



**CENTRO DE PÓS-GRADUAÇÃO E PESQUISA  
CURSO DE MESTRADO EM ODONTOLOGIA  
ÁREA DE CONCENTRAÇÃO EM DENTÍSTICA**

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**INFLUÊNCIA DA SIMULAÇÃO DA TEMPERATURA DENTAL NO  
GRAU DE CONVERSÃO E CINÉTICA DE POLIMERIZAÇÃO DE  
CIMENTOS RESINOSOS AUTOADESIVOS EXPOSTOS A  
DIFERENTES MODOS DE ATIVAÇÃO**

**GUARULHOS**

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Dissertação apresentada à Universidade Guarulhos para obtenção do título de Mestre em Odontologia, Área de Concentração em Dentística.

Orientador: Prof. Dr. Cesar A. Galvão Arrais  
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**GUARULHOS**

**2012**

C433i CHAGAS, Carolina Levatti  
Influência da simulação da temperatura dental no grau de conversão e cinética de polimerização de cimentos resinosos autoadesivos expostos a diferentes modos de ativação / Carolina Levatti Chagas. Guarulhos, 2012.  
47 f. : il. ; 31 cm


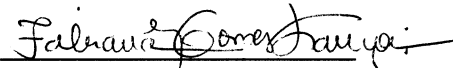

Dissertação (Mestrado em Odontologia) - Centro de pós-graduação e Pesquisa, Universidade Guarulhos, 2012.  
Orientador: Prof. Dr. Cesar A. Galvão Arrais  
Bibliografia: 47 f.

1. Temperatura dental; 2. grau de conversão; 3. polimerização e cimento resinoso; I. Título. II. Universidade Guarulhos.

CDD 22<sup>st</sup> 617.6

A Comissão Julgadora dos trabalhos de Defesa de Dissertação de MESTRADO, intitulada “INFLUÊNCIA DA SIMULAÇÃO DA TEMPERATURA DENTAL NO GRAU DE CONVERSÃO E CINÉTICA DE POLIMERIZAÇÃO DE CIMENTOS RESINOSOS AUTOADESIVOS EXPOSTOS A DIFERENTES MODOS DE ATIVAÇÃO” em sessão pública realizada em 17 de Fevereiro de 2012, considerou a candidata CAROLINA LEVATTI CHAGAS aprovada.

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Guarulhos, 17 de Fevereiro de 2012.

Dedico esta dissertação a Deus e ao meu filho Heitor, que esteve em muitos momentos de sua vida ausente de minha presença e de meu carinho, esperando com muita paciência a minha dedicação e conclusão de um sonho.

Te amo meu filho e muito obrigada.

## Agradecimentos

Agradeço em primeiro lugar ao nosso mestre divino Deus, que me deu a oportunidade da vida e do aprendizado.

Ao Professor César Augusto Galvão Arrais pela extrema eficiência, dedicação como orientador. Em especial pela paciência e profissionalismo na condução desse nosso trabalho.

Ao meu querido mestre e amigo Guto, sendo um exemplo e inspiração para a minha vida, pela sua dedicação inteligência e boa vontade ao ensinar.

Ao Professor André Figueiredo Reis pelo dedicação acadêmica e sua incrível experiência clínica

A professora Alessandra Cassoni pela paciência e dedicação em ensinar .

Aos funcionários da instituição, em especial a Aurea que me auxiliou com tanto carinho

. A todos meus amigos e companheiros de sala e dessa jornada tão intensa de dedicação e trabalho e especial Josi e Micheli que ficamos juntas por 2 anos

.

À **Universidade de Guarulhos**, instituição na qual tive a oportunidade de agregar conhecimentos e valores em minha profissão.

Às coordenadoras do Programa de Pós-graduação em Odontologia da Universidade Guarulhos, **Magda Feres** e **Luciene Cristina de Figueiredo**, pela competência e dedicado trabalho sempre na busca do melhor na condução de todo o curso.

Agradecimentos especiais :

Agradeço ao meu filho Heitor que esta comigo em todos os momentos de minha vida.

Ao meu filho Heitor por acreditar nas minhas escolhas e aceitar a minha ausência para dedicação desse trabalho.

Aos meus avós Lú, Aldo e Maria Igenes Zizo pelo exemplo de vida e amor e empenho na minha vida e orações pra finalização de meus projetos.

A minha Mãe Maisa que me ensinou a ter garra e coragem para enfrentar os mais difíceis obstáculos impostos pela minha vida.

Ao meu irmão Camilo Junior por me tido como meta e determinação nas escolhas de sua profissão, acreditando que tudo pode e é possível, basta acreditar.

A Camila por tanto carinho e cuidado comigo e Heitor.

Aos meus tios Fer e Cidinha pela força e conselhos, a Tia Denise e Gedro por acolherem Heitor na minha ausência.

Ao meu namorado Hildo que dedicou seu tempo para estar comigo nessa finalização de meu trabalho.

A minha secretária Rose, que cuidou tão bem de minha clínica enquanto estive ausente.

Aos meus amigos e pacientes que estiveram do meu lado, me incentivando em não desanimar, em especial Kárytha e Célia.

Obrigada!

“nossa ousadia é do tamanho do nosso futuro”



## RESUMO

Este estudo avaliou o grau de conversão (GC) de duas marcas comerciais de cimentos resinosos autoadesivos (CRA), polimerizados em diferentes valores de temperaturas dentais e sob diversas condições de ativação, utilizando Espectroscopia Infravermelha com Transformada de Fourier (FTIR). RelyX U100 (U100, 3M ESPE) e Maxcem Elite (MX, Kerr Corporation) foram aplicados a 25°C em uma superfície horizontal de uma unidade de reflectância total atenuada (ATR), a 25°C ou aquecidos a 28°C e 32°C e anexado a um espectrômetro infravermelho. Os produtos foram polimerizados usando uma de três condições: a exposição à luz direta através de uma lâmina de vidro (LV), de um disco de 1,5 mm de espessura de cerâmica (EMAX, A2, Ivoclar Vivadent), ou exposto a auto-polimerização (AP). Espectros FTIR foram registrados por 12 min (1 espectro/s, 16 varreduras/espectro, resolução 4 cm<sup>-1</sup>) imediatamente após a aplicação na ATR. O GC foi calculado usando as técnicas padrão observando assim as mudanças nas cadeias alifáticas e aromáticas nos picos de 12 min pré-e pós-polimerização, num intervalo de 1 s. Os dados foram analisados por ANOVA e teste de Tukey (p = 0,05) (n = 7). Nas temperaturas dentais, o GC aumentou significativamente em todos os grupos do MX e nos grupos EMAX e AP do U100. Para MX, o grupo AP exposto à temperatura dental apresentou valores do GC semelhantes aos grupos EMAX. Para U100, grupos EMAX mostraram valores mais elevados do que os grupos AP independentemente da temperatura. O perfil baseado no tempo de conversão variou de acordo com a modalidade do produto, temperatura e tipos de polimerização. A temperatura dental, pode aumentar o GC dos cimentos resinosos auto adesivos mesmo em condições escassas de luz.

**Palavras-chave:** Temperatura dental, grau de conversão, polimerização e cimento resinoso.

## ABSTRACT

This study evaluated the degree of conversion (DC) of two commercial, self-adhesive resin cements (SARCs) polymerized at simulated tooth temperatures and under different curing conditions, using Fourier Transformed Infrared analysis (FTIR). RelyX U100 (U100, 3M ESPE) and Maxcem Elite (MX, Kerr Corporation) were mixed at 25°C and applied to a surface of a horizontal attenuated-total-reflectance unit (ATR), which was at 25°C or heated to 28°C and 32°C, and attached to an infrared spectrometer. The products were polymerized using one of three conditions: direct light exposure through a glass slide (DLE), through a 1.5mm-thick ceramic disc (EMAX, A2 shade, IPS e.max, Ivoclar Vivadent), or were allowed to self-cure (SC). FTIR spectra were recorded for 12 min (1 spectrum/s, 16 scans/spectrum, resolution 4 cm<sup>-1</sup>) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios pre- and 12-min post-curing, as well as during each 1-second interval. DC data (n=7) were analyzed by two-way ANOVA and Tukey's post-hoc test ( $p=0.05$ ). Tooth temperatures significantly increased DC in all groups of MX and in EMAX and SC groups of U100. For MX, SC groups exposed to tooth temperature showed similar DC values to EMAX groups. For U100, EMAX groups showed higher DC values than SC groups regardless of temperature. Time-based conversion profiles ranged according to product, temperature, and curing mode. Tooth temperature can increase DC of SARCs, even in the SC mode.

**Keywords:** resin cements; polymerization; tooth temperature; degree of conversion.

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## 1. INTRODUÇÃO E JUSTIFICATIVA

O sucesso das restaurações indiretas com cerâmicas odontológicas é atribuído à adequada união entre o material restaurador, o cimento resinoso utilizado e os substratos dentais. Em um cimento resinoso, propriedades mecânicas como resistência flexural, dureza e resistência à compressão são cruciais para garantir adequada retenção e longevidade de uma restauração indireta no elemento dental (Inokoshi et al., 1993; Sjogren et al., 1995). Uma vez que tais propriedades mecânicas estão diretamente relacionadas ao grau de conversão (GC) destes materiais (Ferracane, 1985; Ferracane et al., 1998; Lovell et al., 2001), é fundamental que cimentos resinosos sejam capazes de polimerizar adequadamente após o assentamento do elemento protético.

O GC é a medida percentual de ligações duplas de carbono que foram quebradas em ligações simples para formar uma cadeia polimérica (Anusavice, 2005), desta forma quanto maior o GC melhores serão as propriedades mecânicas do material (Imazato et al., 1995; Ruyter e Svendsen, 1978). Uma forma eficaz de se avaliar o GC de um material é a espectroscopia infravermelha com transformada de Fourier (FTIR), capaz de identificar e quantificar as ligações duplas de carbono (C = C) previamente e após a polimerização de compósitos. Para os compósitos usados atualmente no comércio, o GC varia de 35% a 67%, e no sistema fotoativado, o GC diminui à medida que a distância da ponta do fotoativador à superfície aumenta e devido à atenuação da energia radiante que passa através da espessura do material (Sobrinho et al., 2000; Feilzer et al., 1995; ). No caso dos cimentos resinosos, o GC pode ser afetado pela interposição da restauração indireta entre o cimento e a ponta do fotoativador (Arrais et al., 2009). Nestas situações, a luz emitida pelo fotoativador pode ter sua intensidade atenuada em torno de 70% a 90%, dependendo do material e cor da restauração indireta utilizada (Arrais et al., 2009; el mowafy et al., 2004). Por este motivo, as cimentações adesivas de restaurações indiretas são realizadas empregando em sua maioria cimentos de dupla ativação. O cimento de dupla ativação apresenta componentes autopolimerizáveis, o que favorece a conversão de monômeros em polímero mesmo sem a presença de luz (Anusavice, 2005).

A fim de diminuir a sensibilidade pós-operatória e as várias etapas de cimentação, foram introduzidos no mercado os cimentos resinosos autoadesivos (CRA). CRAs são materiais híbridos que combinam as características de resinas

compostas, adesivos autocondicionantes e em alguns casos, cimentos (Ferracane, 2011) Estes cimentos foram projetados para aderir à estrutura dental sem a exigência de um adesivo separado ou condicionamento ácido, ganhando rapidamente em popularidade, com mais de uma dezena de marcas comerciais disponíveis. A principal vantagem destes materiais parece ser a simplicidade na técnica de aplicação. Apresentados na forma de duas pastas ou dois componentes, estes materiais requerem manipulação e mistura, trituração em cápsula ou dosagem em um dispensador com mistura automática. Um componente apresenta monômeros mono-, di-, e/ou multi-funcionais comumente utilizados em uma variedade de materiais restaradores a base de resina. Como exemplo, podemos citar Bis-GMA, UDMA, HEMA, glicerol dimetacrilato (GDMA), trimetilloilpropano trimetacrilato (TMPTMA) entre outros (Ferracane, 2011). Os monômeros funcionais ácidos utilizados são predominantemente monômeros metacrilatos com grupos de ácido carboxílico, como 4-Metacriloxietil Anidro Trimelítico (4-META) e o dimetacrilato glicerol piromelítico (PMGDM). Estes monômeros podem apresentar também grupos com ácidos fosfóricos, como ou com grupos 2- metacriloxietil fenil hidrogênio fosfato (Phenil-P), 10- metacriloxidecil di-hidrogênio fosfato (MDP), bis(2-metacriloxietil) fosfato ácido (BMP) e di-pentaeritritol penta acrilato monofosfato (Penta-P). Muitos destes monômeros foram desenvolvidos especificamente para promover a desmineralização da superfície de esmalte e dentina e formar um sal estável envolvendo cálcio (Ferracane, 2011).

No entanto, o cimento autoadesivo também depende de uma excelente polimerização para obter resultados clínicos e propriedades mecânicas satisfatórias (Llie & Simon, 2011; Piwowarczyk et al., 2007, Piwowarczyk et al., 2005). Uma vez que eles têm mecanismos semelhantes para iniciar a polimerização, em comparação com cimento resinoso convencional, seu modo de polimerização é fundamental para garantir melhor GC (Aguiar et al.; 2010). A maioria das comparações entre as condições de polimerização sobre a conversão de monômero de CRA são baseados em testes *in vitro* em temperatura ambiente aproximadamente 25°C (Aguiar et al., 2010; Moraes et al., 2011; Lohbauer et al.; 2010).

Por outro lado, CRA são aplicados diretamente dente preparado, que tem temperatura variando de 27°C a 32°C dependendo em dente (Rueggeberg et al., 2010; Jafarzadeh et al., 2008). Estudos anteriores demonstraram que a temperatura

elevada do cimento resinoso nos valores de 50°C ou 60°C resulta em maior conversão de monômero independentemente da condição de polimerização (Franca et al., 2011; Daronch et al.; 2005). Portanto, seria razoável esperar algum efeito da temperatura dental no grau de conversão de monômero de CRA. Por exemplo, um estudo anterior demonstrou que a polimerização de CRA a 37°C desenvolve tensão de contração maior do que em 23°C.

Poucos estudos avaliaram os efeitos da temperatura dental sobre CRA, e todos eles têm centrado em valores de resistência de união e análise da microscopia eletrônica sob a tensão-contração. Embora os autores tenham atribuído valores elevados e alta resistência ao GC (Kitzmuller et al., 2011; Cantoro et al., 2008.), não há nenhum estudo disponível na literatura avaliando GC de CRA relacionado ao aumento da temperatura dental.

Este estudo teve como objetivo avaliar os efeitos da temperatura dental no GC dos CRA duais expostos a diferentes condições de polimerização: a exposição à luz direta, exposição à luz atenuada pela restauração indireta de cerâmica, e ativação de auto polimerização dos componentes sem exposição à luz. Duas hipóteses de investigação foram abordadas. A primeira foi que a temperatura dental aumenta GC de cimentos autoadesivos simuladas em cenários clínicos, como que, quando uma restauração cerâmica atenua ou bloqueia totalmente a fotopolimerização. A segunda hipótese de pesquisa é o grau de conversão na temperatura dental será semelhante entre os diferentes modos de ativação simulando as diversas condições clínicas de ativação.

## **2. PROPOSIÇÃO**

O objetivo desse estudo foi avaliar por meio Espectroscopia Infravermelha com Transformada de Fourier (FTIR) o GC de dois tipos de cimento autoadesivos nas temperaturas de 25°C, 28°C e 32°C submetidos a diferentes modos de ativação.

### **3. METODOLOGIA E RESULTADOS**

A presente dissertação está baseada no artigo “Effect of simulated tooth temperature on the degree of conversion of self-adhesive resin cements exposed to different curing conditions”



**EFFECT OF SIMULATED TOOTH TEMPERATURE ON THE DEGREE OF  
CONVERSION OF SELF-ADHESIVE RESIN CEMENTS EXPOSED TO  
DIFFERENT CURING CONDITIONS**

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## EFFECT OF SIMULATED TOOTH TEMPERATURE ON THE DEGREE OF CONVERSION OF SELF-ADHESIVE RESIN CEMENTS EXPOSED TO DIFFERENT CURING CONDITIONS

**Short title:** Monomer conversion of self-adhesive cements exposed to tooth temperatures

### ABSTRACT

**Objectives:** This study evaluated the degree of conversion (DC) of two commercial, self-adhesive resin cements (SARCs) polymerized at simulated tooth temperatures and under different curing conditions, using Fourier Transformed Infrared analysis (FTIR).

**Materials and Methods:** RelyX U100 (U100, 3M ESPE) and Maxcem Elite (MX, Kerr Corporation) were mixed at 25°C and applied to a surface of a horizontal attenuated-total-reflectance unit (ATR), which was at 25°C or heated to 28°C and 32°C, and attached to an infrared spectrometer. The products were polymerized using one of three conditions: direct light exposure through a glass slide (DLE), through a 1.5-thick ceramic disc (EMAX, A2 shade, IPS e.max, Ivoclar Vivadent), or were allowed to self-cure (SC). FTIR spectra were recorded for 12 min (1 spectrum/s, 16 scans/spectrum, resolution 4 cm<sup>-1</sup>) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios pre- and 12-min post-curing, as well as during each 1-second interval. DC data (n=7) were analyzed by two-way ANOVA and Tukey's post-hoc test ( $p=0.05$ ).

**Results:** Tooth temperatures significantly increased DC in all groups of MX and in EMAX and SC groups of U100. For MX, SC groups exposed to tooth temperature showed similar DC values to EMAX groups. For U100, EMAX groups showed higher DC values than SC groups regardless of temperature. Time-based conversion profiles ranged according to product, temperature, and curing mode.

**Conclusions:** Tooth temperature can increase DC of SARCs, even in the SC mode.

**Keywords:** resin cements; polymerization; tooth temperature; degree of conversion.

**Clinical Relevance**

As the degree of conversion of SARCs is higher after exposure to tooth temperature than at room temperature, clinicians may expect differences in setting time and mechanical properties of such products when compared to the results observed at room temperature.

## INTRODUCTION

The success of an indirect restoration relies upon bonding materials to ensure optimum link between prepared tooth and indirect restorative material. Within this context, dual-cured resin cements have been the choice of most clinicians to cement aesthetic indirect restorations. In order to reduce technique sensitivity due to multi-step bonding procedures involved in cementation techniques, manufacturers have developed the so-called self-adhesive resin cements (SARCs). These materials were introduced in the market as a new subgroup of resin cements, which do not require any pretreatment of tooth substrate prior to its application[1,2].

Similarly to conventional dual-cured resin cements, SARCs depend on optimal polymerization to exhibit clinically acceptable mechanical properties and bond strength[3-5]. Once they have similar mechanisms to initiate polymerization in comparison to conventional resin cements, their polymerization mode is crucial to ensure optimal degree of conversion (DC)[6]. With this regard, self-curing mode of such products is less effective than the light activated one[6], and little information is available in the literature regarding the effects of light attenuation due to the presence of an indirect restoration interposed between light-curing unit tip and resin cement layer[7].

Most comparisons between curing conditions on monomer conversion of SARCs are based on *in vitro* tests at room temperature (approximately 25°C)[6,8-10]. Conversely, SARCs are applied directly to prepared tooth, which has temperature ranging from 27.8°C to 32.0°C depending on tooth[11-13]. Previous studies have demonstrated that increased temperature of 50°C or 60°C results in higher monomer conversion in resin cements and resin composites regardless of curing condition[14,15]. Therefore, it would be reasonable to expect some effect of tooth temperature in monomer conversion of SARCs. For instance, a previous study demonstrated that polymerization of SARCs at 37°C develops more shrinkage stress than it does at 23°C[16].

Only few studies have evaluated the effects of tooth temperature on SARCs, and all of them have focused on bond strength values, electron microscopy analysis, and shrinkage stress[17,18,16]. Although the authors have attributed high strength values to elevated DC[17,18,16], there is no study available in the literature evaluating DC of SARCs with increased temperature.

This study aimed to evaluate the effects of tooth temperatures on DC of dual-cured SARC<sub>s</sub> exposed to different curing conditions: direct light exposure, light exposure attenuated by ceramic indirect restoration, and activation by self-curing components with no light exposure involved. Two research hypotheses were addressed. The first one was that tooth temperature increases DC of self-adhesive cements in simulated clinical scenarios, such as that when a ceramic restoration attenuates or totally blocks curing light. The second research hypothesis was that activation conditions simulating clinical situations in which curing light is attenuated or blocked by the presence of an indirect restoration does not impair SARC<sub>s</sub> DC in comparison to the DC observed after direct light exposure.

## **MATERIALS AND METHODS**

### **Specimen preparation**

Two commercially available dual-cure SARC<sub>s</sub> were selected for testing Rely X U100 (U100, 3M ESPE, St. Paul, MN, USA) and Maxcem Elite (MX, Kerr Corp., Danbury, CA, USA). Both SARC<sub>s</sub> were selected because of the differences in composition (Table 1), as one has calcium hydroxide to accelerate pH rise. A ceramic disc with 2 cm diameter by 1.5 mm thickness (EMAX, IPS e.max, A2 shade, Ivoclar Vivadent, Schaan, Liechtenstein) was selected to model indirect restorative material.

Base and catalyst pastes were equally dispensed on a glass plate and were hand-mixed using a metal spatula. The mixture was then applied to the horizontal diamond element of an attenuated total reflectance (ATR) unit attachment (Golden Gate, Specac, Woodstock, GA, USA) in the optical bench of a Fourier transform infrared spectrometer (Tensor Series, Bruker Optik GmbH, Ettlingen, German). For groups simulating tooth temperature, the diamond surface temperature was elevated to 28°C or 32°C using a custom-made heating device, with the surface temperature constantly monitored using the K-type thermocouple (SmartMether, Novus, Porto Alegre, RS, Brazil) during resin cement polymerization. Prior to resin placement, adhesive tape (3M) was placed around the diamond surface to act as a spacer, ensuring standard thickness for all specimens (100 – 120 µm).

The deposited resin cement was covered with a Mylar strip and polymerized using one of three different curing modes. Specimens were exposed directly to light activation without any overlying restorative material (DLE - direct light exposure –

control group) for 20 s for MX and 40 s for U100 (manufacturers' instructions) to a light source emitting approximately  $1500 \text{ mW/cm}^2$  (Radii Plus, SDI Limited, Victoria, Australia). Light intensity from the light-curing units was constantly measured using a radiometer (Cure Rite, Dentsply Caulk, USA). The distal, emitting light guide end was placed directly against a 1-mm thick glass slide, positioned directly over top of the Mylar-covered resin specimen. When exposing cements to light using overlying ceramic disc, the disc was placed directly between the Mylar sheet and the emitting light guide end (Figure 1). In addition, specimens were also allowed to self-cure (SC).

### **Degree of conversion**

Infrared (IR) spectra were collected between  $1680$  and  $1500 \text{ cm}^{-1}$  at a rate of one spectrum per second (16 scans per spectrum) at  $4 \text{ cm}^{-1}$  resolution. Data were counted from the moment the IR scan demonstrated the resin was stabilized on the ATR surface and any overlying objects had been placed. Spectra were recorded continuously during each 1-second interval for 12 min to generate kinetic spectra of monomer conversion. Seven replications were made for each test condition ( $n=7$ ) based on previous studies using the same methodology[19-21,6]. Monomer conversion was calculated using standard methods that evaluated changes in the ratios of aliphatic-to-aromatic C=C absorption peaks ( $1636 \text{ cm}^{-1}/1608 \text{ cm}^{-1}$ ) in the uncured and cured states obtained from the infrared spectra.[22,23] Prior to determining conversion, calibration graphs were made relating the absorbance ratios of known molar concentrations of aliphatic and aromatic C=C to their respective absorbance height ratios. Conversion values among all curing modes were compared statistically within each product only at 12 min from the time the resin cement was stabilized on the ATR surface. All polymerized specimens were carefully removed from the ATR plate and measured for thickness to the nearest 0.01 mm using a digital micrometer (Series 406; Mitutoyo America Corp., Aurora, IL, USA) to ensure similar thickness among all specimens.

### **Statistical analyses**

Degree of conversion data were evaluated within each dual-cure resin product using a two-way ANOVA [factors: temperature (3 levels) and curing condition (3 levels)] followed by Tukey's post-hoc test. Direct comparison of conversion values

between products was not made, because only when resins have the same chemical formulation can such comparisons be made. All testing was performed at a pre-set alpha of 0.05 using personal statistical software (SAS 8.0 for Windows; SAS Institute Inc., Cary, USA). Post-hoc power analysis was performed for the statistical analysis of DC data using IBM SPSS Statistics 19 (SPSS Inc., IBM Company, Armonk, NY, USA).

## RESULTS

### Degree of conversion

The study was adequately powered for both factors, temperature and curing mode (over 95%;  $\alpha=0.05$ ). For both products, two-way ANOVA indicated that the interaction between curing mode and temperature was significant factor in affecting DC for MX ( $p = .0098$ ) and U100 ( $p<.00001$ ).

Table 2 and 3 present the DC of dual-cure cements exposed using the three curing conditions at three temperatures. Temperature increase from 25°C to 28°C resulted in significantly higher DC in all groups of MX, while significant increase in DC of U100 was only observed in groups exposed to light under ceramic restoration and in SC groups ( $p<.0001$ ). No significant increase in DC was observed in control group when temperature increased from 25°C to 28°C. U100 exposed to 32°C showed higher DC values than when it was exposed to other temperatures ( $p<.00001$ ).

For MX, the effect of different curing conditions on DC was temperature-dependent. When exposed to tooth temperatures, the SC mode provided similar DC values to attenuated light (with ceramic restoration interposed between light curing unit tip and resin cement). Self-polymerizing mode at 25°C led to lower DC values than attenuated light-activated mode ( $p=.0427$ ). On the other hand, the effects of curing conditions on DC values were not influenced by temperature when U100 was evaluated. DLE groups showed higher DC values than groups exposed to light attenuated by the indirect restoration ( $p<.0001$ ). Self-polymerizing mode led to the lowest DC values in all temperatures.

Figures 2 and 3 show the effects of tooth temperature on the time-based conversion profiles of U100 and MX, respectively, including comparison to the 12-min DC value from DLE group at 25°C (dashed line) as control. At 28°C and 32°C (Figure 2), time-based conversion changes measured through the ceramic disc of

U100 exhibited polymerization kinetics similar to that obtained in DLE groups at room temperature at 12 min. Polymerization kinetics under ceramic disc exposed to tooth temperatures showed higher rate of conversion until the light curing unit shut off in comparison to the polymerization kinetics at room temperature. Then, a slow, continual increase was noted in all groups under ceramic disc. The slowest initial curing rate was noted for the self-cure only mode (SC) at room temperature, resulting in the lowest DC values. The effects of temperature were more evident in SC groups, as conversion rate at tooth temperatures was apparently much faster than that at room temperature. Less time was required for the SC mode of Rely X U100 to begin polymerization when it was exposed to tooth temperatures.

Overall, time-based conversion profile of MX seemed to be more influenced by tooth temperatures than that of U100. Interestingly, the basic profile of time-based conversion for light-curing through the ceramic disc appeared very much like that of the SC only group, indicating that the overall polymerization reaction when curing through the ceramic disc was related to the self-cure reaction. Besides, time-based conversion profile from SC mode of MX apparently provided faster curing rates than that of U100, regardless of temperature. MX exposure to tooth temperatures promoted faster curing rates than exposure to room temperature in groups relying solely on SC, leading to 12 min DC values as high as those at room temperature.

## **DISCUSSION**

The results validated the first research hypothesis anticipating that tooth temperature increases DC of SARC in simulated clinical conditions. Once these are relatively new products and involve acid-base reaction and radical polymerization, it is difficult to anticipate the effects of such increase in DC on cement mechanical properties and bonding to dentin. It has been shown that high temperature may catalyze the acid-base reaction between acid groups and calcium or aluminum from fillers to promote pH rise[17]. As a consequence, pH would rise more quickly at high temperature than at room temperature. Therefore, polymerization would proceed without the well known detrimental effect from low pH[24,19], but could for this reason compromise bonding between such products and dentin. However, it should be pointed out that studies evaluating the effects of temperature on SARC resin cements have focused on temperature of 60°C[17], which is considerably higher



than tooth temperatures. Thus, it is expected that tooth temperatures may promote similar effects on these products to some extension but with lower magnitude than high temperature does.

The SC mode of SARC at room temperature was less effective than light-activating modes, corroborating with previous findings.[6,8,25] This curing mode was also more influenced by temperature ranging than light-activated modes. More specifically, the DC of MX in SC mode increased approximately 14.2% from 25°C to 28°C, while light activated groups showed around 7.1% to 8.1% increase within the same temperature range. For U100, SC mode groups increased 4.3% from 25°C to 28°C, while light-activated ones increase approximately 1.8%. This finding demonstrates that the effectiveness of self-curing components in SARC exposed to tooth temperature is not as poor as that observed at room temperature. However, it should be noted that Rely X U100 still led to low DC values even at 32°C.

The results invalidated the second hypothesis for MX and U100, which had the DC values significantly decreased in the simulated clinical activating condition at tooth temperatures. Based on time-based conversion profiles (Figures 2 and 3), the effects of tooth temperature on polymerization were clearly related to the amount of self-curing components in resin cements. The degradation rate of benzoyl peroxide into radicals increases with increased temperature,[26] so radicals are created more rapidly when heated. For MX at 25°C (Figure 3A), the importance of self-curing components to compensate for lower light levels reaching the resin cement was evident, as a significant increase in conversion value with time was noted after light exposure. On the other hand, light-activating components in U100 responsible for compensating for light attenuation were seen to work more effectively than in MX. For instance, at 25°C, the conversion-based profile using ceramic disc in U100 produced near the same result as when the SARC was exposed to unattenuated light.

Time-based conversion profiles also demonstrated that the increase in DC values in all curing conditions is a consequence of increased polymerization rate (Figures 2 and 3). In this type of resin cement, high polymerization rate can have detrimental effects on bonding to dentin, as it results in higher shrinkage stress[16]. Besides, high polymerization rates also enhances cross-linking density and network quality[27], resulting in a reduction of resin matrix permeability for fluoride ion

release[28,29]. Conversely, the consequent high DC improves mechanical properties, as previously demonstrated[27,3].

The resin cement polymerization was evaluated for 12 min, which corresponds to approximately the time spent by clinicians to cement an indirect restoration to a prepared tooth. Therefore, the exposure of SARC to tooth temperatures will allow the cement to be better polymerized at the moment of occlusal adjustments and first occlusal stress. However, it must be emphasized that further polymerization along with acid–base reaction may proceed in the following 24 hours[2,30]. Therefore, no assumptions can be made regarding long-term bonding and resin cement mechanical properties, so only evaluation of SARC after all curing reactions are completed can provide evidences of how tooth temperature affects their mechanical properties and bonding to teeth.

## **CONCLUSION**

Within the limitations imposed by the current study, the following conclusions may be made:

1. SARC exposed to tooth temperatures showed increased monomer conversion values in the simulated clinical conditions in comparison to the same products polymerized at room temperature.
2. At tooth temperatures, SARC exposed to attenuated light due to indirect ceramic restoration exhibited DC values as high as those observed when resin cements were exposed to direct light at room temperature.
3. Despite the significant increase in DC promoted by tooth temperature in self-cured SARC, the effectiveness of self-curing components at higher temperatures in comparison to dual-curing ones at room temperature was product-dependent.

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Table 1: Brand, composition and batch number of SARC:

Product (Manufacturer)	Composition (Batch Number)
Rely X U100 (3M ESPE)	Base: glass fiber, methacrylated phosphoric acid esters, dimethacrylates, silanated sílica, sodium persulfate Catalyst: glass fiber, dimethacrylates, silanated sílica, p-toluene sodium sulfate, calcium hydroxide (367405)
Maxcem Elite (Kerr Corporation)	Resin: multifunctional DMAs, GPDM, proprietary Redox initiators and photoinitiators. Filler: barium, fluoroaluminosilicate, fumed silica (66 wt%) (423636)

GPDM: glycerophosphate dimethacrylate; DMAs: dimethacrylates.

Table 2: DC values (SD) of MX at room and tooth temperatures:

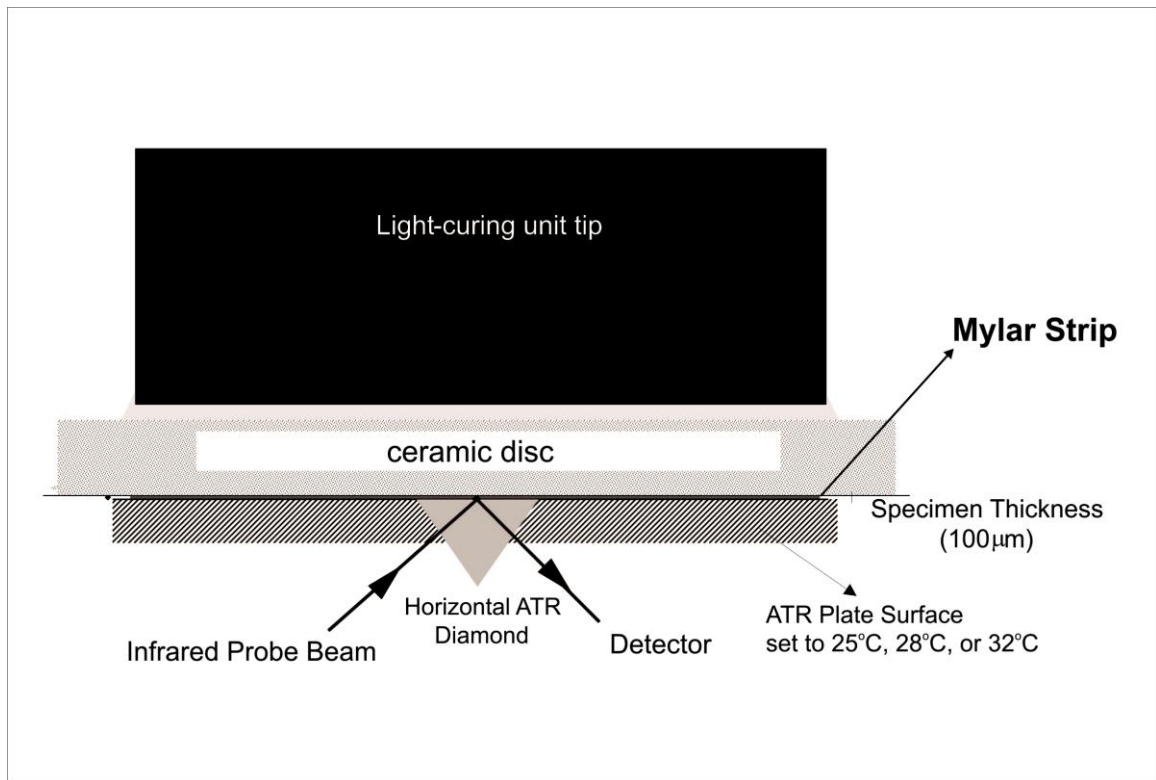
	Direct Light Exposure (DLE)	Under indirect ceramic disc (EMAX)	Self-polymerized (SC)
25°C	58.1 (2.7) Ba	52.4 (1.3) Cb	47.6 (1.8) Bc
28°C	66.2 (2.7) Aa	59.5 (2.4) Bb	61.8 (3.2) Aab
32°C	70.9 (2.0) Aa	66.2 (3.3) Aab	62.1 (1.4) Ab

Mean values followed by different letters (upper case letters within column; lower case letters within row) are significantly different ( $p < 0.05$ ).

Table 3: DC values (SD) of U100 at room and tooth temperatures:

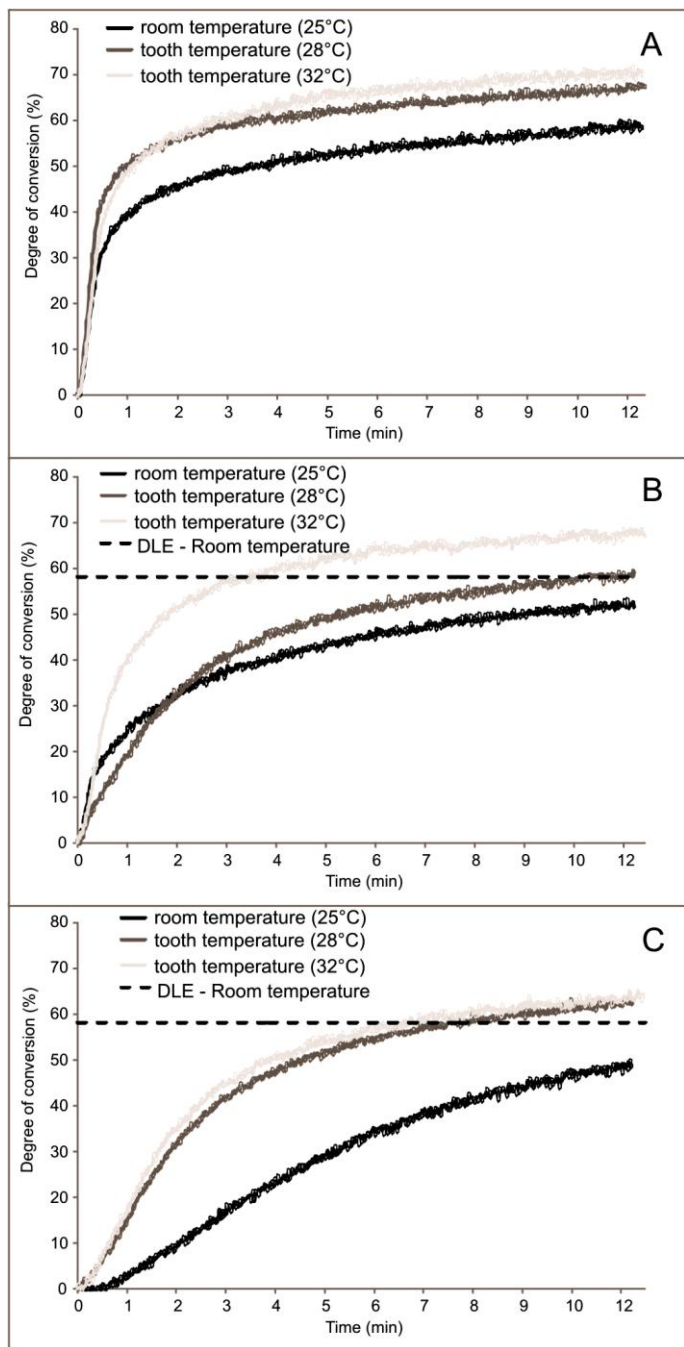
	Direct Light Exposure (DLE)	Under indirect ceramic disc (EMAX)	Self-polymerized (SC)
25°C	49.9 (1.0) Ba	45.1 (2.6) Cb	25.2 (3.7) Cc
28°C	50.7 (2.3) Ba	46.8 (1.0) Bb	29.5 (1.8) Bc
32°C	57.4 (0.9) Aa	47.9 (1.5) Ab	34.1 (1.6) Ac

Mean values followed by different letters (upper case letters within column; lower case letters within row) are significantly different ( $p < 0.05$ ).

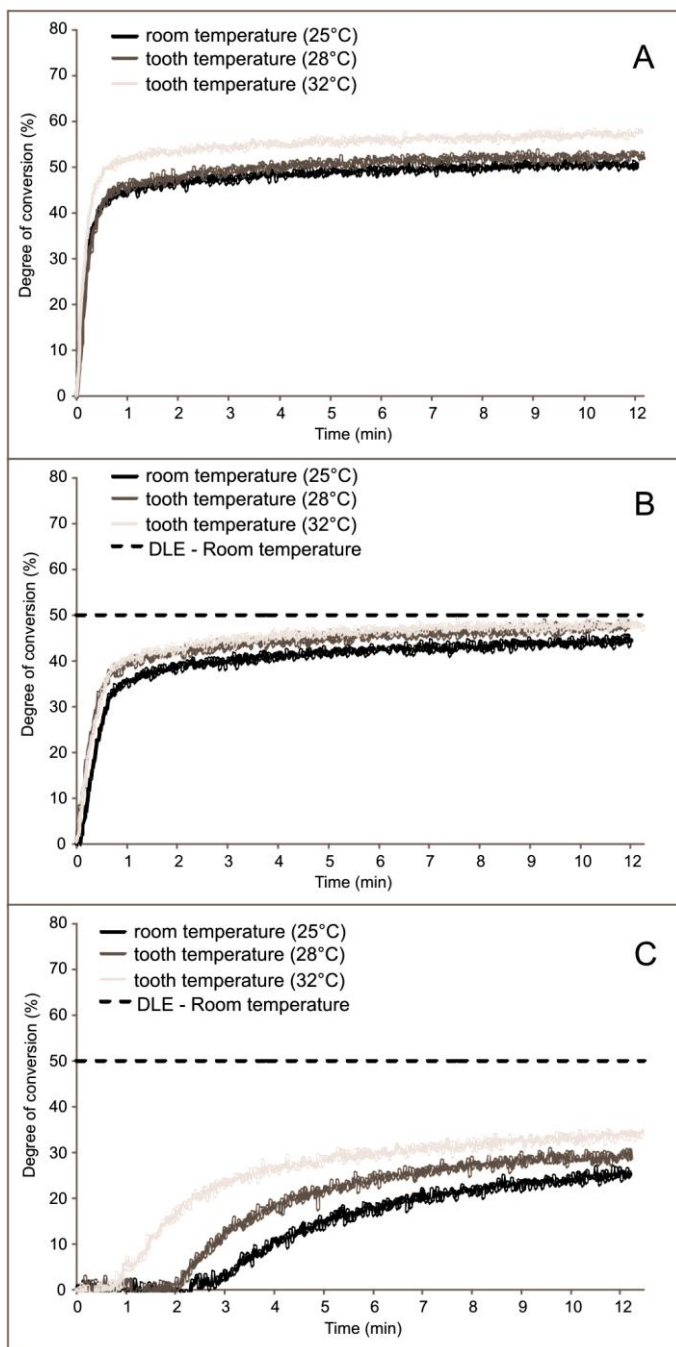


**Figure 1:** Illustrative diagram demonstrating the interaction between the infrared beam and the specimen, as well as the position of ceramic disc and light-curing unit tip.





**Figure 2:** Polymerization kinetics of MX at 25°C, 28°C, and 32°C exposed to 3 curing conditions: direct light exposure (A), light exposure through ceramic disc (B), and self-curing (C). The dashed line represents the degree of conversion after after direct light exposure at 25°C.



**Figure 3:** Polymerization kinetics of U100 at 25°C, 28°C, and 32°C exposed to direct light exposure (A), light exposure through ceramic disc (B), and self-curing (C). The dashed line represents the degree of conversion after direct light exposure at 25°C.

## CONCLUSÃO

Dentro das limitações impostas pelo atual estudo, podemos concluir:

Os cimentos resinosos autoadesivos expostos a temperaturas dentais simuladas, apresentaram maiores valores no grau de conversão do monômero, em comparação com os mesmos produtos polimerizados na temperatura ambiente.

Com a simulação do aumento das temperatura dental, os cimentos resinosos autoadesivos exposto à luz atenuada devido à restauração indireta de cerâmica exibiram valores GC tão elevados quanto os que foram expostos à luz direta em temperatura ambiente.

Apesar do aumento significativo nos valores do GC promovido pelo aumento da temperatura dental na auto-polimerização dos CRA, a eficácia da auto polimerização dos componentes em temperaturas mais elevadas em comparação com os polimerização dual em temperatura ambiente se torna um produto-dependente.

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**ANEXO A – Figuras ilustrativas dos cimentos resinosos de dupla ativação utilizados neste estudo**



Cimentos de dupla-ativação utilizados no estudo, Maxcem Elite (Kerr) (A) e Rely X U100 (3M ESPE) (B).

**ANEXO B – Figuras ilustrativas do espectroscópio infravermelho com transformada de Fourier (FTIR)**

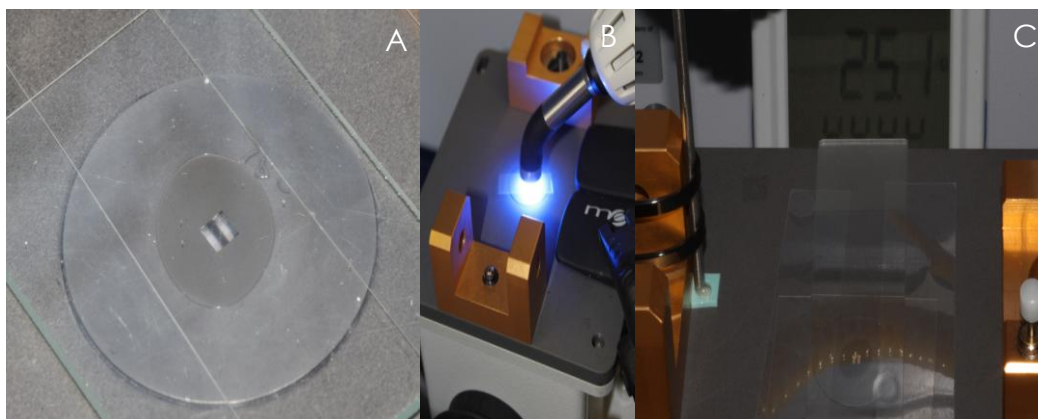


Espectrofômetro infravermelho com transformada de Fourier – FTIR (Tensor 27, Bruker Comp), unidade de reflectância total atenuada – ATR (Satandard Golden Gate. Specac).

## ANEXO C – Preparação dos espécimes



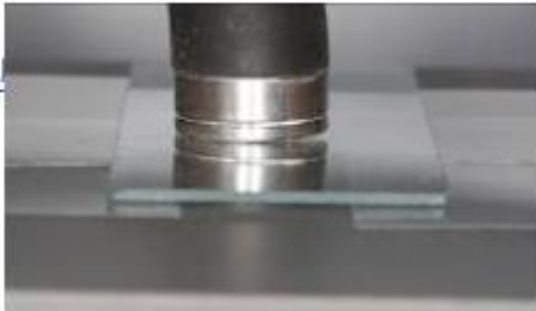
Adequação da mesa de ATR para controle de temperatura. Dispositivo para aquecimento acoplado a mesa (A); adaptação do termômetro com termopar tipo K para controle constante da temperatura (B)



Aplicação do cimento resinoso na superfície de diamante da mesa de ATR (A); fotoativação conforme recomenda o fabricante (B); e proteção do espécime contra a exposição a luz (C).



## ANEXO D – Modos de ativação



Fotoativação através de lâmina de vidro.



Fotoativação através de disco de cerâmica 1,5mm (e.max1,5).



Autopolimerização (DLE).

**ANEXO E – Análise estatística para os resultados de grau de conversão**

The GLM Procedure

Class Level Information

Class	Levels	Values
temp	3	1 2 3
cmode	3	1 2 3

Number of observations 54

NOTE: Due to missing values, only 50 observations can be used in this analysis.

## The GLM Procedure

Dependent Variable: DC

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	8	2319.807133	289.975892	49.84	<.0001
Error	41	238.553667	5.818382		
Corrected Total	49	2558.360800			

R-Square	Coeff Var	Root MSE	DC Mean
0.906755	3.991746	2.412132	60.42800

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	1642.061131	821.030565	141.11	<.0001
cmode	2	588.667615	294.333808	50.59	<.0001
temp*cmode	4	89.078387	22.269597	3.83	0.0098

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	1678.459924	839.229962	144.24	<.0001
cmode	2	538.622118	269.311059	46.29	<.0001
temp*cmode	4	89.078387	22.269597	3.83	0.0098

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

		LSMEAN	
temp	cmode	DC LSMEAN	Number
1	1	58.0500000	1
1	2	52.4400000	2
1	3	47.5666667	3
2	1	66.1500000	4
2	2	59.5000000	5
2	3	61.8500000	6
3	1	70.9000000	7
3	2	66.2000000	8
3	3	62.0833333	9

Least Squares Means for effect temp\*cmode

Pr > |t| for H0: LSMean(i)=LSMean(j)

Dependent Variable: DC

i/j	1	2	3	4	5	6	7	8	9
1		0.0112	<.0001	<.0001	0.9792	0.2905	<.0001	<.0001	0.1195
2	0.0112		0.0427	<.0001	0.0006	<.0001	<.0001	<.0001	<.0001
3	<.0001	0.0427		<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
4	<.0001	<.0001	<.0001		0.0007	0.1586	0.0526	1.0000	0.1135
5	0.9792	0.0006	<.0001	0.0007		0.8447	<.0001	0.0006	0.6468
6	0.2905	<.0001	<.0001	0.1586	0.8447		<.0001	0.1485	1.0000
7	<.0001	<.0001	<.0001	0.0526	<.0001	<.0001		0.0571	<.0001
8	<.0001	<.0001	<.0001	1.0000	0.0006	0.1485	0.0571		0.1048

9	0.1195	<.0001	<.0001	0.1135	0.6468	1.0000	<.0001	0.1048
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## The GLM Procedure

## Class Level Information

Class	Levels	Values
temp	3	1 2 3
cmode	3	1 2 3

Number of observations 81

NOTE: Due to missing values, only 59 observations can be used in this analysis.

10:49 Sunday, April 30, 2000 8

## The GLM Procedure

Dependent Variable: DC1

Source	DF	Sum of		F Value	Pr > F
		Squares	Mean Square		
Model	8	42875485.75	5359435.72	212.30	<.0001
Error	50	1262220.85	25244.42		
Corrected Total	58	44137706.61			

R-Square	Coeff Var	Root MSE	DC1 Mean
0.971403	8.200502	158.8849	1937.502

Source	DF	Type I SS	Mean Square	F Value	Pr > F
temp	2	3131908.19	1565954.09	62.03	<.0001
cmode	2	38950558.07	19475279.03	771.47	<.0001
temp*cmode	4	793019.50	198254.88	7.85	<.0001

Source	DF	Type III SS	Mean Square	F Value	Pr > F
temp	2	2894320.23	1447160.12	57.33	<.0001
cmode	2	38990338.79	19495169.40	772.26	<.0001
temp*cmode	4	793019.50	198254.88	7.85	<.0001

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

		LSMEAN	
temp	cmode	DC1 LSMEAN	Number
1	1	2490.93857	1
1	2	2042.91667	2
1	3	648.67857	3
2	1	2574.95571	4
2	2	2189.17800	5
2	3	872.27429	6
3	1	3298.70571	7
3	2	2294.72833	8
3	3	1163.14429	9

Least Squares Means for effect temp\*cmode

Pr > |t| for H0: LSMean(i)=LSMean(j)

Dependent Variable: DC1

i/j	1	2	3	4	5	6	7	8	9
1	0.0002	<.0001	0.9852	0.0497	<.0001	<.0001	0.4094	<.0001	
2	0.0002	<.0001	<.0001	0.8407	<.0001	<.0001	0.1586	<.0001	
3	<.0001	<.0001	<.0001	<.0001	0.1994	<.0001	<.0001	<.0001	
4	0.9852	<.0001	<.0001	0.0038	<.0001	<.0001	0.0598	<.0001	
5	0.0497	0.8407	<.0001	0.0038	<.0001	<.0001	0.9720	<.0001	
6	<.0001	<.0001	0.1994	<.0001	<.0001	<.0001	<.0001	<.0001	0.0310
7	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
8	0.4094	0.1586	<.0001	0.0598	0.9720	<.0001	<.0001	<.0001	<.0001