



**CENTRO DE PÓS-GRADUAÇÃO E PESQUISA
CURSO DE MESTRADO EM ODONTOLOGIA
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MICHELE DE OLIVEIRA

**EFEITO DA TEMPERATURA NO GRAU DE CONVERSÃO E TEMPO
DE TRABALHO DE CIMENTOS RESINOSOS DUAIS NA SIMULAÇÃO
DA CIMENTAÇÃO DE RESTAURAÇÕES INDIRETAS DE CERÂMICA**

GUARULHOS

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Orientador: Prof. Dr. Cesar A. Galvão Arrais

Co-orientador: Prof. Dr. José Augusto Rodrigues

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DEDICATÓRIA

Dedico este trabalho,

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“Você se fez presente em todos os momentos, e passo a passo pude sentir a sua presença transmitindo-me segurança necessária para enfrentar os desafios e seguir o meu caminho.”

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“O futuro se chama talvez. O importante é não deixar que isso o assuste.”

(Tennessee Williams)

RESUMO

Este estudo avaliou o grau de conversão (GC) e o tempo de trabalho (TT) de dois cimentos resinosos duais polimerizados em diferentes temperaturas, utilizando espectroscopia infravermelha com transformada de Fourier (FTIR). Calibra Regular (Cal/Dentsply Caulk) e Variolink II LV (Var/Ivoclar Vivadent) foram mantidos a 25°C ou aquecidos a 37°C ou 50°C e aplicados na superfície de uma unidade horizontal atenuada (ATR), com temperatura controlada acoplada a um espectroscópio infravermelho. Os produtos foram polimerizados utilizando 4 modos de ativação: fotoativação com luz halógena (600 mW/cm²) através de lâmina de vidro (LV); através de discos de cerâmica (cor A2, IPS e.max/Ivoclar Vivadent) com espessuras de 1,5mm (e.max1,5) e 3,0mm (e.max3,0); e autopolimerização (AP). Espectros foram obtidos (16 *scans*, resolução de 4 cm⁻¹) durante 20 minutos com razão de 1 espectro / segundo (n= 6). O DC foi calculado usando técnicas padrão de mudanças observadas nas taxas de pico alifáticos, aromáticos antes e 20 min após polimerização. A análise de cinética de polimerização foi usada para determinar o seu tempo de trabalho, em cada temperatura. Os dados de GC e TT foram analisados por ANOVA 2 fatores e teste de Tukey (p<0,05). Temperaturas mais altas elevaram o GC de ambos os produtos independente do modo de ativação. Para Cal, apenas o grupo e.max3,0 exibiu GC menor aos demais grupos a 25°C, enquanto não foi observada diferença significativa entre os grupos a 37°C e a 50°C. Para Var, o grupo e.max3,0 apresentou menor GC, quando comparado ao grupo e.max1,5 apenas em 25°C, enquanto o grupo AP apresentou GC inferior aos demais em todas as temperaturas. O TT diminuiu desfavorável para o Cal a 37°C e 50°C, e para Var apenas a 50°C. Para Var a 37°C, o TT diminuiu para um intervalo considerado clinicamente aceitável. O aquecimento aumentou o GC dos cimentos resinosos duais. Apesar da redução crítica do TT na maioria das condições avaliadas, um produto (Var) exibiu um TT clinicamente aceitável a 37°C, equivalente a 1,5 minuto.

Palavras-chave: polimerização, cimentos resinosos, aquecimento, grau de conversão, tempo de trabalho.

ABSTRACT

This study evaluated the degree of conversion (DC) and working time (WT) of dual-cured resin cements polymerized at varying temperatures using Fourier Transformed Infrared analysis (FTIR). Calibra Regular (Cal/Dentsply Caulk) and Variolink II Low Viscosity (VL/Ivoclar Vivadent) were kept at 25°C or heated to 37°C or 50°C and applied to the surface of a horizontal attenuated-total-reflectance unit (ATR) with controlled temperature attached to an infrared spectrometer. The products were polymerized using one of four conditions: light-activation (600 mW/cm²) through a glass slide (DLE), through a 1.5- (e.max1.5) or 3.0-mm (e.max3.0) thick ceramic disc (A2 shade, IPS e.max, Ivoclar Vivadent), or were allowed to self-cure (SC). FTIR spectra were recorded for 20 min (1 spectrum/s, 16 scans/spectrum, with resolution of 4 cm⁻¹) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios pre- and 20-min post-curing. Polymerization kinetics analysis was used to determine their working time (WT) in each temperature. DC and WT data (n=6) were analyzed by two-way ANOVA and Tukey's post-hoc test (p=0.05). Higher temperatures increased the DC regardless of the curing mode and product. For Cal, only e.max3.0 group showed lower DC than the other groups at 25°C, while no significant difference was observed among groups at 37°C and 50°C. For VL, e.max3.0 group showed lower DC than e.max1.5 group only at 25°C, while SC group showed lower DC than the others in all temperatures. The WT decreased unfavorably for Cal at 37°C and 50°C, and for VL only at 50°C. For VL at 37°C, the WT decreased to a clinically acceptable interval. Higher temperature increases the DC of dual-cured cements. Despite the critical reduction in WT for most conditions, one product (VL) showed a clinically acceptable WT at 37°C.

Keywords: polymerization; resin cements; heat; degree of conversion; working time.

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1. INTRODUÇÃO E JUSTIFICATIVA

O sucesso de restaurações indiretas com cerâmicas odontológicas é atribuído, entre outros fatores, à higiene do paciente, tipo de material restaurador, à adequada união entre o material restaurador, o cimento resinoso utilizado e os substratos dentais (Inokoshi et al., 1993; Sjogren et al., 1995). 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. 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., 2001b), é fundamental que cimentos resinosos sejam capazes de polimerizar adequadamente após o assentamento do elemento protético.

Muitos cimentos resinosos apresentam dois modos de ativação e de iniciação da reação de polimerização, recebendo por este motivo o nome de cimentos resinosos de dupla ativação (Nathanson, 1987; Milleding et al., 1995). Neste tipo de produto, um dos modos de ativação é baseado na utilização de um iniciador fotossensível que é convertido em radical livre após exposição à luz com comprimento de onda em torno de 460 nm, sendo este mecanismo chamado de fotoativação. O outro mecanismo, conhecido como autopolimerização, baseia-se na utilização de peróxido de benzoíla, um composto químico capaz de gerar radicais livres responsáveis pelo início da reação de polimerização quando em contato com um ativador, no caso uma amina terciária (Anusavice, 2005). Desta forma, os cimentos resinosos de dupla ativação tornam-se capazes de polimerizar mesmo com a atenuação da luz fotoativadora ao atravessar os materiais restauradores indiretos após sua fixação (Arrais et al. 2008; El-Mowafy et al., 1999; El-Mowafy e Rubo, 2000).

No entanto, tem sido demonstrado que sistemas de cimentação de dupla ativação apresentam em sua maioria GC inferior diante da menor intensidade ou mesmo da ausência de luz fotoativadora devido aos materiais restauradores indiretos opacos, em comparação ao GC observado quando a luz fotoativadora atinge os sistemas de cimentação na irradiância de 400-600 mW/cm² (Arrais et al., 2008; Blackman et al., 1990; Cardash et al., 1993). Como consequência, estes cimentos podem apresentar menor dureza, maior solubilidade, menor resistência à flexão e à compressão, bem como menor resistência de união à dentina quando a

autopolimerização é o único mecanismo disponível diante da ausência de luz (Arrais et al., 2007a; Arrais et al., 2007b; Braga et al., 2002; El-Mowafy e Rubo, 2000; Hofmann et al., 2001; Meng et al., 2008; Piwowarczyk et al., 2007; Sigemori et al., 2005). Tal evidência torna-se um fator preocupante mesmo quando utilizados materiais restauradores indiretos sem estruturas metálicas como em restaurações de cerâmicas puras ou de resinas compostas, considerando-se que estes materiais permitem que apenas aproximadamente 10% a 15% da intensidade de luz atinja o sistema de cimentação (Arrais et al., 2008; El-Mowafy et al., 1999; El-Mowafy e Rubo, 2000). Desta forma, cimentos resinosos de dupla ativação passam a depender principalmente do mecanismo de autopolimerização quando realizada a fixação de peças protéticas.

Uma possível explicação para a menor efetividade do mecanismo de autopolimerização está relacionada a menor velocidade de ativação e propagação da reação de polimerização quando comparado ao mecanismo de fotoativação (Warnock e Rueggeberg, 2004). Após atingir a velocidade máxima de polimerização, a reação passa por uma desaceleração (auto-desaceleração) no momento em que a propagação da reação torna-se controlada pela difusão de radicais reativos (Daronch et al., 2006). Em outras palavras, a desaceleração ocorre devido a redução na mobilidade dos monômeros e radicais livres durante a reação (Andrzejewska, 2001; Cook et al., 1997; Lovell et al., 2001a; Sideridou et al., 2002). Deste modo, os efeitos da inabilidade dos radicais livres migrarem devido a mudança na viscosidade com a formação da rede polimérica tornam-se mais evidentes na autopolimerização, comprometendo a polimerização final (Rueggeberg e Caughman, 1993).

A temperatura na qual a polimerização ocorre também influencia na reação de polimerização e nas propriedades mecânicas dos polímeros (Bajaj et al., 1977; Bausch et al., 1981; Cook et al., 1997). Um aumento na temperatura durante a polimerização promove maior mobilidade dos radicais livres e dos monômeros, promovendo maior velocidade de polimerização e no GC final como consequência (Cook, 1992; Lovell et al., 2001b, Trujillo et al., 2004). Os mesmos achados têm sido observados quando resinas compostas fotoativáveis foram pré-aquecidas em diferentes temperaturas (Daronch et al., 2005; Daronch et al., 2006). Daronch et al. (2005) observaram que resinas compostas pré-aquecidas à temperaturas de 54°C ou 60°C e expostas a densidade de luz inferior àquela recomendada apresentavam

GC similar ou superior àquele exibido pelos mesmos compósitos sem pré-aquecimento e expostos à luz na irradiância recomendada.

Neste contexto, embora saiba-se que a temperatura pode reduzir consideravelmente o tempo de trabalho de resinas autopolimerizáveis (Cantoro et al., 2008; Cantoro et al. 2009), o pré-aquecimento de cimentos resinosos de dupla ativação no momento do assentamento e fixação da restauração indireta pode ser uma opção na tentativa de compensar os efeitos indesejados da atenuação da luz fotoativadora que atravessa a peça protética ou até mesmo a ausência de luz na polimerização dos cimentos resinosos. Além disso, Cantoro et al. (2005, 2006) observaram maiores valores de resistência de união e melhor adaptação de restaurações indiretas em superfícies dentinárias quando os cimentos resinosos eram aquecidos previamente à sua aplicação. No entanto, não há relatos na literatura a respeito da efetividade do pré-aquecimento de cimentos resinosos de dupla ativação quando a luz fotoativadora está ausente ou é reduzida devido à presença da restauração indireta. Tal tipo de avaliação torna-se crucial para se determinar a temperatura ideal de utilização dos cimentos resinosos aquecidos, uma vez que o aquecimento pode também causar uma diminuição significativa no tempo de trabalho destes materiais.

A análise utilizando espectrofotometria infravermelha com transformada de Fourier (FTIR) tem se mostrado uma metodologia eficaz e confiável na identificação das ligações duplas de carbono ($C = C$) previamente e após a polimerização de compósitos, resinas adesivas e no estabelecimento de relações diretas entre o GC e propriedades mecânicas dos polímeros (Eliades et al., 2001; Imazato et al., 1995; Rueggeberg e Craig, 1988; Rueggeberg et al., 1990; Ruyter e Svendsen, 1978). Utilizando esta metodologia, Braga e Ferracane (2002) evidenciaram uma relação direta entre tempo de exposição à luz, GC e contração de compósitos. Eliades et al. (2001), por sua vez, observaram a distribuição dos componentes monoméricos de adesivos aplicados no substrato dentinário. Desta forma, a utilização da análise em FTIR pode contribuir para a melhor compreensão dos efeitos do pré-aquecimento na polimerização de cimentos resinosos de dupla ativação nas diferentes formas de ativação, seja na fotoativação direta em que a luz atinge o cimento resinoso na densidade recomendada, ou quando a densidade de luz é reduzida ou ausente devido à presença da restauração indireta. Deste modo, este estudo avaliou os efeitos da temperatura no GC e no tempo de trabalho de

cimentos resinosos na simulação da cimentação de restaurações indiretas de cerâmica.

2. PROPOSIÇÃO

Os objetivos deste estudo foram avaliar utilizando análise em FTIR:

- Os efeitos da maior temperatura durante a polimerização no grau de conversão e cinética de polimerização de dois cimentos resinosos duais.
- Os efeitos da maior temperatura no GC dos cimentos resinosos após exposição a diferentes modos de ativação e com diferentes cenários de atenuação da luz na simulação da cimentação de restaurações indiretas de cerâmica com diferentes espessuras.
- Os efeitos da maior temperatura no tempo de trabalho dos cimentos resinosos avaliados.

3. METODOLOGIA E RESULTADOS

A presente dissertação está baseada no artigo “Effect of temperature on the degree of conversion and working time of dual-cured resin cements exposed to different curing conditions”.

EFFECT OF TEMPERATURE ON THE DEGREE OF CONVERSION AND WORKING TIME OF DUAL-CURED RESIN CEMENTS EXPOSED TO DIFFERENT CURING CONDITIONS

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ABSTRACT

Objectives: This study evaluated the degree of conversion (DC) and working time (WT) of dual-cured resin cements polymerized at varying temperatures using Fourier Transformed Infrared analysis (FTIR). **Materials and Methods:** Calibra (Cal/Dentsply Caulk) and Variolink II (VL/Ivoclar Vivadent) were kept at 25°C or heated to 37°C or 50°C and applied to the surface of a horizontal attenuated-total-reflectance unit (ATR) with controlled temperature attached to an infrared spectrometer. The products were polymerized using one of four conditions: light-activation (600 mW/cm²) through a glass slide (DLE), through a 1.5- (e.max1.5) or 3.0-mm (e.max3.0) thick ceramic disc (A2 shade, IPS e.max, Ivoclar Vivadent), or were allowed to self-cure (SC). FTIR spectra were recorded for 20 min (1 spectrum/s, 16 scans/spectrum, with resolution of 4 cm⁻¹) immediately after application to the ATR. DC was calculated using standard techniques of observing changes in aliphatic-to-aromatic peak ratios pre- and 20-min post-curing. Polymerization kinetics analysis was used to determine their working time (WT) in each temperature. DC and WT data (n=6) were analyzed by two-way ANOVA and Tukey's post-hoc test (p=0.05). **Results:** Higher temperatures increased the DC regardless of the curing mode and product. For Cal, only e.max3.0 group showed lower DC than the other groups at 25°C, while no significant difference was observed among groups at 37°C and 50°C. For VL, e.max3.0 group showed lower DC than e.max1.5 group only at 25°C, while SC group showed lower DC than the others in all temperatures. The WT decreased unfavorably for Cal at 37°C and 50°C, and for VL only at 50°C. For VL at 37°C, the WT decreased to a clinically acceptable interval. **Conclusion:** Higher temperature increases the DC of dual-cured cements. Despite the critical reduction in WT for most conditions, one product (VL) showed a clinically acceptable WT at 37°C. **Keywords:** resin cements; polymerization; heat; degree of conversion.

INTRODUCTION

Dual-cured resin cements were developed to achieve optimum polymerization even when curing light is attenuated or blocked by the presence of an indirect restoration. For this purpose, their polymerization can be initiated by two activation modes: dual-curing and self-curing modes.^{1,2} However, it has been shown that the self-curing mode of such products is less effective than the dual-curing one, once these cements exhibit lower DC when curing light is attenuated or blocked by the presence of an indirect restoration than when they are properly exposed to the curing light.³⁻⁵ In these detrimental clinical conditions, dual-cured resin cements may present lower hardness, higher solubility, lower flexural and compressive strength values, as well as lower bond strength values to dentin when compared to the values of resin cements properly cured.⁶⁻¹³ Such evidences deserve some concern since only approximately 10 % to 15 % of light intensity remains after the curing light passes through a 2-mm thick indirect restoration with shade varying from A2 to A4.^{3,9,14}

One possible explanation for the lower effectiveness of self-curing components in promoting optimum polymerization in dual-cured resin cements is related to the slow polymerization rate of activation and propagation of radicals in comparison to photoactivation.¹⁵ After reaching its highest rate, this slow polymerization passes through a process called autodeceleration caused by the decrease in mobility of monomers and free radicals by network formation as polymerization proceeds, leading to an increase in viscosity.¹⁶ These changes in polymer viscosity may affect the polymerization more severely when it relies solely on the self-curing components.¹⁷ Another possible explanation for the lower effectiveness of self-curing components is related to the low amount of benzoyl peroxide as well as the inclusion of inhibitors in the cement composition to provide a proper working time (WT) ranging from 2 to 5 minutes according to manufacturers' information. Otherwise, clinicians would not be capable of applying such materials during the cementation of indirect restorations before their polymerization starts.

Temperature can also interfere with the polymerization kinetics and mechanical properties of polymers.¹⁸⁻²⁰ It has been shown that increased resin temperature prior to and during polymerization leads to higher degree of conversion of light-cured resin composites.²¹ An increase in temperature during polymerization promotes higher mobility of free radical and monomers, leading to higher

polymerization rate and higher DC as a consequence.^{21,22} Similar findings were observed with pre-heated resin composites even after they were exposed to a curing light for a shorter time than that recommended by the manufacturers.^{23,24}

Within this scenario, increasing the temperature of dual-cured resin cements during the mixing procedure may be a valuable option to assure optimum polymerization when resin cements rely mostly on the self-curing mode. Nevertheless, it may be difficult to determine the optimum pre-cementation temperature for a specific material as heating can cause a significant decrease in working time. Few studies evaluated the effects of increased temperature of some resin cements on marginal adaptation and bond strength of indirect restorations to the tooth structure.^{25,26} Cantoro et al.^{25,26} attributed the higher bond strength results obtained by the association between higher DC and improved marginal adaptation due to the low viscosity observed in resin cements at higher temperatures. However, there is no information available in the literature regarding monomer conversion and working time of such cements when polymerized at higher temperatures. The aims of this study were to evaluate the effects of increased temperature prior to and during polymerization on the degree of conversion (DC), polymerization kinetics, and working time (WT) of dual-cured resin cements using Fourier Transformed Infrared analysis (FTIR), exposed to different activation modes and with varied light attenuation scenarios. The research hypotheses were that (1) dual-cured resin cements polymerized at 37°C and 50°C show higher DC than the same products polymerized at room temperature when curing light is attenuated by the presence of indirect ceramic restorations with varying thickness or even when no curing light is available; and (2) a clinically acceptable decrease in the WT is observed in the dual-cured resin cements when their temperatures are increased to either 37°C or 50°C.

MATERIALS AND METHODS

Specimen preparation

Two dual-cured resin cements were used (Table 1): Calibra Regular (Dentsply Caulk, Milford DE, USA) and Variolink II Low Viscosity (Ivoclar Vivadent, Schaan, Liechtenstein). Indirect ceramic discs (IPS e.max LT, A2 shade, Ivoclar Vivadent, Schaan, Liechtenstein) were fabricated with two thicknesses: 1.5 mm and 3 mm (2 cm in diameter) to simulate laboratory-processed veneering ceramic restorations.

Table 1: Brand, composition and batch number of the dual-cured resin cementing systems.

Product (Manufacturer)	Composition (Batch Number)
Calibra regular viscosity Shade Medium (Dentsply Caulk)	Base paste: barium boro fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, other colorants are inorganic iron oxides. Catalyst paste: barium boro, fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, benzoyl peroxide. (Base: 081105; Catalyst: 0812011)
VariolinkII low viscosity Shade Yellow (Ivoclar Vivadent/ Schaan, Liechtenstein)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments, Bis-GMA; TEGDMA; UDMA; benzoyl peroxide. (Base: L46354; Catalyst: L36656)

Abbreviations: TEGDMA: triethylene glycol dimethacrylate; Bis-GMA: bisphenol A Diglycidyl ether methacrylate.

For experimental groups of resin cements heated to 37°C or 50°C during polymerization, base and catalyst pastes were equally dispensed on a glass plate resting on a heating stirrer surface (103, Cientec, Piracicaba, São Paulo, Brazil) set at 37±1°C or 50±1°C. Specimen and glass plate temperatures were constantly measured with a K-type thermocouple (SmartMether, Novus, Porto Alegre, RS, Brazil) to assure that base and catalyst pastes reached the temperature stipulated for each experimental group. The resin cements were mixed and applied to a horizontal diamond element from 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). Adhesive tape (3M) was placed around the diamond surface to act as a spacer, ensuring standard thickness for all specimens (100 – 120 μm). For heated groups, the diamond surface was heated to 37°C or 50°C with a custom-made heater device and the surface temperature was constantly monitored with K-type thermocouple (SmartMether, Novus) during resin cement polymerization. For groups of resin cements polymerized at room temperature, all procedures described above were performed at 25°C.

The deposited resin cement was covered with a Mylar strip and polymerized using one of 4 different curing modes: light activation (power density: 600 mW/cm^2 , Optilux 501; Demetron Kerr, Danbury, CA, USA) for 20 s for Calibra and 40 s for Variolink II, according to the manufacturers' instructions, through a glass slide (~ 2 mm thick) (DLE - direct light exposure – control group); through the 1.5- or 3.0-mm thick ceramic discs (e.max1.5 / e.max3.0) (Fig. 1); or they were allowed to self-cure under a Mylar strip and glass slide (SC), with neither exposure to curing light nor exposure to any other light source.

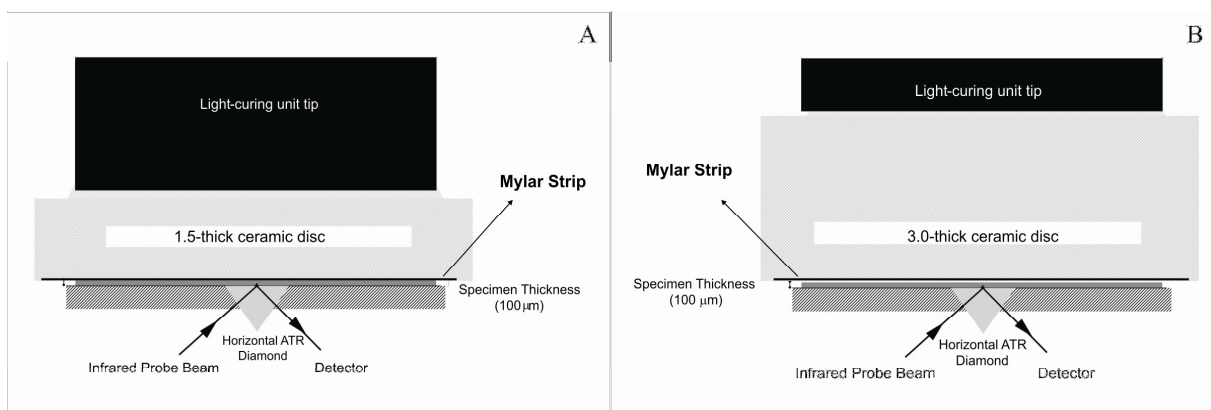


Figure 1: Illustrative diagram demonstrating the interaction between the infrared beam and the specimen, as well as the position of the 1.5- (A) or 3.0-mm (B) thick ceramic disc and the light-curing unit tip.

Degree of conversion and polymerization kinetics

Infrared spectra were collected between 1680 and 1500 cm^{-1} at a rate of 1 spectrum/ s (16 scans per spectrum) at 4- cm^{-1} resolution, from the moment when the resin cement layer was stabilized on the ATR surface by the glass slide or indirect

restorations through the next 20 min (Fig. 2A). Six replications were made for each test condition ($n=6$). Monomer conversion was calculated by standard methods using 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.^{16,17,27,28} All curing modes were compared within each product at 20 min from the time the resin cement was stabilized on the ATR surface (Fig. 2B). 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 that pressure applied to either the microscope slide or ceramic discs provided similar thickness for all specimens.

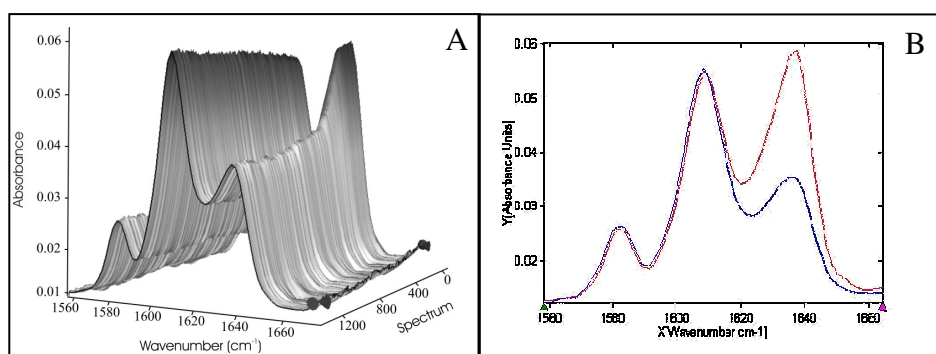


Figure 2: (A) Illustrative graph exhibiting the sequence of FTIR spectra created during kinetic analysis of polymerization of Calibra at 25°C (DLE group). The first and last spectra (B) from the 20-min kinetic analysis were used to calculate the DC.

Working time

In order to determine the WT of self-cured groups of resin cements polymerized after cement mixture at 25°C, 37°C, or 50°C, individual analysis of polymerization kinetic graphs from each specimen was performed to determine the time (in seconds) elapsed between the first FTIR spectrum and the moment when the polymerization started (Figs 3 A and B).

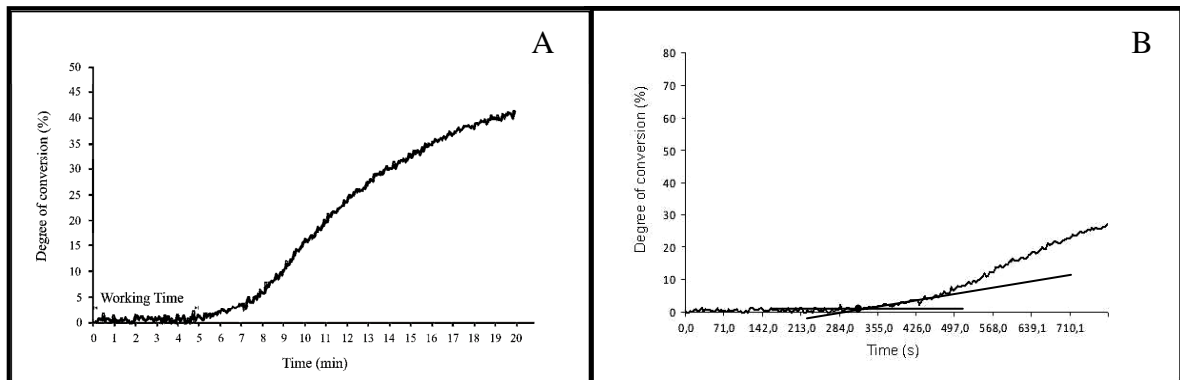


Figure 3: (A) Illustrative polymerization kinetics of Variolink II polymerized by the self-curing mode exhibiting the time elapsed before the beginning of polymerization, and (B) the moment when the polymerization started.

Statistical analysis

For the DC, data were evaluated by two-way ANOVA (“temperature” and “curing mode” factors) followed by Tukey’s post-hoc test (pre-set alpha of 5%). For the WT analysis, the results were subjected to two-way ANOVA (“temperature” and “product” factors) followed by Tukey’s post-hoc test (pre-set alpha of 5%).

RESULTS

Degree of conversion and polymerization kinetics

Table 2 shows the DC (%) of Calibra and Variolink II exposed to four curing modes at three temperatures. Significant interaction between “curing mode” and “temperature” factors for both products ($p=0.00001$). The increase in temperature resulted in significantly higher DC regardless of curing mode and product. The effect of different curing modes on the DC varied according to the temperature for both cements. For Calibra at 25°C, only e.max 3.0 group exhibited lower DC than the other groups, while no significant difference in DC was noted among groups at either 37°C or 50°C. For Variolink II at 25°C, SC group showed lower DC than e.max 3.0 group, which in turn exhibited lower DC than e.max1.5 and DLE group. At 37°C, the DLE group showed higher DC than e.max 1.5 and e.max3.0 groups, which in turn exhibited higher DC than SC group. At 50°C, no significant

difference in DC was noted among light activated groups, which showed higher DC than SC group.

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Table 2. DC (standard deviation) means (standard deviation) of Calibra and Variolink II exposed to three temperatures and four curing modes.

Resin cement	Temperature	DLE	e.max 1.5mm	e.max 3.0mm	SC
Calibra	25°C	55.9 (1.4) Ca	56.0 (1.9) Ca	52.9 (1.6) Cb	56.0 (1.2) Ca
	37°C	63.2 (0.9) Ba	62.4 (0.7) Ba	62.9 (1.1) Ba	61.7 (1.0) Ba
	50°C	67.3 (2.3) Aa	67.7 (1.3) Aa	67.2 (2.6) Aa	66.0 (1.6) Aa
Variolink II	25°C	62.3 (0.7) Ca	59.9 (2.4) Ca	54.5 (3.2) Cb	41.7 (1.0) Cc
	37°C	67.3 (1.4) Ba	64.1 (1.8) Bb	63.6 (1.3) Bb	56.8 (1.3) Bc
	50°C	72.6 (1.1) Aa	72.4 (1.3) Aa	70.3 (1.3) Aa	65.6 (1.0) Ab

Significant differences are indicated by different letters (Upper case letters within column; Lower case letter within row) according to Tukey's post-hoc test at a pre-set alpha of 5%. No comparisons were made between products.

Figures 4 and 5 show the polymerization kinetics of Calibra and Variolink II, respectively, at 25°C (A), 37°C (B), and 50°C (C) in comparison to the 20-min DC value from DLE group at 25 °C (dashed line). At 25°C, e.max1.5, e.max3.0, and SC groups of Calibra exhibited DC values as high as those observed in the DLE groups within 20 min, but lower polymerization rates were observed in e.max1.5, e.max3.0, and SC groups in comparison to that from DLE group. The polymerization of Calibra for e.max1.5 and e.max3.0 groups depended mostly on the self-curing components to lead to DC values as high as those observed in DLE group. At 37°C and 50°C, the polymerization rates of e.max1.5, e.max3.0, and SC groups were almost similar, as well as the 20-min DC values.

Differently from Calibra, the polymerization kinetics of Variolink II (Figure 5) depended mostly on the light intensity reaching the resin cement layer to result in similar DC to that observed in the DLE group at 25°C, as a relatively low polymerization rate was noted in the SC group. At 25°C, polymerization of Variolink II occurred at higher rate in DLE groups than in e.max1.5, which in turn exhibited higher rate than e.max3.0 group. At 37°C, the polymerization rates of Variolink II for e.max1.5 and e.max3.0 were similar to each other, but lower than that of DLE group. At 50°C, the polymerization rates of e.max1.5 group was similar to that of DLE group, which was slightly higher than that measured for 3.0.emax group. A significant increase in the polymerization rate was noted for SC group with increasing temperature from 25°C to 50°C, but the polymerization rate was always lower than that determined for light-cured groups.

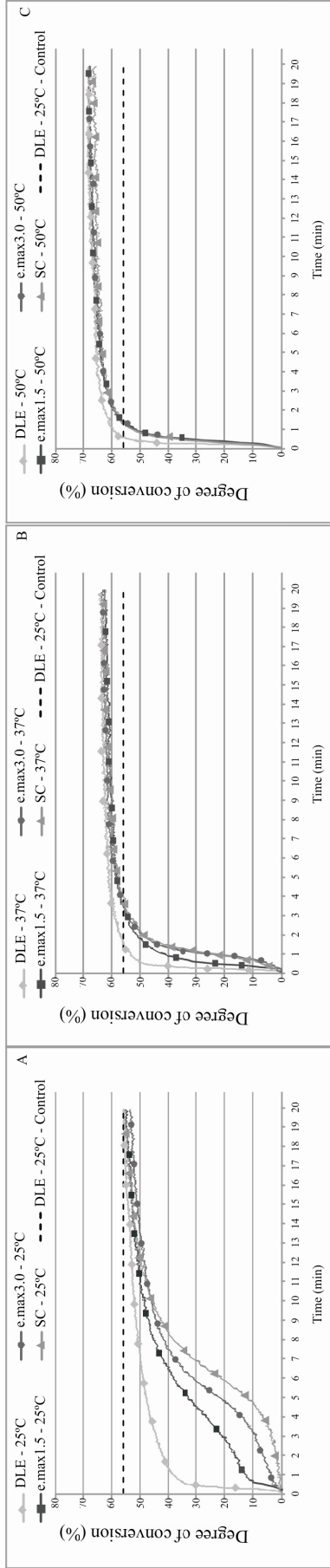


Figure 4: Polymerization kinetics of Calibra at a 25°C (A), 37°C (B), and 50°C (C) exposed to 4 curing conditions. The dashed line represents the degree of conversion after light activation through a glass slide at 25°C

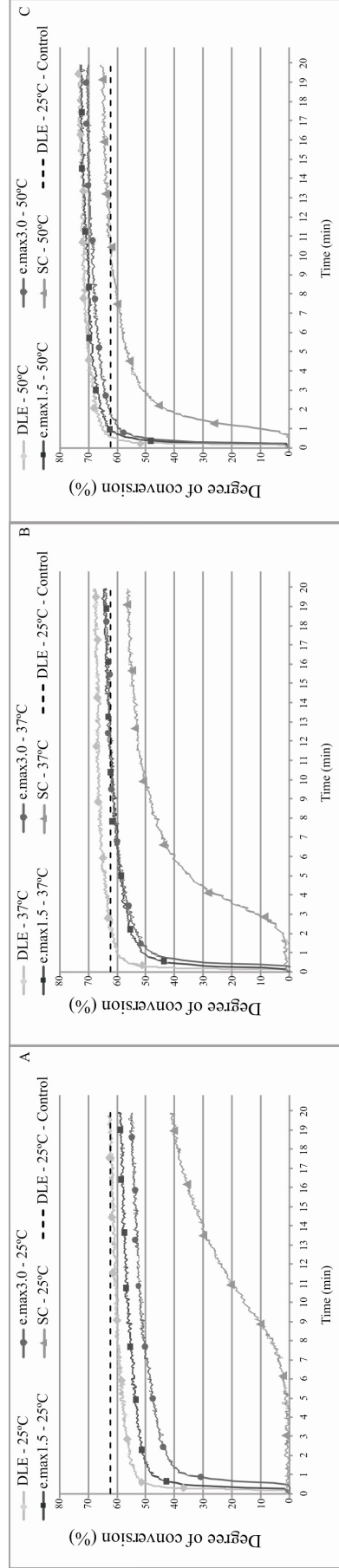


Figure 5: Polymerization kinetics of Variolink II at a 25°C (A), 37°C (B), and 50°C (C) exposed to 4 curing conditions. The dashed line represents the degree of conversion after light activation through a glass slide at 25°C

Working Time

The WT values of both resin cements in the self-curing mode at different temperatures are displayed in Table 3. Significant interaction was detected by the 2-way ANOVA between “product” and “temperature” factors ($p=0.00001$). A significant decrease of approximately 73% in the WT was observed for Calibra when the temperature increased to 37°C. However, while no significant difference was noted between the group polymerized at 37°C and that at 50°C. For Variolink II, significant decreases of approximately 69% and 88% were observed in the WT when the resin cement temperature increased to 37°C and 50°C, respectively. Variolink II exhibited longer WT than Calibra regardless of temperature.

Table 3. Working time in seconds (standard deviation) of Calibra and Variolink II polymerized at three temperatures.

	Calibra	Variolink II
25 °C	83.4 (7.4) Ab	311.4 (80.7) Aa
37 °C	22.5 (10.0) Bb	95.3 (31.0) Ba
50 °C	10.1 (4.2) BB	36.7 (5.3) Ca

Significant differences are indicated by different letters (Upper case letters within column; Lower case letter within row) according to Tukey’s post-hoc test at a pre-set alpha of 5%.

DISCUSSION

The results from the current study demonstrated that higher temperatures led to significant increase in the DC of both dual-cured resin cements, regardless of the activation mode. Similar finding was observed for resin composites by Daronch et al.,^{23,24} who attributed the increased DC to the higher radical mobility and collision frequency of unreacted active groups and radicals due to the decrease in material viscosity promoted by increased resin temperature. Therefore, the first research hypothesis was validated.

In contrast to light-cured resin composites, dual-cured resin cements have not only photoinitiators in their composition but also self-curing components, such as benzoyl peroxide. Once the degradation rate of benzoyl peroxide to radicals increases with increasing temperature,²⁹ radicals are created more quickly from its decomposition and for this reason, the increased DC in both resin cements could

also be attributed to the higher amount of these radicals created during polymerization. In addition, the presence of high amounts of radicals in the early stages of polymerization has also contributed to the evidence that DC values of heated cements were as high as those observed in the DLE groups at 25°C even in clinically unfavorable conditions, such as when a thick indirect restoration was interposed between the curing unit tip and the underlying resin cement layer.

Despite the previously reported severe attenuation in light intensity caused by the presence of indirect restorations between the resin cement layer and curing unit tip,^{3,14} both resin cements from e.max1.5 groups at 25°C showed DC values as high as those observed in DLE groups. The results are in agreement with those reported by other authors,^{3,14} who demonstrated that the use of indirect restorations with thickness lower than 2 mm does not compromise the DC of selected dual-cured resin cements. Based on the analysis of polymerization kinetics of Calibra at 25 °C (Fig. 4), the importance of self-curing components to compensate for the lower light intensity of the curing light reaching the resin cement was evident, as a significant increase in polymerization rate with time was noted after light exposure in both e.max1.5 and e.max3.0 groups (Figure 4A). On the other hand, for Variolink II, light-curing components were responsible for compensating the light attenuation caused by the 1.5-mm thick indirect restoration to generate enough amounts of free radicals to promote similar DC values to those observed in the control group, as evidenced by the proximity of the curves in Figure 5A.

This study also evaluated the simulation of a more critical clinical condition, in which the curing light passes through a thick ceramic layer to reach the resin cement layer at proximal areas. For both resin cements at 25°C, e.max3.0 groups exhibited lower DC than DLE and e.max1.5 groups. These results can be explained by the fact that almost no light can penetrate 3-mm thick indirect restorations,^{14,30} so polymerization relied mostly on the self-curing mode. Nevertheless, for Variolink II, the severely attenuated light reaching the resin cement led to higher DC values than the SC mode alone. Curiously, for Calibra, light activation through 3-mm thick ceramic disc resulted in lower DC than self-polymerization. Although the polymerization of Calibra in e.max3.0 group relied mostly on the self-curing components as demonstrated by the polymerization kinetics analysis (Fig. 4A), the SC group exhibited further increase in DC values from the 10th to 20th min in comparison to the polymerization kinetics of e.max3.0 group within the

same interval. Similar polymerization profile was observed by Moraes et al. (2009)³¹, who compared the polymerization kinetics initiated by short-time light exposure to that initiated by self-curing components. One explanation for this finding is related to the possible initial vitrification promoted by light-activation, which may have led to an increase in resin viscosity. As a consequence, Calibra from e.max3.0 group may have shown higher resin viscosity at the 10th min after resin cement application in comparison with Calibra from SC group. Thus, the initial polymer cross-linking would restrict the mobility of unreacted specimens and radicals created from the self-curing components,¹⁶ resulting in the lower 20-min DC values observed in e.max3.0 group.

In both resin cements, the effects of the higher radical mobility and collision frequency of unreacted active groups and radicals were also observed in the polymerization kinetics analysis of DLE, 15.e.max, e.max3.0, and SC groups (Figures 4 and 5), which showed higher polymerization rates with increased temperatures. As a consequence, no significant difference in DC was noted among DLE, e.max1.5, and e.max3.0 groups at 50°C for both materials. Therefore, as also shown by Daronch et al.,²³ these findings demonstrate that the increased temperature during polymerization compensated for the lower energy delivered from the light-curing unit in these groups. However, for Variolink II, groups e.max1.5, and e.max3.0 at 37°C showed lower DC than DLE group at the same temperature, what indicates that the above-mentioned compensation did not completely occur for these experimental groups. Nevertheless, it should be emphasized that for Variolink II, the DC of the SC, e.max1.5, and e.max3.0 groups increased 52%, 21%, and 29%, respectively, when the temperature increased from 25°C to 50°C, while the DC of the DLE group increased only 16%. For this reason, it is possible to speculate that curing modes responsible for poorer polymerization at 25°C were more influenced by the increase in temperature than those responsible for optimum resin cement polymerization.

In the current study, Calibra at 25°C exhibited a WT of approximately 1.4 minute (83.4 s) while Variolink II had a WT of about 5.5 minutes (311.4 s). The difference in WT between these two products might be mainly attributed to the concentration of inhibitors and benzoyl peroxide in both products, as Calibra has approximately 2% of benzoyl peroxide while Variolink II has approximately 1% (information from MSDS). For the same reason, the SC mode of Calibra provided DC

values as high as those observed in the control group while the SC mode of Variolink II was less effective than the other curing modes.

Similarly to the findings from other studies,^{25,26,32} the increased temperature reduced the WT to clinically unfavorable intervals for both resin cements in most conditions. For instance, the reduction in the WT of Calibra from 83.4 s (25°C) to 22.5 s (37°C) and 10.1 s (50°C) might compromise the seating of the indirect restoration and the success of the restorative procedure as a consequence. Based on this result, it seems that pre-heating of resin cements with higher content of self-curing components should be avoided. Significant reduction in the WT was also observed for Variolink II, as it was reduced from approximately 5.5 min (311.4 s) (25°C) to 1.5 min (95.3 s) (37°C) and 36.7 s (50°C). Although the use of Variolink II at 50°C led to unfavorable WT (36.7 s), the use of this resin cement at a temperature close to that of the oral cavity (37°C) led to a WT (1.5 min) ranging within the time considered by manufacturers as acceptable for clinicians to seat the indirect restoration. Thus, the second research hypothesis was partially rejected.

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.²⁴ For this reason, a drop in cement temperature is expected even if the resin cement is coupled to a heated indirect restoration, so the effects of increased temperature in the WT and DC might not be as evident as those observed in this study. However, it should be emphasized that the resin cement temperature may be further influenced by the tooth and intraoral temperatures, which are close to 37°C. Therefore, it is expected that resin cements when applied to the prepared tooth may present polymerization features similar to those observed in the current study when the temperature of 37°C was used. However, no information was found in the literature regarding the effects of intraoral and tooth temperature on the polymerization features of dual-cured resin cements when they are not pre-heated. Further studies simulating this clinical condition are required to evaluate the effects of temperature drop on the mechanical properties and monomer conversion of resin cements.

CONCLUSION

Based on the results of the current study, it was possible to conclude that:

- The use of dual-cured resin cements with increased temperature can lead to higher DC even in the most critical clinical situation, so the increased resin cement temperature compensated for the lower energy delivered from the light curing unit caused by the presence of indirect restorations with varying thicknesses.
- Caution is recommended before the clinician decides to warm up the resin cement once such procedure may compromise the WT depending on the temperature and product.

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4. CONCLUSÃO

De acordo com os resultados de espectroscopia infravermelha com transformada de Fourier, pode-se concluir que:

- O aquecimento dos cimentos resinosos de dupla ativação Calibra e Variolink II resultou em um aumento no seu GC, independente do modo de ativação utilizado.
- Os efeitos da maior temperatura dos cimentos duais compensou a diminuição da irradiância da luz fotoativadora devido à presença de restaurações indiretas de cerâmica com diferentes espessuras.
- Recomenda-se cuidado ao se decidir pela utilização do pré-aquecimento uma vez que o mesmo pode comprometer o tempo de trabalhos de cimentos resinosos duais dependendo do produto e da temperatura utilizados.

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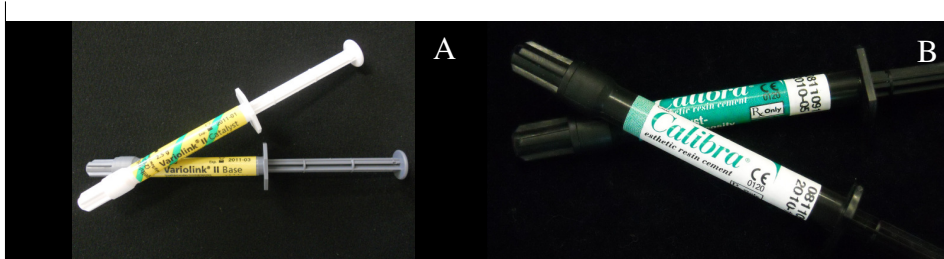
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ANEXOS

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



Cimentos de dupla-ativação utilizados no estudo, VariolinkII (Ivoclar Vivadent) (A) e Calibra (Dentsply Caulk) (B).

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

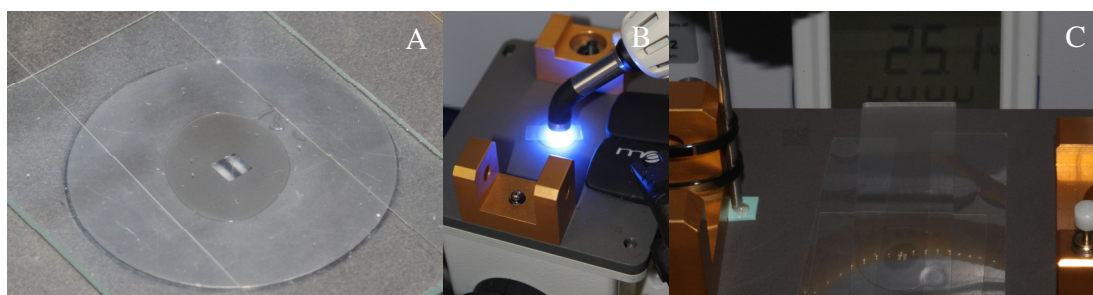


Espectrofômetro infravermelho com transformada de Fourier – FTIR (Tensor 27, Bruker Comp), unidade de reflectância total atenuada – ATR (Satandard Golden Gate, Specac).

ANEXO C – Preparação dos espécimes

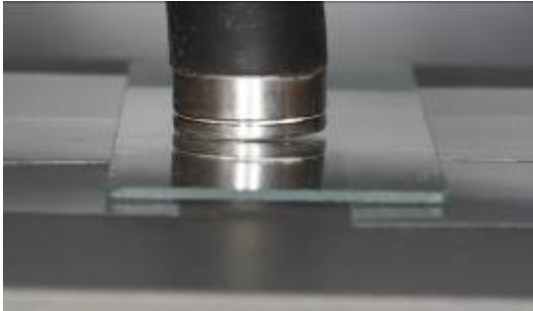


Adequação da mesa de ATR para controle de temperatura. Dispositivo para aquecimento acoplado a mesa (A); adaptação do termômetro com termopar tipo K para controle constante da temperatura (B); agitador mecânico com controle de temperatura utilizado para aquecer os cimentos (C).



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).

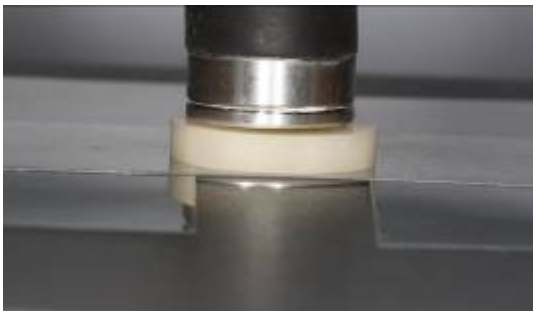
ANEXO D – Modos de ativação



Fotoativação através de lâmina de vidro.



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



Fotoativação através de disco de cerâmica 3,0mm (e.max3,0).



Autopolimerização (DLE).

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

VARIOLINK II

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

CODIGO DO PROJETO:

RESPONSAVEL: CESAR

DELINEAMENTO EXPERIMENTAL: 3x4

OBSERVACOES NAO TRANSFORMADAS

NOME DOS FATORES

```
-----
FATOR  NOME
-----
A  TEMP
B  ATIV
-----
```

QUADRO DA ANALISE DE VARIANCIA

CAUSAS DA VARIACAO	G.L.	S.Q.	Q.M.	VALOR F	PROB.>F
TEMP	2	2938.6232512	1469.3116256	578.3797	0.00001
ATIV	3	1671.0513076	557.0171025	219.2642	0.00001
TEM*ATI	6	374.3594639	62.3932440	24.5605	0.00001
RESIDUO	60	152.4235697	2.5403928		

TOTAL 71 5136.4575924

MEDIA GERAL = 62.594444

COEFICIENTE DE VARIACAO = 2.546 %

TESTE DE TUKEY PARA MEDIAS DE TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%			
1	3	50C	24	70.241666	70.241666	a	A			
2	2	37C	24	62.937503	62.937503	b	B			
			3	1	25C	24	54.604167	54.604167	c	C

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 1.10617 - D.M.S. 1% = 1.39248

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE AUTO DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	65.649999	65.649999	a	A
2	2	37C	6	56.766668	56.766668	b	B
3	1	25C	6	41.733335	41.733335	c	C

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE FOTO LV DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	72.566666	72.566666	a	A
2	2	37C	6	67.266673	67.266673	b	B
3	1	25C	6	62.233332	62.233332	c	C

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE A1.5 DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	72.383331	72.383331	a	A
2	2	37C	6	64.066671	64.066671	b	B
3	1	25C	6	59.833333	59.833333	c	C

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE A3.0 DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	70.366669	70.366669	a	A
2	2	37C	6	63.649999	63.649999	b	B
3	1	25C	6	54.616669	54.616669	c	C

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA
INDICADO

D.M.S. 5% = 2.21235 - D.M.S. 1% = 2.78496

TESTE DE TUKEY PARA MEDIAS DE ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	FOTO LV	18	67.355557	67.355557	a	A
2	3	A1.5	18	65.427778	65.427778	b	B
3	4	A3.0	18	62.877779	62.877779	c	C
4	1	AUTO	18	54.716667	54.716667	d	D

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA
INDICADO

D.M.S. 5% = 1.40503 - D.M.S. 1% = 1.72811

TESTE DE TUKEY PARA MEDIAS DE ATIV
DENTRO DE 25C DO FATOR TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	FOTO LV	6	62.233332	62.233332	a	A
2	3	A1.5	6	59.833333	59.833333	a	A
3	4	A3.0	6	54.616669	54.616669	b	B
4	1	AUTO	6	41.733335	41.733335	c	C

TESTE DE TUKEY PARA MEDIAS DE ATIV
DENTRO DE 37C DO FATOR TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	FOTO LV	6	67.266673	67.266673	a	A
2	3	A1.5	6	64.066671	64.066671	b	B
3	4	A3.0	6	63.649999	63.649999	b	B
4	1	AUTO	6	56.766668	56.766668	c	C

TESTE DE TUKEY PARA MEDIAS DE ATIV
DENTRO DE 50C DO FATOR TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	FOTO LV	6	72.566666	72.566666	a	A
2	3	A1.5	6	72.383331	72.383331	a	A
3	4	A3.0	6	70.366669	70.366669	a	A
4	1	AUTO	6	65.649999	65.649999	b	B

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA
INDICADO

D.M.S. 5% = 2.43358 - D.M.S. 1% = 2.99318

DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 2

CALIBRA

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

CODIGO DO PROJETO:

RESPONSAVEL: CESAR

DELINEAMENTO EXPERIMENTAL: 3x4

OBSERVACOES NAO TRANSFORMADAS

NOME DOS FATORES

 FATOR NOME

 A TEMP
 B ATIV

QUADRO DA ANALISE DE VARIANCIA

CAUSAS DA VARIACAO	G.L.	S.Q.	Q.M.	VALOR F	PROB.>F
TEMP	2	1725.3200282	862.6600141	350.6782	0.00001
ATIV	3	18.4576111	6.1525370	2.5011	0.06682
TEM*ATI	6	41.1871107	6.8645185	2.7905	0.01830
RESIDUO	60	147.5985503	2.4599758		
TOTAL	71	1932.5633002			

MEDIA GERAL = 61.590279

COEFICIENTE DE VARIACAO = 2.547 %

TESTE DE TUKEY PARA MEDIAS DE TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	24	67.037498	67.037498	a	A
2	2	37C	24	62.566668	62.566668	b	B
3	1	25C	24	55.166668	55.166668	c	C

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 1.08853 - D.M.S. 1% = 1.37026

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE AUTO DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	65.983332	65.983332	a	A
2	2	37C	6	61.683334	61.683334	b	B
3	1	25C	6	55.933334	55.933334	c	C

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE FOTO LV DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	67.349996	67.349996	a	A
2	2	37C	6	63.233337	63.233337	b	B
3	1	25C	6	55.866669	55.866669	c	C

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE A1.5 DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	67.666667	67.666667	a	A
2	2	37C	6	62.433334	62.433334	b	B
3	1	25C	6	55.983337	55.983337	c	C

TESTE DE TUKEY PARA MEDIAS DE TEMP
DENTRO DE A3.0 DO FATOR ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	50C	6	67.149999	67.149999	a	A
2	2	37C	6	62.916667	62.916667	b	B
3	1	25C	6	52.883331	52.883331	c	C

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA
INDICADO

D.M.S. 5% = 2.17705 - D.M.S. 1% = 2.74052

TESTE DE TUKEY PARA MEDIAS DE ATIV

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	FOTO LV	18	62.150001	62.150001	a	A
2	3	A1.5	18	62.027779	62.027779	a	A
3	1	AUTO	18	61.200000	61.200000	a	A
4	4	A3.0	18	60.983332	60.983332	a	A

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA
INDICADO

D.M.S. 5% = 1.38261 - D.M.S. 1% = 1.70054

TESTE DE TUKEY PARA MEDIAS DE ATIV
DENTRO DE 25C DO FATOR TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	A1.5	6	55.983337	55.983337	a	A
2	1	AUTO	6	55.933334	55.933334	a	A
3	2	FOTO LV	6	55.866669	55.866669	a	A
4	4	A3.0	6	52.883331	52.883331	b	B

TESTE DE TUKEY PARA MEDIAS DE ATIV
DENTRO DE 37C DO FATOR TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	FOTO LV	6	63.233337	63.233337	a	A
2	4	A3.0	6	62.916667	62.916667	a	A
3	3	A1.5	6	62.433334	62.433334	a	A
4	1	AUTO	6	61.683334	61.683334	a	A

TESTE DE TUKEY PARA MEDIAS DE ATIV
DENTRO DE 50C DO FATOR TEMP

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	3	A1.5	6	67.666667	67.666667	a	A
2	2	FOTO LV	6	67.349996	67.349996	a	A
3	4	A3.0	6	67.149999	67.149999	a	A
4	1	AUTO	6	65.983332	65.983332	a	A

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA
INDICADO

D.M.S. 5% = 2.39476 - D.M.S. 1% = 2.94542

DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 2.39476 - D.M.S. 1% =

ANEXO F – Análise estatística para os resultados do tempo de trabalho

TEMPO DE TRABALHO

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

CODIGO DO PROJETO:

RESPONSAVEL: MICHELE

DELINEAMENTO EXPERIMENTAL:

OBSERVACOES NAO TRANSFORMADAS

NOME DOS FATORES

 FATOR NOME

 A CIMENTO
 B TEMPER

QUADRO DA ANALISE DE VARIANCIA

CAUSAS DA VARIACAO	G.L.	S.Q.	Q.M.	VALOR F	PROB.>F
CIMENTO	1	74428.9062768	74428.9062768	564.0922	0.00001
TEMPER	2	159874.5833545	79937.2916772	605.8399	0.00001
CIM*TEM	2	43114.0761035	21557.0380518	163.3795	0.00001
RESIDUO	30	3958.3375360	131.9445845		
TOTAL	35	281375.9032708			

MEDIA GERAL = 84.141663

COEFICIENTE DE VARIACAO = 13.652 %

TESTE DE TUKEY PARA MEDIAS DE CIMENTO

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	VARIOL	18	129.611104	129.611104	a	A
2	1	CALIBRA	18	38.672222	38.672222	b	B

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 7.82451 - D.M.S. 1% = 10.53196

TESTE DE TUKEY PARA MEDIAS DE CIMENTO
DENTRO DE 25C DO FATOR TEMPER

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	VARIOL	6	270.416646	270.416646	a	A
2	1	CALIBRA	6	83.416667	83.416667	b	B

TESTE DE TUKEY PARA MEDIAS DE CIMENTO
DENTRO DE 37C DO FATOR TEMPER

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	VARIOL	6	81.700002	81.700002	a	A
2	1	CALIBRA	6	22.516665	22.516665	b	B

TESTE DE TUKEY PARA MEDIAS DE CIMENTO
DENTRO DE 50C DO FATOR TEMPER

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	2	VARIOL	6	36.716665	36.716665	a	A
2	1	CALIBRA	6	10.083333	10.083333	b	B

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 13.55246 - D.M.S. 1% = 18.24189

TESTE DE TUKEY PARA MEDIAS DE TEMPER

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	1	25C	12	176.916656	176.916656	a	A
2	2	37C	12	52.108334	52.108334	b	B
3	3	50C	12	23.399999	23.399999	c	C

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 11.57259 - D.M.S. 1% = 14.78904

TESTE DE TUKEY PARA MEDIAS DE TEMPER
DENTRO DE CALIBRA DO FATOR CIMENTO

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	1	25C	6	83.416667	83.416667	a	A
2	2	37C	6	22.516665	22.516665	b	B
3	3	50C	6	10.083333	10.083333	b	B

TESTE DE TUKEY PARA MEDIAS DE TEMPER
DENTRO DE VARIOL DO FATOR CIMENTO

NUM.ORDEM	NUM.TRAT.	NOME	NUM.REPET.	MEDIAS	MEDIAS ORIGINAIS	5%	1%
1	1	25C	6	270.416646	270.416646	a	A
2	2	37C	6	81.700002	81.700002	b	B
3	3	50C	6	36.716665	36.716665	c	C

MEDIAS SEGUIDAS POR LETRAS DISTINTAS DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S. 5% = 16.36611 - D.M.S. 1% = 20.91486

DIFEREM ENTRE SI AO NIVEL DE SIGNIFICANCIA INDICADO

D.M.S