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DEPARTAMENTO DE ODONTOLOGIA
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INTEGRADA

LARISSA COELHO PIRES LOPES

**INFLUÊNCIA DO PRÉ-AQUECIMENTO DE RESINAS *BULK-FILL FLOW* E
CIMENTOS DE IONÔMERO DE VIDRO NAS SUAS PROPRIEDADES FÍSICO-
MECÂNICAS**

Maringá-PR

2020

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CIMENTOS DE IONÔMERO DE VIDRO NAS SUAS PROPRIEDADES FÍSICO-
MECÂNICAS**

Tese apresentada à Universidade Estadual de Maringá, como pré-requisito para obtenção do título de Doutora do Programa de Pós-Graduação em Odontologia Integrada.

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Larissa Coelho Pires Lopes

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nas propriedades físico-mecânicas*

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Dedico esta Tese

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RESUMO

Um desafio frequente na prática clínica com restaurações de resina composta é conseguir um bom selamento e conseqüentemente, boa adaptação marginal. Para as restaurações com cimentos de ionômero de vidro (CIV), especialmente nos períodos iniciais após a mistura, o desafio é assegurar que o material atinja bons níveis de resistência mecânica. Uma das alternativas propostas para contornar esses problemas tem sido o pré-aquecimento das resinas antes da inserção do material na cavidade e o aquecimento do CIV recém-manipulado com fontes de calor. Neste contexto, o objetivo deste trabalho foi avaliar a influência do pré-aquecimento de resinas compostas *bulk-fill* e de CIV nas propriedades físico-mecânicas. Para tanto, foram realizados quatro trabalhos, sendo uma revisão sistemática e três ensaios laboratoriais. Os objetivos de cada estudo foram: 1) realizar uma revisão sistemática sobre a influência do pré-aquecimento e/ou aquecimento de materiais resinosos e ionoméricos nas propriedades físicas e mecânicas e discutir os benefícios e as formas de pré-aquecimento que têm sido realizadas; 2) avaliar o efeito do pré-aquecimento de cimentos de ionômero de vidro no tempo de estabilização (TS) das ligações químicas e na microdureza; 3) avaliar o efeito do pré-aquecimento na liberação e absorção de flúor dos CIV Equia Forte/GC e Ketac Universal/3M ESPE; 4) avaliar o comportamento de fluxo e viscoelasticidade sob tensão de cisalhamento contínuo, a microdureza e o grau de conversão de resinas compostas *bulk-fill* de consistência *flow* (BFF), em diferentes temperaturas de pré-aquecimento. Com base nos achados desses estudos, verificou-se que o pré-aquecimento de BFF e CIV é uma técnica simples, segura e com bons resultados. Com base nos resultados da revisão sistemática verifica-se que o pré-aquecimento melhora as propriedades mecânicas e físicas dos materiais resinosos e ionoméricos. Os resultados dos estudos *in vitro* realizados mostraram que o pré-aquecimento de CIV reduziu o TS para o Equia Forte, mas aumentou para o Ketac Universal, bem como aumentou significativamente a microdureza do Ketac Universal e Equia Forte. O pré-aquecimento dos CIV não alterou o padrão de liberação e absorção de flúor. Com relação ao pré-aquecimento de BFF verificou-se uma redução da viscosidade, melhora da microdureza para alguns materiais e que o procedimento não influenciou o grau de conversão em todas as profundidades da restauração. Conclui-se que as vantagens da indicação de mais uma etapa de pré-aquecimento das resinas compostas *bulk-fill flow* ou ionômeros de vidro ao procedimento restaurador são questionáveis. Para atingir bons resultados é necessário agilidade para que os materiais não percam calor até o momento do procedimento restaurador

e cuidado para não incluir bolhas e evitar a formação de *gaps*, o que compromete o melhor desempenho da restauração.

Palavras-chave: Resinas Compostas; Ionômero de Vidro; Calefação; Testes de Dureza.

ABSTRACT

A frequent challenge in clinical practice with composite resin restorations is to achieve a good seal and, consequently, good marginal adaptation. For restorations with glass ionomer cements (GIC), especially in the initial periods after mixing, the challenge is to ensure that the material reaches good levels of mechanical resistance. One of the proposed alternatives to overcome these problems has been the preheating of the resins before the insertion of the material in the cavity and heating the newly GIC manipulated with heat sources. In this context, the objective of this work was to evaluate the influence of the preheating of *bulk-fill* and GIC composite resins on the physical-mechanical properties. For this purpose, four articles were carried out, one being a systematic review and three laboratory tests. The objectives of each study were: 1) to carry out a systematic review on the influence of the preheating and / or heating of resinous and ionomeric materials on the physical and mechanical properties and to discuss the benefits and the forms of preheating that have been carried out for resinous materials and glass ionomer cements; 2) evaluate the effect of preheating glass ionomer cements on the stabilization time (ST) of chemical bonds and on microhardness; 3) to evaluate the effect of preheating on the fluoride release and absorption of the GIC Equia Forte / GC and Ketac Universal / 3M ESPE; 4) to evaluate the behavior of *flow* and viscoelasticity under continuous shear stress, the microhardness and the degree of conversion of *bulk-fill* resins of flow consistency (BFF), at different preheating temperatures. Based on the findings of these studies, it was found that the preheating of BFF and CIV composite resins is a simple, safe technique and with good results. Based on the results of the systematic review, it appears that preheating improves the mechanical and physical properties of resin and ionomeric materials. According to the *in vitro* studies carried out, the following results were found: the pre-heating of GIC reduced the ST for Equia Forte, but increased for Ketac Universal, as well as significantly increased the microhardness of Ketac Universal and Equia Forte. The preheating of the GIC did not change the fluoride release and absorption pattern. Regarding the preheating of BFF, a reduction in viscosity was observed, improvement of microhardness for some materials and that the procedure did not influence the degree of conversion in all depths of the restoration. It is concluded that the advantages of indicating yet another preheating step for bulk-fill flow resins or glass ionomers still in the restorative procedure are questionable. To achieve good results, agility is required so that the materials do not lose heat until the moment of the restorative procedure and care not to

include bubbles and avoid the formation of gaps, which compromises the best performance of the restoration.

Key words: Composite resin; Glass-ionomer Cements; Heating; Hardness Tests.

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LISTA DE ABREVIATURAS E SIGLAS

$^{\circ}\text{C}$ - Graus Celsius

ANOVA - Análise de Variância

CAPES - Coordenação de Aperfeiçoamento de Pessoal de Nível Superior

CIV - Cimento de Ionômero de Vidro

DC – Degree of conversion

FBF - Filtek *bulk-fill* flow

FR - Fluoride release

FTIR - Fourier TRansform Infrared Spectroscop

g – gramas

GC – Grau de Conversão

GIC - Glass Ionomer Cements

γ - Gradiente de cisalhamento

KBr - Brometo de Potássio

Kg - Kilograma

LED - Light Emitting Diode/Diodo Emissor de Luz

LF - Liberação de fluoretos/flúor

M.O.M. - Manequins Odontológicos de Marília

mm^2 - Milímetro Quadrado

TNC - Tetric N-flow

N - Newtons

n - Índice de fluxo

NaF - Fluoreto de sódio

OBF - Opus *bulk-fill* flow

ppm - parte por milhão

ppmF - parte por milhão de Flúor

PTFE - Politetrafluoretileno

R - Razão entre os Picos

s - segundos

SDR - Surefill SDR flow

ST - Stabilization time

TNF - Tetric N-flow *bulk-fill*

UEM - Universidade Estadual de Maringá

XTR - X-tra base

κ = Índice de consistência

σ = Tensão de cisalhamento

τ_0 = Valor de cedência

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1. CONTEXTUALIZAÇÃO

1.1. Introdução e justificativas

A durabilidade das primeiras restaurações de resina composta em dentes posteriores era bem menor quando comparada às restaurações de amálgama (COLLINS; BRYANT; HODGE, 1998). Com o passar do tempo, as primeiras formulações dos materiais resinosos e de cimentos de ionômero de vidro foram melhoradas e, atualmente, o desempenho clínico de restaurações diretas com esses materiais tem alcançado melhores taxas de sucesso (DE AMORIM; LEAL; FRENCKEN, 2012; DE SOUZA et al., 2015; DIAS et al., 2018; F. MANGANI, S. MARINI, N. BARABANTI, A. PRETI, 2017; LEMPEL et al., 2015; SIDHU, 2010). Este desempenho é resultado de várias tentativas para aumentar a durabilidade, incluindo modificações nas formulações dos materiais, levando ao desenvolvimento das resinas *bulk-fill* (FERRACANE, 2011) e aos novos cimentos de ionômero de vidro (CIV), os cimentos de alta viscosidade (DIAS et al., 2018).

As resinas compostas do tipo *bulk-fill* apresentam características otimizadas como menor contração volumétrica e de tensão de polimerização na interface adesiva e são indicadas especialmente para restauração de dentes posteriores (ABDULRAZZAQ JERRI, 2015). A principal vantagem é que, segundo os fabricantes e os resultados de algumas pesquisas, podem ser inseridas em incrementos de 4 até 5 mm (KIM et al., 2015; LI et al., 2015), diminuindo consideravelmente o tempo de trabalho para confecção das restaurações.

Os CIV modernos, também denominados de CIV de alta viscosidade, apresentam melhoria em seu componente líquido e também no pó, como modificação no tamanho das partículas de carga e distribuição das partículas de vidro. Têm a vantagem sobre os CIV convencionais, pois os CIV são encapsulados e isso permite a padronização na proporção pó/líquido, o que deve reduzir a variação nos resultados clínicos. Com essas melhorias, as restaurações apresentam menor porosidade e maior resistência mecânica, além de apresentarem menor taxa de falha quando comparados com os CIV convencionais (não encapsulados) (FREITAS et al., 2018).

Um dos grandes desafios na prática clínica ainda é conseguir boa adaptação marginal, estável e durável ao longo do tempo, particularmente com incrementos maiores de material. Uma das alternativas propostas para contornar este problema em resinas compostas convencionais tem sido o pré-aquecimento antes da inserção (BAUSCH; DELANGE; DAVIDSON, 1981; LOVELL; NEWMAN; BOWMAN, 1999). Já para os CIV, o uso de luz de LED e ultrassom como fontes de calor têm sido utilizados para melhorar suas propriedades

mecânicas (GAVIC et al., 2016; GORSETA; GLAVINA, 2017; KLEVERLAAN; VAN DUINEN; FEILZER, 2004; MENNE-HAPP; ILIE, 2013; O'BRIEN et al., 2010; TOWLER et al., 2001).

O pré-aquecimento dos materiais restauradores odontológicos é utilizado há quase 40 anos (BAUSCH; DELANGE; DAVIDSON, 1981). Porém, considerando que as resinas *bulk-fill* estão relativamente há pouco tempo no mercado, não existem muitos trabalhos (ABDULMAJEED et al., 2019; DIONYSOPOULOS; TOLIDIS; GERASIMOU, 2016; LEMPEL et al., 2019; TAUBÖCK et al., 2015; THEOBALDO et al., 2017) que avaliaram o efeito do pré-aquecimento nas suas propriedades. Há poucos relatos na literatura (AL-AHDAL; SILIKAS; WATTS, 2014; PAPADOGIANNIS et al., 2015) sobre a reologia do material e o efeito do pré-aquecimento no comportamento de fluxo e de viscoelasticidade. Também há poucos estudos (KHOROUSHI; KARVANDI; SADEGHI, 2012; O'BRIEN et al., 2010) que avaliaram o efeito do pré-aquecimento sobre os cimentos de ionômero de vidro.

A seguir, uma breve revisão da literatura sobre os ensaios e protocolos de pré-aquecimento já disponíveis são apresentados, tanto para resinas compostas quanto para cimentos de ionômero de vidro.

1.2. Pré-aquecimento de resinas compostas

Os primeiros estudos que avaliaram a influência da temperatura na indução de polimerização em monômeros datam de 1977 (BAJAJ; GUPTA; BABU, 1977), mas foi em 1981 que foi publicado o primeiro trabalho avaliando a influência do aumento da temperatura nas propriedades mecânicas de resinas compostas odontológicas (BAUSCH; DELANGE; DAVIDSON, 1981).

Subsequente ao trabalho de Bausch, vieram outros trabalhos que também avaliaram a influência do pré-aquecimento, confirmando uma série de benefícios para os procedimentos restauradores. Os estudos que avaliaram a influência do pré-aquecimento foram feitos com vários tipos e marcas de resinas como: resinas híbridas (DEB et al., 2011; LUCEY et al., 2010; SANTANA et al., 2009), microhíbridas (DA SILVA et al., 2015; JAFARZADEH-KASHI et al., 2011), nanohíbrida (MUNDIM et al., 2011), nanoparticulada (DA COSTA et al., 2009), condensável, *flow* (WAGNER et al., 2008) e também resinas *bulk-fill* (ABDULMAJEED et al., 2019; DIONYSOPOULOS; TOLIDIS; GERASIMOU, 2016; LEMPEL et al., 2019; TAUBÖCK et al., 2015; THEOBALDO et al., 2017).

Daronch; Rueggeberg; Goes, em 2005, estudaram os efeitos do pré-aquecimento de resinas compostas em diferentes condições de temperatura (3°C até 60°C) e a duração da

exposição de luz na polimerização. Ao avaliarem o grau de conversão de uma resina composta encontraram valores de grau de conversão maiores ou iguais aos controles para todas as condições de pré-aquecimento e, mesmo com baixa intensidade luminosa, o aumento da temperatura promoveu uma contração de polimerização adequada. As mesmas condições também foram encontradas por Elhejazi, em 2006.

Outros estudos confirmaram que o pré-aquecimento das resinas compostas melhora a adaptação marginal de restaurações e reduz a microinfiltração quando compara-se com as resinas utilizadas em temperatura ambiente (FRÓES-SALGADO et al., 2010; WAGNER et al., 2008). O estudo de Deb et al. de 2011, além de avaliar a contração de polimerização também estudou a qualidade da adaptação marginal e citocompatibilidade do material após o pré-aquecimento à 60°C de resinas híbridas e *flow*. Os resultados do estudo mostraram o aumento da contração de polimerização, mas sem alterar a adaptação marginal.

Acreditava-se que o tratamento térmico das resinas compostas podia torná-las menos suscetível à degradação e também promover estabilidade de cor. Mundim et al., em 2011 estudaram a influência da temperatura nas mudanças de propriedades ópticas da resina e tiveram como resultados que o pré-aquecimento, embora tenha aumentado o grau de conversão das resinas, não influenciou a estabilidade de cor e as propriedades ópticas.

Davari et al., em 2014, avaliaram o efeito do pré-aquecimento na resistência de união entre resina composta e dentina e tiveram como resultado que o pré-aquecimento à 37°C promoveu a redução da viscosidade do material e aumento da resistência à tração. Esse aumento na resistência de união entre dentina e resina composta ocorre, pois com o aumento da fluidez há penetração mais profunda de resina nas áreas micro-retentivas da dentina e isso aumenta significativamente a resistência da união (PAPACCHINI et al., 2007).

Também é importante citar que as características reológicas das resinas compostas são modificadas com o pré-aquecimento, como o aumento da fluidez. Nos estudos de reologia as resinas foram classificadas como tendo um comportamento pseudoplástico e as variáveis na sua composição influenciam na viscoelasticidade e nas características reológicas desses materiais. O aumento da temperatura atua inversamente na viscosidade (AL-AHDAL; SILIKAS; WATTS, 2014). No estudo de Ayub et al., de 2014, os materiais resinosos pré-aquecidos se mostraram menos viscosos e após a polimerização, microdureza superficial mais elevada quando comparado às resinas sem tratamento térmico. Estes resultados confirmam o trabalho anterior realizado por Lucey et al., em 2010.

Um estudo recente (DA SILVA et al., 2015) mostrou que o pré-aquecimento reduziu a degradação do material resinoso. Encontrou-se diferença significativa na penetração de prata

nos compósitos pré-aquecidos à 60°C quando comparados com o material à temperatura ambiente (25°C). A redução do perfil de degradação do material pré-aquecido foi demonstrada pelo aumento do grau de conversão e reticulação da matriz resinosa, o qual absorve e libera menos solvente. O espaço reduzido entre as cadeias poliméricas e a redução de regiões hidrófilas levaram a resina a se degradar mais lentamente.

Tauböck et al., em 2015, avaliaram a influência da temperatura em resinas *bulk fill* comprovando o aumento do grau de conversão e redução na força de contração induzida pela polimerização, Dionysopoulos; Tolidis; Gerasimou, em 2016, avaliaram o efeito positivo da temperatura na microdureza e Lempel et al., em 2019 observaram aumento do grau de conversão em resinas reforçadas por fibra, porém com efeito negativo no grau de conversão na base das amostras. Abdulmajeed et al., em 2019, relatam que o pré-aquecimento das resinas *bulk-fill* não afeta a resistência flexural e resistência à tração, porém aumenta a resistência à fratura.

Em resumo, as principais vantagens relacionadas ao pré-aquecimento de resinas compostas são:

- maior grau de conversão dos monômeros (AYUB et al., 2014; CANTORO et al., 2008; DA COSTA et al., 2009; DEB et al., 2011; LUCEY et al., 2010; STEINHAUS et al., 2016);
- melhoria das características físicas, químicas (JAFARZADEH-KASHI et al., 2011) e mecânicas (LOVELL; NEWMAN; BOWMAN, 1999);
- melhor adaptação marginal (FRÓES-SALGADO et al., 2010; WAGNER et al., 2008);
- maior profundidade de polimerização (MUÑOZ et al., 2008);
- aumento da microdureza (AYUB et al., 2014; LUCEY et al., 2010; MUÑOZ et al., 2008);
- redução do tempo de polimerização (DARONCH; RUEGGEBERG; GOES, 2005; ELHEJAZI, 2006) e redução de degradação (DA SILVA et al., 2015);
- redução da sorção de água e alteração de cor (FERRACANE, 1985; LOVELL; NEWMAN; BOWMAN, 1999; MUNDIM et al., 2011).

1.3. Pré-aquecimento de cimentos de ionômero de vidro

A partir da técnica de pré-aquecimento dos materiais resinosos, alguns autores avaliaram o efeito do aquecimento nos cimentos odontológicos. Brune em 1982 (BRUNE,

1982) avaliou a resistência a compressão de cimento de ionômero de vidro e cimento de silicato após o aquecimento do pó à 100°C, antes da mistura, e observou um aumento de 10% na resistência à compressão. No ano seguinte, Brune avaliou a influência do aquecimento dos CIV após a mistura à 50°C e a 70°C durante 1 hora e também observou aumento da resistência à compressão à 70°C.

Woolford, em 1994, avaliou o efeito da irradiação infravermelho e luz halógena e observou que a aplicação de irradiação infravermelho (80°C) por 60s teve um efeito ligeiramente maior na dureza superficial do CIV do que a luz halógena (60°C). Porém, o uso da irradiação infravermelho é inviável para uso clínico. Assim, os estudos subsequentes aplicaram luz halógena, luz de LED e também ultrassom para o aquecimento dos CIV. Os estudos se concentraram em várias marcas de CIV restauradores convencionais e poucos trabalhos avaliaram os CIV em cápsula.

A metodologia utilizada para o aquecimento de CIV gira em torno da aplicação de luz de LED, variando o tempo de aplicação e potência do aparelho, ultrassom e também banho maria para às cápsulas. Segundo a literatura a aplicação de luz de LED por 60s pode atingir até 60°C.

Através da metodologia de estudo de calorimetria de varredura diferencial (DSC) observou-se que o aumento da temperatura promove alteração dimensional do CIV (BAN et al., 1990). Porém, se esta temperatura de aquecimento se limitar à 60°C não há alteração dimensional do material (YAN; SIDHU; MCCABE, 2007).

A reação de presa dos CIV ocorre à partir de uma reação ácido/base, portanto acredita-se que supostamente o aquecimento deixa o ácido mais ativo e assim mais rapidamente degrada os vidros acelerando a reação (WOOLFORD, 1994). Os CIV são materiais compostos basicamente por ácidos poliméricos solúveis em água, partículas de vidro e água. Após a mistura dos componentes inicia-se a reação de neutralização entre os poliácidos e as partículas de vidro, com formação imediata de cálcio ou estrôncio poliacrílico e formação tardia de alumínio poliacrílico. Esta reação inicial ocorre dentro de 2-6 minutos, mas a reação continua a sofrer mudanças no primeiro mês até 6 semanas após a mistura. Este processo tardio é denominado de maturação. Com o processo de maturação há aumento da força de compressão, ou seja, o CIV fica mais forte e resistente com o passar do tempo (NICHOLSON, 2018; SIDHU; NICHOLSON, 2016).

O aquecimento ativa os ácidos presentes nos CIV e, quando este entra em contato com a superfície das partículas de vidro, eles reagem mais rapidamente. Um ácido mais reativo e uma maior taxa de liberação de íons levarão à formação mais rápida de cimento por

geleificação dos produtos insolúveis do ácido e das partículas de vidro. O efeito final dessa aceleração será a formação mais rápida da matriz de polialquenoato de cálcio, assim acelerando a reação de presa inicial do material (WOOLFORD, 1994).

Os CIV foram criados no início de 1970 por Wilson e Kent, e desde então sofreram algumas modificações em sua composição para melhora das suas propriedades mecânicas. Os CIV contemporâneos, também chamados de CIV de alta viscosidade, são formulados com partículas de vidro melhoradas e tamanho das partículas aprimorados. Isso confere às novas formulações uma presa mais rápida e restaurações mais resistentes e duráveis (SIDHU, 2011).

Porém, os CIV ainda têm limitações, principalmente logo após a mistura; sabe-se que pelo menos durante 150 minutos o material ainda está vulnerável. É nesse período que acontece o processo de formação das ligações químicas à dentina (YAMAKAMI et al., 2018); e o material apresenta baixa resistência à abrasão, sensibilidade à humidade e longo tempo de presa (NICHOLSON, 2018; SIDHU; NICHOLSON, 2016).

Na tentativa de contornar essas limitações iniciais do CIV, foi proposta a aplicação de calor (ultrassom e luz de LED) para melhoria das propriedades mecânicas do material. Alguns estudos mostraram efeitos benéficos do aquecimento feito com luz de LED:

- Aceleração da reação de formação da matriz (WOOLFORD, 1994);
- Melhoria das propriedades mecânicas (KLEVERLAAN; VAN DUINEN; FEILZER, 2004; WOOLFORD, 1994), com aumento da microdureza em 24 horas (GAVIC et al., 2016; WOOLFORD, 1994);
- Aumento da resistência à compressão (KLEVERLAAN; VAN DUINEN; FEILZER, 2004).

Outros autores também mostraram os efeitos do uso do ultrassom como fonte de calor:

- Aumento da adesão ao dente e aumenta a liberação de flúor (ALGERA et al., 2005; THANJAL et al., 2010);
- Aumento da microdureza (O'BRIEN et al., 2010; TOWLER et al., 2001);
- Redução da porosidade (COLDEBELLA; SANTOS-PINTO; ZUANON, 2011);
- Redução da microinfiltração (GORSETA; GLAVINA; SKRINJARIC, 2012);
- Atinge propriedade mecânicas mais rápidas e melhores (GORSETA; GLAVINA; SKRINJARIC, 2012; TOWLER et al., 2001).

Há também autores que não encontraram diferença significativa nas propriedades mecânicas dos CIV após a aplicação do calor. Acredita-se que a reação de presa e as propriedades mecânicas são influenciadas pelo tamanho das cargas e sua distribuição na

matriz (MENNE-HAPP; ILIE, 2014). DE OLIVEIRA et al., 2019, sugerem que as dificuldades no desempenho do CIV após a aplicação de calor podem estar relacionada com a composição do material, assim como as diferenças na porosidade, hidrofobicidade e propriedades térmicas. A microdureza dos CIV pode estar relacionada com os diferentes formatos e tamanhos das partículas, quanto menor a partícula, maior a dureza.

Ao contrário dos materiais resinosos, para o CIV, o aquecimento não gera aumento na contração no momento da reação de presa. Alguns estudos relatam que o aquecimento melhora a adaptação do material às paredes da cavidade, reduzindo a microinfiltração marginal. Acredita-se também que as moléculas mais reativas formem uma matriz mais rígida, reduzindo até a porosidade da restauração, logo isso reduz a capacidade de liberação e absorção de flúor pelo material.

Também é importante ressaltar que ao contrário dos materiais resinosos, o CIV aumenta sua viscosidade com o aquecimento, pois como demonstrado por Algera et al., em 2006, o aumento da temperatura no CIV reduz o tempo de trabalho e o tempo de presa. Neste contexto, o aquecimento dos CIV mostraram benefícios para às propriedades mecânicas iniciais do material e perda da capacidade de liberação de flúor após o aquecimento do CIV depois da mistura. Entretanto, outras pesquisas precisam ser desenvolvidas para avaliar se estes efeitos também estão observados nos CIV encapsulados.

2. OBJETIVO GERAL

Apresentar quatro artigos sobre a influência do pré-aquecimento de resinas compostas *bulk-fill* e de cimentos de ionômero de vidro nas propriedades físicas e mecânicas.

2.1. Objetivos específicos

- Artigo 1: Realizar uma revisão sistemática sobre a influência do pré-aquecimento e/ou aquecimento de materiais resinosos e ionoméricos nas propriedades físicas e mecânicas e discutir os benefícios e as formas de pré-aquecimento que têm sido realizadas para materiais resinosos e cimentos de ionômero de vidro;
- Artigo 2: Avaliar o efeito do pré-aquecimento de cimentos de ionômero de vidro no tempo de estabilização das ligações químicas e na microdureza;
- Artigo 3: Avaliar o efeito do pré-aquecimento na liberação e absorção de fluoretos dos cimentos de ionômero de vidro Equia Forte/GC e Ketac Universal/3M ESPE;

- Artigo 4: Avaliar o comportamento de fluxo e viscoelasticidade sob tensão de cisalhamento contínuo, a microdureza e o grau de conversão de resinas *bulk-fill*, em diferentes temperaturas de pré-aquecimento.

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3. ARTIGO 1 – Heating and Preheating of dental restorative materials - a review

Abstract:

Objectives: To perform a systematic review on the influence of preheating and/or heating of resinous and ionomeric materials on their physical and mechanical properties and to discuss the benefits and methods of preheating/heating that have been used. *Material and methods:* A search was performed in the Pubmed, Scopus, Scielo and grey literature databases. In vitro studies published from 1980 until now were searched using the descriptors "composite resins OR glass ionomer cements OR resin cements OR adhesives AND heating OR preheating". Data extraction and quality of work evaluation were performed by two independent evaluators. *Results:* At the end of reading the search titles and abstracts, 74 articles were selected. Preheating of composite resins reduces viscosity, facilitates adaptation to cavity preparation walls, increases the degree of conversion and decreases the polymerization shrinkage. Preheating of resin cements improves strength, adhesion and degree of conversion. Dental adhesives showed good results such as higher bond strength to dentin. However, unlike resinous materials, ionomeric materials have an increase in viscosity upon heating. *Conclusions:* Preheating improves the mechanical and physical properties. However, there is a lack of clinical studies to confirm the advantages of preheating technique. *Clinical relevance:* Preheating of dental restorative materials is a simple, safe and successful technique. In order to achieve good results agility and training are necessary so the material would not lose heat until the restorative procedure. Also, care is necessary to avoid bubbles and formation of gaps, which compromises the best restoration performance.

3.1. Introduction

There are currently a wide variety of restorative materials available for dentists. Since early formulations, resinous materials and glass ionomer cements had been improving their clinical behavior, with good success rates [1–5]. On the other hand, these restorative options still present some limitations and more improvements are needed, including the influence of individuals' variables on the quality and longevity of the restoration [6,7].

Several innovations and new techniques have been done in order to increase durability and clinical behavior of restorative materials, including the change in material composition, such as the bulk-fill resins [8] and new glass ionomer cements [9]; the development of alternative photoinitiators and new multi-peak LED light-curing units with a larger spectral emission profile [10], and the minimally invasive approach that conserves more tooth

structures [11]. Another proposed alternative for optimizing the characteristics of dental materials has been preheating [12,13].

The preheating of the resin-based materials has been performed by commercial devices, such as the Calset (AdDent Inc, Danbury CT, USA) [14–16], ENA Heat (Micerium SpA, Avegno GE, Italy) [17,18], Hotset [19], HeatSync [20] and Caps Warmer (VOCO GmbH, Cuxhaven NI, Germany) [21]. Also it is being used with a water bath [22–24], incubator [25–27] and digital wax heaters [28]. The glass ionomer cements have already been heated using [9,29,30] LED light as an externally applied ‘command set’ and ultrasound to mechanically energized GICs rather than directly preheat them [30], which provide energy in the form of heat, as well as obtaining heated capsules by water bath [31]. The heating can be done prior to manipulation and insertion into the cavity / tooth (preheating) or after the restorative materials have been mixed.

Although many studies have addressed the performance of different materials with preheating techniques, there is lack of evidence that restorative materials preheating improves the quality and durability of restorations. Some advantages reported in the literature with the preheating technique of resinous materials include: increased degree of conversion [27], improved marginal adaptation of restorations due to reduce the viscosity [14,16,22] and decreased polymerization contraction [15]. Thus, the objective of this study was: (1) to perform a systematic review on the influence of preheating and / or heating of resinous and ionomeric commercial materials on their physical and mechanical properties and (2) to discuss the benefits and methods of preheating/heating that have been used for resin-based and ionomeric materials.

3.2. Material and Methods

This is a systematic review of the literature to answer the following question: Does preheating / heating of restorative materials (resin, resin cement, adhesive and glass ionomer cement) influence physical and mechanical properties?

3.2.1. Eligibility Criteria

In vitro studies published from 1980 onwards reported the influence of preheating / heating of restorative materials on physical and mechanical properties (degree of conversion, microhardness, viscosity, color, compressive strength, flexural strength, adhesion) without restriction of language were included.

Studies were excluded if: (1) did not evaluate preheated / heated restorative materials; (2) no control group; (3) in vivo studies or clinical study; (4) studies that evaluated orthodontics adhesive systems or experimental materials.

3.2.2. Database and Search Strategy

A search was conducted in the PubMed (US National Library of Medicine National Institutes of Health), Scopus (Elsevier) Scielo and gray literature databases until July 2020. MeSH terms were used along with the listed entry terms to construct a highly sensitive search strategy. The search strategy used for PubMed was: ("composite resins"[MeSH Terms] OR ("composite"[All Fields] AND "resins"[All Fields]) OR "composite resins"[All Fields]) OR ("glass ionomer cements"[MeSH Terms] OR ("glass"[All Fields] AND "ionomer"[All Fields] AND "cements"[All Fields]) OR "glass ionomer cements"[All Fields])) OR ("resin cements"[MeSH Terms] OR ("resin"[All Fields] AND "cements"[All Fields]) OR "resin cements"[All Fields])) OR ("adhesives"[Pharmacological Action] OR "adhesives"[MeSH Terms] OR "adhesives"[All Fields])) AND ("heating"[MeSH Terms] OR "heating"[All Fields])) OR preheating[All Fields], and complemented with references being cited in the selected papers.

3.3. Selection of studies and calibration of investigators

Initially, titles and abstracts were selected and evaluated by two independent researchers (LCPL and FMT). Selected studies were included for reading the full article. Each selected article was independently analyzed by the researchers and included or not in the review, based on the inclusion and exclusion criteria. In case of disagreement between the investigators, a third reviewer (RSST) evaluated the article to reach a consensus.

3.4. Risk of bias and quality of work

Data extraction and quality of work evaluation were performed by two independent evaluators (LCPL and FMT). The risk of bias assessment was performed following the guidelines of the Guidelines OHAT Risk of Bias Tool (National Health and Medical Research Council, 2015) [32], taking into account 11 criteria. Each item analyzed received the answers according to the guideline: ++**Definitely Low** risk of bias; +**Probably Low** risk of bias; -NR **Probably High** risk of bias; --**Definitely High** risk of bias.

3.5. Results

We found 1921 articles in the Pubmed database, 179 articles in the Scopus database and 288 in Scielo. At the end of reading the search titles and abstracts, 83 articles from the Pubmed database and 71 articles from the Scopus database and 1 article from Scielo database were selected. After reading the full texts, excluding repeated titles and evaluating the eligibility criteria, 65 articles were selected and 9 references cited from the selected articles were included, totaling 74 articles at the end (Figure 1). After analyzing the risk of bias based on the guidelines of the OHAT Risk of Bias Tool Guidelines [32], it was found that the included articles were classified as probably low risk of bias, since most studies presented at least 2 items assessed as "definitely low risk of bias" and at least 5 or more items rated "probably low risk of bias". Only 4 papers had at least 1 "probably high risk of bias" response and only 1 paper had 5 items rated "definitely high risk of bias".

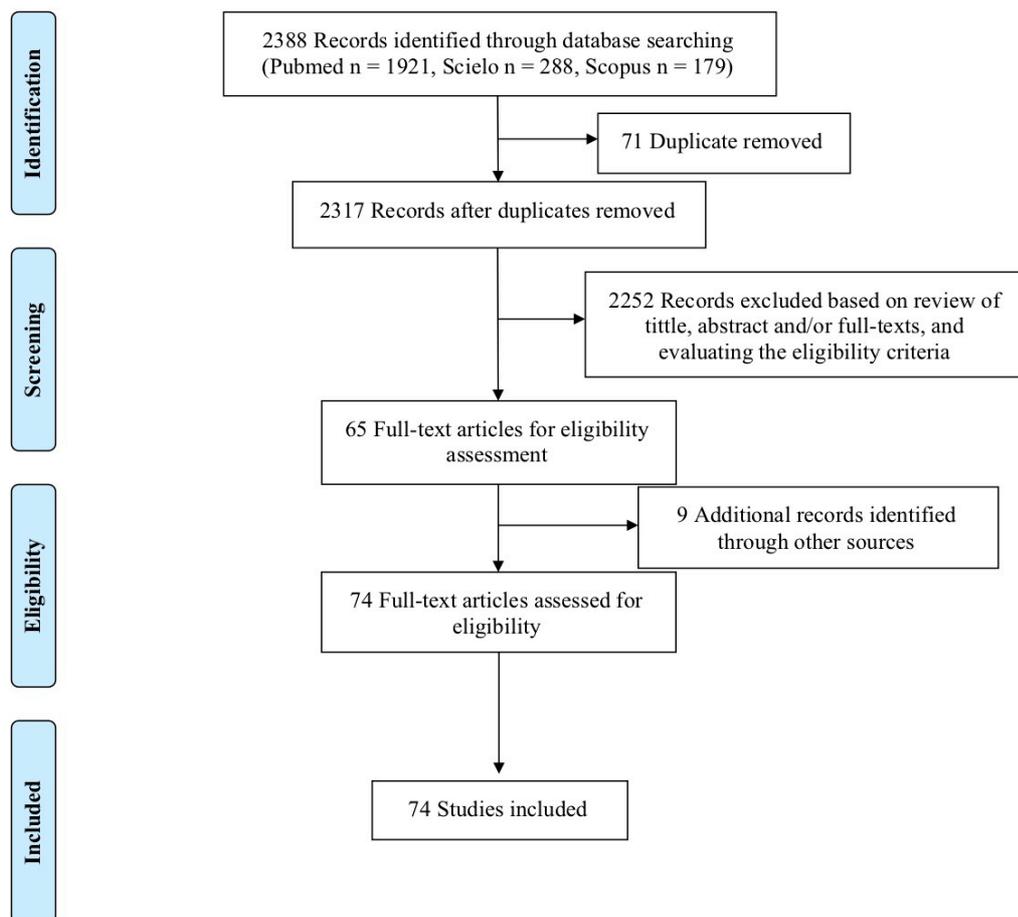


Figure 1. Flowchart showing the number of publications identified, retrieved, extracted, and included in the final analysis.

The articles selected were from 17 different countries (Table 1). Most of the selected studies evaluated composite resin (73.9%), followed by glass ionomer cement (11.5%), resin cements (10.1%) and adhesives (4.3%).

Table 1 - Countries of origin of articles selected for analysis.

Continent (Country of Origin)	Percentage (%)
América (Brazil 23.2%; USA 11.6%; Argentina 1.4%)	36.2%
Europe (Greece 8.7%, Holland 4.3% Italy, Turkey and Croatia 17.4%; England 5.8%, Germany 2.9%, Switzerland, Ireland and Poland 4.5%)	43.6%
África (Egypt 5.8%)	5.8%
Asia (Iran 10.1%, India 2.9%, Korea 1.4%)	14.4%

Table 2 presents a summary of the heating methods that have been employed depending on the restorative material and the heating protocol and Table 3 the main results obtained.

Table 2 - Publication characteristics of the articles included in the analysis.

Material Type	Manufacturer	Commercial Name	Heating Type	Preheating/Heating Time	Temperatures	
Adhesive	Dentsply Caulk	Prime&Bond 2.1 [33]	Waterbath [23] Calset [34] Oven [25] Halogen Light [33]	30 min [23] 1 h [33] 2 h [25]	4°C [23] 5°C [33] 20°C [33] 22°C [34] 25°C [23,25] 37°C [33] 40°C [23] 50°C [33] 58°C [34] 60°C [25]	
	Ivoclar Vivadent	Excite [25] Tetric N-Bond [25] XP Bond [25]				
	Kuraray Noritake	Clearfil SE Bond [23]				
	3M ESPE	Adper Single Bond 2 [23,25,33] Adper Easy One [25] Scotchbond Multi-Purpose [34]				
Compos ite resin	Bisico	Micro Esthetic [35]	Caps Warmer [21,65] Calset [14–16,37,42–45,57–60,62,66,70,71,77,78] Differential scanning calorimetry [40] Digital Wax [28] EASE-IT [64] Ena Heat [17,18,35,39,48,49] Incubator [26,38,68] Infrared heating	1 h [75] 2 min [54] 3 min [55,65] 5 min [15,19,28,66,76] 10°C/min [36] 10 min [20,39,58,70] 8 h [47] 12 min [48] 15 min [24,26,46,51,67] 20 min [21,42] 24 h [44] 30 s [38,41,62,65] 30 min [22,37,71] 40 min [60,73]	0°C [40,72] 3°C [41] 4°C [22,26,52,54,78] 5°C [20] 8°C [12,62] 10°C [41,61] 15°C [40] 20°C [41,54] 21°C [39,42,48] 22°C [14,21,28,40,41,47] 23°C [12,15,17,18,22,35,50,56,58,61,65,73,77] 24°C [45,46,60,66,76] 25°C [16,24,26,41,53,57,62,63,69,72] 27°C [41]	
	Bisco	Aelite LS Posterior [35]				
	Coltene	Synergy [36,37]				
	Dentsply Caulk	CeramX [37–39] Core Max II [40] Dyract Extra [35] Dyract Flow [35] Esthet-X [37,41–45] Esthet-X Flow [37,43] QuixFil [15] SDR Bulk fill [27] Spectrum TPH [14,35,46] Surefil [47] TPH [21,37,42] Vytol [12]				
		FGM				Opallis [39,48]

GC Corp.	EverX Posterior [49] Grandia Direct [37,50] Kalore [50]	source [12] Heated platform [52]		30°C [41,56] 36°C [37] 37°C
Heraeus Kulzer	Charisma [35,51,52] Charisma Diamond [35] Charisma Opal Flow [35] Durafil VS [53,54,55] Venus [28,50,56,57] Venus Bulk fill [49]	Heater [22] HeatSync [20] Hotset [19] Non-commercial heater [53,72] Oven [54,55,61] Programmable temperature		[12,17,21,22,24,40,42,45,47,50,52,54,56-59,68,70] 39°C [39,61] 40°C [38,41,64] 44°C [56] 45°C [26,48,64,69] 50°C [38,64,72] 54°C
Ivoclar Vivadent	Ceram X [37] Compoglass F [35] Compoglass flow [35] Heliomolar [14,35,37] Helioseal F [51] Isocap [12] IPS Empress Direct [19] Matrixx [37] Tetric Ceram [58-60] Tetric EvoCeram[15,21,37,51,57,61] Tetric EvoCeram Bulk fill [15,35, 49] Tetric Evo Flow [35,51] Tetric Flow [51,58] Tetric N- Ceram [62] Tetric N- Ceram Bulk fill [63] Te-Econom Plus [35] 4 Seasons [37]	controller (type 680) [51] Therma-flo™ [76] Thermal mechanical analyzer [36] Water bath [24,63,67]		[35,37,59,67,68,70,73,77,41,43-45,47,49,54,57,58] 55°C [17,18,20,69] 58°C [24] 60°C [12,14,35,38,40,42,44,46,52,53,55,62,67,69,73,74] 64°C [28] 68°C [15,37,45,54,57-61,63,65,66,70,71,76-78] 70°C[47] 75°C [72] 100°C [72] 25-69°C [36] 25-250°C [51]

Kerr Dental	Herculite Classic [55] Herculite XRV [14,37,44,47,51] Point 4 Flow [37] Point [37] Premise [37,56] Sonic Fill Bulk fill [15] Harmonize [21]			
King Dental Corp.	King Dental [40]			
Kuraray	Clearfil AP-X [35,37,56] Clearfil Majesty Posterior [45]			
Micerium	Enamel Plus HFO [39,48] Enamel Plus HRI [48]			
SDI	Conseal [51] Wave [14,35]			
Shofu	Beautifil II [35] Beautifil Bulk Restoration [49] Beautifil Bulk flowable [49]			
Ultradent	PermaFlo [37] Vit-I-essence [37,60]			
VOCO GmbH	Admira Fusion [21] Grandio [17,35,37,50,64] Viscalor [21,65] Xtra base [49,63] Xtra fill Bulk fill [15,49,63]			

	Tokuyama	Estelite Omega [19]			
	3M ESPE	Concise [12] F2000 [14] Filtek A110 [47] Filtek Bulk fill [49, 66] Filtek Bulk fill Posterior [67] Filtek Flow [35,37] Filtek One Bulk Fill [20] Filtek P60 [14,22,35,68] Filtek P90 [26,69] Filtek Z100 [19,37,55,70] Filtek Z250 universal/XT [17,18,22,24,26,28,35,37,51,5 3,56,64,66, 69,71, 72] Filtek Z350Flow/XT1 [16,53,68,73,74,75] Filtek Z350XT [76] Filtek Z 550 [49] Filtek Silorane [18,24,35,77,78], Filtek Supreme XT/Ultra [20,21,35,37,50,57,60,70,71,7 8], Silar [12]			
Ionomeri Material	Dentsply Caulk	Chemfil Rock [79]	External heat source [30,82] Led [9,29,79,83] Thermal mechanical analyzer [36] Ultrasonic [30,82] Reometro [81] Water Bath [80]	40 s [9, 82] 60 s [79] 90 s [80]	20°C [81] 30°C [81] 24°C -54°C [9] 32°C -57°C [79] 40°C [80,81] 50°C [81] 60°C [81] 70°C [81] 25°C-70°C [36]
	GC Corp.	Equia Fil [29,79] Fuji II LC [36,80] Fuji VII [30] Fuji IX [9,30,36,79,81,82] Fuji Triage capsule [82]			
	Megadenta	Megacem [30]			

	VOCO GmbH	Ionofil Molar [29,30,82] IonoStar Molar [83]			
	3M ESPE	Ketac Cem [36] Ketac fil Plus Aplicap [83] Ketac Molar [9,29,36,79,81]			
Resin Cement	Bisco Dental	BisCem [84]	Digital wax heater [28] Hotset [19] Programmable temperature controller (type 680) [51] Water Bath [87] Incubator [75] Heating stirrer surface [86] Oven [84,85]	1 day [79, 82] 1 min [80]	4°C [84,85] 22°C [28] 23°C [75] 24°C [84,85] 25°C [19,86] 37°C [84,85] 50°C [86] 54°C [75] 55°C [87] 60°C [84,85,87] 64°C [28] 69°C [19] 25°C-69°C [19] 25°C-250°C [51]
	Dentsply Caulk	Dyract Extra [51] XP Bond/Calibra [84,86]			
	GC Corp.	G-Cem [84]			
	Ivoclar Vivadent	Compoglass F [51] Excite DSC [86] Multilink Sprint [84]			
	Kuraray Noritake	Panavia 2.0 [85,87] SAC-A [84]			
	3M ESPE	RelyX ARC [28,75] RelyX Ultimate [75] RelyX Veneer [19,75] RelyX Unicem [85]			

Table 3 - Advantages and disadvantages of preheating.

Material	Advantage	Disadvantage
Adhesives	<p>Improve of dentin bond strength [23]</p> <p>Increase of penetration rate and high evaporation of monomers [23]</p> <p>Increase of degree of conversion [25]</p> <p>Reduction of sorption and solubility [25]</p>	<p>Increase of water sorption and solubility [25]</p>
Composite resin	<p>Increase of microhardness [12,17,18,24,46,59,67]</p> <p>Increase of degree of conversion [27,38,41,47,58,59,61,68]</p> <p>Increase of fluidity [19,37,46,59,64,70]</p> <p>Improvement of marginal adaptation [14,16,37,43,45,70,73]</p> <p>Microleakage Reduction [49]</p> <p>Reduction in the extrusion force and increased extruded mass [21]</p>	<p>Modification in resin color [61,65]</p> <p>Increase of volumetric contraction [25,30,47,52]</p> <p>Reduction of flexural strength [48]</p>
Ionomeric Material	<p>Reduction of setting time and working time [9,76]</p> <p>Increase of microhardness [31,78]</p> <p>Improve of adhesion [77]</p> <p>Improvement in marginal adaptation and reduction in microleakage [30]</p>	<p>Fluoride release reduction [78]</p>
Resin cement	<p>Water sorption reduction and solubility [71]</p> <p>Increased dentin adhesion [80,82]</p>	<p>Reduction of root canal bond strength [81]</p>

3.6. Discussion

Preheating dental restorative materials has been used for almost forty years. The first material to be subjected to the preheating technique was a composite resin of regular consistency [12] and subsequently fluid resins and resin cements. Preheating apparently increases the flowability of regular consistency composites [60], which improves the adaptation of the material in the cavity walls [42,43]. Another situation that preheating would be indicated was for dentists who store the resins in a refrigerator, following the manufacturer's guidelines. In this context, some authors report that the cooling of composite resins may disrupt some characteristics and it is important that they return to environment temperature before use [44]. Also, incomplete polymerization and unreacted monomers may leach into saliva promoting undesirable consequences and acting plasticizers that decrease mechanical strength and dimensional stability, color change and allow bacterial growth. Unreacted monomers can also cause allergic and sensitivity reactions [14].

The heating technique has been applied to glass ionomer cements after manipulation using external heat energy as a command set to improve mechanical properties too. Some studies [31,80] have shown that the application of heat in glass ionomer cements after mixing increases surface microhardness by up to 4 mm, improves marginal adaptation, reduces working time and crack propagation.

Various types of resin-based materials (hybrid composite resin, methacrylates, silorane, resin cements) have been tested in laboratory to evaluate the influence of preheating on their physical, mechanical and photoactivation properties. The average preheating temperature found in the literature is 54-68°C, considered a safe temperature for some authors [29,67,77], since it does not cause damage to the pulp tissue. Clinically, other situations can commonly cause increased pulp temperature such as the use of diamond burs during cavity preparation and photoactivation of resin materials. Possibly, the heating caused by the use of high irradiance from light-curing units is similar or greater than the heating of restorative materials. The temperature of the heated material placed into the cavity is not the same, as there is a rapid dropping of approximately 50% in 2 minutes counted after removing the material from the heating device [44]. A pulp temperature rise of 5.5°C is considered as the potential damaging threshold for human pulp tissue [88] and the remaining dentin thickness still appears to be one of the most important factors for the protection of the pulp since dentin acts as a thermal barrier against harmful stimuli [67]. However, Knezevic et al. [70], when assessing cellular toxicity resulting from preheating of resins at 68°C suggested that this procedure may not be safe.

Another important consideration about preheating is the required time to achieve good fluidity and improvement of restorative material properties. Not all papers mention this information. From studies that mentioned the required time for material heating, the minimum and maximum times found were 40 seconds to 24 hours, i.e., there is a very wide variation. However, a reasonable clinical time is approximately 15 min, as used in some studies [24, 26, 46, 51, 67]. For another study, 11 minutes was enough to reach the temperature required [44].

The most common device for preheating is Calset (AdDent Inc, Danbury, CT, USA). The manufacturer's instructions recommend it to preheating many types of instruments and materials like compules or syringes of composite resins, composite dispenser, anesthetics, spatulas, and laminate veneers. The device is presented with different trays, depending on what the clinician needs for preheating. It offers three different temperatures and permits preheat or maintain the temperature in: 37°C, 54°C or 68°C, as Caps Warmer (VOCO) [21]. Another device, ENA Heat (Micerium SpA, Avegno GE, Italy), offers two different temperatures: 39°C and 55°C, Hotset 39°C and 69°C [19] and HeatSync [20].

The composite resins reduce their viscosity when heated, facilitating the adaptation to the walls of the cavity preparation [14,16,22,37,43,44,56,57,68,74] and there is an improvement of many physical properties [17,18,24,26,42,46,51,60,62,72], such as a higher degree of conversion [27,38,41,47,53,59,72] and lower polymerization shrinkage [15]. Preheating of resin-based materials enhances conversion without hastening the time at which maximum cure rate occurs. This enhancement is probably attained by increased molecular mobility and collision frequency of reactive species. The phenomenon involves a postponement of diffusion-controlled propagation, reaction-diffusion-controlled termination, and autodeceleration, thereby allowing the system to reach higher limiting conversions before vitrification [89]. It is further known that, in addition to preheating, resin properties can be improved due to other situations such as increased light-activation time and the power of the LED light-curing units [42,60,69,74]. Usually, the temperature used to enhance these properties is 54°C to 68°C, depending on the type of device available. At this moment no work searched the differences between preheating at 54°C or 68°C.

It should be considered that resin composites with different compositions may take different times to reach stable temperature and sufficient time is mandatory for they reach and maintain the temperature [44]. Also, when the effect of compules/composite types on temperature values was evaluated it seems that different compule types did not affect temperature values and maximum compule temperature attained was $48.3 \pm 0.7^{\circ}\text{C}$ when the Calset unit was preset to 54°C, and $54.7 \pm 1.9^{\circ}\text{C}$ when preset to 60°C [44]. But the composite

compule already loaded into a delivery syringe was more efficient: higher temperatures were attained with this method as opposed to preheating the compule separately.

Although much work has shown the benefits of preheating composite resins, others have shown that preheating did not influence on some physical and mechanical properties of resin [71], such as flexural strength [16,39], microhardness [52], degree of conversion [61,66,90], polymerization shrinkage [54] and marginal microleakage [73]. Repeated heating of the resin may not be detrimental to flexural strength [48] but can cause color change [69]. Also, re-heating of unused composite may not affect its degree of conversion, thus decreasing material waste [44]. These results may be a function of different methodologies, but the benefits of preheating are achieved when light-activation is performed with the resin still warm [43,45]. Thus, to succeed with this technique, it is important to insert the material into the cavity quickly and efficiently, also avoiding the formation of bubbles and gaps [45]. The success of the technique also depends on other variables, such as the formulation of the material itself [49,91], quantity and organic matrix type [92], inorganic load filling [17], heating time and temperature, light-activation technique [42,71], in addition to the operator variability [40,43].

The same preheating technique has also been applied to adhesives with incongruent results. Some studies have reported dentin bond strength of Adper Single Bond improvement [23], degree of conversion increasing and less solubility for Adper Single Bond 2 [25], solubility and water sorption increasing for XP Bond adhesive [25] and others have not found significant difference in dentin bond strength, using Scotchbond Multipurpose Adhesive [34], Prime&Bond 2.1 and Adper Single Bond 2 [33], as well as Clearfil SE Bond [23].

There are many manufacturers developing resin cements with color and consistency appropriate for aesthetic and efficient cementation. However, there are alternative materials to be used in prosthetic cementation. The greatest benefit of preheating composite resins is the reduction in viscosity, enabling the use for cementation of indirect restorations [28]. Clinically, it looks easier to apply than resin cements. Preheating of regular consistency resins also appears to reduce cement line thickness by 24% [28,35]. Composite resins may perform better than resin cement on restoration margins due to more inorganic load filling and the long term color stability should be better because they don't have the autopolymerization reaction [28]. On the other hand, the benefits of preheating resin cements are still controversial. Lima et al. [75] observed that preheating of luting agents at 54°C for 15s reduced water sorption and oral solubility. Improvement in microtensile strength has been reported for dual-cure resin cements at 50°C [86], because the monomeric conversion increasing following a specific

light-activation, condition [93]. However, some authors have reported that heating at 60°C was not beneficial [84], leading to hardening of RelyX Unicem cement before being dispensed from the syringe [85] or reduction of resin cement bonding in the root canal [87]. In contrast, other types of resin cements such as Panavia 2.0 and self-adhesives had their bond strength improved [85]. The divergence of results is probably due to differences in research methodologies and material composition, light-activation time or even technical and operator variability.

Although some studies have reported that intraradicular temperature [94] and relative humidity [95] do not interfere in the bond strength, it is important to consider that despite the fact laboratory studies were careful and well conducted, they do not bring clinical evidence. There are few case reports or clinical trials showing the advantages of preheating resinous materials in these conditions. Also, as mentioned above, according to Daronch et al. [44], when a compound is heated to 60°C and removed from the heat source, its temperature drops 50% after 2min and 90% after 5min. So, the clinician must work very fast to ensure the least temperature drop possible. The clinician should dispense the material, adapt it, remove the excess and sculp it if necessary and light-cured while the material is still heated to obtain the advantages of higher monomeric conversion.

Another concern is related with time necessary for the composite resins stored in the refrigerator to reach room temperature. The clinician should wait at least eleven minutes before using composite within a compule stored in a refrigerator [46]. This time should be higher when the clinician uses a bigger compule or a syringe, for example.

Unlike resin materials, heating of glass ionomer cements promotes an increase in viscosity. Heating is believed to increase the ion diffusion rate, accelerating the reaction, reducing working time and hardening time [81]. However, it can be seen that heating glass ionomer cements after mixing promotes an improvement in their physical and chemical properties. The heating of the ionomeric materials have been performed with LED light or mechanically energized with ultrasound and there was improvement in marginal adaptation; reduction in microleakage [30]; increase in flexural strength [9,79], increase in microhardness [83], increase in bond strength [82] and acceleration of gelification reaction that protects the material in the early periods that are most critical against contamination with saliva [9]. Glass ionomer cement showed the smallest dimensional change when heated to 50°C [36]. O'Brien, 2010 [31] observed that the preheating of glass ionomer capsules before mixing had better influence on the depth microhardness than heat application after mixing with ultrasound and LED light.

It is suggested that the heating of the glass ionomer cement after mixing promotes water evaporation and this promotes acceleration of the chemical reaction of the material [96]. The positive effect of preheating on the glass ionomer cement is not well established and clear yet, because there are few reports on this matter. The differences in the results can be attributed to complexity of the material setting reaction. It is known that the reaction of glass ionomer cement happens not only by the neutralization of polyacids, but also the phosphates proved to be key components in the reaction [97,98]. Also, any change in the proportion of components, such as the polyacid concentration, size and shape of the glass particles may influence the end result of the reaction [99]. Preheating glass ionomer cement is also considered to be a safe procedure as it does not raise the pulp temperature significantly [29]. There are still few studies that make it clear whether heating of glass ionomer cement is really beneficial, so more research is needed to confirm this promising technique.

Despite the fact that the investigated commercial materials are not specifically designed for preheating/heating and more clinical results are necessary, heating or preheating is still a technique to be more investigated. There are new resins in the market like Viscalor [21,65] designed specifically for preheating/heating with easy manipulation due to enhanced handling properties. Because the indication of injectable composite resin technique is increasing, further *in vitro* and *in vivo* studies are necessary to answer the performance of these new techniques and heated materials.

3.7. Conclusions

Based on the results of laboratory studies, preheating procedures for dental restorative materials is a simple, safe and relatively successful technique. In general, for resinous materials there is an increase in microhardness and degree of conversion, reduction in viscosity and better adaptation to cavity walls. For ionomeric materials, heating promotes reduction of setting time, working time and porosity and increase of microhardness. However, there is a lack of clinical research proving the advantages of indication of pre-heating technique. In order to achieve good results agility and training are necessary so the material would not lose heat until the restorative procedure. Also care is necessary to avoid bubbles and formation of gaps, which compromises the best restoration performance.

3.8. REFERENCES

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4. ARTIGO 2 - *In vitro* evaluation of the stabilization time of chemical bonds during setting reaction and microhardness of preheated glass-ionomer cements

Clinical Relevance: The preheating of glass-ionomer cements can influence the performance of the restoration, potentially maintaining it in the mouth for longer.

Running Title: Preheating of glass-ionomer cements

4.1. ABSTRACT

OBJECTIVES: To evaluate the effect of preheating of glass-ionomer cement (GIC) restorative materials on stabilization time (ST) of their metal carboxylate bonds and on microhardness. **METHODS AND MATERIALS:** Two conventional high viscosity GICs, Ketac Universal (3M ESPE) and Equia Forte (GC), were evaluated. The thermographic camera was used to measure the temperature inside the glass-ionomer cement capsules before and after heating. The preheating of capsules was performed at 54°C over 30s, in a commercial device. Characterization of ST in the GICs was determined by Fourier Transform Infrared (FTIR) spectroscopy. For this, 10 samples of each material were prepared, 5 in the non-preheated group (control) and 5 with preheating. FTIR spectra were obtained 10 min after mixing (control group) or after heating and then every 10min over 120min. For the microhardness test, 20 cylindrical specimens (3mm height x 6mm diameter) were prepared for each material (10 preheated, 10 control). The microhardness was determined at three time intervals: 10min after mixing, after the ST as detected through the FTIR part of the study, and after one week. Knoop microhardness was assessed using a diamond indenter with a 25g load and 15s dwell time. **RESULTS:** Ketac Universal showed increase in temperatures of 15.7°C for powder and 3.6°C for liquid, while Equia Forte showed 16.4°C for powder and 8.5°C for liquid. FTIR spectra indicated that preheating reduced the ST for Equia Forte but increased it for Ketac Universal. Preheating increased the initial microhardness (T_1) of Equia Forte. With maturation over one week, it was observed that preheating significantly improved the microhardness of both materials compared to the control specimens. **CONCLUSION:** Preheating influenced the ST and the microhardness of Ketac Universal and Equia Forte. The ST and microhardness of Ketac Universal increased after 7 days. On the other hand, Equia Forte showed a reduced ST and increased microhardness from the outset.

Keywords: heating; glass-ionomer cement; microhardness; Spectroscopy, Fourier Transform Infrared

4.2. INTRODUCTION

Glass-ionomer cement (GIC) is a widely-used material due to its properties such as biocompatibility in the mouth, fluoride release, ability to promote remineralization of dental structures,¹ chemical adhesion to tooth² and bioactivity without shrinkage during the setting reaction.³ It is a material that hardens following an acid-base reaction between fluoroaluminosilicate glass powder and an aqueous solution of polyacid.⁴ According to the ISO standard (ISO 9917-1),⁵ restorative GICs have setting times in the range of 1.5 to 6min. The hardening reaction occurs in a short period and despite this, it does not mean that the material has reached complete chemical stability.² After its initial hardening, the GIC material continues to undergo changes (maturation process) for some time.^{4,6} A recent study about the dynamics of the setting process of GIC demonstrated that the time required for Ketac Molar Easy Mix (3M ESPE) to acquire stabilization time of its chemical bonds takes up to 150 min.² The manufacturer of this material states that the setting time is only 5 min. The setting time is different from the time to reach chemical bond stability (stabilization time), as evaluated by FTIR spectroscopy.^{2,7}

Contemporary GICs have improved over previous versions and have a wide range of uses.⁸ Modern glass-ionomers for clinical use, the so-called high-viscosity materials, are formulated with improved particle size and particle size distribution glasses, which have more rapid set than previous types, and result in mechanically strong and durable restorations. However, glass-ionomer cements do have limitations, including their physical resistance, sensitivity to humidity, opacity and relatively slow setting reaction.^{1,4}

In an attempt to reduce these limitations, some authors have suggested applying external energy such as ultrasound, LED light or hot metal elements after mixing the material so as to speed up the setting reaction and improve the mechanical properties of the set GIC.^{7,9-13} Preheating has been used successfully with composite resin materials to improve the mechanical and physical properties,^{14,15} but only a few studies evaluated heating before mixing the GIC.^{10,16} Currently, it is not clear if there is an effect of heating before mixing GIC restorative materials. Although GICs and composite resins show different behavior due to

their individual composition, heating GICs before mixing may similarly improve the mechanical and physical properties.

For composite resins, preheating increases the monomer's degree of conversion by reducing the viscosity of the material, increasing the microhardness and flowability,¹⁷ increasing diametral tensile strength,¹⁸ improving marginal adaptation, as well as reducing microleakage.¹⁹ Previous researchers have found that external energy applied to hand-mixed GIC after mixing potentially increases benefits, after applying thermo-light curing for 60s,¹³ such as increased superficial microhardness up to a depth of 4mm, reduction of crack propagation and working time,^{9,10} as well as an improvement in marginal microleakage.¹¹

The aim of this study was to evaluate the influence of preheating (before mixing the material) on stabilization time of the metal carboxylate bonds and surface microhardness in capsulated restorative glass-ionomer cement materials. The null hypotheses tested were 1) the application of extrinsic thermal energy does not modify the time needed to stabilize the metal carboxylate bonds during the setting reaction of restorative glass-ionomer cement materials, 2) the preheating of glass-ionomer does not change the physical property of surface microhardness.

4.3. METHODS AND MATERIALS

This was an *in vitro* study to assess the effect of preheating on two restorative GIC materials on the dynamics of their setting reaction, as evaluated by infrared spectroscopy, and on the microhardness, as assessed by the Knoop hardness test. Preheating of capsules was performed at 54°C for 30s. For each of these analysis methods (spectroscopy and microhardness), 10 and 20 samples respectively of each material were made, half without preheating (control) and half with preheating of the material.

The GICs, batch numbers and manufacturers of the products evaluated in this study are listed in Table 1.

Table 1 - Brand names, batch numbers and manufacturers of the products.

Material	Chemical Composition	Batch n°	Manufacturer
Ketac Universal	Water 40-60% Acrylic – maleic acid copolymer 30-50% Tartaric acid 1-10% Benzoic acid <0.2% Glass oxide >95%	3817763	3M ESPE, Seefeld, Germany
Equia Forte	Polybasic carboxylic acid 5-10% Iron oxide (III) <0.5%	1706191	GC, Europe N.V., Leuven, Belgium

4.3.1. Measurement of glass-ionomer cement temperature inside the capsules

To determine the actual or eventual temperature of the glass-ionomer cement material inside the capsules when preheated, an additional analysis was conducted. A thermographic camera (ImageIR® 7300, InfraTec, Dresden, Germany), which operates in the wavelength range from 2 to 5.7µm was used; this was mounted upright and the sample positioned in its focal lens position at 25cm. The temperature measurements of this camera ranges from -40°C to 300°C, with a resolution of ±0.002°C. Measurements were performed at room temperature, which was around 22°C. The samples were placed on a waterproof paper (SDI, Victoria, Australia) suitable for GIC handling. First, three intact capsules were preheated and the internal thermographic images of both precursors, powder and liquid, were collected. After that, three new capsules were selected for the GIC preparation. They were heated for 30s and immediately transferred to the mechanical agitator for mixing. Then, immediately after removal from the capsule the thermal image of each GIC was collected, using the IRBIS® 3 software (Infratec, Dresden, Germany). The same software calculated the average temperature variations of the samples (n=3) of each material.

4.3.2. Sample preparation

Samples of each material were prepared and divided into two equal groups: one group with preheating (test group) and one group without preheating (control). For preheating the GICs, the capsule tips of the materials were slightly modified to enable them to be fitted into a heating device (Figure 1) (Calset; AdDent, Danbury, CT, USA) using a tungsten carbide bur

n°1251 (American Burrs, Pedra Branca, Palhoça, Brazil) in a slow-speed handpiece. The material capsules were either preheated to 54°C for 30s prior to mixing or not preheated (control). The mixing of each capsule was conducted according to each manufacturer's instructions for both groups.

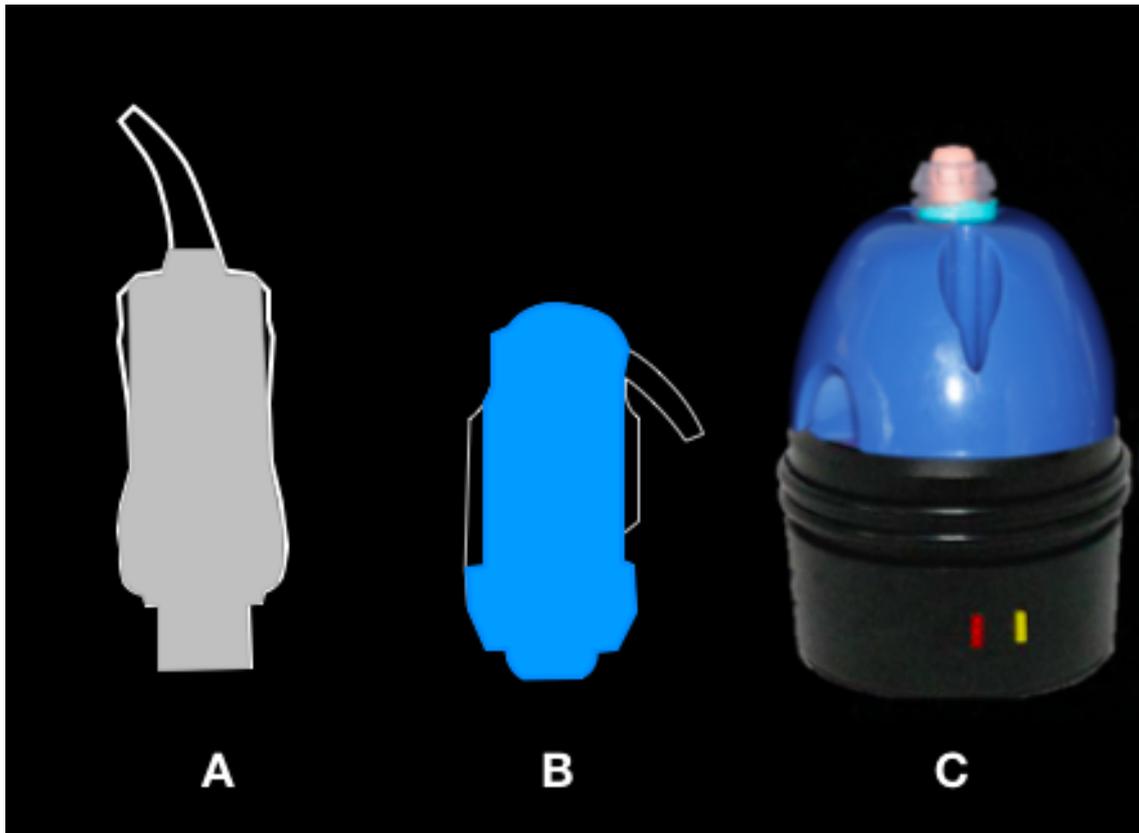


Figure 1 - A and B) Schematic drawings showing the original capsules and the slightly modified capsules overlapped. The filled area in each drawing represents the final modified capsule (A: Equia Forte, GC Corp.; B: Ketac Universal, 3M ESPE). C) The modified capsule inside the heating device.

For the *Fourier Transform Infrared (FTIR) spectroscopy* analysis, 10 samples of each material were prepared and divided into 2 groups: 5 for the non-preheated group (control) and 5 for the preheated group (test group). For preparation of these samples, each mixed material was placed between two polyester tapes, pressed between two plates of glass and loaded with a constant force of 0.4N for 30s generated vertically to the specimen via the upper glass plate. After the first 5min of setting, the samples of GIC were prepared for measurements. The GICs were ground in a mortar using an agate pestle. After that, they were mixed with potassium

bromide (KBr) powder and pressed with a manual hydraulic press (Specac, Orpington, Kent, UK) under 10 SI (tons), for 2min to obtain pellets for analysis.⁷

Following the protocol of Xie *et al*²⁰ for microhardness measurement, 20 cylindrical specimens of each material were prepared using a split steel mold with internal dimensions of 3 mm height and 6 mm diameter. To prepare each specimen, the mixed GIC was placed into the mold, and compressed with polyester tapes on both sides of the mold with a screw clamp for 10min. Control (non-preheated) specimens (n=10) of each material were mixed and fabricated at room temperature, according to the manufacturer's instructions. In the test group (preheated) specimens (n=10) of each material, the capsules were preheated to 54°C in the heating unit as in the previous section for 30s prior to mixing according to the manufacturer's instructions.

4.3.3. Analysis of the stabilization time (ST) of the chemical bonds by Fourier Transform Infrared (FTIR) spectroscopy

The characterization of the molecular stabilization time (ST) of GICs was determined with a FTIR research spectrometer (Vertex 70v, Bruker Optik GmbH, Ettlingen, Germany). To produce pellets (n=5) for this analysis, 0.002g of GICs was diluted with 0.198g potassium bromide (KBr), weighed with an analytical scale (GH-202, A&D Weighing, San José, CA, USA), and compressed with a manual hydraulic press (Specac, Orpington, Kent, UK) under 10 SI (tons) for 2min. The spectra of the KBr-sample pellets were collected by making an average of 128 scans with a 4cm⁻¹ resolution in the spectral range of 4000-400cm⁻¹. FTIR spectra were collected 10min after mixing the GIC in both preheated and non-preheated samples, and again every 10min for 120min thereafter.

The samples were kept in the FTIR spectrometer, maintaining the vacuum condition throughout the whole analysis. The setting process was analyzed in the spectra by the ratio of the intensities of the bands associated with the formation of the COO⁻ carboxylate salts of the polyacid (1637cm⁻¹) and C=O acid group (1720cm⁻¹) as a function of time, following the same methodology described by De Oliveira *et al* (2019).⁷ Analysis was performed by the variation of the ratio band intensities as a function of time. The fitting was done using an exponential decay-type function written as (Equation 1):

$$y(t) = y_0 + y_1 e^{(-t/\tau)}$$

with y_0 being the area of the bands after the material reached stabilization, y_1 the amplitude of the area variation, t the instant times of the measurements, and τ the characteristic decay time

representing the instant t when the area of the bands decay to $1/e$ (approximately 37% of y_1 value). In this type of mathematical analysis, the curve shape tends to a constant value with time (approximately five times the value of τ), meaning that 99.95% of the decay variation has been reached and the material can be considered stabilized in terms of the changes of the chemical bond.² This analysis was performed to observe the dynamics of possible changes in the chemical bonds of the restorative material after preheating.

4.3.4. Microhardness measurement: Knoop microhardness test

For each specimen, the microhardness test was performed at three time intervals: 10min after mixing, after the stabilization time of chemical bonds as detected through the FTIR spectroscopy (Table 2) and after one week. During this period, the specimens were stored in a dry, closed environment at a room temperature of 22°C.

The microhardness was determined using a microhardness tester (FM-ARS 900, FUTURE-TECH Corp., Tokyo, Japan). The Knoop hardness test was performed using a diamond indenter with a 25g load and 15s dwell time. Three measurements were made on the surface of each of the ten specimens for the preheated and non-preheated materials investigated.

4.3.5. Statistical Analysis

Data collected from FTIR spectra were submitted to Shapiro-Wilk normality, Levene homogeneity and Student t-test at the 5% significance level. To analyze the influence of preheating on microhardness by the time (initial, ST and 7 days) the repeated measures factorial One-way Analysis of Variance (ANOVA) and post hoc comparison of means (Tukey test) were performed to determine any significance over time ($p=0.05$). To analyze the influence of preheating on GIC the t-test was applied. The statistical analysis was carried out with SigmaPlot 12.0 (Systat Software Inc., San Jose, California, USA) software.

4.4. RESULTS

Figure 2 shows the thermal images obtained with the thermographic camera. On the left side and in the middle are the images for room or ambient temperature in the capsule and after preheating at 54°C for 30s, respectively, for both powder and liquid precursors positioned inside the capsules. On the right side are the images for the capsule after preheating and mixing. Ketac Universal (A) showed an increase in temperature from the ambient temperature in the capsule of 15.7°C for powder and 3.6°C for liquid, after 30s of

preheating, while Equia Forte (B) showed increases of 16.4°C for powder and 8.5°C for liquid. On the right, after preheating and mixing, the temperature rise in the two GICs in relation to room temperature was 7.3°C for Ketac Universal and 5.7°C for Equia Forte.

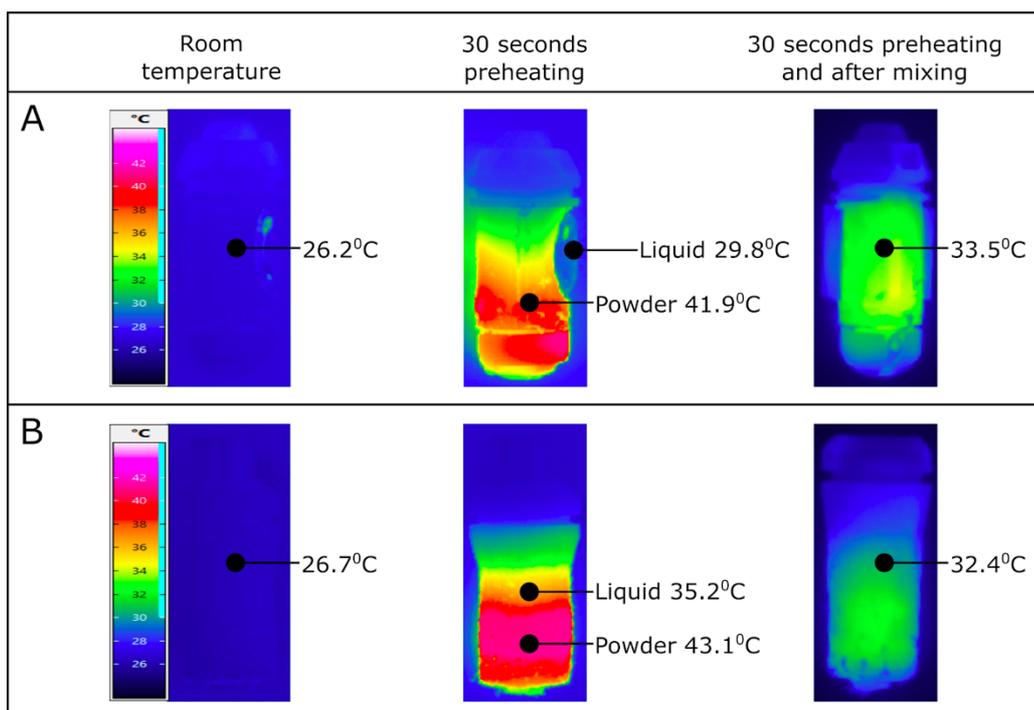


Figure 2 – Thermographic images of the precursors, powder and liquid, at room temperature (left) and 30s after preheating at 54°C (middle). On the right are images for the heated and mixed GICs, Ketac Universal (A) and Equia Forte (B).

4.4.1. FTIR spectroscopy

The FTIR results (Figure 3) showed that preheating the capsules of Ketac Universal increased the time of stabilization of chemical bonds and reduced the ST for Equia Forte (Table 2).

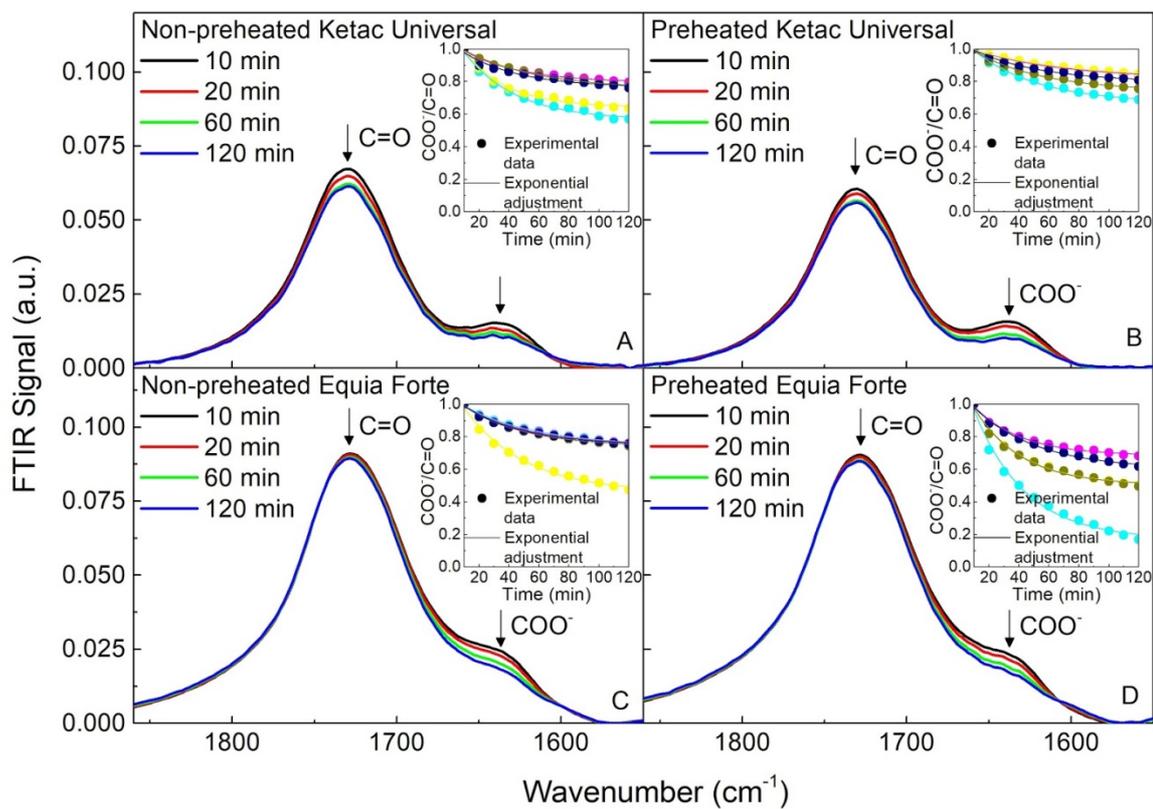


Figure 3 - FTIR spectra: (A) non-preheated Ketac Universal (control), (B) preheated Ketac Universal, (C) non-preheated Equia Forte (control) and (D) preheated Equia Forte. The inserts show the changes of the COO⁻/C=O ratio for the samples (n=5) as a function of time adjusted with the exponential function (Equation 1).

Table 2 - Mean stabilization times (in mins with standard deviations) of chemical bonds obtained with Fourier Transform Infrared (FTIR) Spectroscopy

Time in minutes				
Groups	Room temperature (22°C)		Preheated	
	τ	Stabilization time (ST) (5× τ)	τ	Stabilization time (ST) (5× τ)
Ketac Universal	26.26±5.49	131.30±27.46 ^A	35.55±6.02	177.75±30.08 ^B
Equia Forte	29.61±2.23	148.08±11.14 ^A	23.79±3.14	118.99±15.72 ^B

Means followed by the same letter comparing the columns (materials preheated and non-preheated), are not statistically different ($p>0.05$). $n=5$ specimens/group. τ is the time characteristic of the chemical reaction and 5x this time was considered the time of stabilization, 99.95% of the reaction occurred in that period.

4.4.2. Microhardness measurement: Knoop microhardness test

Based on the stabilization time of chemical bonds, the microhardness test was performed on the materials with and without preheating, at 3 different time intervals: 10min after mixing (t_1), after the stabilization time of chemical bonds (t_s) and after 7 days (t_7). The means and standard deviation values of the microhardness test are shown in Table 3 and Table 4.

Table 3 - Means and standard deviations of the surface microhardness (Kg/mm²) values of the groups studied at 3 different time intervals

Group	Initial (at t ₁)	At stabilization time (t _s)	At 7 days (t ₇)
Ketac Universal	36.0±10.4 ^a	64.3± 7.2 ^b	74.3±7.8 ^c
Preheated Ketac Universal	39.9±7.2 ^a	69.0±7.6 ^b	87.6±9.6 ^c
Equia Forte	32.1±6.7 ^a	61.1±6.3 ^b	68.5±7.2 ^b
Preheated Equia Forte	37.8± 3.9 ^a	58.6±9.1 ^b	77.0±7.2 ^c

Means followed by the same letter (*i.e.* time: initial, after stabilization and after 7 days of each material) are not statistically different ($p>0.05$). n=10 specimens/group.

Table 4 - Means and standard deviations of the surface microhardness (Kg/mm²) values of materials preheated or not preheated at 3 different time intervals

Group	Initial (at t ₁)	p	At stabilization time (t _s)	p	At 7 days (t ₇)	p
Ketac Universal	36.0±10.4	0.464	64.3±7.2	0.171	74.3±7.8	0.003*
Preheated Ketac Universal	39.9±7.2		69.0±7.6		87.6±9.6	
Equia Forte	32.1±6.7	0.041*	61.1±6.3	0.504	68.5±7.2	0.017*
Preheated Equia Forte	37.8±3.9		58.6±9.1		77.0±7.2	

* Statistical significance ($p>0.05$).

Analyzing the microhardness values over time (Table 3), for Equia Forte non-preheated there was a significant increase in microhardness between t₁ to t_s, and t₁ to t₇, but not

between t_n to t_r . For each GIC, preheating showed a significant difference in microhardness between all time intervals.

It was observed that preheating increases significantly the microhardness of Equia Forte at t_r ($p < 0.05$; Table 4). With maturation after one week, it was observed that the microhardness increased significantly comparing non-preheated with preheated Ketac Universal ($p < 0.05$; Table 4) and Equia Forte ($p < 0.05$; Table 4).

4.5. DISCUSSION

The null hypotheses tested were rejected because the application of heat before mixing modified the time of stabilization of the chemical bonds and the surface microhardness of the GICs tested. With the spectroscopy analysis, a significant increase in the stabilization time of the chemical bonds for Ketac Universal and a significant reduction in the ST of Equia Forte were observed. It was also observed that preheating significantly increased the microhardness of the two materials. Menezes-Silva *et al*²¹ reported that the longer the stabilization time of the chemical bonds the greater will be the mechanical properties of the material. In the present study, it was observed that preheating Ketac Universal increased the ST by approximately 40min, with a significant increase in surface microhardness over time.

The differences between the two materials tested can be attributed to the complexity of the setting reaction. It was initially previously shown that the setting of glass-ionomer cements involved not only neutralization of the polyacid component, but also reaction of inorganic species arising from the ion-depleted glass particles.²² Subsequent studies suggested that the key components were phosphate species.²³ Preheating the cement components is likely to affect both possible setting reactions with two possible consequences. Depending on the relative speeds, both reactions may speed up and result in increased rates of formation of both the ionically-crosslinked polyacid chains and the inorganic network. Alternatively, if the inorganic network forms too quickly, this will reduce the mobility of polymer chains, and could reduce the rate at which the acid functional groups react to form carboxylate salts. It is this latter reaction which was observed using FTIR and on which the calculation of stabilization time was based. Consequently, it is possible that either an increase or decrease in stabilization time may occur with heat; also we would not expect these changes to be correlated with variations in hardness determined mechanically.

Although preheating has different effects on the stabilization time of the chemical bonds within GICs, the heating is believed to increase the rate of diffusion of ions, accelerating the overall setting reaction, with consequent reduction in work time and reaction

time.¹⁹ Therefore, GIC heating would be beneficial for improving its initial properties, when the material is more susceptible. It is suggested that the application of heat to GIC, especially in the hand-mixed versions, evaporates part of the water in the cement matrix and accelerates the chemical reaction of the material.²⁴ However, it is important to point out that the hardening time (setting time) of the material presented by the manufacturer is different from the time to reach chemical stabilization²¹. There are certainly advantages of a shorter working time, i.e. reduction of contamination with saliva exposure and increase of the initial resistance of the material.²⁵ Although it has been observed that GIC loses mass when it is heated in a dry environment after mixing, at the same time there is heat absorption that generates expansion of the material.²⁶ Nevertheless, heating up to 50°C promotes minimal dimensional changes in the material, keeping in balance the volume.²⁷ With the application of external energy and consequent temperature rise in the GIC after mixing, weak water bonds are the first to be lost promoting mass loss and small structural changes, which can be reversed after contact with water.²⁷

The analysis of spectra data showed a difference in the intensity of the peaks with no displacement (Figure 3). This suggests that the preheating promotes a modification in the time of the chemical reaction of the cement, but it was not possible to observe the presence of damage or changes to its molecular structure. GIC formulations consist of glass particles with complex structure and several components.²⁸ Any change in the proportion of these components as well as the polyacid concentration, the size and shape of the glass particles, may influence the final reaction result.²⁰ De Oliveira *et al*⁷ suggest that the variation in the behavior of GIC after heating can be attributed to the different compositions of the material, as well as porosity, hydrophilicity and thermal properties.

The first few hours after handling and insertion of the material into the cavity are the most critical. The material is more vulnerable to moisture, which influences the susceptibility to fracture and initial wear.² Clinically, a protective agent is used to avoid the problems of syneresis and imbibition. This aspect was not investigated in this work because the protective material could interfere in the hardening process of the GIC and also may present additional FTIR bands that could overlap with those from the GIC. Furthermore, for the FTIR test, protection was not possible because the reading was performed during the hardening of the material. For the microhardness test, the protection was also not performed because it was intended to maintain the same condition in both tests. To prevent dehydration during the microhardness test, the samples were maintained dry in a small closed environment controlling the dehydration of the material, with only the water present in the liquid allowing

the material to harden after 7 days. It is felt that maintaining the water balance of the cement is important. The specimens were maintained for 10 minutes inside the mold, without contact with oxygen. In cement, the water undergoes some kind of interaction with other chemical species present and is strongly linked. There seem to be several ways in which water gets attached to ionomer cements over time.⁴ One is the hydration of the cations released by the glass, they are all present in the anhydrous state, but they are capable of strongly "coordinating" the water and all will form highly hydrated ions under appropriate conditions.²⁹ It seems like this occurs inside the cement and that the hydrated ions formed are stable and capable of retaining their water molecules, even under desiccation conditions.⁴

Clinically, the faster the material hardens, the better its initial mechanical properties will be and the less susceptible it will be to the early masticatory load.³⁰ However, recently it has been demonstrated that the slower the ST of GIC, the more the chemical bonds will be formed, consequently improving the final mechanical properties of the material.²¹ Therefore, with the reduction of working time and setting of GIC, the restorations are expected to have increased durability and for patients with difficult handling, GICs may be the material of choice. Some authors also found an increase in the microhardness of GICs after applying external energy in the form of heat after mixing, and attributed this to the composition of the material.^{9,12,31} O'Brien *et al*¹⁰ observed that the preheating of capsules prior to mixing had a greater influence on the microhardness depth of the GIC compared to the application of heat with ultrasound and LED light after mixing. Unlike composite resins, the viscosity of GICs apparently increased with preheating, which could impair the adaptation of the material to the cavity and encourage the inclusion of air voids.

To date there is little work^{10,16} on preheated GICs, making it difficult to compare the data. Most of the studies involved warming the materials after mixing. In the present study, the time required for preheating was established from a pilot study conducted by the authors. It was observed that 30s was sufficient to promote changes in the stabilization time of the chemical bonds of the material and allow a sufficient working time. With time greater than 30s, the material became very viscous and was difficult to express from the capsules.

Analyzing the results of the thermographic camera, it was observed that the material inside the capsule did not reach the temperature recommended by the heating device. This may have occurred because the capsule prevented the transmission of heat. The temperature of 54°C was determined from the literature,¹⁴ and it was observed that there is significant improvement of the superficial initial hardness when GIC is heated to a maximum of 60°C. Previous research on external energy applied with LED light, showed that the light

temperature reaches a maximum of 60°C after 60s of activation. As GIC has low thermal conductivity, heating is considered a safe procedure.³² Therefore, training and working rapidly are necessary so that the material does not lose heat before the restorative procedure is carried out. Also, care is necessary to avoid bubbles and formation of gaps. The literature shows that composite resin loses 50% of temperature approximately 2min after preheating.³³ This may also occur with GICs.

Commercial GIC capsules are not uniform in construction and contain a volume of air in the powder region of the capsule. Furthermore, the location of the GIC liquid is different in each product. The Equia Forte liquid is in a more central region of the capsule, while the Ketac Universal capsule has the liquid in a small compartment more at the capsule periphery. Thus, there are unequal internal heating patterns between the two capsulated materials and this was demonstrated with the thermographic camera data. The thermographic camera was found to be a useful method for measure the temperature changes in this kind of research.

Preheating is an easy method that could be used in a dental office. The devices used for preheating composites could also be used with GIC capsules with slight modification. This may prompt manufacturers to modify the design of their GIC capsules to fit into standard preheating devices or alternatively, to produce devices specifically for their capsulated materials.

A huge benefit of using GIC as a restorative material is its ability to release fluoride. If preheating will be able to improve the mechanical properties and the ability to release fluoride, the preheating technique could be adopted to improve the material properties. Future research should be conducted to further clarify the benefits of preheating GIC.

4.6. CONCLUSIONS

Preheating influences the stabilization time of the chemical bonds and increases the microhardness of the GICs tested. For the microhardness test, the heating influenced the maturation of the cements in the stabilization time and in 7 days. These results demonstrate that preheating is a promising technique.

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5. ARTIGO 3 - Influence of preheating of glass-ionomer cement on fluoride release and uptake: An *in vitro* study

5.1. Abstract

Objectives: To evaluate the effect of preheating on the release and uptake of fluoride (F⁻) in two glass-ionomer cements. **Materials and Methods:** Sixty specimens (5.5mm x 3mm) from Equia Forte (GC) or Ketac Universal (3M ESPE) were prepared for three storage times (1 day, 7 days and 1 month). Half were preheated at 54 °C for 30 s and the other half were not. After mixing, each specimen was weighed, immersed in 5 cm³ of deionized water and stored at 37 °C. After each time period the specimens were dried, weighed and immersed in 5 cm³ of NaF solution (1000 ppmF) for 24 h and the F⁻ was determined. The specimens were transferred to 5cm³ of deionized water and stored for an additional 24 h after which the F⁻ was measured. Data were analyzed by ANOVA and Tukey (p<0.05). **Results:** For both materials, there was mass gain after immersion in deionized water and mass loss after immersion in NaF solution. All materials showed uptake of F⁻ after immersion in NaF solution, but without significant difference in uptake between groups. Despite the F⁻ uptake, there was no change in F⁻ release on subsequent storage in deionized water. Also, F⁻ release showed no difference between preheated and unheated samples at any storage time. **Conclusion:** Preheating of the glass-ionomers Equia Forte and Ketac Universal does not alter their pattern of F⁻ release and uptake. **Clinical Relevance:** Preheating glass ionomer cements does not compromise fluoride release.

Keywords: Heating, Glass-ionomer Cements, Fluoride Release, Uptake

5.2. Introduction

Glass-ionomer cement (GIC) was launched onto the market in 1975, and since then it has been the material of choice for several clinical situations due to its physical and biological properties. It is biocompatible in the mouth [1,2], has chemical adhesion to tooth [3], possesses coefficients of thermal expansion close to that of the teeth, releases fluoride and is able to promote remineralization of tooth structure [4].

Modern glass-ionomers are greatly improved compared with the earliest versions of these cements. In particular, the particle size and size distribution of the glass powder is different, which makes them [5]. However, the first hours after placement of the material into the cavity are still critical, as it is during this period that the material is vulnerable to water

loss/gain and is also when the chemical bonds with dentin are formed [3]. This has led to proposals for methods of accelerating the initial hardening (setting) so that the material can resist water loss/gain and achieve high strength more rapidly. Possible means of accelerating setting include application of external energy in the form of heat from a halogen or LED dental curing lamp, or ultrasonication once the cement has been placed [6-11]. Another possibility involves preheating the cement components prior to mixing using a water bath [10,12]. Preheating in this way does not lead to simple increase in setting, and it has been observed that some GICs show a longer stabilization time for the carboxylate chemical bonds within the cement when heated [6], which implies that this aspect of setting may be inhibited by heating in certain cement formulations. On the other hand, preheating has always been found to lead to increased microhardness [7], reduced crack propagation [10,11] and better resistance to marginal microleakage [8].

Although the effect of preheating of glass-ionomers on mechanical properties is known, the effect on fluoride release and uptake has not been reported. By contrast, where heating is applied after placement, using an LED lamp, the amount of fluoride released was found to be reduced [13,14]. This is potentially a disadvantage, as release of fluoride by GIC is assumed to have clinical benefits, especially the initial relative high levels of release [15]. According to Menezes-Silva et al. [16], application of heat in this way increases the stabilization time of chemical bonds, specifically those associated with neutralization and formation of carboxylate salts from the polyacrylic acid component. For reasons which are not clear, these changes are associated with mechanical properties and may be responsible for the reduced amount of fluoride released.

The current work has been undertaken to address the question of the effect of preheating glass-ionomers on their fluoride release and also subsequent fluoride uptake. Two commercial glass-ionomer cements were studied, with storage under different conditions and time intervals. The null hypothesis was that the application of heating to GICs before mixing does not alter the fluoride release or uptake in these materials.

5.3. Materials and Methods

This was an *in vitro* study in which 40 specimens of each material were made for 3 different storage times (1 day, 7 days and 1 month), giving a total of 120 specimens. Half of the specimens were made using preheated material and the other half were not heated. Preheating of capsules was performed at 54 °C for 30 s, as described below. The materials used in this study are listed in Table 1.

Table 1: Brand names, batch numbers and manufacturers of the products

Material	Composition	Batch n°.	Manufacturer
Ketac Universal	Water 40-60%		
	Acrylic - maleic acid copolymer	3817763	
	30 - 50%	651704	3M ESPE, Seefeld, Germany
	Tartaric acid 1 - 10%		
	Benzoic acid <0,2%		
	Glass oxide > 95%		
Equia Forte	Polybasic carboxylic acid 5 - 10%	1801251 1809061	GC, Europe N.V., Leuven, Belgium
	Iron oxide (III) < 0,5%	1706091	

For preheating, the tips of the GIC capsules were modified slightly to fit a device and then preheated to 54 °C in a dental heating unit designed for use with composite resins (Calset; AdDent, Danbury, CT, USA). Heating was carried out for 30 s prior to mixing.

The capsules (preheated or non-heated) were activated and immediately transferred to a rotating mixer (RotoMix™; 3M ESPE) for mixing. Subsequently, the material was extruded into a PTFE mold to prepare discs of dimensions 5.5 mm diameter and 3.0 mm height. The material was pressed flat with polyester strips on both sides of the mold. After 10 min, the samples were removed carefully from the molds and weighed using an analytical balance (Mettler HK 160 - Greifensee - Zurich). They were then immersed individually in 5 cm³ volumes of deionised water and stored at 37 °C for 1 day, 7 days and 1 month.

After the relevant time of storage (1 day, 7 days and 1 month) the samples were again weighed, then placed in 5 cm³ volumes of sodium fluoride (NaF) at 1000 ppm in fluoride (Fluoride standard, Fisher Scientific, UK) for 24 hours. After this, the specimens were removed, dabbed dry with a tissue, weighed and the fluoride concentration was determined from the storage solution. The specimens were then transferred to individual 5 cm³ volumes of deionized water and stored for an additional 24 hours the specimens were removed, dabbed dry with a tissue, weighed and after that the fluoride concentration was measured from the storage solution.

In all cases, the fluoride concentrations were determined using a calibrated fluoride-selective electrode (type 4048, Thermo Orion Europe, Witchford, Cambridgeshire, UK). The electrode was connected to a pH/ISE meter analyser (model 710A, also Thermo Orion Europe, Witchford, Cambridgeshire, UK) via a cable with dual electrode head (ELIT 201, NICO2000 Ltd, Harrow, Middlesex, UK). The electrode was calibrated using standards prepared by serial dilution of a commercially available 1000 ppm fluoride solution (as NaF) (supplied by Sigma-Aldrich, Poole, Dorset, UK). All fluoride determinations were carried out with samples or standards diluted by 1:1 addition of TISAB IV to ensure complete decomplexation of all available fluoride.

Following the above experimental work, data obtained were analysed for statistical significance using ANOVA followed by the Tukey HSD test ($p < 0.05$).

5.4. Results

Mass changes for specimens in different storage solutions and for different time intervals are shown in Table 2. Results showed a consistent pattern of mass gain on initial storage in water followed by mass loss on subsequent storage in fluoride solution. Although all specimens lost mass during the storage in fluoride solution, the value did not drop below the initial mass over the total storage period.

Table 2: Mass changes for glass-ionomer specimens (%). Standard deviations are given in parentheses

Material	Storage time	Medium	Change (%) compared with initial mass*	
Equia Forte	1 day	Water	1.98 (0.34)	A
		NaF solution	1.50 (0.28)	a
	7 days	Water	2.54 (0.30)	B
		NaF solution	2.21 (0.30)	b
	1 month	Water	3.26 (0.21)	AB
		NaF solution	2.74 (0.28)	b
Equia Forte preheated	1 day	Water	2.12 (0.37)	C
		NaF solution	1.84 (0.29)	c
	7 days	Water	2.54 (0.30)	C
		NaF solution	2.21 (0.20)	d
	1 month	Water	3.26 (0.21)	C
		NaF solution	2.74 (0.28)	d
Ketac Universal	1 day	Water	2.82 (0.28)	D
		NaF solution	3.07 (0.25)	d
	7 days	Water	2.95 (0.24)	D
		NaF solution	3.27 (0.29)	d
	1 month	Water	2.77 (0.36)	D
		NaF solution	2.67 (0.38)	d
Ketac Universal preheated	1 day	Water	2.03 (0.23)	E
		NaF solution	2.31 (0.25)	e
	7 days	Water	2.74 (0.46)	F
		NaF solution	3.02 (0.80)	ef
	1 month	Water	3.05 (0.48)	F
		NaF solution	2.94 (0.44)	f

*Means followed by the same letter indicate no significant difference in the group ($p > 0.05$), (uppercase compares storage medium water, and lowercase compares NaF solution storage, values based on initial mass before immersion).

Amounts of fluoride released after storage in deionized water are shown in Table 3. All samples released fluoride under these conditions, and in all cases there was no significant difference in release between unheated and preheated samples of the same

material. In the case of Equia Forte, there were significant differences between the various storage times, i.e. between 1 day and 7 days and between 7 days and 1 month, showing that longer storage times progressively increased the amount of fluoride that was released by the cements. For Ketac Universal, the difference between the 1 day and 7 days storage periods was significant, but the difference between 7 days and 1 month was not. However, for both unheated and preheated specimens, there was an increase in the amount of fluoride released after 1 month of storage compared with 7 days. These results show that for this material too, longer storage increases the amount of fluoride that can be released from the cement.

Table 3: Means and (standard deviations) of fluoride release at 1 day, 1 week and 1 month (ppm).

Material	Storage time	Fluoride release/ppm*	
Equia Forte	1 day	25.0 (4.7)	A
	7 days	43.6 (7.8)	B
	1 month	88.7 (4.2)	C
Equia Forte preheated	1 day	23.8 (5.0)	A
	7 days	40.6 (6.9)	B
	1 month	90.2 (8.8)	C
Ketac Universal	1 day	16.6 (2.4)	A
	7 days	35.5 (3.8)	B
	1 month	45.4 (7.9)	B
Ketac Universal preheated	1 day	12.9 (1.9)	D
	7 days	28.7 (4.8)	B
	1 month	48.9 (6.8)	B

*Means followed by the same letter are not statistically different ($p>0.001$).

The fluoride concentrations in storage solutions after the samples had been stored in them for 24 hours are shown in Table 4. In all cases, the initial concentrations were 1000 ppm, so all of these values represent a reduction in fluoride concentration. This shows that these specimens take up fluoride from the storage solution. Although the values varied slightly, none of the differences was significant.

Table 4: . Mean and (standard deviations) of fluoride uptake by samples (ppm)

Material	Storage time	Amount of fluoride absorbed *	
Equia Forte	1 day	783.2 (17.0)	A
	7 days	811.4 (17.7)	A
	1 month	829.2 (13.3)	A
Equia Forte preheated	1 day	806.2 (12.2)	A
	7 days	806.2 (12.9)	A
	1 month	811.0 (14.4)	A
Ketac Universal	1 day	801.1 (47.6)	A
	7 days	831.3 (28.3)	A
	1 month	838.4 (34.2)	A
Ketac Universal preheated	1 day	834.6 (15.8)	A
	7 days	836.6 (30.2)	A
	1 month	834.2 (31.0)	A

*Means followed by the same letter are not statistically different ($p>0.001$).

Table 5 shows the results for fluoride release following 24 hours storage in NaF solution, together with the corresponding figures for % release and % retention. These figures are based on the amount of fluoride taken up during storage in NaF solution, so that % retention figures show that, in all cases, the majority of the fluoride taken up is retained by the cements. There was no significant difference between unheated and preheated specimens of either material at any storage time, and no consistent trends as storage time increased. The smallest retention amounts were shown by Ketac Universal preheated at 7 days storage, but even these specimens retained over 70% of the fluoride taken up on exposure to NaF solution.

Table 5: Means and (standard deviations) of fluoride exchange after exposure to NaF solution.

Material	Storage time	Fluoride release after storage in NaF solution/ppm	Proportion released/%	Proportion retained/%
Equia Forte	1 day	32.0 (4.7)	14.7	85.3
	7 days	23.7 (3.7)	12.6	87.4
	1 month	26.3 (3.9)	15.4	84.6
Equia Forte preheated	1 day	33.0 (10.2)	15.5	84.5
	7 days	21.1 (4.3)	15.5	84.5
	1 month	24.0 (2.8)	12.7	87.3
Ketac Universal	1 day	37.3 (8.1)	18.8	81.2
	7 days	45.5 (7.8)	27.0	73.0
	1 month	28.7 (8.3)	17.8	82.2
Ketac Universal preheated	1 day	32.6 (4.7)	19.7	80.3
	7 days	48.9 (6.8)	29.9	70.1
	1 month	30.9 (8.8)	18.6	81.4

5.5. Discussion

The null hypothesis was accepted because both the fluoride release and subsequent uptake were unaltered by preheating the GIC material. Preheating made no difference to either fluoride release or fluoride uptake at any storage time; it also made no difference to mass change on storage.

Specimens for all storage times and whether unheated or preheated showed a gain in mass on initial storage in water. The subsequent storage in fluoride solution caused a slight loss relative to the final value after 24 hours in water, but this was still higher than the initial mass prior to immersion. The third immersion, this time in deionized water once more, showed a further reduction in mass compared with the value after 24 hours in water, but there was still a gain in all cases relative to the pre-immersion masses. This is similar to behavior previously reported for glass-ionomer cements, where storage in aqueous media leads to an initial increase in mass at 24 hours, followed by a gradual loss for some time afterwards [17].

The application of heat after mixing and/or preheating the glass-ionomer cements has been previously undertaken in an attempt to improve their mechanical properties. Some authors have shown that warming brings about improved microhardness [10]. It also seems to cause a reduction in porosity [18] and also improved adhesion to dental structures [19]. Prior to the present work, there have been no studies on the effect of preheating glass-ionomers on their fluoride release.

However, heating of glass-ionomer samples following initial hardening has been studied and shown to have an adverse effect on their fluoride release. Woolford [20] showed that the application of radiant heat led to reduced ability to release fluoride. More recently, the application of light from LED lamps has also been shown to reduce fluoride release [15,21,14]. By contrast, ultrasound applied to a glass-ionomer cement while setting leads to a material which shows increased levels of fluoride release [22]. This is despite the fact that ultrasound, like external heat, speeds up the setting process and leads to stronger cements.

It is not clear why heating the components of the cement prior to mixing does not have similar effects on fluoride release. Our results, though, are quite clear; preheating the cement capsule causes no significant differences in fluoride for either material at any storage time. Further work is necessary in order to explain these observations.

Our results show another feature that has not been reported previously, namely, that no matter how much fluoride is released by the cement the uptake does not vary, at least not significantly. Both brands show increased fluoride release at longer storage times, yet there is no increase in the amount of fluoride later taken up by those samples matured for longer times. Furthermore, as shown in previous studies [23], the amount of fluoride taken up is much greater than the amount previously released. This shows that fluoride exchange does not simply happen from specific, well-defined sites on the cement surface. It is not the case that fluoride ions are released, leaving vacancies in the surface and that subsequent immersion in fluoride solution leads to these vacancies being re-occupied. Rather, there are sites on the surface that are capable of taking up fluoride ions independently of any prior loss of fluoride. Moreover, these sites generally bind that fluoride irreversibly. Our results confirm previous observations in this regard and confirm that most of the fluoride taken up from a fluoride solution is retained by the cement when it is later placed in deionised water [24,23].

5.6. Conclusion

The effect of preheating on fluoride release and uptake from the glass-ionomer brands Equia Forte and Ketac Universal has been studied. The amount of fluoride released varies

with storage time, but increased fluoride release at longer storage times did not change the amount of fluoride subsequently taken up. It follows that fluoride release and fluoride uptake are independent events, and take place at different sites on the cement surface. Preheating made no difference to the amount of fluoride released or taken up, regardless of storage time, from which we conclude that the rate of setting does not affect the sites of fluoride exchange. Application of external heat after mixing has been previously shown to affect fluoride release, and the difference between these observations and ours needs further investigation.

5.7. REFERENCES

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6. ARTIGO 4 - Avaliação da influência do pré-aquecimento de resinas *bulk-fill* nas propriedades reológicas e de fotoativação

6.1. Resumo

OBJETIVOS: Avaliar o comportamento de fluxo e viscoelasticidade sob tensão de cisalhamento contínuo, a microdureza em profundidade e o grau de conversão de resinas *bulk-fill* de consistência *flow* (BFF), em diferentes temperaturas de pré-aquecimento.

MATERIAIS E MÉTODOS: Trata-se de um estudo *in vitro* realizado com cinco resinas BFF: Tetric N-Flow Bulk fill (TNF); Opus Bulk fill flow (OBF); X-tra base/Voco (XTR); Filtek bulk fill flow (FBF) e Surefill SDR Flow (SDR). e uma resina *flow* convencional, Tetric N-Flow (TNC). As resinas foram avaliadas quanto ao comportamento reológico de cisalhamento contínuo nas temperaturas de 24, 37, 54 e 68°C, utilizando um reômetro de gradiente e tensão de cisalhamento controlado no modo de fluxo, geometria cone-placa de 35 mm de diâmetro, ângulo de 2°, separadas por uma distância fixa de 0.052 mm. Para a análise da microdureza e do grau de conversão foram realizadas restaurações mésio-ocluso-distais em terceiros molares humanos, com o material pré-aquecido à temperatura de 24° ou 68°C em uma unidade de aquecimento. A microdureza foi avaliada com um indentador Knoop, após 30 dias, com carga de 25 g por 10 s, sendo 4 indentações na caixa mesial e 6 na distal. Para a análise do grau de conversão, em profundidade, as leituras dos espectros das restaurações foram realizadas por meio de um espectrofotômetro Raman, com laser 532 nm, objetiva de 20x, fenda de 50x1000 µm, 20 *coaditions* e 3 s. Foram realizadas 12 leituras na superfície interna da restauração da caixa mesial e 18 leituras da caixa distal.

RESULTADOS: Todas as formulações apresentaram redução significativa da viscosidade com o aumento da tensão de cisalhamento (índice de fluxo <1), ou seja, apresentaram comportamento pseudoplástico. Com o aumento da temperatura, para todos os materiais testados ocorreu redução nos índices de consistência e valor de cedência. À 24°C todos os materiais apresentaram área de histerese significativamente maior que às demais temperaturas, com exceção do grupo controle (TNC) que apresentou área de histerese menor à 24°C. TNC, TNF XTR apresentam aumento significativo da microdureza, OBF apresentou redução significativa do valor da microdureza com o aquecimento. O pré-aquecimento não influenciou o grau de conversão dos materiais testados.

CONCLUSÃO: O pré-aquecimento de BFF reduz a viscosidade, melhora a microdureza de alguns materiais (TNC, TNF, XTR) e não influencia o grau de conversão em todas as profundidades da restauração.

6.2. Introdução

A durabilidade das primeiras restaurações de resina composta em dentes posteriores era bem menor quando comparada às restaurações de amálgama. (1) Atualmente, a taxa de sucesso de restaurações em dentes posteriores com resina composta pode atingir até 97.9% após 10 anos de acompanhamento. (2) Um dos fatores que contribui para a longevidade das restaurações de resina composta é a qualidade e a durabilidade da adaptação marginal. Por sua vez, a adaptação marginal é influenciada pela contração volumétrica do material. (3,4)

Dentre as resinas compostas utilizadas para restauração de dentes posteriores, as resinas *bulk-fill* apresentam características otimizadas como menor contração volumétrica e de tensão de polimerização na interface adesiva (5) e são utilizadas especialmente em dentes posteriores. (5) A principal vantagem desses materiais é que, segundo os fabricantes e os resultados de algumas pesquisas, (6,7) elas podem ser inseridas em incrementos de 4 mm até 5 mm, diminuindo consideravelmente o tempo clínico.

Particularmente com incrementos maiores e o uso de resinas compostas de consistência regular, há a possibilidade de inclusão de bolhas no interior das restaurações e uma boa adaptação pode ficar ainda mais difícil. Uma das alternativas propostas para contornar este problema em resinas compostas convencionais tem sido o pré-aquecimento antes da restauração. (8,9) A principal fonte ativadora da reação de polimerização da resina composta é a luz, porém o calor também pode ser usado como tratamento para melhorar o desempenho. (10,11) O aquecimento aumenta a fluidez, o que melhora a adaptação do material ao preparo cavitário. (12,13)

O pré-aquecimento de resinas compostas convencionais melhora as propriedades físicas e químicas, promove maior grau de conversão dos monômeros influenciada pela redução da viscosidade do material, (14–19) aumenta a microdureza e fluidez, (18) aumenta a resistência à tração diametral, (20) melhora a adaptação marginal, bem como reduz significativamente a microinfiltração. (12,21) Em resinas *bulk-fill*, Tauböck *et al.* (22), avaliaram a influência da temperatura e observaram melhora na contração de polimerização e no grau de conversão e Dionysopoulos *et al.* (23) avaliou o efeito da temperatura na microdureza; Lempel *et al.* (2) observou aumento do grau de conversão em resinas reforçadas por fibra, porém efeito negativo no GC na base das amostras e Abdulmajeed *et al.* (24) relatam que o pré-aquecimento das resinas *bulk-fill* não afeta a resistência flexural e resistência à tração, porém aumenta a resistência à fratura.

Entretanto, até onde sabemos, há poucos relatos na literatura (25,26) sobre a reologia do material e o efeito do pré-aquecimento no comportamento de fluxo e de viscoelasticidade e

ainda há controvérsias sobre a influência do pré-aquecimento nas propriedades físicas das resinas compostas *bulk-fill* com consistência fluida. Neste contexto, o objetivo deste trabalho foi avaliar o comportamento de fluxo e viscoelasticidade sob tensão de cisalhamento contínuo, a microdureza e o grau de conversão de resinas *bulk-fill*, em diferentes temperaturas de pré-aquecimento. As hipóteses nulas testadas foram: H0₁ - o aquecimento de resinas *bulk-fill* de consistência *flow* não influencia a viscoelasticidade; H0₂ - o pré-aquecimento de resinas *bulk-fill* de consistência *flow* não influencia a microdureza; H0₃ - o pré-aquecimento de resinas *bulk-fill* de consistência *flow* não influencia o grau de conversão; H0₄ - o pré-aquecimento de resinas *bulk-fill* de consistência *flow* não aumenta a profundidade de polimerização.

6.3. Materiais e Métodos

6.3.1. Delineamento experimental

Trata-se de um estudo *in vitro* para avaliar a influência do aquecimento de cinco resinas *bulk-fill* de consistência *flow* (Tabela 1) nas propriedades reológicas, na microdureza e no grau de conversão em profundidade. Quatro temperaturas diferentes (24°C, 37°C, 54°C e 68°C) foram utilizadas na análise de índice de fluxo e duas (24°C e 68°C) para avaliar o grau de conversão e microdureza em profundidade. Para a análise da microdureza e do grau de conversão foram realizadas restaurações méso-ocluso-distais em terceiros molares humanos e os materiais foram pré-aquecidos em uma unidade de aquecimento Calset (AdDent, Danbury, CT, USA). Esta pesquisa foi aprovada pelo Comitê Permanente de Ética em Pesquisa com Seres Humanos da Universidade Estadual de Maringá - (CAEE No. 87404918.6.0000.0104).

Tabela 1 - Materiais avaliados e informações dos fabricantes

Classificação	Abreviação	Nome comercial	Fabricante	Composição	Tipo de Carga	Conteúdo total de carga (peso, %)	Escala de cor	Tempo de fotoativação (s)	Número de lote
Resina composta convencional "flow"	TNC	Tetric N-Flow	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Bis-GMA, Bis-EMA, UDMA (27.8%)	Vidro de bário, trifluoreto de itérbio, óxidos mistos, dióxido de silício e copolímeros	63.8%	A3.5	20	X12552
Bulk-fill: baixa viscosidade	TNF	Tetric N-Flow Bulk fill	Ivoclar Vivadent, AG, Schaan, Liechtenstein	Bis-GMA, Bis-EMA, UDMA	Vidro de bário, trifluoreto de itérbio, óxidos mistos e copolímeros	68.2%	IVB	20	W15996
	OBF	Opus Bulk fill flow	FGM, Joinville, SC	Bis-GMA, TEGDMA, UDMA (26%)	Vidro de Bário Silanizado, Sílica silanizada, 4-etil dimetil aminobenzoato (DABE), Trifluoreto e Ytérbio, Canforoquinona, Tinuvin P, Butil hidroxitolueno e corantes	68.0%	A2	20	290517 190618

	XTR	X-tra base/Voco	Voco	Bis-EMA, MMA	Vidro de Silício	75.0%	A2	20	1724340
	FBF	Filtek Bulk Fill Flow/3M ESPE	3M	Bis-GMA, Bis-EMA, UDMA, TEGDMA, Procrilat	Silica/zirconia, trifluoreto de itérbio	64.5%	A2	40	N886476 1808600331
	SDR	Surefill SDR Flow/Dentsply	Dentsply	EBPADMA TEGDMA, UDMA modificado	Vidro de bário alumínio boro flúor silicato; Vidro de estrôncio alumínio flúor silicato	68.0%	Universal	20	1610251 1607291
<p>Abreviações: AFM, Monómero de fragmentado de adição; UDMA, dimetacrilato uretano aromático; Bis-GMA, bisfenol-A Dimetacrilato de éter diglicidílico; Bis-EMA, Dimetacrilato de bisfenol-A etoxilado; Bis-MPEPP,2,2-bis (4-metacriloxipolietoxifenil) propano; DDDMA, Dimetacrilato de 1,12-dodecanodiol; EPDMA, Dimetacrilato de bisfenol-A etoxilado; PMMA, metacrilato polimetil; TEGDMA, dimetracrilato trietilenoglicol; TMGDMA, Dimetacrilato de trimetileno glicol; UDMA, Dimetacrilato de uretano.</p>									

6.3.2. Análise da viscoelasticidade

Para mensurar as propriedades dinâmicas viscoelásticas das resinas compostas *bulk-fill* (Tabela 1) foi utilizado um reômetro de gradiente e tensão de cisalhamento controlado MARS II, Haake® (Thermo Fisher Scientific. Waltham, MA USA), no modo de fluxo. As resinas foram avaliadas quanto ao comportamento reológico de cisalhamento contínuo nas temperaturas de 24, 37, 54 e 68°C, analisando os seguintes parâmetros: valor de cedência (τ_0), índice de consistência (κ), índice de fluxo (n) e área de histerese.

Utilizou-se a geometria cone-placa de 35mm de diâmetro, ângulo de 2°, separadas por uma distância fixa de 0.052 mm. (27,28) Uma porção pequena de resina suficiente para realizar a leitura, de aproximadamente 0.1g, foi cuidadosamente aplicadas à placa inferior do reômetro diretamente da seringa comercial sem a ponteita, assegurando o mínimo de cisalhamento da formulação e permitindo um tempo de repouso de 5 min antes de cada análise. As curvas ascendentes de fluxo foram obtidas com um gradiente de cisalhamento partindo de $0s^{-1}$ a $110s^{-1}$ e descendente de $110s^{-1}$ até $0s^{-1}$, de acordo com cada formulação. O gradiente de cisalhamento foi aumentado durante um período de 150 s, mantido no limite superior por 10 s e posteriormente diminuído durante um período de 150 s. (29) Em cada caso, as propriedades de cisalhamento contínuo foram determinadas em triplicatas. As curvas ascendentes e descendentes foram analisadas utilizando a equação de *Oswald de Waele* (Lei da Potência - Power Law; Equação 1), para obtenção dos índices κ e n (27,29–31):

$$\sigma = \kappa \cdot \dot{\gamma}^n \quad (1)$$

onde: σ = Tensão de cisalhamento; κ = Índice de consistência; $\dot{\gamma}$ = Gradiente de cisalhamento; n = Índice de fluxo.

Os modelos reológicos de Casson e Herschel-Buckley foram utilizados para obtenção do valor de cedência e estão respectivamente apresentadas abaixo pelas equações 2 e 3 (32):

$$\tau = \sqrt[n]{\left(\tau_0^n + (\dot{\gamma} \cdot \eta_p)\right)^n} \quad (2)$$

$$\tau = \tau_0^n + k\dot{\gamma}^n \quad (3)$$

onde: τ = tensão de cisalhamento; τ_0 = valor de cedência (valor de rendimento); k = índice de consistência; $\dot{\gamma}$ = gradiente de cisalhamento; n = índice de fluxo; η_p = viscosidade plástica de Casson.

Ademais, foram calculadas as áreas de histerese por meio do programa RheoWin 4.10.0000 (Haake® - Thermo Fisher Scientific. Karlsruhe, Germany).

6.3.3. *Preparo dos espécimes para análise do grau de conversão e microdureza*

Para a análise do grau de conversão em profundidade e para a microdureza foram realizadas restaurações simulando a posição do dente na arcada e os grupos foram compostos por 7 espécimes, totalizando 84 terceiros molares livres de cárie.

Foram selecionados 84 terceiros molares humanos extraídos e livres de cárie, armazenados após a extração em solução de timol 0.2%, em temperatura ambiente, por um período de tempo inferior à 6 meses. (33) Para cada material (Tabela 1) e cada temperatura de pré-aquecimento (24°C ou 68°C) foram utilizados 7 dentes humanos. As pontas de cúspide foram previamente removidas com o auxílio de uma cortadora de precisão a fim de padronizar a distância da incidência da luz do fotoativador à cavidade.

Foram realizados preparos de classe II complexa envolvendo as faces oclusal, mesial e distal. As dimensões na caixa oclusal foram de 3 mm de profundidade à partir da margem oclusal e 4 mm de extensão vestibulo-lingual; a extensão méso-distal foi determinado até o rompimento da crista marginal, com caixa mesial com distância cérvico-oclusal de 4 mm e distal com distância cérvico-oclusal de 6 mm e profundidade áxio-proximal de 1.5mm. Os preparos foram realizados com ponta diamantada 3145 (KG Sorensen - San José, Cotia - SP) em alta rotação, e polimento da cavidade com a ponta diamantada 3145 FF (KG Sorensen - San José, Cotia - SP) com Contra Ângulo Multiplicador 1:5 (Kavo – Joinville, SC). Para a realização dos procedimentos restauradores os dentes foram acoplados à um manequim odontológico (M.O.M. - Manequins Odontológicos Marília Ltda, Marília, SP). A abertura bucal do manequim foi determinada pela média de abertura bucal da população. (34)

Os dentes foram distribuídos aleatoriamente entre os grupos. Foi inicialmente realizado o ataque ácido em esmalte durante 30 s em todos os dentes, os quais foram lavados abundantemente e secos com jato de ar, com o auxílio de um microbrush foi aplicado o adesivo Single Bond Universal (3M – ESPE, Sumaré/SP) em todo o preparo e fotoativado por 20 s. Após esta etapa os dentes foram inseridos no manequim para a realização do procedimento restaurador. Para tal, foi utilizada a matriz Unimatrix (TDV - Pomerode, SC) e cunhas de madeira. As resinas compostas foram mantidas sob as temperaturas de 24 °C (temperatura ambiente) ou durante 15 min em uma unidade de aquecimento (Calset; AdDent, Danbury, CT, USA) à 68 °C. Assim, a cavidade foi restaurada com as resinas *bulk-fill* que sofreram ou não a alteração de temperatura. Para todas as resinas *bulk-fill* a cavidade foi

preenchida em apenas em um único incremento, para a resina TNC, o material foi inserido e fotoativada a cada 2 milímetros até o preenchimento de toda a cavidade. O tempo máximo entre a remoção do material na unidade de aquecimento e a fotoativação foi de 40s. (13) O material foi fotoativado com um aparelho Valo (1000 mW/cm² - Ultradent Product Inc., South Jordan, UT, USA), por 20 ou 40 s, seguindo a orientação do fabricante (Tabela 1). Os dentes foram cortados ao meio no sentido méso-distal com o auxílio de uma cortadora de precisão (IsoMet™ Low Speed, Buehler. Lake Bluff, Illinois, EUA) e um disco de diamante, por meio de um corte perpendicular ao longo do eixo do dente. Os dentes restaurados foram armazenados em estufa à seco 37 °C, por 7 dias (35) antes da leitura de para o grau de conversão e armazenados por 30 dias nas mesmas condições para o teste de microdureza. A metade vestibular de cada espécime foi utilizada para o teste de grau de conversão e a metade lingual/palatina foi destinada ao teste de microdureza.

Os espécimes que foram reservados para análise da microdureza receberam um tratamento de superfície. As superfícies foram polidas com discos de óxido de alumínio com granulação de 400, 600, 800 e 1200 e pastas de diamante de 6-, 3- e 1µm com o auxílio de uma politriz (APL 4, Arotec; Cotia, SP, Brasil).

6.3.4. Análise da microdureza

A microdureza do material restaurador foi avaliada através de um indentador Knoop (HVM-2, Shimadzu, Tóquio, Japão), após 30 dias, com uma carga de 25 g por 10 s. As indentações foram feitas à partir do topo da superfície da cavidade restaurada mais próxima à ponta do fotoativador até o fundo da cavidade, deixando uma margem de 0.3mm de resina entre o dente e a indentação, sendo que na caixa mesial foram realizadas 4 leituras e na distal 6 leituras, totalizando 10 leituras por espécime (Figura 1).

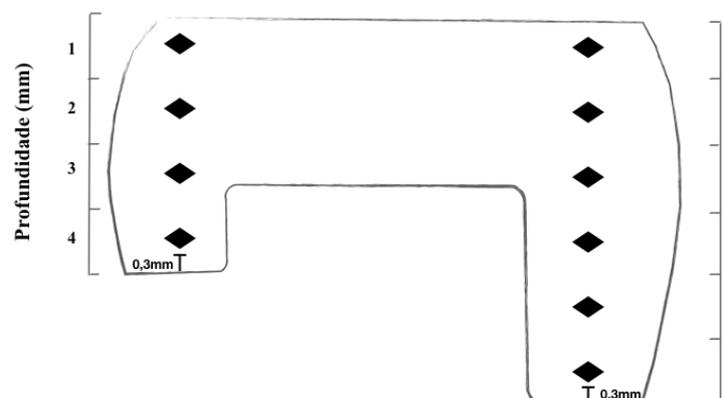


Figura 1 - Desenho esquemático do local das indentações realizadas para análise da microdureza. Total de leituras = 10.

6.3.5. Análise do grau de conversão

As leituras dos espectros das restaurações foram realizadas por meio de um espectrofotômetro Raman (VERTEX 70v - RAM II module - Bruker®, Illinois, Chicago, USA), laser 532 nm, objetiva de 20 x, fenda de 50 x1000 µm, 20 *coaditions* e 3 s. Para cada espécime foram realizadas 12 leituras na superfície interna da restauração da caixa mesial e 18 leituras da caixa distal. Partindo-se do topo da superfície da cavidade restaurada mais próxima à ponta do fotoativador até o fundo da cavidade, para cada milímetro em profundidade foram realizadas 3 leituras. Assim, para cada espécime realizou-se 30 leituras no total. Os espectros foram tratados no programa OPUS (Bruker Optik GmbH, Ettlingen, Germany) e os picos em 1610 e 1640 cm^{-1} foram selecionados para avaliação do grau de conversão. Para tanto, foi utilizada a fórmula:

$$\text{GC (\%)} = \left[1 - \frac{\text{R Polimerizada}}{\text{R Não Polimerizada}} \right] \times 100$$

Onde: R= Razão entre os picos 1610 e 1640 cm^{-1}

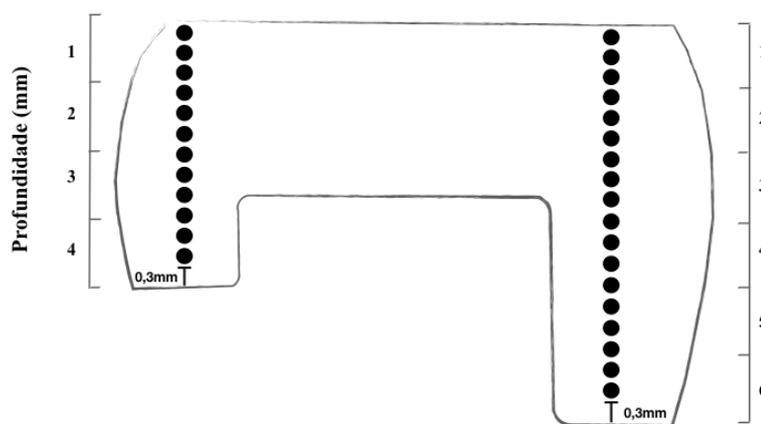


Figura 2 - Desenho esquemático dos pontos de leitura realizados para a análise do grau de conversão. Total de leituras = 30.

6.3.6. Análise estatística

Os dados da análise da reologia foram tabulados em programas específicos e analisados posteriormente no programa StatSoft Statistica 12 (Dell, Eldorado do Sul, RS). Para cada hipótese a ser testada foi realizado o teste de aderência à curva normal para verificar a presença ou não de distribuição normal através dos testes Shapiro-Wilks, Kolmogorov-Smirnov e Cramer-von Mises. Para os dados de grau de conversão e microdureza utilizou-se o programa R (R Foundation for Statistical Computing, Vienna, Austria), no qual foi utilizado um modelo ajustado de equações de estimativas generalizadas (GEE). Para todos os dados foram a análise de variância (ANOVA) e posteriormente test Tukey. Foi também aplicado análise de resíduos para o ajuste do modelo.

6.4. Resultados

6.4.1. Reometria de cisalhamento contínuo

Todas as formulações apresentaram redução significativa da viscosidade com o aumento da tensão de cisalhamento (índice de fluxo (n) <1), ou seja, apresentaram comportamento *shear-thinning* (pseudoplástico). Os reogramas de tensão por gradiente de cisalhamento (Figura 1) evidenciaram o comportamento *shear-thinning* dos materiais. Avaliando o índice de fluxo (n) (Tabela 2), apenas a resina SDR não apresentou diferença significativa entre as temperaturas; já TNF apresentou diferença significativa entre todas as temperaturas.

Com relação ao parâmetro índice de consistência (k) (Tabela 3), para todos os materiais testados ocorreu redução no k com o aumento da temperatura. Verificou-se que à 24°C o TNF apresentou o maior k e TNC o menor, a 37°C, 54°C e 68°C a resina XTR apresentou o maior k . Já a SDR mostrou o menor k à 68°C.

Com o aumento da temperatura houve redução no valor de cedência (Tabela 3), ou seja, o material fluiu mais facilmente com o aquecimento. Apenas a resina SDR não apresentou diferença significativa entre as temperaturas. Os demais materiais apresentaram diferença significativa entre 24°C e as outras temperaturas, ou seja, à partir de 37°C o material fluiu com menos resistência.

Observou-se também que em geral a área de histerese (Tabela 2) diminuiu com o aumento da temperatura. A 24 °C a área de histerese é significativamente maior para todas das resinas *bulk-fill* testadas. Isto se deve provavelmente pelo sistema estar mais estruturado

à 24 °C. Apenas o grupo controle (TNC) apresentou área de histerese menor à 24 °C, com diferença significativa entre às demais temperaturas e também diferença entre 54 °C e 68 °C.

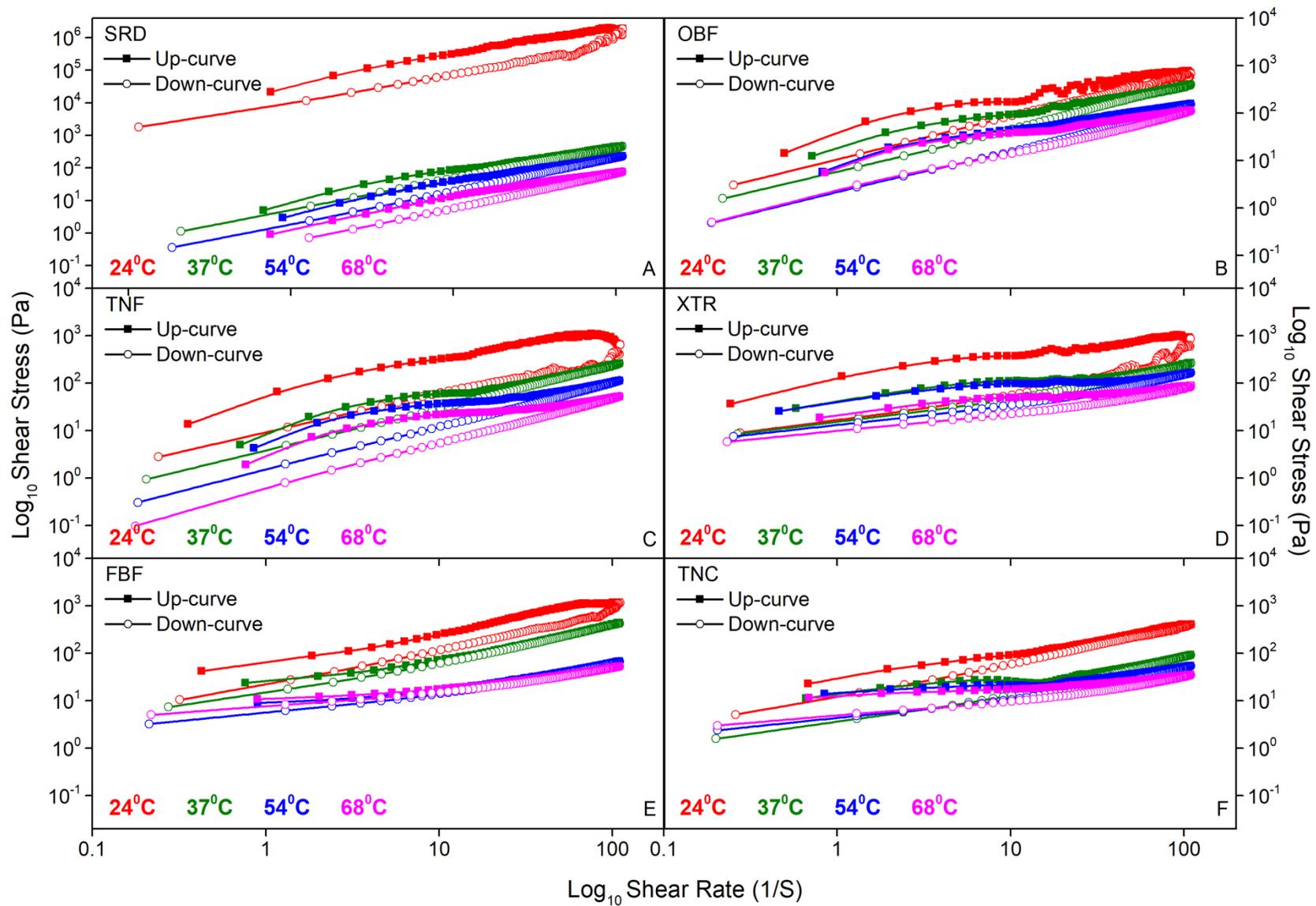


Figura 3 – Gráficos dos reogramas de tensão por gradiente de cisalhamento nas temperaturas de 24, 37, 54 e 68°C

Tabela 2 - Efeito das diferentes temperaturas nos valores de índice de fluxo (n) e na área de histerese em Pa/s das diferentes resinas compostas.

Material	n ± s				Área de histerese			
	24°C	37°C	54°C	68 C	24°C	37°C	54°C	68°C
FBF	0.66±0.08 ^A	0.83±0.00 ^B	0.66±0.0 ^A	0.52±0.01 ^C	38,695.00±9,708.57 ^A	533.00±75.94 ^B	228.70±11,17 ^B	181.26±16.45 ^B
SDR	0.91±0.08 ^A	0.86±0.09 ^A	0.86±0.015 ^A	0.84±0.03 ^A	493.00±104.00 ^A	3,31±0,32 ^B	2,08±0,31 ^B	1.13±0.02 ^B
TNF	0.42±0.02 ^A	0.80±0.01 ^B	0.73±0.02 ^C	0.64±0.01 ^D	92.15±38.24 ^A	2.13±0.24 ^B	1.41±0,08 ^B	1,03±0,1 ^B
XTR	0.48±0.11 ^A	0.60±0.04 ^{AB}	0.42±0.04 ^A	0.39±0.67 ^{AC}	71.48±4.73 ^A	3.98±0.10 ^B	2.97±0.38 ^B	1.76±0.23 ^B
OBF	0.64±0.00 ^A	0.73±0.03 ^B	0.71±0.02 ^{BC}	0.66±0.03 ^{AC}	17,800.00±183.84 ^A	3.59±1.63 ^B	2.96±0,25 ^B	2.01±0.51 ^B
TNC	0.71±0.01 ^A	0.73±0.00 ^A	0.56±0.01 ^B	0.41±0.01 ^C	3.56±0.21 ^A	584.76±62.26 ^B	749.33±80.59 ^{BC}	576.13±76.27 ^B

N=3. Médias seguidas da mesma letra comparam as colunas (temperaturas). s = desvio-padrão

Tabela 3. Efeito das diferentes temperaturas no valor de cedência (τ_0), em Pa, e no índice de consistência (k), em (mPa.s)ⁿ das diferentes resinas compostas.

Resina	$\tau_0 \pm s^*$				k $\pm s^*$			
	24°C	37°C	54°C	68°C	24°C	37°C	54°C	68°C
FBF	27.80±6.54 ^A	6.05±0.33 ^B	3.96±1.03 ^B	6.54±0.78 ^B	45.83±5.83 ^A	8.67±0.59 ^B	2.91±0.62 ^B	4.39±0.40 ^B
SDR	4.37±4.29 ^A	4.43±0.50 ^A	1.02±0.17 ^A	0.32±0.16 ^A	26.39±1.17 ^A	9.98±0.95 ^B	4.25±0,80 ^C	1.39±0.16 ^D
TNF	59.35±15.89 ^A	5.05±0.32 ^B	3.18±0.31 ^B	2.57±0.28 ^B	95.89±2.88 ^A	7.49±0.32 ^B	3.48±0,31 ^C	2.48±0.28 ^C
XTR	40.80±9.89 ^A	22.12±9.06 ^B	25.07±1.81 ^{AB}	15.42±2.04 ^B	66.73±11.69 ^A	14.85±6.00 ^B	20.03±1.14 ^B	12.17±1.54 ^B
OBF	20.72±2.98 ^A	8.95±2.95 ^B	3.85±0.18 ^B	4.47±0.70 ^B	34.38±5.13 ^A	12.69±2.83 ^B	5.12±0.15 ^{BC}	4.84±0.59 ^C
TNC	13.14±1.47 ^A	3.75±0.66 ^B	5.50±0.57 ^{BC}	6.78±0.70 ^C	13.98±1.78 ^A	2.76±0.26 ^B	3.57±0.30 ^B	4.81±0.47 ^B

N=3. Médias seguidas da mesma letra comparam as colunas (temperaturas) para a mesma linha (resina). s=desvio-padrão.

6.4.2. *Microdureza*

A Tabela 4 apresenta os resultados de microdureza para os materiais estudados, nas temperaturas de 24°C e 68°C. Apenas o grupo TNC e TNF apresentam diferenças significativamente maior no valor da microdureza na caixa mesial. Apenas os grupos OBF e XTR apresentam diferença significativa com maior valor da microdureza na caixa distal. OBF diminuiu significativamente o valor da microdureza com o aquecimento, principalmente nos últimos milímetros. XTR aumentou significativamente o valor da microdureza com o aquecimento, além de ter sido o material com maior dureza. A Figura 4 representa graficamente os resultados.

6.4.3. *Grau de conversão*

A Tabela 5 apresenta os resultados de grau de conversão para todos os materiais, nas temperaturas de 24°C e 68°C. Os resultados não apresentaram diferença significativa no grau de conversão com o aquecimento. Houve apenas diferença significativa no grau de conversão em profundidade em todos os materiais na caixa mesial entre o 1^o e 4^o milímetro, já na caixa distal não houve diferença significativa entre as profundidades 1^o e 2^o, 3^o e 4^o, 4^o e 5^o. A Figura 5 representa graficamente os resultados.

Tabela 4 – Média (desvio-padrão) dos valores de microdureza (kg/mm²) de restaurações mésio-ocluso-distais, em profundidade (mm).

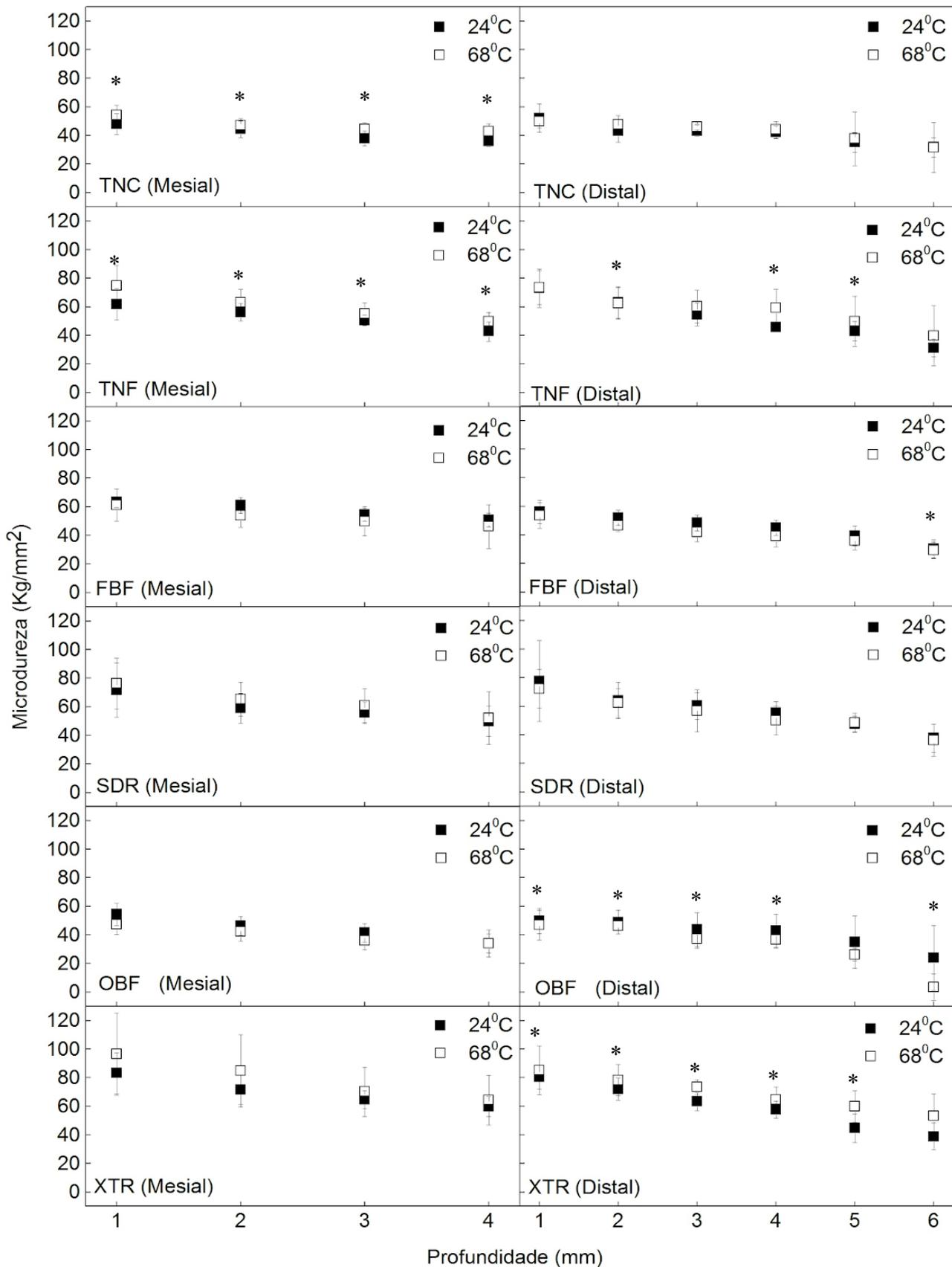
	TNC		TNF		FBF		SDR		OBF		XTR		
	24 ^o C	68 ^o C	24 ^o C	68 ^o C	24 ^o C	68 ^o C	24 ^o C	68 ^o C	24 ^o C	68 ^o C	24 ^o C	68 ^o C	
Mesial	1	47.7 (7.3) ^{Aa}	54.3 (6.9) ^{Ab}	61.8 (11.1) ^{Aa}	74.8 (14.2) ^{Ab}	63.3 (3.8) ^{Aa}	61.3 (11.1) ^{Aa}	71.7 (19.1) ^{Aa}	76.3 (17.9) ^{Aa}	54.4 (7.7) ^{Aa}	47.4 (7.2) ^{Aa}	83.2 (14.4) ^{Aa}	96.6 (28.8) ^{Aa}
	2	44.3 (5.9) ^{Ba}	46.9 (4.9) ^{Bb}	56.2 (6.1) ^{Ba}	63.2 (8.9) ^{Bb}	61.1 (5.6) ^{Ba}	53.9 (8.4) ^{Ba}	59.0 (10.5) ^{Ba}	65.5 (11.8) ^{Ba}	46.3 (6.7) ^{Ba}	42.3 (6.6) ^{Ba}	71.5 (10.3) ^{Ba}	84.9 (25.2) ^{Ba}
	3	37.9 (5.2) ^{Ca}	44.4 (4.6) ^{Cb}	50.5 (3.6) ^{Ca}	55.1 (7.6) ^{Cb}	54.6 (4.5) ^{Ca}	49.9 (10.3) ^{Ca}	55.8 (7.6) ^{Ca}	60.8 (11.9) ^{Ca}	41.5 (6.5) ^{Ca}	35.8 (6.2) ^{Ca}	64.6 (6.4) ^{Ca}	70.3 (17.2) ^{Ca}
	4	35.9 (3.9) ^{Da}	42.9 (5.4) ^{Db}	42.8 (6.9) ^{Da}	49.8 (6.5) ^{Db}	50.7 (4.7) ^{Da}	46.2 (15.3) ^{Da}	49.8 (10.6) ^{Da}	52.2 (18.4) ^{Da}	34.2 (6.6) ^{Da}	34.0 (9.5) ^{Da}	59.9 (7.1) ^{Da}	64.4 (17.4) ^{Da}
Distal	1	52.2 (10.1) ^{Aa}	49.9 (4.3) ^{Aa}	73.1 (13.3) ^{Aa}	73.5 (12.1) ^{Aa}	56.2 (8.3) ^{Aa}	53.7 (9.1) ^{Aa}	77.6 (28.3) ^{Aa}	72.3 (13.4) ^{Aa}	49.8 (8.8) ^{Aa}	47.0 (10.4) ^{Ab}	80,5 (8.3) ^{Aa}	85,2 (17.0) ^{Ab}
	2	43.2 (8.1) ^{Ba}	47.6 (6.3) ^{Ba}	63.2 (11.1) ^{Ba}	62.5 (11.1) ^{Bb}	52.0 (5.3) ^{Ba}	46.7 (4.1) ^{Ba}	64.2 (12.7) ^{Ba}	62.5 (10.1) ^{Ba}	48.9 (8.3) ^{Ba}	46.3 (5.5) ^{Bb}	72,0 (7.7) ^{Ba}	78,3 (10.9) ^{Bb}
	3	43.2 (3.9) ^{Ca}	46.0 (2.1) ^{Ca}	54.6 (8.1) ^{Ca}	60.3 (11.5) ^{Ca}	48.5 (5.5) ^{Ca}	41.8 (6.4) ^{Ca}	60.4 (9.4) ^{Ca}	56.9 (14.8) ^{Ca}	43.8 (11.8) ^{Ca}	37.3 (6.3) ^{Cb}	63,5 (6.3) ^{Ca}	73,5 (4.9) ^{Cb}
	4	42.1 (4.4) ^{Da}	44.0 (5.9) ^{Ca}	45.6 (2.9) ^{Da}	59.4 (12.9) ^{Cb}	45.0 (5.4) ^{Da}	39.2 (7.3) ^{Ca}	55.4 (8.1) ^{Da}	50.2 (10.0) ^{Ca}	42.8 (11.9) ^{Da}	36.4 (4.8) ^{Cb}	57,8 (6.0) ^{Da}	64,7 (8.9) ^{Cb}
	5	35.2 (6.8) ^{Ea}	37.7 (18.9) ^{Da}	43.0 (6.8) ^{Ea}	49.9 (17.5) ^{Db}	39.5 (7.0) ^{Ea}	35.9 (6.3) ^{Da}	47.7 (5.7) ^{Ea}	48.6 (6.5) ^{Da}	34.9 (18.4) ^{Ea}	26.2 (4.6) ^{Da}	44,7 (9.9) ^{Ea}	60,1 (10.9) ^{Db}
	6	31.7 (6.9) ^{Fa}	31.6 (17.6) ^{Ea}	31.3 (6.1) ^{Fa}	39.8 (21.0) ^{Ea}	30.2 (6.4) ^{Fa}	29.1 (5.7) ^{Eb}	37.7 (10.0) ^{Fa}	36.4 (11.2) ^{Ea}	23.9 (22.8) ^{Fa}	3.5 (9.2) ^{Eb}	38,9 (9.3) ^{Fa}	53,1 (15.7) ^{Ea}

As letras minúsculas representam diferença significativa entre as colunas (temperatura) e as maiúsculas representam diferença entre as linhas (profundidade), p<0,05.

Tabela 5 - Média (desvio-padrão) dos valores do grau de conversão (em %), nas caixas mesial e distal, em diferentes profundidades (mm).

	TNC		TNF		FBF		SDR		OBF		XTR		
	24 ⁰ C	68 ⁰ C	24 ⁰ C	68 ⁰ C	24 ⁰ C	68 ⁰ C	24 ⁰ C	68 ⁰ C	24 ⁰ C	68 ⁰ C	24 ⁰ C	68 ⁰ C	
Mesial	1	62.0 (2.0) ^{Aa}	55.0 (6.0) ^{Aa}	62.0 (12.7) ^{Aa}	56.0 (7.1) ^{Aa}	55.0 (4.3) ^{Aa}	51.0 (5.6) ^{Aa}	62.0 (5.6) ^{Aa}	63.0 (3.2) ^{Aa}	72.0 (3.9) ^{Aa}	71.0 (3.6) ^{Aa}	53.0 (5.2) ^{Aa}	54.0 (5.7) ^{Aa}
	2	57.0 (5.3) ^{Ba}	53.0 (5.0) ^{Ba}	55.0 (11.8) ^{Ba}	53.0 (6.4) ^{Ba}	51.0 (6.3) ^{Ba}	53.0 (4.7) ^{Ba}	56.0 (8.0) ^{Ba}	62.0 (9.2) ^{Ba}	69.0 (4.5) ^{Ba}	70.0 (3.9) ^{Ba}	52.0 (6.8) ^{Ba}	56.0 (6.7) ^{Ba}
	3	55.0 (5.6) ^{Ba}	52.0 (6.8) ^{Ba}	56.0 (12.5) ^{Ba}	50.0 (11.7) ^{Ba}	54.0 (7.0) ^{ABa}	56.0 (8.2) ^{Ba}	55.0 (12.0) ^{Ba}	54.0 (9.4) ^{Ba}	61.0 (8.6) ^{Ba}	62.0 (9.2) ^{Ba}	56.0 (7.4) ^{Ba}	56.0 (7.4) ^{Ba}
	4	41.0 (24.3) ^{Ca}	52.0 (17.1) ^{Ca}	42.0 (15.0) ^{Ca}	47.0 (10.7) ^{Ca}	47.0 (8.6) ^{Ca}	49.0 (7.7) ^{Ca}	50.0 (14.0) ^{Ca}	56.0 (5.7) ^{Ca}	47.0 (15.0) ^{Ca}	62.0 (12.3) ^{Ca}	52.0 (13.3) ^{Ca}	53.0 (7.6) ^{Ca}
Distal	1	59.0 (7.8) ^{Aa}	60.0 (8.8) ^{Aa}	54.0 (13.2) ^{Aa}	57.0 (10.0) ^{Aa}	56.0 (4.5) ^{Aa}	53.0 (11.8) ^{Aa}	63.0 (6.2) ^{Aa}	62.0 (5.8) ^{Aa}	71.0 (11.3) ^{Aa}	75.0 (4.1) ^{Aa}	56.0 (6.2) ^{Aa}	61.0 (10.7) ^{Aa}
	2	56.0 (7.8) ^{Aa}	54.0 (7.6) ^{Aa}	58.0 (6.8) ^{Aa}	56.0 (4.7) ^{Aa}	57.0 (0.5) ^{Aa}	51.0 (10.0) ^{Aa}	61.0 (6.0) ^{Aa}	55.0 (10.4) ^{Aa}	59.0 (23.9) ^{Aa}	73.0 (2.9) ^{Aa}	55.0 (3.6) ^{Aa}	61.0 (5.5) ^{Aa}
	3	55.0 (13.3) ^{Ba}	56.0 (3.9) ^{Ba}	41.0 (22.4) ^{Ba}	49.0 (13.5) ^{Ba}	55.0 (3.7) ^{Ba}	44.0 (6.8) ^{Ba}	54.0 (5.6) ^{Ba}	54.0 (8.1) ^{Ba}	59.0 (16.1) ^{Ba}	68.0 (11.0) ^{Ba}	54.0 (4.8) ^{Ba}	53.0 (6.0) ^{Ba}
	4	58.0 (5.4) ^{BCa}	56.0 (6.0) ^{BCa}	49.0 (16.2) ^{BCa}	45.0 (16.0) ^{BCa}	39.0 (21.3) ^{BCa}	38.0 (13.4) ^{BCa}	55.0 (21.4) ^{BCa}	50.0 (13.0) ^{BCa}	52.0 (25.7) ^{BCa}	68.0 (6.6) ^{BCa}	52.0 (5.6) ^{BCa}	54.0 (8.3) ^{BCa}
	5	52.0 (13.6) ^{Ca}	51.0 (6.9) ^{Ca}	42.0 (19.4) ^{Ca}	52.0 (10.9) ^{Ca}	39.0 (9.0) ^{Ca}	48.0 (8.8) ^{Ca}	51.0 (12.2) ^{Ca}	49.0 (17.0) ^{Ca}	42.0 (29.5) ^{Ca}	64.0 (2.3) ^{Ca}	43.0 (3.7) ^{Ca}	50.0 (4.8) ^{Ca}
	6	39.0 (15.9) ^{Da}	46.0 (18.4) ^{Da}	43.0 (17.8) ^{Da}	36.0 (8.6) ^{Da}	30.0 (10.9) ^{Da}	35.0 (9.5) ^{Da}	42.0 (15.1) ^{Da}	47.0 (19.1) ^{Da}	31.0 (30.7) ^{Da}	39.0 (6.5) ^{Da}	46.0 (11.3) ^{Da}	48.0 (7.2) ^{Da}

* Letras minúsculas representam diferença significativa entre as colunas (temperatura) e maiúsculas representam diferenças entre as linhas (profundidade)



* diferença significativa entre as temperaturas 24°C e 68°C.

Figura 4 - Apresentação gráfica dos valores encontrados na análise de microdureza Knoop (kg/mm²) de resinas *bulk-fill flow*, em diferentes profundidades (mm).

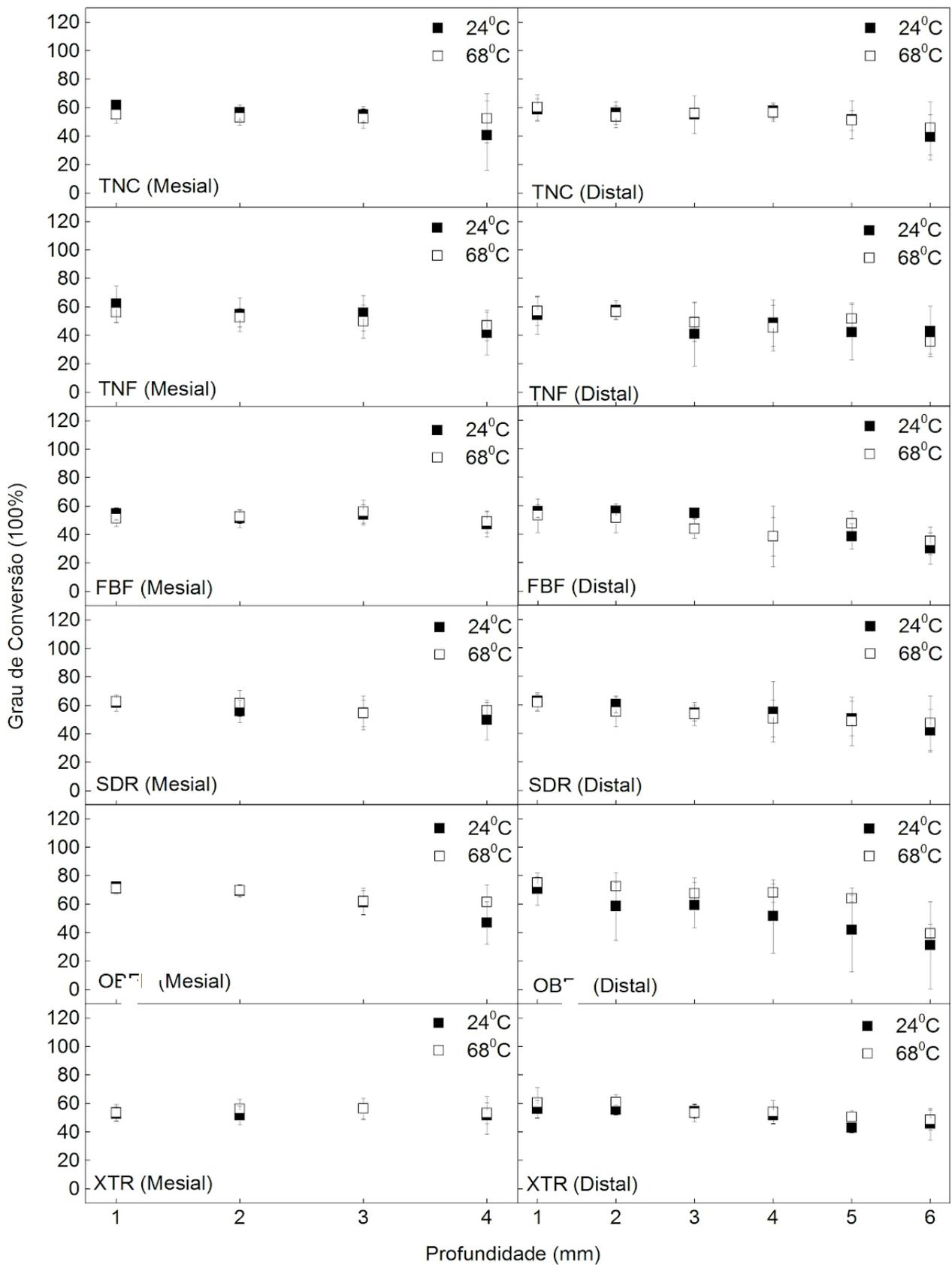


Figura 5 - Apresentação gráfica das variações encontradas na análise do grau de conversão (%) de resinas *bulk-fill flow*, em diferentes profundidades (mm).

6.5. Discussão

A investigação das propriedades reológicas de resinas compostas ajuda a compreender melhor os diferentes comportamentos de fluxo durante o seu manuseio. (36) As resinas testadas apresentaram certa resistência para serem removidas das seringas e maior rigidez à temperatura ambiente; porém, com o aumento da temperatura, tornaram-se menos viscosas e mais fáceis de serem acomodadas ao preparo cavitário, o que pode melhorar a adaptação marginal como já demonstrado em outros estudos. (37,38) O aumento da temperatura promoveu maior liberdade de movimentação dos monômeros da matriz polimérica da resina devido às forças vibracionais térmicas. (37) Além disso, somada à característica de fluxo *shear thinning*, o valor de cedência e a linearização dos gráficos de tensão por taxa de cisalhamento, pode-se classificar as resinas *bulk-fill flow* em materiais pseudoplásticos, com valor de cedência ou plástico de *Bingham*, o que indica que as resinas analisadas necessitam atingir um limiar de tensão para começar a extrair da seringa, assim como outros materiais já testados. (39) Este modo de fluxo também foi observado em trabalhos anteriores. (25,40,41)

O valor de cedência diminuiu com o aumento da temperatura, evidenciando diferença significativa entre a temperatura de 24 °C e as demais ($p < 0.05$), como observa-se na Tabela 3. Desta forma, pode-se inferir que, na prática, a 37 °C as resinas compostas já apresentam um manuseio melhor, o que também já foi demonstrado em um outro trabalho recente. (42) Comparando as temperaturas de 37, 54 e 68 °C, observou-se que de maneira geral não há diferença significativa ($p > 0.05$) entre as temperaturas mais altas (54 e 68 °C), ou seja, aquecer o material acima de 54 °C não denota alterações significativas na resistência ao fluxo. Corroborando com os resultados encontrados para o índice de consistência (k) a 24 °C, a resina TNF apresentou o maior valor de cedência, sendo o material com maior resistência a fluir, provavelmente devido ao alto volume de carga (68.2%) e por apresentar em sua composição BisGMA, BisEMA e UDMA, monômeros de alto peso molecular. (19) Por outro lado, a SDR, com 68% de carga, EBPADMA e TEGDMA (monômeros de peso molecular mais baixo) e UDMA modificado na sua composição (19,43) apresentou um menor valor de k , fluindo mais facilmente. Desta forma, foi possível observar que a composição do material influencia o índice de consistência de um material resinoso, como observado por outros autores (25,41). A interação entre as partículas de carga e a matriz resinosa são fundamentais para as propriedades viscoelásticas das resinas compostas. (44)

Durante a análise reológica de fluxo contínuo, as amostras são submetidas a um gradiente de cisalhamento de zero até um valor máximo, resultando em um valor extremo de

tensão de cisalhamento configurando a curva ascendente. Quando este processo é reduzido, de modo gradual, até zero, obtém-se a curva descendente, possibilitando a determinação da área de histerese, um processo tempo-dependente. (39,45,46) Analisando este parâmetro para as temperaturas, nota-se que a restauração da configuração molecular relaxada resultou em uma viscosidade aparente menor após a remoção da tensão de cisalhamento, indicando que as resinas apresentam tixotropia. (27,40,47,48) Esta característica pode indicar maior flexibilidade e resistência à quebra, influenciando diretamente em melhores características mecânicas do material. À 24 °C, a área de histerese para todas as amostras é maior em relação às demais temperaturas, com exceção da TNC. Isso se deve provavelmente pelo sistema estar mais estruturado nesta temperatura. A resina TNC apresentou área de histerese bem menor à 24 °C e houve aumento da área com o aumento da temperatura. Isso pode ser explicado pelo fato de a resina apresentar um modificador reológico que desconhecemos, mas que segundo o fabricante (Ivoclar Vivadent, AG, Schaan, Liechtenstein) faz com que haja um deslocamento linear da estrutura polimérica quando é aplicada uma força sobre ela. O tempo de recuperação da resina composta pode variar conforme o tamanho da carga e o próprio tipo de carga, estando a viscosidade do material estritamente relacionada com o tempo necessário para o relaxamento das cadeias poliméricas. (49) Portanto, a hipótese nula 1 foi rejeitada, pois o pré-aquecimento influenciou o comportamento de fluxo e a viscoelasticidade das resinas testadas.

Um dos parâmetros mais importantes que influencia a propriedades mecânicas e físicas das resina é o processo de polimerização. (50) A polimerização das resinas depende de uma série de fatores como: composição do material (fotoiniciadores, tamanho e quantidade de partícula de carga, tipo de matriz orgânica), fonte de luz fotoativadora, condições de fotoativação e espessura do incremento. (51) Na prática clínica, muitas vezes nos deparamos com cavidades complexas e extensas nos dentes posteriores, o que demanda mais tempo durante o processo restaurador. Com o desenvolvimento das resinas *bulk-fill* esta etapa pode ser acelerada com a inserção de incrementos maiores (4 mm). De acordo com os nossos achados, a resina TNC, inserida de acordo com as orientações do fabricante (2 mm de espessura e 20 s de ativação) apresentou os valores mais baixos de microdureza. Esta resina tem a menor quantidade de carga (63,8%) dentre os materiais testados. Sabe-se que a quantidade de carga dentro da matriz resinosa pode ser um obstáculo no processo de polimerização da matriz resinosa. (52) Em contrapartida, observamos um aumento significativo na microdureza com o pré-aquecimento deste material.

Trabalhos recentes mostram que o pré-aquecimento de resinas *bulk-fill* promove aumento da microdureza dos materiais (22,23) No presente trabalho, observamos aumento significativo da microdureza apenas para as resinas TNC, TNF e XTR. As resinas TNC e TNF apresentam alta translucidez e a presença de um fotoiniciador mais reativo (Ivocerim), além de partículas de carga pequenas que variam de 0.1 μm à 30 μm . Essas características favorecem a passagem e difusão da luz na matriz resinosa, o que ajuda na fotoativação dos materiais. Já a resina XTR apresenta a maior porcentagem de carga (75%) entre os materiais testados. Entretanto, para a resina OBF houve redução significativa da microdureza, principalmente nas camadas mais profundas. Os diferentes resultados podem ter ocorrido em função da quantidade, tipo e tamanho das partículas de carga do material, pois quanto maior a quantidade de carga de uma resina composta, maior a microdureza. (53) Por outro lado, uma maior quantidade de carga pode reduzir a dispersão da luz através do material, dificultando a conversão dos monômeros em polímeros. (54) Assim, a composição do material certamente influenciou nos resultados encontrados e a hipótese nula 2 foi aceita parcialmente, pois o pré-aquecimento alterou positiva e significativamente a microdureza de 3 resinas *bulk-fill*.

O pré-aquecimento provavelmente promove maior mobilidade dos monômeros devido à energia térmica, tornando a resina mais fluida e promovendo maior mobilidade das cadeias poliméricas. (16) Com isso, esperava-se que o grau de conversão aumentasse e a microdureza que representa indiretamente esta propriedade, também. No entanto, com o presente trabalho não podemos fazer esta afirmação pois o pré-aquecimento dos materiais não promoveu aumento no grau de conversão para nenhuma resina. Podemos hipotetizar que possivelmente a resina tenha resfriado antes mesmo de ser fotoativada, em função do contato do material com as paredes cavitárias ou mesmo pelo tempo despendido entre a remoção da seringa do dispositivo de aquecimento até o procedimento restaurador. Todos os procedimentos restauradores foram realizados pelo mesmo operador (treinado e com experiência clínica) e o tempo entre a remoção da resina do aquecedor até a ativação com a luz foi de até 40s, como preconizado na literatura. (22) Alguns estudos demonstraram que a temperatura da resina reduz rapidamente depois de removida do aquecedor. (2,22,55,56) De acordo com Daronch et al. (57), quando a resina é aquecida a 60°C e removida da fonte de calor, sua temperatura cai 50% após 2min e 90% após 5min. Neste presente trabalho o procedimento restaurador foi realizado em temperatura ambiente controlada a 23°C com aparelho ar-condicionado, e isso pode ter contribuído para o rápido resfriamento do material não alcançando resultados significativamente maiores para os valores de grau de conversão. A temperatura intra-oral e a umidade relativa são maiores que a temperatura ambiente o que poderia favorecer a

manutenção da temperatura da resina até a fotoativação. (58) Um outro fator que pode justificar os resultados é a temperatura do Calset. O aparelho utilizado foi adquirido para o desenvolvimento desta pesquisa, mas trabalhos futuros poderiam avaliar se, de fato, a temperatura estipulada pelos fabricantes é de fato alcançada. Yang et al. (42), relata que "a resina se apresentou mais fria do que a temperatura do aquecedor Caps Warmer" (VOCO, Germany), o que pode ter acontecido no nosso estudo. Portanto, a hipótese nula 3 foi aceita, pois o pré-aquecimento das resinas *bulk-fill* não alterou o grau de conversão dos materiais.

Os primeiros milímetros apresentaram os maiores valores de microdureza e grau de conversão e houve redução significativa desses valores com o aumento da profundidade, porém sem aumento significativo dos valores para os grupos pré-aquecidos. Estes resultados estão de acordo com outros estudos. (23,59) Isto pode acontecer pois a luz é dispersa pelas partículas de carga e absorvida pelos fotoiniciadores e pigmentos, o que reduz a penetração de luz. (10,60,61) Logo, a hipótese nula 4 foi aceita, pois o pré-aquecimento de resinas *bulk-fill* de consistência *flow* não aumenta a profundidade de polimerização.

Estudos laboratoriais diferem da realidade clínica e têm suas limitações, porém são os precursores de possíveis estudos clínicos subsequentes, pois avaliam se será viável aplicar a metodologia *in vivo*. Considerando a metodologia adotada no presente trabalho que possibilite comparações com os resultados da literatura verifica-se que há poucos estudos que avaliaram as propriedades viscoelásticas das resinas *bulk-fill*. (25,26) Para o teste da microdureza adotou-se a dureza Knoop após 30 dias, pois alguns estudos mostraram que a microdureza aumenta em até 30 dias. (62,63) A avaliação do grau de conversão foi realizada após 7 dias de confecção dos espécimes e armazenagem a 37°C, já que a maior parte da reação de polimerização acontece nos primeiros minutos (64) ou nas primeiras 24 horas. (65) Na tentativa de criar uma padronização da profundidade de penetração da luz, as pontas de cúspide dos dentes foram removidas. Clinicamente, sabemos que na maioria das vezes a luz não é colocada perpendicularmente ou diretamente em contato com superfície do material por questão de dificuldade de acesso de algumas pontas mais curvas e biossegurança. Um auxiliar que efetua a fotoativação mais desatento, bem como a presença de uma cavidade mais profunda, dentes mal posicionados, diferenças de morfologia das fóssulas e cúspides, são outros fatores que podem prejudicar a eficácia da polimerização. Assim, a confecção de caixas mais amplas para cervical na parte distal justifica-se por simular situações como essas. Ainda, optou-se por utilizar resinas *flow* pois elas apresentam maior capacidade de adaptação às paredes do preparo e facilidade de inserção, principalmente nos casos de cavidades complexas e mais extensas em dentes posteriores.

Com o pré-aquecimento da resina *bulk-fill flow* pode-se observar que a viscosidade diminui muito, tornando o material bem fluido e difícil de manter-se estável no interior da cavidade. Devido à maior fluidez, durante a etapa restauradora observamos que quando o material é inserido na cavidade há uma tendência de escoamento para a distal, principalmente em dentes superiores em função da posição supina da cabeça. Nessas condições, é imprescindível a indicação de um bom conjunto de matriz, cunha e anel, muito bem adaptados à parede gengival da caixa proximal do preparo, para o material não fluir além da cavidade. Assim, para atingir bons resultados é necessário que o profissional tenha agilidade e treinamento para que o material não perca calor até a fotoativação. Também é necessário muito cuidado para evitar a inclusão de bolhas e formação de *gaps*, o que compromete o desempenho e sucesso da restauração. Atualmente, as taxas de sucesso obtidas com resinas compostas são altas. (66) Neste contexto, com base nos resultados do nosso trabalho, é precoce afirmar que devemos adicionar uma nova etapa como o pré-aquecimento de resinas *bulk-fill flow* no procedimento restaurador. Mais estudos sobre a reologia de outros materiais como as resinas *Bulk-fill* de consistência regular, análise da adaptação marginal em microCT e ensaios clínicos deveriam ser conduzidos para confirmar a indicação do pré-aquecimento das resinas *bulk-fill* nas mesmas condições.

6.6. Conclusão

O pré-aquecimento de resinas *bulk-fill* reduz a viscosidade, melhora a microdureza para alguns materiais (TNC, TNF, XTR) e não influencia o grau de conversão em todas as profundidades da restauração.

6.7. REFERENCIAS

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ANEXO A

TERMO DE CONSENTIMENTO LIVRE E ESCLARECIDO

Gostaríamos de convidá-lo a participar da pesquisa intitulada **Avaliação da influência do pré-aquecimento de resinas nas propriedades reológicas e de fotoativação**, que faz parte do curso de pós graduação em odontologia, nível de doutorado e é orientada pelo prof Dra. Raquel Sano Suga Terada da Universidade Estadual de Maringá. O objetivo da pesquisa é avaliar a influência do pré-aquecimento de resinas compostas bulk-fill nas propriedades reológicas, físicas e de adesividade. A adesividade será avaliada à partir da união do material restaurado Bulk-fill e dente humano que é conseguido através dos adesivos dentários, que são materiais que desenvolvem um mecanismo de união físico/química entre a resina composta e o dente, por meio da permeação nos espaços existente na superfície do dente. Os testes serão realizados em laboratório (in vitro) com a utilização de dentes humanos extraídos que não apresentem a lesão cariosa, após a aprovação do projeto pelo Comitê de Ética em Pesquisa.

Os resultados obtidos servirão como base de dados para a realização, publicação e apresentação de trabalhos científicos. É direito do participante o esclarecimento de qualquer dúvida relacionada à realização da pesquisa. A participação será isenta de qualquer gasto ou recompensa, sendo totalmente voluntária. Além disso, o participante terá a liberdade de retirar seu consentimento a qualquer momento e deixar de participar do estudo.

Para isto a sua participação é muito importante, e ela se daria da seguinte forma: Após a exodontia dos seus dentes indicados para extração por motivos periodontais, ortodônticos e/ou retenção dental, você assinará um termo de cessão desses dentes, estando ciente de que eles serão utilizados com a finalidade de realização de pesquisas laboratoriais. Quando desejar, você poderá requisitar novamente os dentes que foram cedidos, mesmo que estejam restaurados ou seccionados.

Informamos que após o procedimento de extração existe a possibilidade de ocorrência de alguns riscos como hemorragia, dor e inchaço, e é de extrema importância que você siga todas as orientações e recomendações após o

procedimento de extração a fim de minimizar estes riscos. Entretanto os benefícios compensam os riscos, pois complicações futuras poderiam surgir do dente não extraído. Gostaríamos de esclarecer que sua participação é totalmente voluntária, podendo você: recusar-se a participar, ou mesmo desistir a qualquer momento sem que isto acarrete qualquer ônus ou prejuízo à sua pessoa. Informamos ainda que as informações serão utilizadas somente para os fins desta pesquisa, e serão tratadas com o mais absoluto sigilo e confidencialidade, de modo a preservar a sua identidade. Após a utilização do dente cedido, este material biológico será descartado da mesma forma como seria após sua extração, ou seja, em lixo hospitalar de material contaminado.

Os benefícios serão indiretos, e espera-se que com os resultados da pesquisa possamos estabelecer padrões de temperatura de armazenamento e protocolos de restaurações de resinas odontológicas que aumentem a longevidade clínica de restaurações dentárias. Caso você tenha mais dúvidas ou necessite maiores esclarecimentos, pode nos contatar nos endereços abaixo ou procurar o Comitê de Ética em Pesquisa da UEM, cujo endereço consta deste documento. Este termo deverá ser preenchido em duas vias de igual teor, sendo uma delas, devidamente preenchida e assinada entregue a você.

Além da assinatura nos campos específicos pelo pesquisador e por você, solicitamos que sejam rubricadas todas as folhas deste documento. Isto deve ser feito por ambos (pelo pesquisador e por você, como sujeito ou responsável pelo sujeito de pesquisa) de tal forma a garantir o acesso ao documento completo.

Eu,.....(nome por extenso do sujeito de pesquisa) declaro que fui devidamente esclarecido e concordo em participar VOLUNTARIAMENTE da pesquisa coordenada pela Prof^a Dra. Raquel Sano Suga Terada, aceitando ceder _____ dente(s) permanente(s) para o Departamento de Odontologia da UEM.

No caso de menor de idade, eu,(nome por extenso do responsável pelo sujeito de pesquisa), responsável pelo menor _____ declaro que fui

devidamente esclarecido e concordo em que ele (a) participe VOLUNTARIAMENTE da pesquisa coordenada pela Prof^a Dra. Raquel Sano Suga Terada , aceitando ceder _____ dente(s) permanente(s) para o Departamento de Odontologia da UEM.

Estou ciente de que este(s) dente(s) foi (foram) extraído(s) por indicação terapêutica, como documentado em meu prontuário e de que o(s) mesmo(s) será(ão) utilizado(s) para pesquisa.

_____ Data:.....
Assinatura ou impressão datiloscópica

Eu,.....(nome do pesquisador ou do membro da equipe que aplicou o TCLE), declaro que forneci todas as informações referentes ao projeto de pesquisa supra-nominado.

_____ Data:.....
Assinatura do pesquisador

Qualquer dúvida com relação à pesquisa poderá ser esclarecida com o pesquisador, conforme o endereço abaixo:

Nome: Larissa Coelho Pires Lopes e Raquel Sano Suga Terada

Endereço: Av. Mandacarú, 1550, Maringá,PR (telefone/e-mail): 3011-9051

E-mail: larissapires.uem@gmail.com

Qualquer dúvida com relação aos aspectos éticos da pesquisa poderá ser esclarecida com o Comitê Permanente de Ética em Pesquisa (COPEP) envolvendo Seres Humanos da UEM, no endereço abaixo:

COPEP/UEM

Universidade Estadual de Maringá.

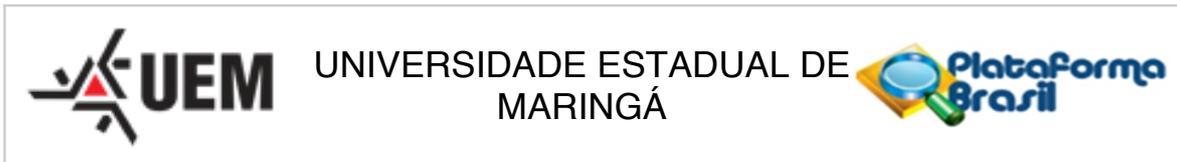
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E-mail:

copep@uem.br

ANEXO B



PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Avaliação da influência do pré-aquecimento de resinas nas propriedades reológicas e de fotoativação

Pesquisador: Raquel Sano Suga Terada

Área Temática:

Versão: 1

CAAE: 87404918.6.0000.0104

Instituição Proponente: Universidade Estadual de Maringá

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 2.648.664

Apresentação do Projeto:

Trata-se de projeto de pesquisa proposto por pesquisador vinculado à Universidade Estadual de Maringá.

Objetivo da Pesquisa:

Avaliar a influência do pré-aquecimento de resinas compostas bulk-fill nas propriedades reológicas e físicas. Objetivos secundários: 1. Avaliar as características viscoelásticas de resinas bulk-fill; 2. Avaliar a microdureza de resinas bulk-fill inseridas em diferentes temperaturas de pré-aquecimento; 3. Avaliar o grau de conversão em profundidade em função da variação da temperatura de pré-aquecimento de resinas bulk-fill.

Avaliação dos Riscos e Benefícios:

Avalia-se que os possíveis riscos a que estarão submetidos os sujeitos da pesquisa serão suportados pelos benefícios apontados.

Comentários e Considerações sobre a Pesquisa:

Trata-se de projeto de pesquisa proposto por pesquisador vinculado ao Programa de Pós-Graduação (doutorado) em Odontologia da Universidade Estadual de Maringá. O estudo pretende avaliar a influência do pré-aquecimento de resinas bulk-fill utilizadas no processo de restaurações dentárias, a partir de uma amostra de 126 terceiros molares livres de cáries. 1. Desenho/Metodologia: Estudo in vitro para avaliar a influência do pré-aquecimento nas

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UNIVERSIDADE ESTADUAL DE
MARINGÁ



PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Avaliação da influência do pré-aquecimento de resinas nas propriedades reológicas e de fotoativação

Pesquisador: Raquel Sano Suga Terada

Área Temática:

Versão: 1

CAAE: 87404918.6.0000.0104

Instituição Proponente: Universidade Estadual de Maringá

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 2.648.664

Apresentação do Projeto:

Trata-se de projeto de pesquisa proposto por pesquisador vinculado à Universidade Estadual de Maringá.

Objetivo da Pesquisa:

Avaliar a influência do pré-aquecimento de resinas compostas bulk-fill nas propriedades reológicas e físicas. Objetivos secundários: 1. Avaliar as características viscoelásticas de resinas bulk-fill; 2. Avaliar a microdureza de resinas bulk-fill inseridas em diferentes temperaturas de pré-aquecimento; 3. Avaliar o grau de conversão em profundidade em função da variação da temperatura de pré-aquecimento de resinas bulk-fill.

Avaliação dos Riscos e Benefícios:

Avalia-se que os possíveis riscos a que estarão submetidos os sujeitos da pesquisa serão suportados pelos benefícios apontados.

Comentários e Considerações sobre a Pesquisa:

Trata-se de projeto de pesquisa proposto por pesquisador vinculado ao Programa de Pós-Graduação (doutorado) em Odontologia da Universidade Estadual de Maringá. O estudo pretende avaliar a influência do pré-aquecimento de resinas bulk-fill utilizadas no processo de restaurações dentárias, a partir de uma amostra de 126 terceiros molares livres de cáries. 1. Desenho/Metodologia: Estudo in vitro para avaliar a influência do pré-aquecimento nas

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Continuação do Parecer: 2.648.664

Tipo Documento	Arquivo	Postagem	Autor	Situação
Informações Básicas do Projeto	PB_INFORMAÇÕES_BÁSICAS_DO_PROJETO_1084065.pdf	10/04/2018 09:28:18		Aceito
Outros	autorizacao.pdf	10/04/2018 09:27:32	LARISSA COELHO PIRES LOPES	Aceito
TCLE / Termos de Assentimento / Justificativa de Ausência	TCLE.pdf	27/03/2018 09:38:39	LARISSA COELHO PIRES LOPES	Aceito
Folha de Rosto	folhaderosto.pdf	14/03/2018 15:51:10	LARISSA COELHO PIRES LOPES	Aceito
Projeto Detalhado / Brochura Investigador	ProjetoDoutorado_LarissaPires.pdf	14/03/2018 15:50:23	LARISSA COELHO PIRES LOPES	Aceito

Situação do Parecer:

Aprovado

Necessita Apreciação da CONEP:

Não

MARINGÁ, 10 de Maio de 2018

Assinado por:
Ricardo Cesar Gardiolo
(Coordenador)

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