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ALEXANDRE MORAIS

EFEITO DO PRÉ-AQUECIMENTO DE CIMENTOS RESINOSOS DE DUPLA-ATIVAÇÃO NA RESISTÊNCIA DE UNIÃO DE RESTAURAÇÕES INDIRETAS DE COMPÓSITO

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

Co-orientador: Prof. Dr. André Figueiredo Reis

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"... buscai antes o Reino de Deus e a sua justiça e todas as coisas vos serão dadas por acréscimo"

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RESUMO

O objetivo deste estudo foi avaliar os efeitos do pré-aquecimento de cimentos resinosos polimerizados na resistência à tração por meio do método de microtração (MTBS) de restaurações indiretas à dentina. Superfícies oclusais de dentina de 40 terceiros molares humanos foram expostas e aplainadas. Os dentes foram divididos em 8 grupos (n = 5) de acordo com a temperatura dos cimentos resinosos (25ºC e 50ºC), modos de polimerização (fotoativação ou autopolimerização) e produtos [Excite DSC / Variolink II (VII) e XP Bond / Calibra (Cal)]. Adesivos foram aplicados nas superfícies dentinárias de acordo com as instruções dos fabricantes. Para os materiais pré-aquecidos, os cimentos resinosos e os discos de resina pré-polimerizados (2 mm de espessura / TPH-Spectrum) foram aquecidos a 50ºC previamente à mistura sobre uma superfície aquecida de um agitador mecânico. Os cimentos resinosos foram misturados e aplicados nos discos de resina composta, que posteriormente foram aderidos às superfícies dentinárias. O material foi foto-ativado de acordo com instruções do fabricante (PP / Optilux 501), ou foi deixado para auto-polimerizar (AP). Em seguida, os dentes restaurados foram armazenados em umidade relativa de 100% a 37ºC por 7 dias e foram ambos seccionados mésio-distalmente e buco-lingualmente para obtenção de múltiplos 'palitos' (aproximadamente 1 mm^2 de área transversal). Cada amostra foi testada em tensão a uma velocidade de 1 mm/min até o momento da fratura. Os dados (MPa) foram analisados por ANOVA três fatores e teste Tukey (α = 5%). Os padrões de fratura dos espécimes testados foram analisados usando lupa estereoscópica. VII pré-aquecido mostrou MTBS superior ao VII à 25ºC, independentemente do modo de ativação (p = 0,02476). Não houve diferença significativa entre Cal à 50ºC e Cal à 25ºC, independentemente do modo de ativação. A utilização de cimentos resinosos duais com o aumento da temperatura pode melhorar a resistência de união de restaurações indiretas à dentina. No entanto, os efeitos do aumento da temperatura de cimento são produto-dependentes.

Palavras-chave: cimentos resinosos duais, ensaio de microtração, temperatura, restauração indireta

ABSTRACT

The aim of this study was to evaluate the effects of resin cements polymerized with increased temperature on microtensile bond strength (µTBS) of indirect restorations to dentin. Occlusal dentin surfaces of 40 human third molars were exposed and flattened. Teeth were assigned to 8 groups (n=5) according to the temperature of the resin cements (25^{o}C or 50^{o}C), polymerizing modes (dual- or self-curing modes), and products [(Excite DSC/Variolink II (VII) and XP Bond / Calibra (Cal)]. Bonding agents were applied to dentin surfaces according to manufacturers' instructions. For pre-heated materials, resin cements were heated to 50^{o}C on a heated stirrer surface prior to mixture. Resin cements were mixed and applied to pre-polymerized resin discs (2-mm thick/TPH-Spectrum), which were subsequently bonded to the dentin surfaces. The restored teeth were light-cured according to manufacturers' instructions (Optilux 501), or were allowed to self-cure. Restored teeth were stored in relative humidity at 37°C for 7 days and were both mesio-distally and bucco-lingually sectioned to obtain multiple bonded beams (approximately 1 mm^2 of cross-sectional area). Each specimen was tested in tension at a crosshead speed of 1 mm/min until failure. Data (MPa) were analyzed by 3-way ANOVA and Tukey's post-hoc test (pre-set alpha of 5%). Failure patterns of tested specimens were analyzed using stereoscopic microscope. Pre-heated VII showed higher µTBS than VII at 25^{o}C regardless of activation mode (p=0.02476). No significant difference in µTBS was noted between Cal at 50 ^{o}C and Cal at 25^{o}C regardless of activation mode. The use of dual-cured resin cements with increased temperature may improve bond strength of indirect restorations to dentin. However, the effects of increased cement temperature were product-dependent.

Keywords: Dual-cured resin cements, Microtensile bond strength, temperature, indirect restoration

SUMÁRIO

Página

1. INTRODUÇÃO E JUSTIFICATIVA 11
2. PROPOSIÇÃO 14
3. METODOLOGIA E RESULTADOS 15
REFERÊNCIAS BIBLIOGRÁFICAS 35
ANEXOS.. 38

1. INTRODUÇÃO E JUSTIFICATIVA

Restaurações indiretas em metal, cerâmica ou compósito necessitam ser unidas ao dente por meio de um agente cimentante. Frente às exigências estéticas, a odontologia vem optando por restaurações diretas em compósitos e indiretas em compósitos ou cerâmica ao invés das metálicas. O uso dos cimentos resinosos está presente não somente na cimentação de restaurações indiretas tipo *metal-free*, como também na cimentação de *brackets* ortodônticos, pinos metálicos ou de fibras, e próteses parciais adesivas. Sendo assim, o desenvolvimento de cimentos resinosos com propriedades mecânicas superiores às do cimento de fosfato de zinco é um dos fatores mais importantes para se garantir a longevidade dessas restaurações.

A retenção de restaurações convencionais indiretas e próteses fixas podem ser melhoradas com o uso dos cimentos resinosos (EL-MOWAFY et al., 1996). A indicação e utilização dessas restaurações indiretas em cerâmicas odontológicas ou compósitos têm crescido consideravelmente nos últimos anos. Além da melhora nas propriedades mecânicas desses materiais restauradores, como o aumento na resistência ao desgaste, na resistência à compressão e flexão (LEINFELDER, 2005, MANHART et al., 2004, VAN DIJKEN, 1994), o desenvolvimento e aprimoramento dos sistemas de cimentação (agentes de união/cimentos resinosos) contribuiu para uma melhor união das restaurações indiretas à estrutura dental, promovendo maior segurança aos clínicos no momento da cimentação (INOKOSHI et al., 1993, SJOGREN et al., 1995). A obtenção de uma união efetiva entre os materiais e os tecidos dentários duros tem sido um dos principais objetivos de investigação de muitos pesquisadores (CARVALHO et al., 2004, FUSAYAMA et al., 1979, GIANNINI et al., 2004, REIS et al., 2003). Para se garantir o sucesso do procedimento restaurador indireto, é interessante que os agentes cimentantes sejam biocompatíveis, cariostáticos e capazes de selar as margens da restauração, além de apresentar propriedades mecânicas adequadas (DE LA MACORRA and PRADIES, 2002, LEINFELDER, 2005, MANHART et al., 2004).

Sendo assim, a utilização de cimentos resinosos na cimentação de peças protéticas tem aumentado consideravelmente, por apresentarem algumas vantagens como baixa solubilidade, consistência e espessura da película adequada, propriedades mecânicas melhores do que as apresentadas pelos cimentos convencionais como o fosfato de zinco, adesão otimizada às estruturas dentais e aos materiais restauradores e reduzida microinfiltração (CAUGHMAN et al., 2001, FONSECA et al., 2005, LEINFELDER, 2005, ROSENSTIEL et al., 1998).

A maioria dos cimentos resinosos é composta por uma matriz de resina de Metacrilato Diglicidil Bisfenol A (Bis-GMA) ou de dimetacrilato uretano (UDMA) reforçada com partículas inorgânicas em diferentes concentrações. Os cimentos resinosos podem ser classificados em fotopolimerizáveis, autopolimerizáveis, ou duais, os quais apresentam tanto componentes fotopolimerizáveis como autopolimerizáveis. A escolha dentre essa classificação é primariamente baseada na sua utilização. Na comparação entre estes diferentes tipos de cimentos, os fotopolimerizáveis oferecem vantagens clínicas de maior tempo e melhor controle do tempo de trabalho e maior estabilidade de cor (BRAUER et al., 1979, ROSENSTIEL et al., 1998). Entretanto, o uso dos cimentos apenas fotopolimerizáveis está limitado às situações em que a espessura e a cor da restauração indireta não afetam drasticamente a transmissão da luz para polimerizar o cimento, como cimentação de facetas ou incrustações muito finas (BREEDING et al., 1991, CHAN and BOYER, 1989, LEE et al., 2008). Os cimentos resinosos duais são indicados quando, no processo restaurador, a opacidade do material a ser cimentado pode atenuar ou inibir a luz fotoativadora que alcança o cimento (ARRAIS et al., 2008, BRAGA et al., 2002, MYERS et al., 1994).

Nessas situações, a intensidade de luz que chega até o cimento pode ser suficiente para iniciar o processo de polimerização, mas um iniciador autopolimerizável é necessário para contribuir para que ocorra a polimerização mesmo quando a luz fotoativadora é severamente atenuada ao atravessar a restauração indireta. Estudos sugerem que a passagem da luz é possível somente quando a espessura do material restaurador indireto é menor que três milímetros (EL-BADRAWY e EL-MOWAFY, 1995, EL-MOWAFY e RUBO, 2000). Procedimentos restauradores nos condutos radiculares e cimentação de peças protéticas são exemplos de indicações de cimentos que apresentem também autopolimerização, uma vez que a ativação pela luz é deficiente (CAUGHMAN et al., 2001).

Tem sido mostrado que resinas compostas aquecidas a uma temperatura entre 40-60ºC previamente à polimerização apresentam maior grau de conversão quando comparadas às mesmas resinas quando polimerizadas em temperatura ambiente

(DARONCH et al., 2005, DARONCH et al., 2006). Alguns autores atribuem esse aumento no grau de conversão à diminuição na viscosidade do material, aumentando a mobilidade de radicais livres e a cinética de polimerização dos compósitos (DARONCH et al., 2005, DARONCH et al., 2006, TRUJILLO et al., 2004). Diferenças significantes a esse respeito têm sido levantadas entre a temperatura de estocagem em refrigeração e a temperatura intraoral (DARONCH et al., 2005, DARONCH et al., 2006). Os compósitos estocados em refrigeração obtiveram valores de conversão mais baixos. Ainda com menos tempo de exposição da fonte ativadora, o compósito aquecido teve resultados maiores de conversão do que o compósito em temperatura ambiente (DARONCH et al., 2005). No entanto, ainda são poucos os trabalhos que têm avaliado os efeitos do pré-aquecimento de cimentos resinosos de dupla-ativação na resistência de união de restaurações indiretas em dentina (CANTORO et al., 2009, CANTORO et al., 2008). Uma vez que maior grau de conversão tem efeitos positivos em várias propriedades mecânicas de materiais resinosos, como dureza de superfície, resistência flexural e módulo de flexão, resistência à fratura, resistência à tensão e resistência ao desgaste (FERRACANE, 1985, FERRACANE et al., 1998, PEUTZFELDT, 1995), é esperado que características como resistência à fratura e resistência à tração sejam melhoradas com o pré-aquecimento dos cimentos resinosos.

2. PROPOSIÇÃO

O objetivo desse estudo foi analisar os efeitos do pré-aquecimento de cimentos resinosos de dupla ativação na resistência de união desses sistemas de cimentação entre dentina e restaurações indiretas de resina composta. A hipótese de pesquisa foi que o pré-aquecimento dos cimentos aumenta a resistência de união quando o cimento é fotoativado ou autopolimerizado.

3. METODOLOGIA E RESULTADOS

A presente dissertação está baseada no artigo “Effects of Pre-Heating of Dual-Cured Resin Cements on Tensile Bond Strength of Indirect Restoration to Dentin”

EFFECTS OF PRE-HEATING OF DUAL-CURED RESIN CEMENTS ON TENSILE BOND STRENGTH OF SIMULATED INDIRECT RESTORATION TO DENTIN

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ABSTRACT

The aim of this study was to evaluate the effects of resin cements polymerized with increased temperature on microtensile bond strength (µTBS) of indirect restorations to dentin. Occlusal dentin surfaces of 40 human third molars were exposed and flattened. Teeth were assigned to 8 groups (n=5) according to the temperature of the resin cements (25°C or 50°C), polymerizing modes (dual- or self-curing modes), and products [(Excite DSC/Variolink II (VII) and XP Bond / Calibra (Cal)]. Bonding agents were applied to dentin surfaces according to manufacturers' instructions. For pre-heated materials, resin cements were heated to 50°C on a heated stirrer surface prior to mixture. Resin cements were mixed and applied to pre-polymerized resin discs (2-mm thick/TPH-Spectrum), which were subsequently bonded to the dentin surfaces. The restored teeth were light-cured according to manufacturers' instructions (Optilux 501), or were allowed to self-cure. Restored teeth were stored in relative humidity at 37°C for 7 days and were both mesio-distally and bucco-lingually sectioned to obtain multiple bonded beams (approximately 1 mm^2 of cross-sectional area). Each specimen was tested in tension at a crosshead speed of 1 mm/min until failure. Data (MPa) were analyzed by 3-way ANOVA and Tukey's post-hoc test (pre-set alpha of 5%). Failure patterns of tested specimens were analyzed using stereoscopic microscope. Pre-heated VII showed higher µTBS than VII at 25°C regardless of activation mode (p=0.02476). No significant difference in µTBS was noted between Cal at 50 °C and Cal at 25°C regardless of activation mode. The use of dual-cured resin cements with increased temperature may improve bond strength of indirect restorations to dentin. However, the effects of increased cement temperature were product-dependent.

Keywords: Dual-cured resin cements, Microtensile bond strength, Temperature, Indirect restoration

INTRODUCTION

The use of indirect ceramic/composite restorations has increased considerably in the last years. Besides the improved mechanical properties, such as resistance to wear, flexural and compressive strength, in comparison to direct restorative materials,[1-3] indirect restorations can be reliably bonded to the prepared tooth with cementing systems (bonding agents / resin cements). For this reason, clinicians have trusted on the bonding procedures for indirect restorations.[4,5] In order to ensure a successful indirect restoration, the cement must present proper mechanical properties, biocompatibility, cariostatic effects, and ability of sealing the restoration margins.[1,2,6] Resin cements are widely applied for luting the indirect restoration to tooth because of their low solubility, proper consistence and film thickness, better mechanical properties than conventional cements, bonding to both tooth and restorative material, and lower microleakage.[1,7-9]

Resin cements can be classified according to their activation mode, so three types of resin cements are available: light-, self-, and dual-cured resin cements. Light-cured products depend exclusively on curing light to polymerize and have as advantages the working time controlled by the clinician and better color stability.[9,10] However, the use of light-cured resin cements is limited to the clinical situations where the translucency of the indirect restoration does not severely attenuate the curing light that reaches the resin cement layer.[11,12] On the other hand, dual-cured resin cements have both light- and self-curing components to ensure proper polymerization of the resin cement when curing light is attenuated or even totally blocked by the indirect restoration.[13-15] In these clinical situations, the self-curing components can continue the polymerization that was poorly initiated by the curing light reaching the resin cement layer at low intensity.[15-17] However, some studies have demonstrated that the self-curing components are not as effective as the light-curing components to provide proper polymerization.[13-17]

Recently, some studies have shown that pre-heating of resin composites at 40-60ºC leads to higher degree of conversion (DC) than composites polymerized at room temperature even when the pre-heated composites were exposed to curing light for a shorter period than that recommended by the manufacturers.[18,19] Daronch et al.[18,19] attributed the higher DC values to the decrease in resin viscosity before curing, which in turn would allow increased free radical mobility. Therefore, it is reasonable to assume that pre-heated dual-cured resin cements would show higher degree of conversion than the same materials polymerized at room temperature. As monomer conversion is directly related mechanical properties of polymers,[20-22] improved bond strength of indirect restorations to dentin would be expected even when the curing light is totally blocked by the indirect restoration and the cure of resin cement layer depends mostly on the self-curing mode. However, few studies have evaluated the effects of pre-heating resin cements on bond strength of indirect restorations to dentin.[23,24]

Therefore, the aim of the current study was to evaluate the effects of pre-heating dual-cured resin cements on the bond strength of indirect restorations to dentin. The research hypothesis was that pre-heated resin cements provide higher bond strength to dentin than cements at room temperature regardless of the activating mode.

MATERIALS AND METHODS

Indirect restorative procedures

Forty freshly extracted, human third molars were used in this study. The research protocol was approved by the Human Assurance Committee of Guarulhos University (90/09). Teeth were stored in saturated thymol (Symrise GmbH & Co, Holzminden, Germany) at 5°C for no longer than 3 months. Teeth were sectioned perpendicular to their long axis using diamond double face discs (7020, KG sorensen, SP, Brazil) to expose middle-depth occlusal dentin surface. The exposed dentin surfaces were wet ground (APL 4; Arotec, Cotia, SP,Brazil) with 600-grit SiC papers (Carborundum, Saint-Gobain Abrasivos, Guarulhos, SP, Brazil) to create a flat surface with standard smear layer.

Forty pre-cured, photo-polymerized composite resin discs (2-mm thick and 10 mm in diameter – A2 shade – TPH Spectrum, Dentsply Caulk, Milford, Del, USA) were prepared to simulate overlying laboratory-processed composite resin restorations. One surface of each pre-cured resin disc was airborne-particle abraded with 50 μm aluminum oxide particles (Asfer Indústria Química LTDA, São Caetano do Sul, SP, Brazil) for 10 s (distance from the tip: 1.5 cm) (Microjato Puls, Bio-Art, São Carlos, SP, Brazil). The dentin surfaces were acid etched with 35% phosphoric acid (Brazil Dentsply, Rio de Janeiro, RJ, Brazil) for 15 s, thoroughly water-rinsed, and excess water was removed with absorbent paper. Two dual-cured cementing systems were used (Table 1): Excite DSC/ Variolink II (Ivoclar Vivandent, Schaan, Liechtenstein) and XP Bond / Calibra (Dentsply Caulk, Milford, Del, USA). Adhesive systems were applied and light-activated according to manufacturers' instructions. The resin cements were applied at 25°C or 50°C, and were subjected to two activating modes, dual- and self-curing mode, so 8 experimental groups were evaluated (n=5). For the experimental groups with resin cements heated to 50°C, base and catalyst pastes of resin cements were equally dispensed on a glass plate laying on heating stirrer surface (Cientec, Piracicaba, SP, Brazil) set at 50°C. The cement and glass plate temperatures were constantly measured with a K-type thermocouple (SmartMether, Novus, Porto Alegre, RS, Brazil) to ensure that base and catalyst pastes reached 50°C. The pre-polymerized composite resin disc was also placed on the heated plate until its temperature reached 50°C, so both resin cement and indirect restoration had the same temperature during cementation. For experimental groups with resin cements at 25°C, base and catalyst pastes of resin cements were equally dispensed on a glass plate at 25°C. Base and paste were mixed and applied to the airborne-particle abraded surface of the pre-polymerized composite resin discs, which were positioned and fixed to the adhesive-coated dentin surface under load of 500 g.

The restored teeth were either exposed to curing light (dual-cured groups) or were allowed to self-cure (self-cured groups). When the cementing materials were light-cured through the indirect restoration, the composite disc was positioned and fixed to the adhesive-coated dentin surface under load of 500 g for 5 seconds and the light-curing unit tip was positioned against the composite resin disc for 40 seconds (power density: 600 mW/cm2, Optilux 501; Demetron Kerr, Danbury, CA, USA). The light intensity of the curing unit was constantly measured with a radiometer (Cure Rite, Dentsply Caulk). For restored teeth that were allowed to self-cure, the composite disc was positioned and fixed to the adhesive-coated dentin surface under load of 500 g for 5 min. A 3-mm thick layer of auto-polymerizing composite resin (Alpha Plast, DFL Indústria e Comércio S.A., Rio de Janeiro, RJ, Brazil) was placed on the untreated, polymerized composite resin surface to facilitate specimen gripping during microtensile testing. For groups using the self-curing mode, the auto-polymerizing composite resin was applied on the composite resin disc only after the auto-polymerization reaction of the cement had passed (5 min).

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

Product (Manufacturer)	Composition (Manufacturer supplied) (Batch Number)
Calibra (resin cement) (Dentsply Caulk)	Base paste: barium boron fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, other colorants are inorganic iron oxides. Catalyst paste: barium boron, fluoroalumino silicate glass, Bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, benzoyl peroxide. (Base: 081105; Catalyst: 0812011)
Variolink II (resin cement) (Ivoclar - Vivadent)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments Bis-GMA; TEGDMA; urethane dimethacrylate; benzoyl peroxide. (Base: L46354; Catalyst: L 36656)
XP Bond (bonding agent) (Dentsply Caulk)	HEMA; Tert-butyl alcohol; Dipentaerythritol pentaacrylate phosphate; acrylates; carboxylic acid modified dimethacrylate urethane dimethacrylate resin; triethyleneglycol dimethacrylate (Lot:0804002271) Self Cure Activator: Aromatic Sodium Sulfinate, (Self cure initiator), Acetone, Ethanol (Lot: 041110)
Excite DSC (bonding agent) (Ivoclar - Vivadent)	Mixture of dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, initiators and stabilizers (Lot: 59594)

Abbreviations: TEGDMA: triethylene glycol dimethacrylate, Bis-GMA: Bisphenol A diglycidyl ether methacrylate, HEMA: hydroxyethyl methacrylate, SiO_2: silicon dioxide

Microtensile Bond Strength Test (µTBS)

Restored teeth were stored in the dark in 100% relative humidity at 37° C for 7 days and were vertically, serially sectioned into several 1.0-mm thick slabs using a diamond saw (Isomet 1000; Buehler Ltd, Lake Bluff, Illinois, USA). Each slab was further sectioned to produce beams with a cross-sectional area of approximately 1.0 mm^2 at the bonded interface. Each bonded stick was attached to the grips of a microtensile testing jig with cyanoacrylate glue (Loctite Super Bonder Gel; Henkel Ltda, Itapevi, SP, Brazil) and tested in tension on a universal testing machine (EZ Test; Shimazu Co, Kyoto, Japan) at a crosshead speed of 1 mm/min until failure. After testing, the specimens were carefully removed from the fixtures with a scalpel blade and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital micrometer (Series 406; Mitutoyo America Corp., Aurora, Illinois, USA). Specimen cross-sectional area was calculated in order to present µTBS data in units of stress: MPa. Five beams were tested per each tooth, and the average obtained from these beams represented the µTBS values for that restored tooth.

Statistical Analysis

A 3-way analysis of variance (ANOVA) (product, temperature, and polymerizing mode factors) was performed. Tukey's post-hoc test was used to detect pair-wise differences among experimental groups. All statistical testing was performed at a pre-set alpha of 0.05.

Failure mode analysis

Fractured surfaces of tested specimens were allowed to air-dry overnight at 37^{o} C. The fractured surfaces were examined with a stereoscopic microscope (Panambra Ind. e Tec. S.A., São Paulo, SP, Brazil) and failure patterns were classified as follows: adhesive along the dentin surface, cohesive within dentin, cohesive within resin cement and mixed when simultaneously exhibiting remnants of both hybrid layer and resin cement.

RESULTS

Microtensile Bond Strength

The µTBS results for each product, temperature, and activation mode are displayed in Table 2. A double interaction between "product" and "activation mode" factors was detected by ANOVA. Teeth restored with Variolink II at 50°C showed higher µTBS values than those restored with Variolink II at 25°C in both activation modes ($p=0.02476$). On the other hand, no significant difference in µTBS values was noted between groups of Calibra at 50 °C and those groups at 25°C regardless of activation mode. Light-activation promoted higher µTBS values than the self-curing mode in Variolink II groups regardless of temperature ($p<0.05$). When Calibra was tested, no significant difference in µTBS values was observed between dual- and self-cured groups for all temperatures.

Table 2 – µTBS values of dual-cured resin cements submitted to different curing modes and temperature:

	Variolink II		Calibra	
	Light-cured	Self-cured	Light-cured	Self-cured
25°C	33.4 (8.7) Aa	13.9 (11.0) Ab	37.5 (9.6) Aa	31.6 (10.3) Aa
50°C	47.1 (8.6) Ba	21.7 (10.2) Bb	36.2 (8.0) Aab	25.4 (11.7) Aab

Different letters (upper case letter = column; lower case letter = row) indicate significant difference among means.

Regarding the comparison between products, the self-cured groups of Calibra at 25°C exhibited higher µTBS values than those of Variolink II ($p=0.02809$). Within the light-cured groups, no significant difference in µTBS values was noted between the resin cements. However, pre-heating of resin cements to 50°C resulted in no significant difference in µTBS values between Calibra and Variolink II for all activation modes.

Failure pattern analysis

The distribution of failure modes among groups is shown in Figure 1. Mixed failure was predominantly observed in most groups. Only dual-cured groups of Calibra polymerized at 25°C exhibited failures predominantly located within dentin. The percentage of cohesive failures in dentin observed in dual-cured groups restored with Calibra at 25°C decreased considerably in dual-cured groups restored with Calibra at 50°C. For Variolink II, the percentage of cohesive failures in dentin observed in both dual-cured and self-cured groups at 25°C reduced to zero when both groups were polymerized at 50°C.

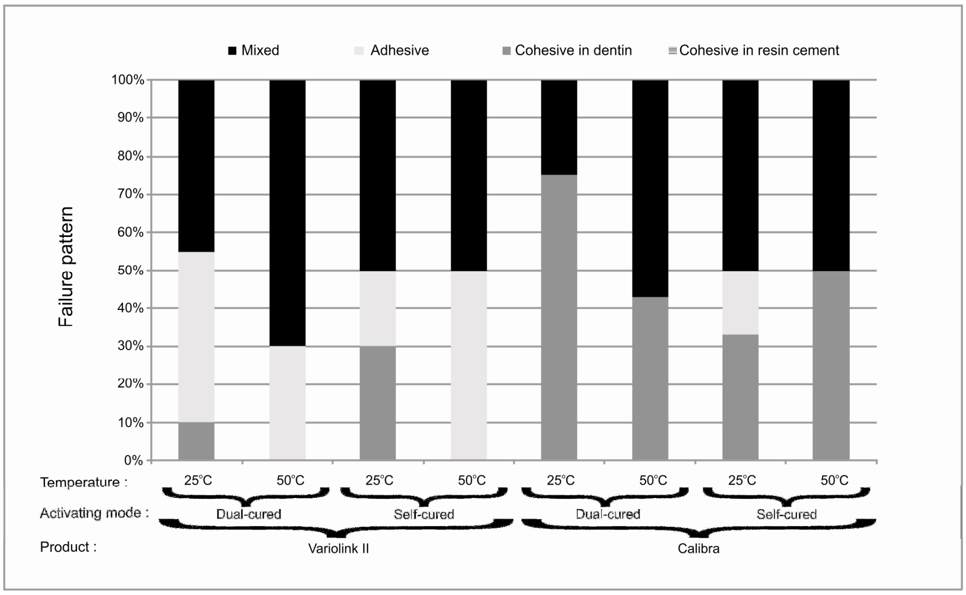

Figure 1: Failure pattern of fractured specimens from all experimental groups.

DISCUSSION

Based on the results of the current study, Variolink II resin cement pre-heated to 50°C promoted higher μTBS values than resin cements at 25°C, regardless of the activating mode. Therefore, the research hypothesis was accepted for Variolink II. Such finding is in agreement with the results from some studies that evaluated pre-heated self-adhesive resin cements.[23,24] Daronch et al.[18,19] demonstrated that resin composites cured with increased temperature during polymerization presents higher degree of conversion than composites polymerized at room temperature. The authors attributed the differences in monomer conversion to the low viscosity of pre-heated resin composites, which allowed higher radical mobility and collision frequency of unreacted active groups and radicals.[18,19] Therefore, the lower viscosity of Variolink II might have resulted in higher monomer conversion with improved mechanical properties as a consequence. Once there is a severe attenuation in curing light caused by the presence of an indirect restoration during cementation, the DC of the resin cement layer under an indirect restoration is not as high as that of resin composites that are exposed to high intensity curing light.[8,25] For this reason, it is expected that the effects of increased temperature on monomer conversion and mechanical properties may be more evident in resin cements than in resin composites.

When polymerization was initiated by the dual- or self-curing modes in pre-heated Variolink II, the decrease in resin viscosity and increased radical mobility were probably not the only factors contributing to the increase in μTBS values. Variolink II presents benzoyl peroxide as the initiator of the self-curing reaction. Benzoyl peroxide is unstable and can be activated by heat,[26,27] so increased temperature decomposes benzoyl peroxide on radicals that initiate resin polymerization. Therefore, benzoyl peroxide decomposes faster at higher temperatures, originating more radicals more rapidly than it does at room temperature.[27] As a consequence, more radical formation from decomposed benzoyl peroxide along with lower resin viscosity at increased temperature may have been responsible for the increased μTBS values when Variolink II was used.

However, the self-cured groups of Variolink II showed lower μTBS values than the dual-cured groups, despite the significant increase in μTBS of self-cured groups at 50°C. Therefore, the self-curing mode was not as effective as the light-curing one even at higher temperature. This finding might be explained by the low content of benzoyl peroxide in the composition of Variolink II. For this reason, this resin cement is not indicated by the manufacturer in clinical situations where the curing light is totally blocked by the indirect restoration.[28] However, it is conceivable to assume that Variolink II may be subjected to such situations even when the curing light is able to pass through an indirect restoration. For instance, there are deep regions in the prepared tooth where the resin cement layer is under thick indirect restorative material or very distant from the curing unit tip. Some studies have shown that indirect restorations with thickness higher than 5 mm can block the curing light,[17] so it is expected that the resin cement relies solely on the self-curing components to polymerize at those regions.

The results observed for Calibra were different from those observed for Variolink II regarding the effects of temperature on µTBS values. In groups involving the use of Calibra, no significant difference in µTBS values was noted between groups polymerized at 25°C and those polymerized at 50°C regardless of the activation mode. Therefore, the research hypothesis was rejected for Calibra. Curiously, no significant difference in µTBS was noted between dual-cured and self-cured groups cured at 25°C, so the self-curing components in Calibra were as effective as the light-curing components. Based on this finding, it is possible to speculate that Calibra has higher content of benzoyl peroxide than other dual-cured resin cements. This speculation may also explain why the increased temperature did not promote the same effects on µTBS values as those observed when Variolink II was tested. As previously discussed, the increased temperature decomposes benzoyl peroxide more quickly, so resin cements with higher content of benzoyl peroxide may set faster than those with low content of benzoyl peroxide. Similar finding was observed by Cantoro et al (2009)[23,24], who noted that the catalyst paste of Calibra set at 60°C before base and catalyst pastes were mixed. This was also observed during specimen preparation, when the cement started setting right after the indirect restoration was cemented on the dentin surface. Thus, it is possible that polymer network was rigid already during light exposure, so the benefits of light-activation at 50°C were not observed in this resin cement.

The fast cure of Calibra at 50°C in the self-curing mode may also be responsible for the lack of significant differences between self-cured groups cured at 25°C and those cured at 50°C. Although higher µTBS values were expected because of the increased monomer conversion, it is possible that the cement polymerization started while the restoration was seated on the tooth. As a consequence, more tension was created at the bonding interface and the expected effects of increased monomer conversion were masked. The short working time of Calibra at 50^{o}C deserves some concern since the working time should be long enough to not only allow proper seating of the indirect restoration on the tooth, but also allow the clinician to remove all excess of resin cement at the restoration margins. For this reason, manufacturers should provide more specific information for clinicians regarding the content of self-curing components, so the clinician could choose when pre-heated resin cements should be used.

In this study, both resin cement and indirect restoration were heated to 50^{o}C, which was chosen based on other reports.[18,19,29] The use of a heated indirect restoration coupled to a heated resin cement layer may delay the drastic drop in temperature that was observed in pre-heated resin composites when they were placed in tooth cavities.[30] However, a drop in temperature during cement polymerization is expected, but even the slight increase in the cement temperature improved the µTBS of an indirect restoration to dentin for one specific dual-cured resin cement. Besides, the analysis of failure pattern showed differences in failure patterns between groups at 25^{o}C and those at 50^{o}C. According to the results, the main change in failure pattern was related to the amount of cohesive failures in dentin, as such pattern ranged according to the resin cement temperature. Considering that cohesive failure in dentin may correspond to the bottom of the hybrid layer, it is possible that any increase in cement temperature may improve polymerization and mechanical properties of adhesive resin within the demineralized dentin. For this reason, a decrease in the amount of cohesive failure at the bottom of the hybrid layer was observed in dual-cured and self-cured groups restored with Variolink II at 50^{o}C and dual-cured groups restored with Calibra at 50^{o}C. However, only further evaluation of monomer conversion of bonding agents associated with pre-heated resin cements and degradation of bonding interface in this clinical situation may confirm this speculation.

Although most studies evaluated resin composites and resin cements at 50^{o}C,[18,19,23,24,31] some concern may arise regarding the effects of high temperature on the pulp tissue. It has been demonstrated that the increase in temperature of resinous materials to temperatures ranging from 54^{o}C and 60^{o}C does not produce significant changes in intrapulpal temperature.[29] However, the effects of heated resin cements coupled with heated indirect restorations on pulp tissue deserve further evaluation.

CONCLUSION

Within the limitations of the current study, the increase in temperature prior to polymerization may promote higher bond strength to dentin in simulated indirect restorations. However, the effectiveness of resin cement pre-heating on bond strength may be product-dependent, since more evident effects are observed in resin cements with low content of self-curing components.

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4. CONCLUSÃO

Dentro das limitações desse estudo, o aumento da temperatura dos cimentos resinosos de dupla ativação antes da polimerização pode promover maior resistência de união à dentina em restaurações indiretas com compósitos. No entanto, a eficácia do cimento resinoso pré-aquecido na resistência de união pode ser material-dependente, sendo os melhores resultados observados em cimentos com baixa concentração de componentes autopolimerizáveis.

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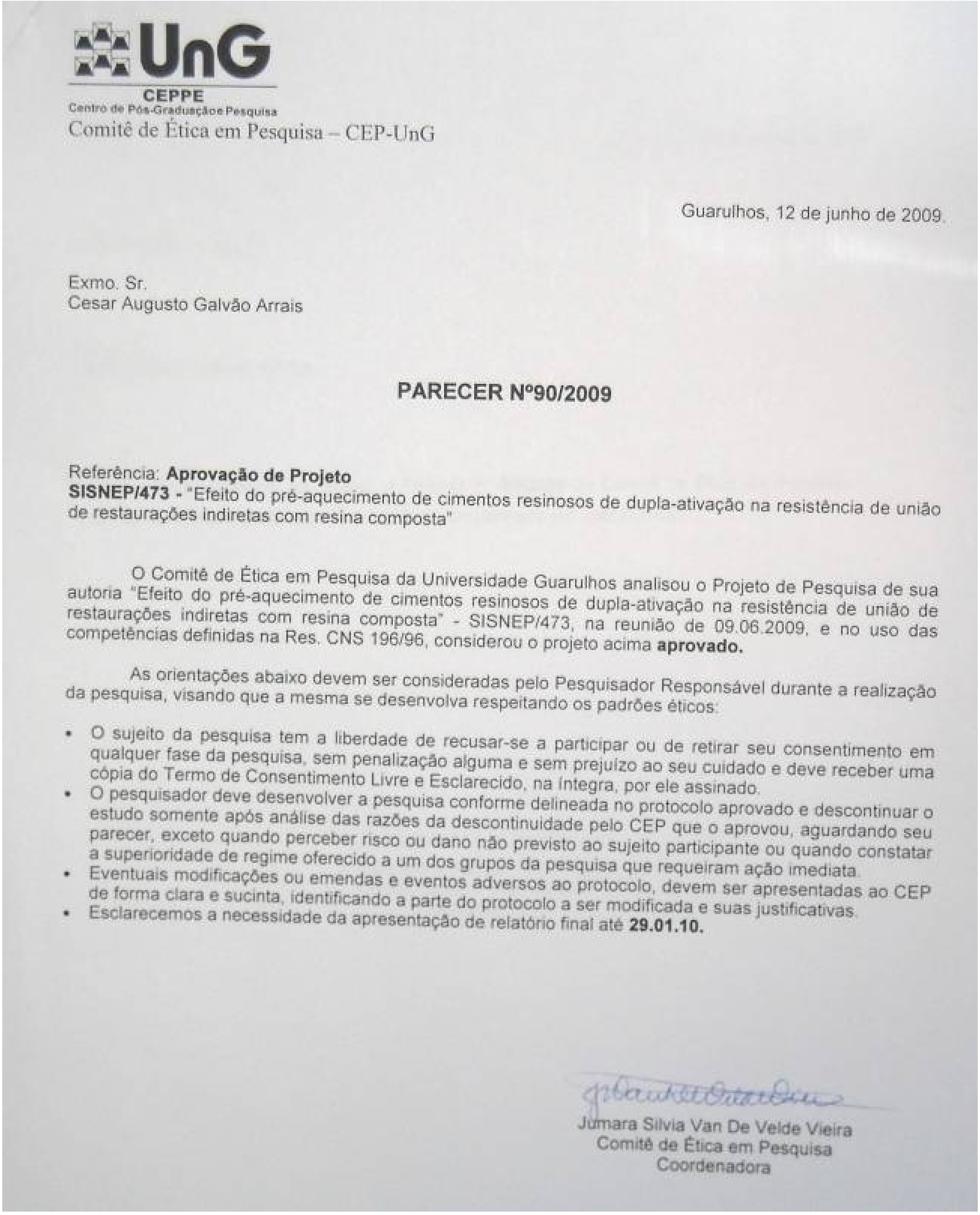
ANEXOS

ANEXO 1 – Declaração de aprovação do Comitê de Ética



UnG
CEPPE
Centro de Pós-Graduação e Pesquisa
Comitê de Ética em Pesquisa – CEP-UnG

Guarulhos, 12 de junho de 2009.

Exmo. Sr.
Cesar Augusto Galvão Arrais

PARECER Nº90/2009

Referência: **Aprovação de Projeto**
SISNEP/473 - "Efeito do pré-aquecimento de cimentos resinosos de dupla-ativação na resistência de união de restaurações indiretas com resina composta"

O Comitê de Ética em Pesquisa da Universidade Guarulhos analisou o Projeto de Pesquisa de sua autoria "Efeito do pré-aquecimento de cimentos resinosos de dupla-ativação na resistência de união de restaurações indiretas com resina composta" - SISNEP/473, na reunião de 09.06.2009, e no uso das competências definidas na Res. CNS 196/96, considerou o projeto acima **aprovado.**

As orientações abaixo devem ser consideradas pelo Pesquisador Responsável durante a realização da pesquisa, visando que a mesma se desenvolva respeitando os padrões éticos:

- O sujeito da pesquisa tem a liberdade de recusar-se a participar ou de retirar seu consentimento em qualquer fase da pesquisa, sem penalização alguma e sem prejuízo ao seu cuidado e deve receber uma cópia do Termo de Consentimento Livre e Esclarecido, na íntegra, por ele assinado.
- O pesquisador deve desenvolver a pesquisa conforme delineada no protocolo aprovado e descontinuar o estudo somente após análise das razões da descontinuidade pelo CEP que o aprovou, aguardando seu parecer, exceto quando perceber risco ou dano não previsto ao sujeito participante ou quando constatar a superioridade de regime oferecido a um dos grupos da pesquisa que requeiram ação imediata.
- Eventuais modificações ou emendas e eventos adversos ao protocolo, devem ser apresentadas ao CEP de forma clara e sucinta, identificando a parte do protocolo a ser modificada e suas justificativas.
- Esclarecemos a necessidade da apresentação de relatório final até **29.01.10.**

Jumara Silvia Van De Velde Vieira
Comitê de Ética em Pesquisa
Coordenadora

ANEXO 2 – Figuras ilustrativas dos procedimentos restauradores e ensaio de microtração

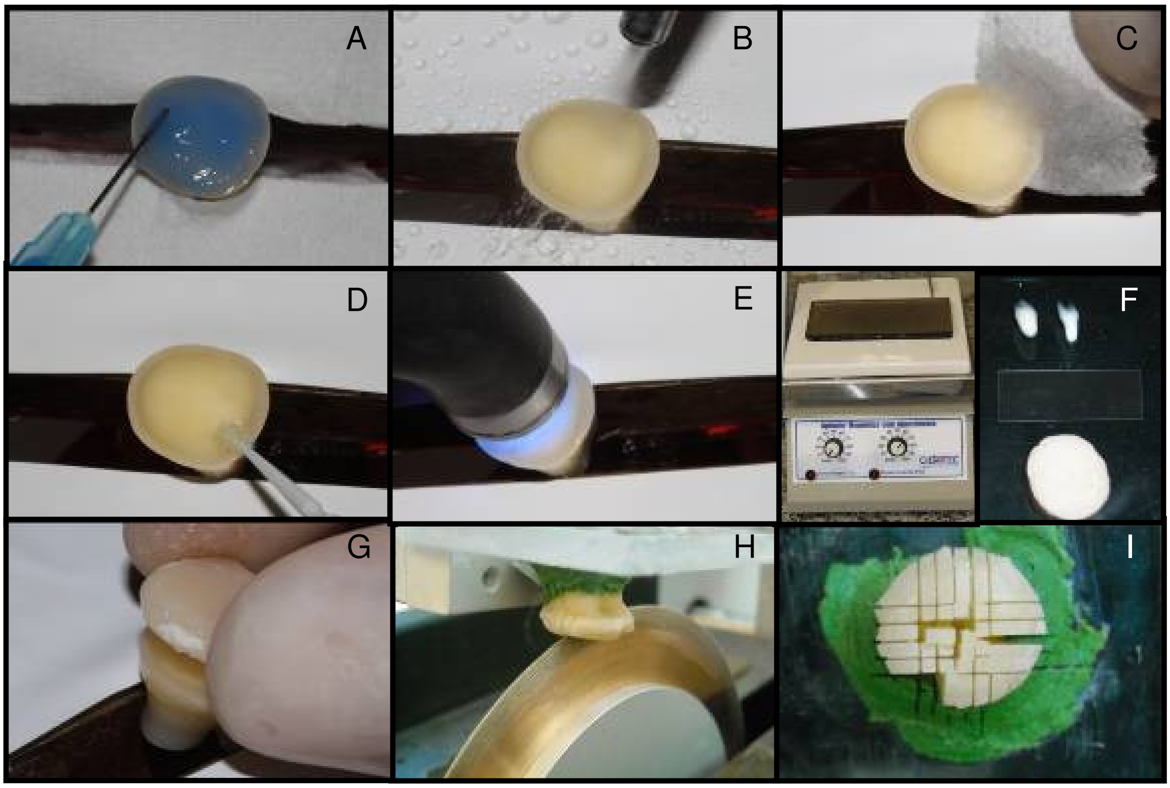


Figura: Condicionamento da superfície dentária com ácido fosfórico 35% (A), lavagem com água (B) e remoção do excesso de umidade com papel absorvente (C), aplicação (D) e fotoativação (F) do sistema adesivo, aquecimento do cimento resinoso e do disco de resina composta (F), fixação da restauração indireta (G); secção do dente restaurado (H) para obtenção dos espécimes (I)

ANEXO 3

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

Codigo do projeto:

Responsavel: cesar arrais

Delineamento experimental: 2x2x2

Observacoes nao transformadas

Nome dos fatores

Fator	nome
A	product
B	activat
C	temper

Quadro da analise de variancia

Causas da variacao	g.l.	S.q.	Q.m.	Valor f	prob.>f
Product	1	133.3350504	133.3350504	1.3739	0.24848
Activat	1	2371.1386293	2371.1386293	24.4321	0.00010
Temper	1	122.4655030	122.4655030	1.2619	0.26899
Pro*act	1	502.0425620	502.0425620	5.1730	0.02809
Pro*tem	1	527.1479524	527.1479524	5.4317	0.02476
Act*tem	1	74.5556328	74.5556328	0.7682	0.60872
Pro*act*tem	1	0.6032839	0.6032839	0.0062	0.93558
Residuo	32	3105.5984021	97.0499501		
Total	39	6836.8870159			

Media geral = 30.829750

Coeficiente de variacao = 31.954 %

Teste de tukey para medias de product

Dentro de foto do fator activat

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	1	variol	10	40.246001	40.246001	a	a
2	2	calibra	10	36.812000	36.812000	a	a

Teste de tukey para medias de product

Dentro de auto do fator activat

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	2	calibra	10	28.499000	28.499000	a	a
2	1	variol	10	17.762000	17.762000	b	a

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado

D.m.s. 5% = 8.98448 - d.m.s. 1% = 12.07484

Teste de tukey para medias de product

Dentro de 25c do fator temper

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	2	calibra	10	34.536000	34.536000	a	a
2	1	variol	10	23.624001	23.624001	b	a

Teste de tukey para medias de product

Dentro de 50c do fator temper

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	1	variol	10	34.384000	34.384000	a	a
2	2	calibra	10	30.774999	30.774999	a	a

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado

D.m.s. 5% = 8.98448 - d.m.s. 1% = 12.07484

Teste de tukey para medias de activat

Dentro de variol do fator product

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	1	foto	10	40.246001	40.246001	a	a
2	2	auto	10	17.762000	17.762000	b	b

Teste de tukey para medias de activat

Dentro de calibra do fator product

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	1	foto	10	36.812000	36.812000	a	a
2	2	auto	10	28.499000	28.499000	a	a

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado

D.m.s. 5% = 8.98448 - d.m.s. 1% = 12.07484

Teste de tukey para medias de temper

Dentro de variol do fator product

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	2	50c	10	34.384000	34.384000	a	a
2	1	25c	10	23.624001	23.624001	b	a

Teste de tukey para medias de temper

Dentro de calibra do fator product

Num.ordem	num.trat.	Nome	num.repet.	Medias	medias originais	5%	1%
1	1	25c	10	34.536000	34.536000	a	a
2	2	50c	10	30.774999	30.774999	a	a

Medias seguidas por letras distintas diferem entre si ao nivel de significancia indicado

D.m.s. 5% = 8.98448 - d.m.s. 1% = 12.07484