity when pre-cured resin disc was used or for

the lower effectiveness of self-curing components when polymerization depended solely on the self-curing mode. However, it should be emphasized that the DC values of Rely X ARC cured at 50°C through indirect restoration were apparently similar to or even higher than those observed when Rely X ARC was cured through glass slide at 25°C. Thus, it is expected that the longevity of RCs cured at 50°C may be increased as higher DC decreases the material solubility.¹²

When Variolink II was polymerized at 50°C, no significant difference in DC was noted between the LC and A2 groups, while A2 group showed lower DC than LC group at 25°C. This finding might be explained by the difference in DC increase for each group after temperature was changed. Based on the current results, pre-heated LC group exhibited DC values approximately 8.3% higher than the DC of LC group at 25°C, while pre-heated A2 group showed DC values 19% higher than the A2 group at 25°C. Therefore, considering that approximately only 10% of the light intensity reaches the RC after the curing light passes through a 2-mm thick indirect resin composite restoration,⁴ it seems that the effects of increased temperature were more evident when curing light was severely attenuated by the resin disc.

Although the increased temperature resulted in significantly higher DC values in both RCs, no significant difference in UTS values was noted between A2 groups polymerized at 25°C and those polymerized at 50°C. Thus, the second research hypothesis was invalidated for both products. In spite of the evidences that little increase in DC is capable of improving some mechanical properties, such as fracture toughness and flexural strength,²³ it seems that the 8% to 10% increase in DC values had no influence on the UTS of cements light-activated at 50°C. As previously established, mechanical properties of polymers do not depend solely on the DC, but also on the network formation features in these materials.²⁴ Resin cements such as those used in the current study have low filler content and high content of diluents monomers to ensure proper viscosity. At increased temperature, these materials may present fast cure rate when exposed to curing light. As a consequence, the material develops high shrinkage strain and residual stress during polymerization,²⁵ so the UTS was probably not only influenced by the DC but also by the stress level into the material.

The SC groups of Variolink II cured at 50°C exhibited higher UTS than SC groups cured at 25°C. When polymerization is initiated by this activating mode, the

decrease in resin viscosity and increased radical mobility due to increased resin temperature during polymerization were probably not the only factors contributing to the higher DC and UTS values than those from SC groups polymerized at 25°C. The RCs evaluated in this study have benzoyl peroxide as the initiator of the self-curing reaction. Benzoyl peroxide is unstable and may be activated by heat.²⁶ For this reason, increased temperature decomposes benzoyl peroxide on radicals that initiate the polymerization, so benzoyl peroxide decomposes faster at higher temperature. As a consequence, more radicals created more quickly from benzoyl peroxide along with lower resin viscosity at increased temperature were responsible for the higher DC and UTS values. The effects of increased DC on the UTS values in SC groups were observed only in Variolink II probably because of the poor effectiveness of self-curing components at 25°C, which led to low UTS values at 25°C.

The temperature of 50°C was chosen based on other studies^{14,15,27} and was maintained during light-activation for 5 min in the self-cured specimens. This methodology may differ from the clinical situation where a drastic drop in temperature of heated resin composites is observed when they are placed in tooth cavities.²⁸ However, only a controlled laboratorial condition would provide the real effect of increased temperature on DC and UTS values of RCs. Besides, slighter decrease in cement temperature is expected in comparison to the reported temperature drop in resin composites once it was suggested in this study that the RC should be coupled with a heated indirect restoration. With this regard, it is believed that even a slight increase in cement temperature may lead to higher DC and bond strength values when RCs depend solely on its self-curing mode because of the significant effect that temperature has on self-curing components. However, only further studies simulating this clinical condition may confirm such speculation.

Although most studies evaluating pre-heated resin composites and RCs used temperatures of 50°C,^{14,15,18-20} some concern may arise regarding the effects of high temperature on pulp tissue. It has been demonstrated that the increase in temperature of resinous materials to temperatures ranging from 54°C and 60°C does not change intrapulpal temperature significantly.²⁷ However, the effects of heated RCs cements coupled with heated indirect restorations on pulp tissue deserve further evaluation.

Conclusion

The increased temperature of Rely X ARC and Variolink II prior to polymerization promoted higher cement DC regardless of the activating mode. However, the increase in temperature to 50°C only improved the ultimate tensile strength of only Variolink II polymerized by the self-curing mode solely. Although long-term effects of pre-heating dual-cured resin cements are still unknown, the pre-heating of resin cements might be a reliable option when dual-cured resin cements depend on the self-curing mode to bond indirect restorations to tooth.

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4. CONCLUSÃO

Com base nos resultados de espectroscopia infravermelha com transformada de Fourier (FTIR) e nos testes de resistência máxima à tração obtidos por meio de microtração, podemos concluir que o pré-aquecimento dos cimentos resinosos duais Rely X ARC e Variolink II pode ser viável, uma vez que ocorreu um aumento no grau de conversão de ambos os cimentos, independente do modo de ativação utilizado, autoativação ou fotoativação. Mesmo com a atenuação da luz ativadora pelos discos de resina interpostos entre o cimento e a ponta do fotopolimerizador, não foi necessário o aumento do tempo de exposição à luz para que ocorresse a polimerização dos cimentos. A atenuação da luz ativadora não diminuiu a resistência máxima à tração dos cimentos testados, mas o pré-aquecimento aumentou os valores de resistência máxima à tração dos cimentos testados quando eles foram autoativados.

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ANEXOS

ANEXO 1 – Figuras ilustrativas da análise de grau de conversão

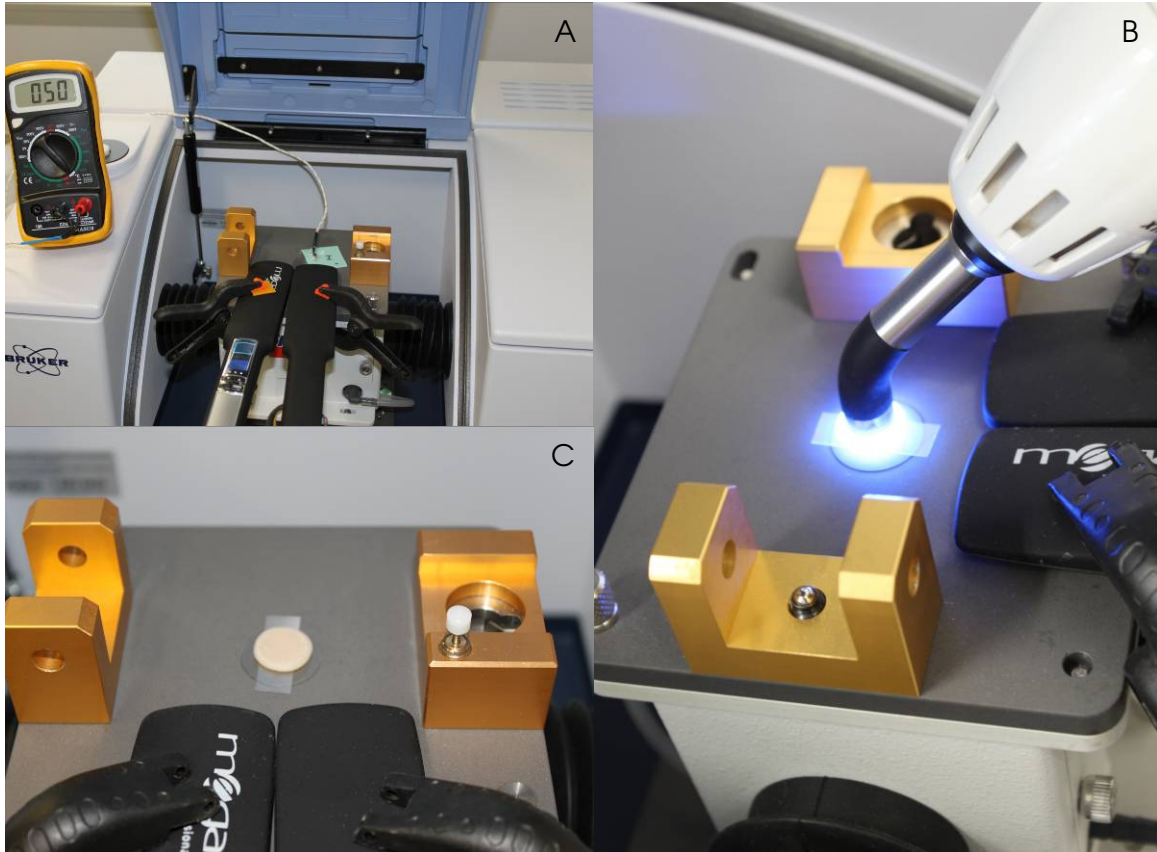


Figura1: Avaliação da temperatura do cimento na mesa de ATR aquecida (A); Aplicação do cimento e posicionamento do disco de resina composta sobre o espécime (B) e fotoativação do espécime (C).

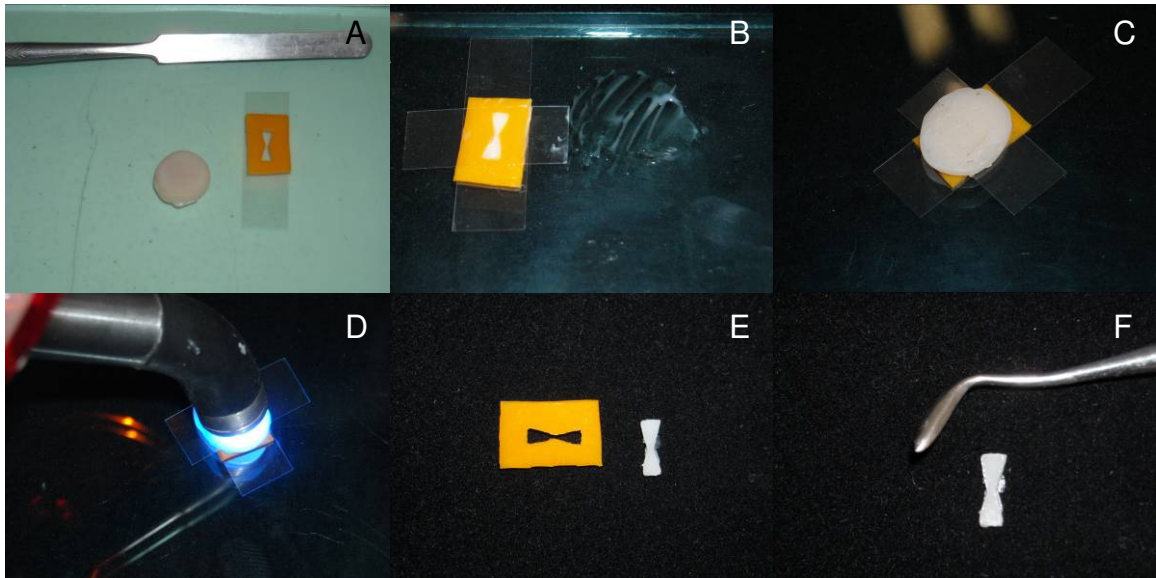
ANEXO 2 - Figuras ilustrativas da análise de resistência máxima de tração

Figura 2: Colocação do molde de silicone e do disco de resina composta sobre a placa aquecida (A); manipulação e aplicação do cimento resinoso aquecido no molde de silicone (B); posicionamento do disco de resina composta sobre o espécime (C); e fotoativação do espécime através do disco (D); ou através de uma lâmina de vidro; espécime removido do molde (E); e remoção de excessos do espécime com instrumento cortante (F).

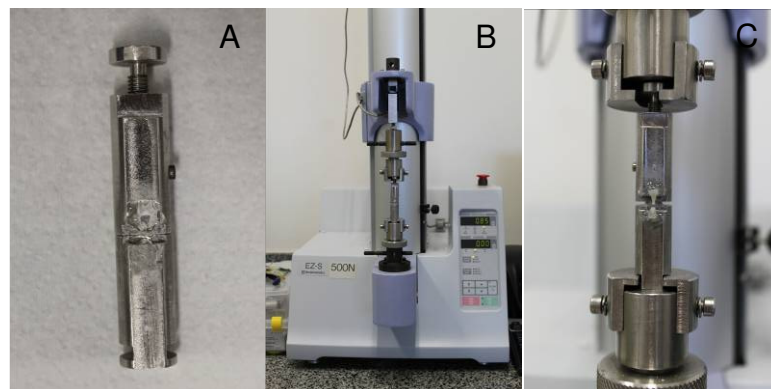


Figura 3: Colagem do espécime no dispositivo de microtração; posicionamento do dispositivo na máquina de ensaio universal (B) e realização do ensaio de tração no espécime (C).

ANEXO 3 – Análise estatística para os resultados de grau de conversão e resistência máxima à tração

 * SANEST - SISTEMA DE ANALISE ESTATISTICA *
 * Autores: Elio Paulo Zonta - Amauri Almeida Machado *
 * Instituto Agronomico de Campinas - I A C *
 * ANALISE DA VARIABEL DC - ARQUIVO: DCRX *

Codigo do projeto: xx

Responsavel: cesar arrais

Delineamento experimental: 2*3

Observacoes nao transformadas

Nome dos fatores

 Fator nome

A temp
 B cmode

Quadro da analise de variancia

| Causas da variacao | g.l. | S.q. | Q.m. | Valor f | prob.>f |
|--------------------|------|--------------|--------------|----------|---------|
| Temp | 1 | 1143.5740915 | 1143.5740915 | 118.0543 | 0.00001 |
| Cmode | 2 | 2328.4351166 | 1164.2175583 | 120.1854 | 0.00001 |
| Tem*cmo | 2 | 142.0485125 | 71.0242562 | 7.3320 | 0.00288 |
| Residuo | 30 | 290.6053415 | 9.6868447 | | |
| Total | 35 | 3904.6630621 | | | |

Media geral = 64.336113

Coeficiente de variacao = 4.838 %

Teste de tukey para medias de temp

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 18 | 69.972224 | 69.972224 | a | a |
| 2 | 1 | 25c | 18 | 58.700000 | 58.700000 | b | b |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
 D.m.s. 5% = 2.12008 - d.m.s. 1% = 2.85368

Teste de tukey para medias de temp

Dentro de foto do fator cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 6 | 76.416667 | 76.416667 | a | a |
| 2 | 1 | 25c | 6 | 68.199997 | 68.199997 | b | b |

Teste de tukey para medias de temp
Dentro de fotoa2 do fator cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 6 | 71.733337 | 71.733337 | a | a |
| 2 | 1 | 25c | 6 | 63.016668 | 63.016668 | b | b |

Teste de tukey para medias de temp
Dentro de auto do fator cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 6 | 61.766668 | 61.766668 | a | a |
| 2 | 1 | 25c | 6 | 44.883336 | 44.883336 | b | b |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
D.m.s. 5% = 3.67209 - d.m.s. 1% = 4.94271

Teste de tukey para medias de cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|--------|------------|-----------|------------------|----|----|
| 1 | 1 | foto | 12 | 72.308332 | 72.308332 | a | a |
| 2 | 2 | fotoa2 | 12 | 67.375003 | 67.375003 | b | b |
| 3 | 3 | auto | 12 | 53.325002 | 53.325002 | c | c |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
D.m.s. 5% = 3.13564 - d.m.s. 1% = 4.00715

Teste de tukey para medias de cmode
Dentro de 25c do fator temp

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|--------|------------|-----------|------------------|----|----|
| 1 | 1 | foto | 6 | 68.199997 | 68.199997 | a | a |
| 2 | 2 | fotoa2 | 6 | 63.016668 | 63.016668 | b | a |
| 3 | 3 | auto | 6 | 44.883336 | 44.883336 | c | b |

Teste de tukey para medias de cmode
Dentro de 50c do fator temp

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|--------|------------|-----------|------------------|----|----|
| 1 | 1 | foto | 6 | 76.416667 | 76.416667 | a | a |
| 2 | 2 | fotoa2 | 6 | 71.733337 | 71.733337 | b | a |
| 3 | 3 | auto | 6 | 61.766668 | 61.766668 | c | b |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
D.m.s. 5% = 4.43446 - d.m.s. 1% = 5.66696
diferem entre si ao nivel de significancia indicado
D.m.s.

 * sanest - sistema de analise estatistica *
 * autores: elio paulo zonta - amauri almeida machado *
 * instituto agronomico de campinas - i a c *
 * analise da variavel dc - arquivo: variofra *

Codigo do projeto: 2x

Responsavel: cesar arrais

Delineamento experimental: 2x3

Observacoes nao transformadas

Nome dos fatores

 Fator nome

 A temp
 B cmode

Quadro da analise de variancia

| Causas da variacao | g.l. | S.q. | Q.m. | Valor f | prob.>f |
|--------------------|------|--------------|-------------|----------|---------|
| Temp | 1 | 964.0957979 | 964.0957979 | 125.2474 | 0.00001 |
| Cmode | 2 | 1099.4679245 | 549.7339623 | 71.4169 | 0.00001 |
| Tem*cmo | 2 | 141.7467927 | 70.8733963 | 9.2073 | 0.00105 |
| Residuo | 30 | 230.9260140 | 7.6975338 | | |
| Total | 35 | 2436.2365292 | | | |

Media geral = 60.219444

Coefficiente de variacao = 4.607 %

Teste de tukey para medias de temp

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 18 | 65.394443 | 65.394443 | a | a |
| 2 | 1 | 25c | 18 | 55.044442 | 55.044442 | b | b |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
 D.m.s. 5% = 1.88989 - d.m.s. 1% = 2.54384

Teste de tukey para medias de temp

Dentro de glass do fator cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 6 | 68.799998 | 68.799998 | a | a |
| 2 | 1 | 25c | 6 | 63.483327 | 63.483327 | b | b |

Teste de tukey para medias de temp

Dentro de a2 shade do fator cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 6 | 67.033330 | 67.033330 | a | a |
| 2 | 1 | 25c | 6 | 56.316666 | 56.316666 | b | b |

Teste de tukey para medias de temp
Dentro de self do fator cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|------|------------|-----------|------------------|----|----|
| 1 | 2 | 50c | 6 | 60.350001 | 60.350001 | a | a |
| 2 | 1 | 25c | 6 | 45.333333 | 45.333333 | b | b |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
D.m.s. 5% = 3.27339 - d.m.s. 1% = 4.40605

Teste de tukey para medias de cmode

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|----------|------------|-----------|------------------|----|----|
| 1 | 1 | glass | 12 | 66.141663 | 66.141663 | a | a |
| 2 | 2 | a2 shade | 12 | 61.674998 | 61.674998 | b | b |
| 3 | 3 | self | 12 | 52.841667 | 52.841667 | c | c |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
D.m.s. 5% = 2.79519 - d.m.s. 1% = 3.57207

Teste de tukey para medias de cmode
Dentro de 25c do fator temp

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|----------|------------|-----------|------------------|----|----|
| 1 | 1 | glass | 6 | 63.483327 | 63.483327 | a | a |
| 2 | 2 | a2 shade | 6 | 56.316666 | 56.316666 | b | b |
| 3 | 3 | self | 6 | 45.333333 | 45.333333 | c | c |

Teste de tukey para medias de cmode
Dentro de 50c do fator temp

| Num.ordem | num.trat. | Nome | num.repet. | Medias | medias originais | 5% | 1% |
|-----------|-----------|----------|------------|-----------|------------------|----|----|
| 1 | 1 | glass | 6 | 68.799998 | 68.799998 | a | a |
| 2 | 2 | a2 shade | 6 | 67.033330 | 67.033330 | a | a |
| 3 | 3 | self | 6 | 60.350001 | 60.350001 | b | b |

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado
D.m.s. 5% = 3.95299 - d.m.s. 1% = 5.05167
diferem entre si ao nivel de significancia indicado
D.m.s. 5% =

The GLM Procedure

Class Level Information

| Class | Levels | Values |
|-------|--------|--------|
| temp | 2 | 1 2 |
| activ | 3 | 1 2 3 |

Number of observations 60

The GLM Procedure

Dependent Variable: mpa1

| Source | DF | Sum of Squares | Mean Square | F Value | Pr > F |
|-----------------|----|----------------|-------------|---------|--------|
| Model | 5 | 187.0934603 | 37.4186921 | 52.04 | <.0001 |
| Error | 54 | 38.8305387 | 0.7190840 | | |
| Corrected Total | 59 | 225.9239989 | | | |

| | | | |
|----------|-----------|----------|-----------|
| R-Square | Coeff Var | Root MSE | mpa1 Mean |
| 0.828126 | 11.62997 | 0.847988 | 7.291406 |

| Source | DF | Type I SS | Mean Square | F Value | Pr > F |
|------------|----|-------------|-------------|---------|--------|
| temp | 1 | 3.0543099 | 3.0543099 | 4.25 | 0.0441 |
| activ | 2 | 165.4411439 | 82.7205720 | 115.04 | <.0001 |
| temp*activ | 2 | 18.5980065 | 9.2990033 | 12.93 | <.0001 |

| Source | DF | Type III SS | Mean Square | F Value | Pr > F |
|------------|----|-------------|-------------|---------|--------|
| temp | 1 | 3.0543099 | 3.0543099 | 4.25 | 0.0441 |
| activ | 2 | 165.4411439 | 82.7205720 | 115.04 | <.0001 |
| temp*activ | 2 | 18.5980065 | 9.2990033 | 12.93 | <.0001 |

The GLM Procedure
Least Squares Means
Adjustment for Multiple Comparisons: Tukey

| | | LSMEAN | |
|------|-------|-------------|--------|
| temp | activ | mpa1 LSMEAN | Number |
| 1 | 1 | 8.67311625 | 1 |
| 1 | 2 | 8.50181330 | 2 |
| 1 | 3 | 4.02242296 | 3 |
| 2 | 1 | 7.81947800 | 4 |
| 2 | 2 | 8.84210816 | 5 |
| 2 | 3 | 5.88949672 | 6 |

Least Squares Means for effect temp*activ
Pr > |t| for H0: LSMean(i)=LSMean(j)

Dependent Variable: mpa1

| i/j | 1 | 2 | 3 | 4 | 5 | 6 |
|-----|--------|--------|--------|--------|--------|--------|
| 1 | | 0.9975 | <.0001 | 0.2323 | 0.9977 | <.0001 |
| 2 | 0.9975 | | <.0001 | 0.4748 | 0.9455 | <.0001 |
| 3 | <.0001 | <.0001 | | <.0001 | <.0001 | 0.0001 |
| 4 | 0.2323 | 0.4748 | <.0001 | | 0.0926 | <.0001 |
| 5 | 0.9977 | 0.9455 | <.0001 | 0.0926 | | <.0001 |
| 6 | <.0001 | <.0001 | 0.0001 | <.0001 | <.0001 | |

The GLM Procedure

Class Level Information

| Class | Levels | Values |
|-------|--------|--------|
|-------|--------|--------|

| | | |
|------|---|-----|
| temp | 2 | 1 2 |
|------|---|-----|

| | | |
|-------|---|-------|
| activ | 3 | 1 2 3 |
|-------|---|-------|

Number of observations 60

The GLM Procedure

Dependent Variable: mpa

| Source | Sum of | | Mean Square | F Value | Pr > F |
|-----------------|--------|-------------|-------------|---------|--------|
| | DF | Squares | | | |
| Model | 5 | 15159.94333 | 3031.98867 | 21.02 | <.0001 |
| Error | 54 | 7790.84000 | 144.27481 | | |
| Corrected Total | 59 | 22950.78333 | | | |

| R-Square | Coeff Var | Root MSE | mpa Mean |
|----------|-----------|----------|----------|
| 0.660541 | 19.22344 | 12.01145 | 62.48333 |

| Source | DF | Type I SS | Mean Square | F Value | Pr > F |
|------------|----|-------------|-------------|---------|--------|
| temp | 1 | 27.74400 | 27.74400 | 0.19 | 0.6628 |
| activ | 2 | 14238.77433 | 7119.38717 | 49.35 | <.0001 |
| temp*activ | 2 | 893.42500 | 446.71250 | 3.10 | 0.0533 |

| Source | DF | Type III SS | Mean Square | F Value | Pr > F |
|------------|----|-------------|-------------|---------|--------|
| temp | 1 | 27.74400 | 27.74400 | 0.19 | 0.6628 |
| activ | 2 | 14238.77433 | 7119.38717 | 49.35 | <.0001 |
| temp*activ | 2 | 893.42500 | 446.71250 | 3.10 | 0.0533 |

The GLM Procedure

Tukey's Studentized Range (HSD) Test for mpa

NOTE: This test controls the Type I experimentwise error rate, but it generally has a higher Type II error rate than REGWQ.

| | |
|-------------------------------------|----------|
| Alpha | 0.05 |
| Error Degrees of Freedom | 54 |
| Error Mean Square | 144.2748 |
| Critical Value of Studentized Range | 3.40824 |
| Minimum Significant Difference | 9.154 |

Means with the same letter are not significantly different.

| Tukey Grouping | Mean | N | activ |
|----------------|--------|----|-------|
| A | 73.870 | 20 | 2 |
| A | | | |
| A | 72.875 | 20 | 1 |
| B | 40.705 | 20 | 3 |