

**Universidade Federal de Pelotas**  
**Faculdade de Odontologia**  
**Programa de Pós-Graduação em Odontologia**



**Tese**

**Resina composta restauradora pré-aquecida como agente de cimentação  
adesiva de restaurações indiretas**

**Rogério Luiz Marcondes**

Pelotas, 2021

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**Resina composta restauradora pré-aquecida como agente de cimentação  
adesiva de restaurações indiretas**

Tese apresentada ao Programa de Pós-Graduação em Odontologia da Faculdade de Odontologia da Universidade Federal de Pelotas, como requisito parcial à obtenção do título de Doutor em Odontologia, área de concentração Biomateriais e Biologia Oral, ênfase Materiais Odontológicos.

Orientador: Prof. Dr. Rafael Ratto de Moraes

Co-orientador: Prof. Dr. Marco Aurélio de Carvalho

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Data da defesa: 01/10/2021

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## **DEDICATÓRIA**

À minha querida esposa, Carolina dos Santos Marcondes, o grande amor da  
minha vida e meu porto seguro.

Aos meus dois filhos, Guilherme e Gabriel, pessoas que me fazem aprender e  
crescer dia a dia e enriquecem minha vida intensamente, com eles aprendi a amar  
incondicionalmente.

À memória de meu irmão, Ricardo Kochinski Marcondes, um exemplo fraterno de  
inteligência e tranquilidade. Meu irmão, saudades eternas!

Ao meu pai, Genilton Luiz Marcondes, um pai presente e dedicado a vida dos filhos  
À minha mãe, Anísia Kochinski Marcondes, pessoa que me inspira e acima de tudo,  
sempre MÃE.

## **Agradecimentos**

À Universidade Federal de Pelotas e ao Programa de Pós-Graduação em Odontologia (PPGO) pela oportunidade de aprimorar minha formação.

Ao meu orientador, Prof. Dr. Rafael Ratto de Moraes, pela incansável dedicação. Muito obrigado mestre, sua ajuda tornou simples até as fases mais complexas. E mesmo quando eu estava desistindo, me deu força para seguir em frente e, só por isso, foi possível a realização deste trabalho.

Ao meu co-orientador, Prof. Dr. Marco Aurélio de Carvalho, pela oportunidade de aprender contigo. Um verdadeiro mestre no conhecimento e na simplicidade. Muito obrigado pelas discussões que enriqueceram a elaboração teórica e pela ajuda nas tarefas descritivas deste trabalho. Meu grande amigo e professor., te considero muito!

A Prof<sup>a</sup>. Dr<sup>a</sup>. Giana da Silveira Lima, pela ajuda em diversas disciplinas e por ajudar a instigar em mim a vontade, já adormecida, dentro da didática aplicada à odontologia. Obrigado, mais uma vez, por esta oportunidade de aprender contigo.

Aos demais professores do PPGO, por todos os conhecimentos divididos.

Aos alunos do PPGO, Verônica Lima, Fabíola Barbon, Matheus Kinalski e Peterson Boeira pela ajuda nos ensaios no laboratório, assim como à pós-doutoranda Cristina Isolan.

Aos colegas Dr. Tiago Veras Fernandes, Dr. Adriano Lima, Dr. Fabricio Ogliari, Dra. Aline Ogliari, Dr. Jefferson Ricardo Pereira e Dr. Gregori Boeira pelo apoio e ajuda nessa jornada.

## **Notas Preliminares**

A presente tese foi redigida segundo o Manual de normas UFPel para trabalhos acadêmicos de 2019, adotando o Nível de Descrição em Capítulos não convencionais, descrito no Apêndice C do manual (último acesso em 28/08/2021) <<https://wp.ufpel.edu.br/sisbi/files/2019/06/Manual.pdf>>. O projeto de pesquisa que originou esta tese foi submetido a exame de qualificação em 19/10/2018 e aprovado pela banca examinadora composta pelos professores doutores Rafael Ratto de Moraes, Aline Oliveira Ogliari e Gregori Franco Boeira.

## Resumo

MARCONDES, Rogério Luiz. **Resina composta restauradora pré-aquecida como agente de cimentação adesiva de restaurações indiretas**. Orientador: Rafael Ratto de Moraes. 2021. 68f. Tese (Doutorado em Odontologia) – Programa de Pós-Graduação em Odontologia, Universidade Federal de Pelotas, Pelotas, 2021.

O uso de resina composta restauradora pré-aquecida para cimentação adesiva de restaurações indiretas como alternativa aos tradicionais cimentos resinosos é cada vez mais popular. No entanto, ainda há espaço para melhorar a técnica clínica, entender como selecionar o compósito corretamente e reduzir a espessura de película, bem como relatar resultados clínicos de longo prazo. Essa tese abordou o tema por meio de três estudos. O primeiro estudo foi uma investigação *in vitro* dos efeitos do pré-aquecimento a 69°C sobre viscosidade, espessura de película e perda de temperatura de 10 compósitos contemporâneos, além do efeito da energia do ultrassom na espessura do filme resultante. Este estudo mostrou que materiais com formulações distintas reagem de forma diferente ao pré-aquecimento, afetando viscosidade e espessura do filme. O tempo de trabalho ideal dos compósitos pré-aquecidos foi curto, sugerindo que clínicos devem adequar a sequência de cimentação para aproveitar as temperaturas mais altas encontradas nos primeiros 15s após o pré-aquecimento. Além disso, observou-se que a aplicação do ultrassom foi eficaz na redução da espessura de película e pode ajudar resinas compostas restauradoras a alcançar películas abaixo de 50µm. O segundo estudo foi uma técnica clínica relatando a cimentação de facetas laminadas cerâmicas com composto pré-aquecido, descrevendo um procedimento passo a passo que pode ser utilizado por clínicos em sua rotina de trabalho, incluindo aplicação de ultrassom sobre a cerâmica para otimizar a espessura do filme. O terceiro estudo foi um relato de caso de tratamento clínico no qual os laminados cerâmicos foram cimentados adesivamente a dentes superiores anteriores de uma paciente utilizando resina composta pré-aquecida e mostrou excelente desempenho clínico e notável integridade marginal após 123 meses de acompanhamento. Uma transição marginal suave entre cerâmica, agente de cimentação e dente (área de continuidade adesiva) e a ausência de lacunas marginais e valamento indicaram que o compósito restaurador foi capaz de suportar os desafios abrasivos e superficiais impostos pelo ambiente oral em longo prazo. Em geral, esta tese mostra que resina composta pré-aquecida para a cimentação de restaurações indiretas pode ser considerado uma excelente opção clínica e que o desempenho geral da técnica clínica depende da seleção adequada de um compósito que responde adequadamente ao pré-aquecimento. Ainda há espaço para estudos clínicos controlados sobre o tema.

Palavras-chave: Cimentação. Resinas compostas. Restauração dentária permanente. Ultrassom.

## Abstract

MARCONDES, Rogério Luiz. **Preheated restorative resin composite as adhesive luting agent of indirect restorations.** Advisor: Rafael Ratto de Moraes. 2021. 68p. Thesis (PhD in Dentistry) – Graduate Program in Dentistry, Universidade Federal de Pelotas, Pelotas, 2021.

The use of restorative preheated resin composite for adhesive luting of indirect restorations as an alternative to traditional resin-based cements is increasingly popular. However, there is still room for improving the clinical technique, understanding how to select the composite properly and reduce film thickness, as well as on reporting long-term clinical results. This thesis addressed the topic by means of three studies. The first study was an *in vitro* investigation of the effects of preheating at 69°C on viscosity, film thickness, and temperature loss of 10 contemporary resin composites, in addition to the effect of ultrasound energy on the resulting film thickness. This study showed that materials with distinct formulations react differently to preheating, affecting viscosity and film thickness. Optimal working time of the preheated composites was short, suggesting that clinicians should adequate the luting sequence to take advantage of higher temperatures found in the first 15s after preheating. In addition, it was observed that application of ultrasound energy was effective in reducing film thickness and may aid restorative resin composites to achieve films below 50µm. The second study was a clinical technique reporting the luting of ceramic laminate veneers with preheated composite, describing a step-by-step procedure that may be used by clinicians in their working routine, including the application of ultrasound energy over the ceramic to optimize film thickness. The third study was a case report of a clinical treatment in which ceramic laminate veneers were adhesively luted to maxillary anterior teeth of a patient using preheated resin composite and showed excellent clinical service and remarkable marginal integrity after 123 months of follow up. A smooth marginal transition between ceramic, luting agent, and tooth (area of adhesive continuity) and the absence of marginal gaps and ditching indicated that the restorative resin composite was able to withstand the abrasive and surface challenges imposed by the oral environment in the long term. In general, this thesis shows that preheated resin composite for luting indirect restorations may be considered an excellent clinical option and that the overall performance of the clinical technique depends on proper selection of a resin composite that responds properly to preheating. There is still room for further controlled clinical studies on the topic.

Keywords: Cementation. Composite resins. Dental restoration, Permanent. Ultrasonics.



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## 1 Introdução

A alta demanda social por estética dentofacial tem implicações significantes na prática clínica de dentistas, aumentando consideravelmente os tratamentos com restaurações indiretas, especialmente facetas cerâmicas, também conhecidas como laminados cerâmicos ou lentes de contato dentárias. As facetas de cerâmica oferecem uma modalidade de tratamento previsível e bem-sucedida, proporcionando ótima preservação de estrutura dentária sadia (D'ARCANGELO et al., 2012). No intuito de diminuir os efeitos da intervenção restauradora, os preparos tendem a ser pouco invasivos, aproveitando a efetividade de materiais adesivos e obtendo margens íntegras, o que favorece a saúde periodontal e a longevidade dos trabalhos protéticos (HELVEY, 2009; MAGNE e DOUGLAS, 1999). O sucesso e sobrevivência no tratamento com facetas cerâmicas depende de diversos fatores, incluindo planejamento, preparo, moldagem, cimentação adesiva, acabamento e preservação entre fatores-chave para o sucesso e longevidade estética em longo prazo (AHMAD, 2010; D'ARCANGELO et al., 2012; MAGNE et al. 2002).

O sucesso a longo prazo das facetas pode também estar relacionado às propriedades do material escolhido para confeccionar a restauração (BAGHERI et al., 2007; BOVERA, 2016; FERRACANE, 2006; MCLAREN e WHITEMAN, 2010; PETRIDIS e MALLIARI, 2012) e agentes cimentante utilizados (KAWAI et al., 1994; MANHART et al., 2004). Outros são o desenho do preparo do dente, a condição funcional e morfológica do dente suporte e a adaptação marginal da restauração (PEUMANS et al., 2000). A deterioração da linha de cimentação adesiva deve a ser considerada no sucesso clínico de restaurações indiretas, sendo um dos elos fracos. A descoloração severa de margem já foi reportada em uma metanálise como uma importante causa de insucesso, com incidência de 2% (MORIMOTO et al., 2016). Uma revisão sistemática mostrou que a taxa de descoloração marginal inaceitável foi de 2,8% em 5 anos e 5,4% em 10 anos para restaurações cerâmicas. Os autores associaram a descoloração marginal ao valamento da margem (degradação marginal) associado à pigmentação extrínseca.

Neste sentido, pode ser observada na literatura uma busca por melhor adaptação marginal e menores linhas de cimentação em restaurações indiretas. A

maioria dos autores sugere uma linha de cimentação de no máximo 120 micrômetros (GOIJAT et al., 2019). No entanto, a espessura da linha de cimentação já foi avaliada clinicamente com valores que vão de 100 a 315 micrômetros (AKIN et al., 2015; KARAGÖZOĞLU et al., 2016; YUCE et al., 2019). Essa discrepância observada clinicamente em margens pode favorecer o valamento marginal, visto que o cimento resinoso, agente tradicionalmente usado para cimentar restaurações indiretas, pode sofrer desgaste por abrasão (KAWAI et al., 1994). Além disso, com o tempo, o cimento exposto na margem estará sujeito à sorção de água e fluidos (FERRACANE, 2006), degradação superficial (BAGHERI et al., 2007) e desgaste, que podem acelerar o valamento e descoloração marginal (MANHART et al., 2004), o que pode ocasionar falhas clínicas, especialmente por questões estéticas.

Tradicionalmente os cimentos resinosos são os mais utilizados para a cimentação adesiva de restaurações indiretas, como facetas cerâmicas, devido à simplicidade de aplicação associada à sua baixa viscosidade, que facilita o assentamento rápido da peça. No entanto, resinas compostas restauradoras têm sido utilizadas para procedimentos de colagem desde a introdução das restaurações indiretas parciais não-retentivas (BELSER et al., 1997; CHRISTENSEN, 1985; D'ARCANGELO et al., 2012; DARONCH et al., 2006; FRIEDMAN, 1998; HELVEY, 2009; SCHULTE et al., 2005). As possíveis vantagens que justificariam o uso de resinas compostas como agente de cimentação estão relacionado ao potencial menor valamento marginal por ser um material mais resistente ao desgaste, maior estabilidade de cor e maior resistência mecânica (BARBON et al., 2019; COELHO et al., 2019; DONG et al., 2016; DUARTE et al., 2011; GRESNIGT et al., 2017; GUGELMIN et al., 2020; SCHNEIDER et al., 2020; SPAZZIN et al., 2017; TOMASELLI et al., 2019; VAN DEN BREEMER et al., 2021).

Resinas compostas restauradoras são mais viscosas que cimentos resinosos e, dessa forma, normalmente são pré-aquecidas antes da cimentação para aproveitar o ganho de fluidez obtido no aquecimento, que pode ser feito até ~70°C (DA COSTA et al., 2009; FRÓES-SALGADO et al., 2010; HELVEY, 2009; LOHBAUER et al., 2009). Assim, uma característica da técnica que usa resinas composta não fluidificada corretamente como agente de cimentação é a maior espessura do agente cimentante na comparação da técnica com cimento resinoso (AL-DWAIRI et al., 2019; SAMPAIO et al., 2017). Esta informação poderia levar a uma contraindicação precoce do uso de resinas compostas como agente cimentante, visto que a margem é o elo fraco

atualmente das restaurações indiretas (SAMPAIO et al., 2017). De fato, o uso de resinas compostas restauradoras que apresentam alta viscosidade mesmo após o aquecimento deve ser evitado, por poderem criar filmes muito grandes, chegando a mais de 300 micrômetros (SAMPAIO et al., 2017). Da mesma forma, devem ser evitadas técnicas de aquecimento que não permitam adequado controle da temperatura do material e procedimentos clínicos de fotopolimerização do agente cimentante sem uso de pressão.

Apesar das resinas compostas restauradoras não compartilharem das propriedades mecânicas inerentes aos cimentos resinosos que os fazem mais susceptíveis à degradação marginal (DA COSTA et al., 2010; DUARTE et al., 2011; SHINKAI et al., 2001) maiores espessuras de linha de cimentação podem gerar maiores ajustes clínicos. No intuito de se diminuir a linha de cimentação, o fluxo ideal para cimentação adesiva com resina composta restauradora envolve pelo menos a correta seleção da resina apropriada, aquecimento previsível da resina e técnica de cimentação adesiva com a resina aquecida. O aquecimento de algumas resinas compostas restauradoras com propriedades reológicas ideais favorece a diminuição da viscosidade do material, possibilitando o uso previsível do mesmo para colagem de laminados cerâmicos finos (MAGNE et al., 2018; MARCONDES et al., 2020). Espera-se que esse compósito aquecido possa ser utilizado como agente de cimentação sem perder suas propriedades de resistência mecânica (NIKOLAOS-STEFANOS, 2019).

Há ainda dúvidas clínicas sobre quais resinas seriam mais indicadas para a técnica de cimentação adesiva com resina composta termomodificada (RTM), principalmente se há alguma correlação da viscosidade após aquecimento com a composição de matriz orgânica e partículas de carga. Não se sabe se a reologia das resinas compostas restauradoras é influenciada pela quantidade de partícula de carga na sua composição, por exemplo. A dinâmica de perda de calor e diminuição da temperatura com aumento da viscosidade após cessar o aquecimento de diferentes resinas também é um fator importante no procedimento de cimentação e faltam dados na literatura acerca deste aspecto. Além do tipo de resina e do aquecimento, o uso de energia ultrassônica tem sido recomendado para facilitar a remoção de excessos e melhorar o assentamento de restaurações indiretas (PEUTZFELDT, 1994; SCHMIDLIN et al., 2005; WALMSLEY e LUMLEY, 1995, 1999). No entanto, ainda não

se sabe do efeito de associar o aquecimento de diferentes resinas compostas com energia ultrassônica para diminuição de espessura de filme.

Há muitas formas de se aquecer resina composta restauradora para cimentação adesiva que vão desde do banho-maria, ao uso de um dos aparelhos específicos para aquecimento controlado. Há poucas informações sobre as diferentes técnicas e os benefícios e limitações do fluxo clínico de cada uma. Muitas vezes o cirurgião-dentista não sabe selecionar o melhor fluxo para cimentação com RTM para sua realidade. Além disso, poucos são os relatos de caso na literatura que mostram a sobrevivência e sucesso de restaurações cimentadas com RTM. Sendo assim o objetivo desta tese foi avaliar o comportamento reológico de várias resinas compostas restauradoras e cimentos resinoso após aquecimento, o efeito do uso do ultrassom da diminuição de filme, a perda de calor após aquecimento (capítulo 1); relatar o fluxo clínico da técnica de cimentação adesiva com RTM (capítulo 2) e exemplificar, por meio de relato de caso, o acompanhamento clínico de restaurações cimentadas com RTM após 10 anos (capítulo 3).

## 2 Capítulo 1<sup>1</sup>

Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness

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Acknowledgements: This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Brazil (Finance Code 001). The sponsor had no role in study design, collection, analysis or interpretation of data, writing the report, or decision to submit for publication. V.P.L., F.J.B., C.P.I., and M.V.S. are grateful to CAPES/Brazil for scholarships.

Declarations of interest: None.

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<sup>1</sup> Artigo publicado no periódico Dental Materials (Anexo A).

## ABSTRACT

**Objectives:** This study investigated viscosity and thermal kinetics of 10 selected preheated restorative resin composites and the effect of ultrasound energy on film thickness.

**Methods:** A range of different resin composites was tested: Charisma Diamond, IPS Empress Direct, Enamel Plus HRi, Essentia, Estelite Omega, Filtek Z100, Filtek Z350 XT, Gradia, TPH Spectrum and VisCalor. A flowable resin composite (Opallis Flow) and two resin cements (RelyX Veneer, Variolink Esthetic LC) also were tested. Viscosity (Pa.s) was measured at 37°C and 69°C (preheating temperature) using a rheometer. Film thickness (µm) was measured before and after application of ultrasound energy. Temperature loss within resin composite following preheating (°C/s) was monitored. Data were statistically analyzed ( $\alpha=0.05$ ).

**Results:** Viscosity at 69°C was lower than at 37°C for all materials except the flowable resin composite. Preheating reduced viscosity between 47% and 92% for the restorative resin composites, which were generally more viscous than the flowable materials. Film thickness varied largely among materials. All preheated resin composites had films thicker than 50 µm without ultrasound energy. Application of ultrasound reduced film thickness between 21% and 49%. Linear and nonlinear regressions did not identify any relationship between filler loading, viscosity, and/or film thickness. All materials showed quick temperature reduction following preheating, showing maximum temperature loss rates after approximately 10 s.

**Significance:** Distinct restorative resin composites react differently to preheating, affecting viscosity and film thickness. The overall performance of the preheating technique depends on proper material selection and use of ultrasound energy for reducing film thickness.

**Keywords:** luting, temperature, flowability, ultrasonics, resin cement, flowable resin composite.

## 1. INTRODUCTION

Use of preheated restorative resin composite as luting agent for veneers and other thin indirect restorations is increasingly popular. The topic has been investigated in clinical and laboratory studies [1–9]. When compared to photopolymerizable resin cements and flowable resin composites, potential advantages of preheated restorative

resin composites may include increased shade availability, lower cost, less polymerization shrinkage and marginal degradation, and improved mechanical performance due to their higher filler content [8–19].

Preheating intends to reduce viscosity and increase flowability of restorative resin composite pastes [20], but thicker films compared to resin cements are commonly observed [3,6,7]. It has been reported that a poor marginal fit of indirect restorations could lead to resin cement dissolution and marginal discoloration [21–24]. There is still no consensus, however, for limits of clinically acceptable film thickness. As a laboratory screening method, the ISO 4049 standard considers 50  $\mu\text{m}$  as a limit for resin-based luting agents [25]. Most authors suggest that films should be thinner than 120  $\mu\text{m}$  in the clinics [26–28], whereas clinical studies indicate that average marginal discrepancies in indirect restorations may vary between 100 and 315  $\mu\text{m}$  [29–31]. The film thickness yielded by different preheated restorative resin composites should be evaluated in order to aid the proper selection of an adequate material for the technique.

A new resin composite claiming a ‘thermoviscous technology’ (VisCalor, Voco, Cuxhaven, Germany) was recently introduced. VisCalor is primarily a bulk-fill restorative, but perhaps it could generate a thin film if used as luting agent. Recent reports observed that preheating reduced up to 66% the force required to extrude VisCalor from its compule, whereas the degree of C=C was not affected [32] and no adverse effect of premature polymerization was observed [33]. Another alternative to reduce film thickness, raised in previous work [3], is the use of ultrasound energy, which could increase flowability of the restorative resin composite if applied over the ceramic restoration [34–36].

Several restorative resin composite options are available in the market. Since most materials are not primarily intended to be preheated, chances are that dentists will choose anyone at hand. However, a recent study [3] reported that different formulations of resin composites may react differently to preheating, affecting viscosity and film thickness, and ultimately influencing the mechanical performance of luted ceramic structures. Thermal loss after preheating is ceased will likely play a role on those aspects. Since not all clinical preheating techniques may provide adequate working time, the cooling patterns of different resin composites should be further studied. The best-case scenario would be understanding how a range of restorative resin composites react to preheating and the resulting flowability and film thickness, guiding proper material selection and the clinical procedures.



This study investigated the effects of preheating on viscosity, film thickness, and temperature loss of 10 contemporary restorative resin composites. The effect of ultrasound energy application on film thickness also was investigated. Two resin cements and a flowable resin composite were included for comparison. The hypotheses tested were: (i) film thickness, viscosity and thermal loss would be material dependent, (ii) use of ultrasound would reduce film thickness.

## 2. MATERIALS AND METHODS

### 2.1. Study design and materials tested

This in vitro study evaluated the effect of preheating different restorative resin composites on their viscosity and film thickness as primary response-variables. Ten restorative resin composites (Table 1) were selected considering their range in classifications, formulations, and manufacturers. Dentins shades A1, A2, or similar were tested. A flowable resin composite and two resin cements were tested for comparison, and are herein referred as flowable materials. A 69°C temperature was used as clinical desired temperature for luting with preheated restorative resin composites. The effect of ultrasound energy application on film thickness was also tested. Thermal kinetics within resin composite increments following preheating was monitored, with temperature loss and cooling rates as response-variables.

### 2.2. Viscosity

Viscosity ( $n=5$ ) was measured using a dynamic oscillation rheometer (R/S-CPS+; Brookfield, Middleboro, MA, USA). Two temperatures were tested: 69°C, as the initial temperature obtained clinically after preheating on the specific heater device used here (HotSet; Technolife, Joinville, SC, Brazil), and 37°C (body temperature) as final temperature, simulating the clinical condition after seating the restoration. It was not possible to test the materials at 25°C because some resin composites were too viscous at room temperature and exceeded the rheometer measuring range. The resin composites were taken from their original packages (i.e. syringe or compule) with a spatula and placed in a half-circle mold for standardizing a 0.5 mL volume. The test material was dispensed on the lower plate of the rheometer and positioned with a 0.05 mm gap between the plates. Heating was provided by the rheometer itself. Viscosity (Pa.s) was measured until reaching the designated temperature and for additional 45

s, at a constant shear rate of  $2 \text{ s}^{-1}$ . The flowable resin composite and resin cements were also tested in both temperatures.

### 2.3. Film thickness

Film thickness ( $n=3$ ) was measured based on the ISO 4049 standard [25]. Only restorative resin composites were preheated in this analysis. Two optically flat, square glass plates with  $200 \text{ mm}^2$  contact surface area were used. The combined thickness of the two glass plates stacked in contact was measured with a digital caliper (Mitutoyo, Tokyo, Japan) with  $1 \text{ }\mu\text{m}$  accuracy. Increments of restorative resin composites were preheated to  $69^\circ\text{C}$  for 10 min in order to achieve and stabilize this temperature before testing [37]. The increment was placed directly on the preheating device using a spatula. A standard  $0.1 \text{ mL}$  volume of the preheated material was dispensed on the center of a glass plate and the other plate was placed on top. A  $150 \text{ N}$  force was centrally and vertically applied via the upper plate using a loading device (Odeme Dental Research; Joaçaba, SC, Brazil). After 180 s, the loading system was released and the combined thickness of the two glass plates was measured again. Film thickness was calculated as the difference between the two readings. Three different specimens were tested for each material. The thickness of each specimen was read three times and the average value was recorded as the film thickness for that specimen. No light-polymerization was carried out because the same specimen was used next for testing the effect of ultrasound energy, in accordance with the clinical workflow of seating indirect restorations [35]. The ultrasound energy was applied through the upper glass plate for 30 s using a polyacetal tip. The tip was positioned statically at the center of the glass plate with slight hand pressure, the ultrasound equipment operated at 40% power (DentSurg Pro; CVdentus, São José dos Campos, SP, Brazil). It should be highlighted that the resin composite between the glass plates was not warm anymore during the ultrasound application step, simulating what happens in the clinical scenario when luting indirect restorations with pre-heated resin composite. Film thickness after ultrasound application was measured anew.

### 2.4. Thermal kinetics

Resin composite increments (2 mm in thickness, mass  $\sim 130 \text{ mg}$ ) were placed over a polyester stripe and inside the preheating device. The preheating device has spaces that allowed the increments to be placed without overflowing during preheating.

This was important to avoid reduction in increment thickness that could affect the temperature measurements. A type-K thermocouple was used (TM902C, Yarboly, China), the tip (diameter = 1 mm) was inserted within the increment to monitor temperature. When it reached  $70\pm1^{\circ}\text{C}$ , the polyester stripe with increment was removed from the preheating device and placed over the bench at room temperature ( $25^{\circ}\text{C}$ ). Temperature ( $^{\circ}\text{C}$ ) within the increment was recorded every second for 2 min after placing the resin composite over the bench ( $n=3$ ). This time was enough for all resin composites to approximately reach room temperature. Plotted temperature vs. time data were adjusted by curve fitting ( $R^2 > 0.997$ ) and temperature loss rates were calculated using these fitted plots.

## 2.5. Statistical analysis

Viscosity data were submitted to a Two-Way Analysis of Variance – ANOVA (material vs. temperature). Viscosity data were transformed to ranks before the analysis. Data for film thickness without use of ultrasound were analyzed using One-Way ANOVA. Film thickness data of restorative resin composites including the use of ultrasound were analyzed using Repeated Measures ANOVA (one factor repetition). All pairwise multiple comparison procedures were carried out using the Tukey method. Regression analysis were used to investigate the relationship between filler load (wt% and vol%), viscosity, and/or film thickness. Significance level was set at  $\alpha=0.05$  for all analyses. Thermal loss within resin composite and cooling rates were analyzed descriptively.

## 3. RESULTS

Results for viscosity at  $37^{\circ}\text{C}$  and  $69^{\circ}\text{C}$  are shown in Figure 1. Materials are listed in ascending order of viscosity at  $69^{\circ}\text{C}$  (top to bottom). Average reductions in viscosity by preheating (%) are presented. Both factors and their interaction were statistically significant ( $p<0.001$ ). The viscosity at  $69^{\circ}\text{C}$  was significantly lower than at  $37^{\circ}\text{C}$  for all materials ( $p\leq 0.027$ ) except the flowable resin composite ( $p=0.45$ ). Significant differences in viscosity were observed in almost all comparisons between materials, including at  $69^{\circ}\text{C}$  (Table 2). In either temperature, all restorative resin composites were significantly more viscous than the flowable resin composite and Variolink Esthetic LC resin cement. When preheated, four resin composites had lower viscosity compared with RelyX Veneer resin cement (at room temperature): Essentia, Gradia, VisCalor,

and Estelite Omega. Filtek Z350 XT showed remarkably higher viscosity than all other materials in both temperatures tested. At 69°C, Filtek Z350 XT showed viscosity around 14 kPa.s, whereas all other preheated materials were at least 3-fold less viscous. Preheating also reduced viscosity of the resin cements. VisCalor (92%), TPH Spectrum (82%), and Essentia (81%) showed the highest viscosity reductions by preheating.

Figure 2 presents the results for film thickness before and after use of ultrasound energy. Materials are listed in ascending order of film thickness after ultrasound application (top to bottom). Average reductions in film thickness by use of ultrasound (%) also are shown. The dashed line indicates the 50- $\mu$ m film thickness limit defined by ISO 4049 standard. The statistical analysis revealed significant differences between groups ( $p < 0.001$ ) and the results varied largely among materials. In the regular test (no ultrasound), all preheated restorative resin composites had films thicker than 50  $\mu$ m, and all flowable materials thinner than the ISO limit (Table 2). The use ultrasound energy significantly reduced film thickness ( $p < 0.001$ ), the reductions varied between 21% and 49%. Five restorative resin composites had film thicknesses below or approximate 50  $\mu$ m after use of ultrasound: Estelite Omega, Filtek Z100, Enamel Plus HRi, VisCalor, and Gradia. Two resin composites showed films thicker than 70  $\mu$ m even after ultrasound: Filtek Z350 XT and TPH Spectrum. Linear and nonlinear regression analyses were not able to identify any trend or relationship between filler loading, viscosity, and film thickness of the materials tested. Figure 3 presents plots for linear regression analyses of filler load (wt%) vs. viscosity at 69°C (Fig.3A), filler load vs. film thickness without ultrasound (Fig.3B), and viscosity vs. film thickness (Fig.3C). The coefficients of determination ( $R^2$ ) were below 0.2.

Results for the thermal analysis are presented in Figure 4 (temperature loss) and Figure 5 (cooling rate). The materials were separated in higher viscosity and lower viscosity restorative resin composites in these figures. All materials showed quick temperature reduction after placed in the bench. Cooling rate analysis showed that, for most higher viscosity materials, maximum temperature loss rates were reached 7 to 8 s after the heating was ceased. Lower viscosity resin composites took slightly longer (about 10 s) to reach maximum temperature loss rates. Table 2 shows the temperature within resin composite increments 15, 30, and 60 s after preheating. Fifteen seconds after the heating was ceased, all resin composites had average temperature within increment below 50°C, with temperature losses varying between 45% and 61%. This

calculation considers that the temperature loss is 100% when the increment reaches room temperature. The average temperature within the increments was below 37°C after 30 s (average 84% temperature loss), and below 29°C for all resin composites after 60 s (average 96% loss).

#### 4. DISCUSSION

The first hypothesis was accepted as film thickness, viscosity and thermal loss were material dependent. The 10 restorative resin composites tested have distinct formulations, including monomers and fillers, which affect their response to preheating as each component has a specific heat capacity. The resin phase is less thermal conductive than filler particles but it is expected to react most to preheating by increasing monomer mobility. The filler particles play an important role on thermal conductivity as well [38]. A study with dental resin composites [39], for instance, showed a nonlinear increase in the system enthalpy by increasing the concentration of fillers. In this study, no relationship was observed between filler content, viscosity, and/or film thickness. This may have occurred because not only filler content but also particle type, shape, size, nature of particle surface, and filler spatial arrangement within the resin composite are relevant aspects for thermal conductivity [38,39]. Those features are expected to differ among the tested resin composite materials. Since manufacturers do not disclose formulation details, experimental materials should be used in future studies for further understating how different monomers and filler features might influence the resin composite reaction to preheating.

Characteristics of the inorganic particles may also influence flowability. Preheating to 69°C was able to reduce between 47% and 92% the viscosity of restorative resin composites in comparison to 37°C. Another study reported that preheating increased between 23% and 55% the flowability of four restorative resin composites [40], also showing a negative correlation between filler content and flowability. In the present study, preheated resin composites hardly showed viscosity values in the range of flowable composite and resin cements. This is likely a result of the higher filler content leading to increased filler-to-filler interactions and interfacial friction between fillers and resin matrix, affecting flowability. Findings of the present study suggest that viscosity at preheating temperature (69°C) or change in viscosity upon preheating (%) are not adequate parameters for selecting a restorative resin composite for luting purposes. This can be illustrated by the behavior of VisCalor, a

resin composite designed by the manufacturer to be preheated. VisCalor showed the highest reduction in viscosity at 69°C among all materials tested (92%), but it was not able to yield films thinner than 50 µm without the use of ultrasound. Essentia is another good example, as it showed low viscosity after preheating but generated thicker films than other materials with higher viscosity.

It has been reported that preheating restorative resin composites may reduce their film thickness between 4% and 77% depending on the tested material [41]. The same authors observed no significant correlation between either weight or volume of fillers and film thickness. Similarly, the thinning of preheated restorative resin composite films was not a result of their filler content alone. In addition, this study demonstrates that use of ultrasound energy significantly reduced film thickness (between 21% and 57%). Thus, the second hypothesis also was accepted. For most materials, an optimal film thickness may be achieved by combining preheating and ultrasound application. The influence of ultrasound is more evident as its application occurred at least 3 min after preheating, when the resin composite was already at room temperature. During the clinical luting of indirect restorations, application of ultrasound occurs specifically after restoration seating and removal of major excesses. Therefore, the temperature of the luting agent during ultrasound use is lower than in the initial seating step [35]. The glass plates used in the test also are thicker than indirect restorations, thus the use of ultrasound energy could be even more beneficial in the clinical scenario. However, the glass plates are flat and smooth, whereas the intaglio surface of indirect restorations may not be. Although ultrasound energy has been previously used by clinicians to reduce film thickness [34–36], this is the first study to evaluate the effect of ultrasound on a variety of restorative resin composites. Finally, considering the relevant film thinning observed with ultrasound energy even at room temperature, the working time may not be a significant issue when preheated resin composite is used as luting agent. Provided that ultrasound is applied afterwards, clinicians can take their time for proper excess removal before final seating and light polymerization.

Different from clinical luting procedures, the method used here to evaluate film thickness requires the simulated luting load to be released before measurements. In addition, the materials were not light polymerized, whereas a reduction in film thickness after light polymerization has been reported [4]. In the clinical scenario, it is suggested that indirect restorations should be maintained under slight hand pressure during light

polymerization. This would avoid possible restoration displacement arising from the viscoelastic response of the resin composite in the event pressure is removed. The use of ultrasound is also important in the control of film thickness, although there is no consensus on the limits for clinically acceptable film thicknesses. Perhaps the 50  $\mu\text{m}$  value defined by ISO 4049 should not be considered a limit when restorative resin composite is the luting agent, especially because up to 6 times higher marginal discrepancy values have been reported for indirect restorations in clinical studies [29–31]. In addition, resin composites are direct restorative materials designed to withstand intraoral challenges; a thicker film may not be of clinical significance provided that it does not interfere with adaptation of the indirect restoration.

The restorative resin composites presented a rapid temperature reduction in the cooling rate analysis. Therefore, clinicians have between 10 to 15 s of ideal working time with preheated resin composites, when temperature and viscosity are still optimal. This working time should be taken into consideration in the selection of a proper luting sequence. Several techniques are available, some requiring less time from the moment the preheated composite is removed from the heating device until it is placed at the prepared tooth [37]. Warm water bath has been used for preheating resin composites, but a study reported an up to 2-fold increase in film thickness compared to flowable materials [6]. This is likely explained by the water bath technique being more time consuming, which may have affected flowability and film thickness. Other quicker luting sequences have been proposed, including preheating compules already attached to delivery syringes [37], or placing the resin composite into the intaglio surface of indirect restorations and preheating them simultaneously [14]. These two latter techniques seem to take more advantage of the optimal working and flowability of preheated resin composites. Placing the preheated resin composite over the bench before luting is not advised.

Taking all results of the present study into account, it seems reasonable to suggest that Charisma Diamond, Essentia, Filtek Z350 XT, and TPH Spectrum should not be used as luting agents since these resin composites yielded films with ~70-80  $\mu\text{m}$  in average even when preheating was associated with ultrasound. This study shows that there are better resin composite options for the preheating luting technique. Gradia and IPS Empress Direct showed intermediary results. However, it should be noted that a thicker film of resin composite may not be a clinical issue because this material is designed to have color stability and abrasion resistance, as shown in

laboratory and clinical studies [10,42,43]. In addition, recent studies [3,11] raised the question whether thicker films could have a positive effect on the strengthening of thin feldspar ceramic structures. Finally, clinicians could consider other aspects they find relevant for resin composites, including handling, stickiness and cost, which will depend on the selected resin composite brand.

## 5. CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions can be drawn:

- Restorative resin composites with distinct formulations react differently to preheating, affecting viscosity and film thickness;
- Optimal working time of preheated composite is short and clinicians should adequate the luting sequence to take advantage of higher temperatures found in the first 15 s;
- Application of ultrasound energy is effective in reducing film thickness and may aid restorative resin composites to achieve films below 50  $\mu\text{m}$ ;
- The overall performance of the preheating resin composite technique depends on proper material selection.

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Table 1. Characteristics and formulation of the resin-based agents tested as informed by manufacturers

Materials tested	Type	Manufacturer	Formulation	
<i>Restorative resin composites</i>			Resin phase	Filler wt% (vol%)
Charisma Diamond	Nanohybrid	Kulzer, Hanau, Germany	Bis-GMA, UDMA, TEGDMA, TCD-DI-HEA	77
IPS Empress Direct	Nanohybrid	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, TCDDMA	60 or 79.6*
Enamel Plus HRi	Nanohybrid	Micerium, Avegno, Italy	Bis-GMA, UDMA, BDDMA	80 (63)
Essentia	Microhybrid	GC, Tokyo, Japan	Bis-GMA, UDMA, TEGDMA, Bis-EMA, Bis-MEPP	81 (65)
Estelite Omega	Supranano	Tokuyama, Tokyo, Japan	Bis-GMA, TEGDMA	82 (78)
Filtek Z100	Microhybrid	3M ESPE, St. Paul, MN, USA	Bis-GMA, TEGDMA	80 (66)
Filtek Z350 XT	Nanohybrid	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA	72.5 (55.6)
Gradia	Microhybrid	GC	UDMA	80
TPH Spectrum	Nanohybrid	Dentsply Sirona, York, PA, USA	Bis-GMA, Bis-EMA, TEGDMA	75 (57)
VisCalor	Nanohybrid	Voco, Cuxhaven, Germany	Bis-GMA, aliphatic dimethacrylate	83
<i>Resin cements</i>				
RelyX Veneer	Light-cured cement	3M ESPE	Bis-GMA, TEGDMA	66
Variolink Esthetic LC	Light-cured cement	Ivoclar Vivadent	UDMA, DDMA	(38)
<i>Flowable resin composite</i>				
Opallis Flow	Microhybrid	FGM, Joinville, SC, Brazil	Bis-GMA, TEGDMA, Bis-EMA	72

Bis-GMA, bisphenol-A glycidyl dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; TCD-DI-HEA, Bis-(acryloyloxymethyl) tricyclodecane; TCDDMA: tricyclodecane dimethanol dimethacrylate; BDDMA: 1,4-butandiol dimethacrylate; Bis-EMA, bisphenol-A ethoxylated dimethacrylate; Bis-MEPP, bisphenol-A polyethoxy methacrylate; PEGDMA, polyethylene glycol dimethacrylate; DDMA: 1,10-decandiol dimethacrylate. \*Including prepolymer as filler.

Table 2. 95% confidence intervals for viscosity at 69°C (n=5) and film thickness after use of ultrasound (n=3), and means  $\pm$  standard deviations for temperature within resin composite increments with time following preheating (n=3)

Material	Viscosity, kPa.s	Film thickness, $\mu$ m	Temperature within increment, °C		
			15 s	30 s	60 s
Charisma Diamond	2.91–3.01 <sup>c</sup>	48–106 <sup>ab</sup>	43 $\pm$ 2	35 $\pm$ 4	28 $\pm$ 2
IPS Empress Direct	2.45–2.48 <sup>e</sup>	37–83 <sup>bc</sup>	42 $\pm$ 8	30 $\pm$ 2	25 $\pm$ 1
Enamel Plus HRi	2.85–2.91 <sup>c</sup>	40–54 <sup>bc</sup>	49 $\pm$ 9	37 $\pm$ 5	29 $\pm$ 3
Essentia	0.34–0.36 <sup>i</sup>	61–119 <sup>ab</sup>	43 $\pm$ 3	33 $\pm$ 4	27 $\pm$ 2
Estelite Omega	0.71–0.73 <sup>g</sup>	30–44 <sup>c</sup>	45 $\pm$ 4	29 $\pm$ 1	25 $\pm$ 1
Filtek Z100	2.59–2.63 <sup>d</sup>	25–60 <sup>bc</sup>	41 $\pm$ 9	29 $\pm$ 7	26 $\pm$ 2
Filtek Z350 XT	14.0–14.3 <sup>a</sup>	71–109 <sup>ab</sup>	41 $\pm$ 2	31 $\pm$ 2	25 $\pm$ 1
Gradia	0.41–0.43 <sup>h</sup>	13–61 <sup>cd</sup>	45 $\pm$ 4	33 $\pm$ 1	28 $\pm$ 1
TPH Spectrum	3.77–3.80 <sup>b</sup>	71–123 <sup>a</sup>	46 $\pm$ 3	31 $\pm$ 1	27 $\pm$ 2
VisCalor	0.43–0.47 <sup>h</sup>	30–64 <sup>bc</sup>	43 $\pm$ 9	31 $\pm$ 4	27 $\pm$ 2
RelyX Veneer*	0.92–1.01 <sup>f</sup>	15–39 <sup>cd</sup>	-	-	-
Variolink Esthetic LC*	0.22–0.24 <sup>j</sup>	6–20 <sup>d</sup>	-	-	-
Opallis Flow*	0.12–0.14 <sup>k</sup>	14–38 <sup>cd</sup>	-	-	-

\*Viscosity at 37°C; in film thickness analysis, ultrasound was not applied for these materials.

Different letters in same column indicate statistical differences between materials ( $\alpha=0.05$ ).

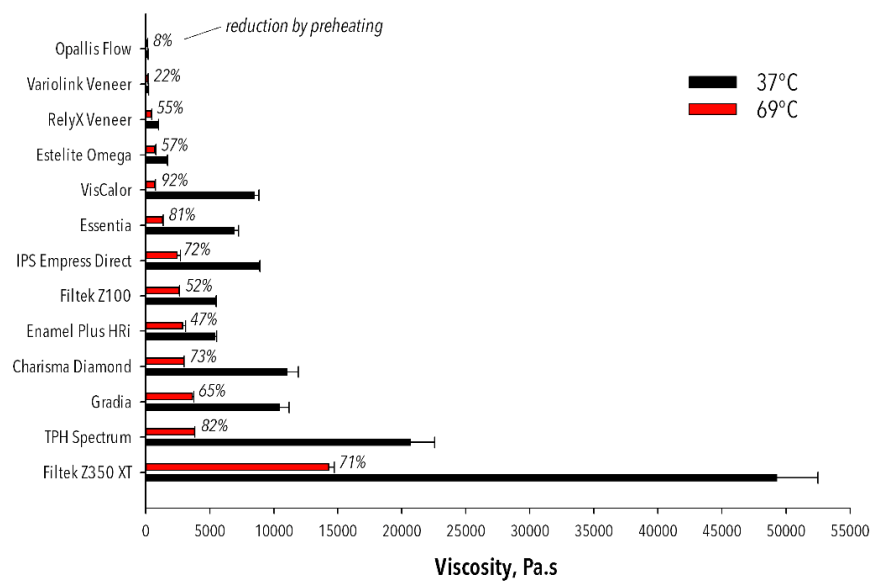


Figure 1. Means + standard deviations for viscosity at 37°C and 69°C (n=5). Materials are listed in ascending order of viscosity at 69°C (top to bottom). Change in viscosity by preheating (%) is shown for each material.

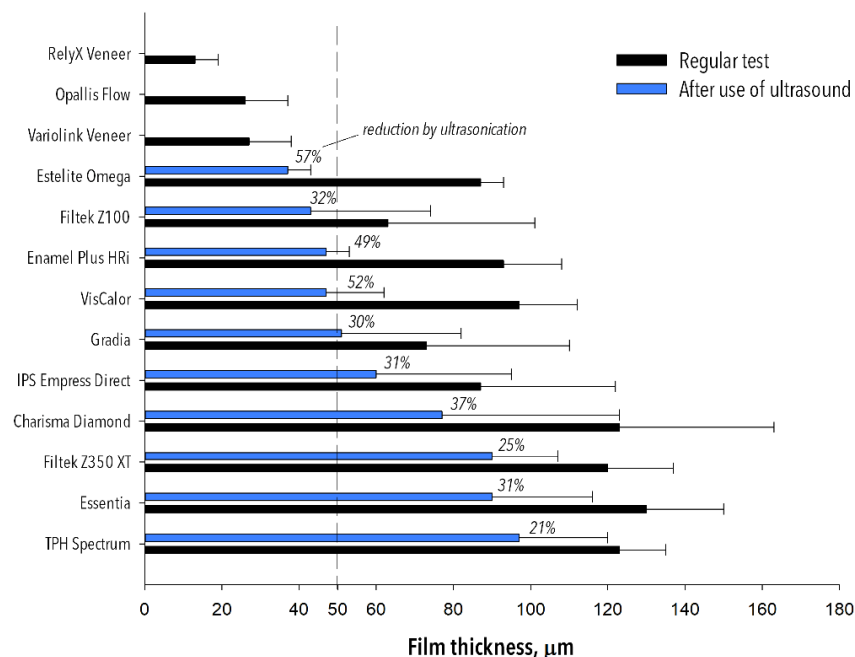


Figure 2. Means + standard deviations for film thickness with and without use of ultrasound energy (n=3). Materials are listed in ascending order of film thickness after use of ultrasound (top to bottom). Note that only restorative resin composites were preheated and subjected to ultrasound energy. Change in film thickness by ultrasound application (%) is shown for each material. Dashed line indicates 50-μm film thickness limit defined by ISO 4049 standard.

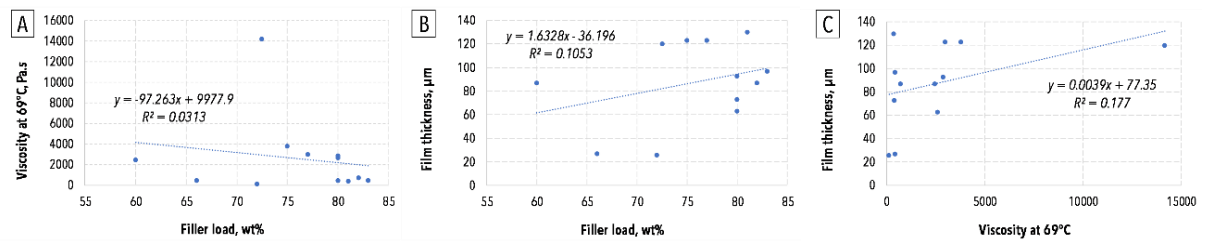


Figure 3. Plots for linear regression analyses of filler load (wt%) vs. viscosity at 69°C (A), filler load vs. film thickness without ultrasound (B), and viscosity vs. film thickness (C). The coefficients of determination ( $R^2$ ) were below 0.2. These and other linear or nonlinear regressions were not able to identify any trend or relationship between filler load, viscosity, and film thickness of the materials tested.

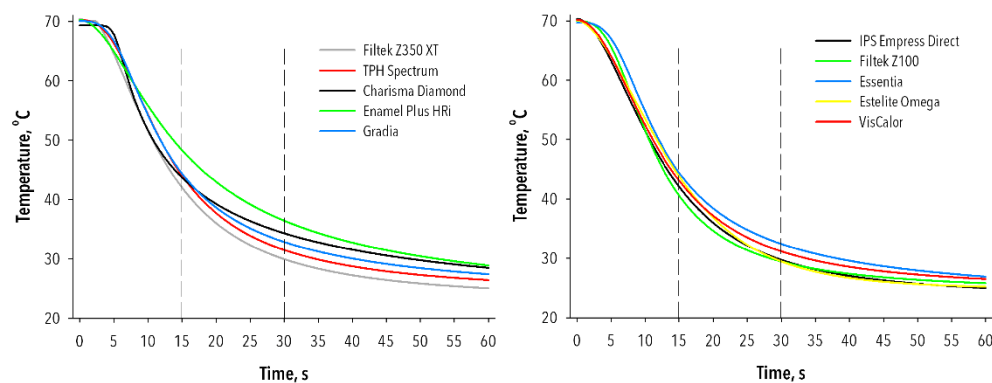


Figure 4. Temperature reduction within resin composite increments placed on bench following preheating ( $n=3$ ). Restorative resin composites were separated in higher viscosity (left hand) and lower viscosity (right hand). Dashed lines indicate 15- and 30-second marks.



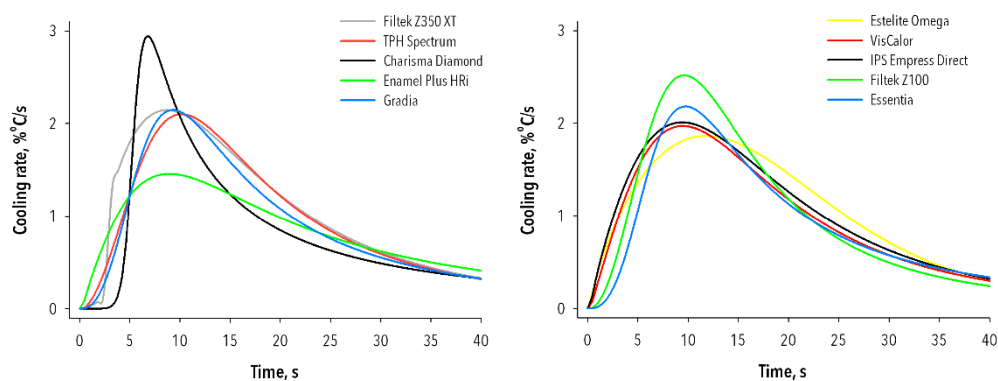


Figure 5. Cooling rates following preheating (n=3). Restorative resin composites were separated in higher viscosity (left hand) and lower viscosity (right hand). Maximum rates of temperature loss were typically achieved up to 10 s after preheating.

### 3 Capítulo 2<sup>2</sup>

#### **Preheated restorative composite for luting ceramic laminate veneers: A technique report**

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#### **Abstract**

Resin cements are traditionally used to lute ceramic laminate veneers due to their lower viscosity, which facilitates a fast restoration seating. However, resin cements have lower wear resistance than restorative composites. Thus, restorative composite resin is an alternative luting agent with lower marginal degradation as a potential advantage for clinical longevity. This article presents an application of preheated restorative composite resin for adhesive luting of laminate veneers with a predictable clinical technique for seating and marginal quality.

#### **Introduction**

Bonding of indirect restorations, known as adhesive cementation, comprises one of the most critical steps of adhesive treatment and responds to the majority of clinical failures reported in literature.<sup>1-3</sup> The main causes of failure reported for indirect restorations are marginal discoloration, marginal degradation, and debonding of the restoration.<sup>4-6</sup> Thus, there is a constant need for better marginal adaptation while

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<sup>2</sup> Artigo que será enviado para potencial publicação no periódico The Journal of Prosthetic Dentistry.

attaining film thickness below 120  $\mu\text{m}$ ,<sup>7</sup> although clinical studies found marginal discrepancies in indirect restorations between 100 and 315  $\mu\text{m}$ .<sup>8,9</sup>

The gap between indirect restorations and tooth surfaces filled by luting material is known as area of adhesive continuity.<sup>10</sup> A thick line of exposed cement could over time be subject to sorption<sup>11</sup>, surface degradation,<sup>12</sup> and wear, leading to marginal ditching and discoloration.<sup>13</sup> Even tooth brush abrasion can lead to marginal ditching influenced by dentifrice abrasiveness,<sup>14</sup> tooth brushing force,<sup>15</sup> and direction of the bristles.<sup>16</sup> When subject to tooth brushing, resin cements with larger filler particles have shown increased wear than those with smaller particles.<sup>14,17</sup> In addition, resin cements showed greater marginal degradation than resin composites.<sup>18</sup>

Resin cements are traditionally used for luting indirect restorations due to the simplicity of application associated with their lower viscosity, which enables fast restoration seating. However, restorative composite resins have been increasingly used for bonding non-retentive partial restorations.<sup>19-25</sup> The benefits that would justify the use of composites are related to lower marginal degradation, greater color stability, and improved mechanical strength.<sup>26-33</sup> Preheating of restorative composites with appropriate rheological properties enables its predictable use for bonding indirect restorations.<sup>34-36</sup> Associated with preheating, the use of ultrasonic devices favors a faster excess removal during the seating of restorations and may aid in reducing the luting agent film.

Considering the clinical evidence that the adhesive interface between the dental substrate and restoration is the weak link of adhesive indirect restorations, bonding the restoration with pre-heated restorative composite resin may provide an interface filled with a restorative resin material presenting optimized mechanical properties. Therefore, an area of adhesive continuity that is more resistant to degradation and staining is expected to provide improved restoration prognosis. This article reports a clinical technique for luting ceramic laminate veneers with preheated composite.

## Technique

Figure 1 to 8 illustrate the technique, which was carried out using the following clinical steps:

1. Ceramic laminate veneers were prepared for ten maxillary teeth to recover incisal length and esthetic enhancement of the smile.

2. Removal of provisional restorations, dry and wet try-in of the laminate veneers on the prepared teeth.
3. The operative field was isolated with rubber dam for moisture control.
4. Dry try-in of the laminate veneers after rubber dam isolation to assess correct restoration seating even with a clamp;
5. For luting, the intaglio feldspar ceramic surfaces (Creation CC; Willi Geller International GmbH, Meiningen, Austria) were etched with 9.5% hydrofluoric acid for 60 seconds (Porcelain Etchant; Bisco, Schaumburg, IL), cleaned with 35% phosphoric acid for 15 seconds (Ultra Etch; Ultradent, South Jordan, UT), silanated (Bis-Silane; Bisco), and filled with hydrophobic adhesive (OptiBond FL; Kerr, Brea, CA).
6. Enamel was etched with 35% phosphoric acid gel for 30 seconds and the same adhesive was used.
7. Compules of restorative composite resin Estelite Omega, shade BL2 (Tokuyama, Tokyo, Japan) were preheated to 156°F/69°C for 10 minutes (HotSet warmer, Technolife; Joinville, SC, Brazil) and used as luting material. The composite was applied to the veneers with Centrix syringe. Restorations were positioned on prepared teeth, and seating by hand pressure was applied.
8. Initial removal of composite resin excesses.
9. Ultrasonic activation applied over ceramic with ultrasonic unit and polyacetal tip at 40% power (Dentsurg; CVDentus, São José dos Campos, SP, Brazil) to further increase composite flowability and reduce film thickness. More excesses are removed on this stage.
10. Light-curing under pressure for 60 seconds on each face (20 seconds × 3, with intervals of 10 seconds between applications);
11. Additional 10 seconds marginal light-curing using water-soluble gel to reduce oxygen-inhibited layer;
12. Finishing with scalpel blades and polishing with diamond polishers (D.Fine; Clinician's Choice, New Milford, CT).

## Summary

This article proposes a predictable clinical sequence for using preheated restorative composite resin to lute indirect restorations. This technique may reduce

degradation, wear, ditching, and discoloration at the restorative margins by improving the mechanical properties of the luting material at the adhesive interface.

## Discussion

Preheated restorative composite resin may be considered an excellent clinical option for luting ceramic laminate veneers due to its improved mechanical properties,<sup>26-33</sup> but care should be taken when choosing this technique as it needs training and adequate apparatus, and sequence to achieve a temperature (156°F/69°C) for decreasing viscosity and allowing predictable seating. Preheating approaches that do not provide predictable seating of the restoration should be avoided. Wrong composite resin selection and/or not reaching the ideal temperature could jeopardize the quality of the restorative composite resin flowability. Associated with the preheating of the composite, the use of ultrasonic device over the ceramic allows more accurate fitting of these restorations, reducing film thickness, thus this instrument should be considered when using the technique.<sup>34</sup> To ensure the final restoration seating, initial photopolymerization should be carried out under pressure. This ensures that the restoration will not dislocate, which would generate marginal misfit and greater need for occlusal adjustments.<sup>36</sup>

Choosing a composite with poor response to preheating may prevent optimal flowability and proper seating of restorations. Some restorative composites have been shown to be contraindicated as luting agent, as they provide unacceptable film thickness.<sup>34,36</sup> In contrast, there are composites resins with a high amount of inorganic fillers, excellent mechanical and optical properties that could be indicated for the luting technique using preheated composite. A proper selection would not compromise the restoration seating<sup>35</sup>, provide adequate film thicknesses<sup>34</sup> and could be beneficial to lute an indirect restoration, reaching lower marginal degradation, greater color stability, and greater mechanical strength.<sup>26-33</sup>

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## Figure Legends



Fig. 1. Dry try-in test is performed to assess the fitting of laminate veneers, including marginal adaptation and insertion axis of each restoration.

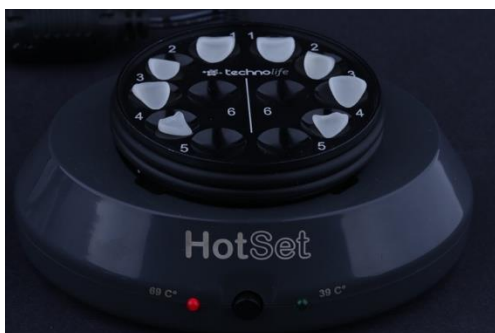


Fig. 2. Feldpar ceramic laminates on the preheating tray. Veneers were previously acid-etched, silanated and treated with adhesive.



Fig. 3. Ceramic laminate veneer was loaded with composite resin before preheating to 69°C/156°F for 10 minutes.



Fig 4. Application of adhesive system on teeth surfaces, excesses should not be removed at this stage and adhesive should not be photopolymerized.



Fig. 5. Removal of restorative composite excesses right after initial seating of the veneer. Excess removal is easy to perform because the preheated composite acquires higher viscosity within seconds after seating.



Fig. 6. Use of ultrasound polyacetal tip applied from incisal edge to cervical as an auxiliary mean to improve flowability of the restorative composite resin and reduce luting agent film thickness.



Fig. 7. Photopolymerization of composite resin using hand pressure to ceramic veneer to ensure best seating.



Fig. 8. Finishing with scalpel blades followed by polishing of restorative composite resin at margins using diamond polishers.

## 4 Capítulo 3<sup>3</sup>

Ceramic laminate veneers luted with preheated resin composite: A 10-year clinical report

*Running title: Ceramic veneers luted with preheated composite*

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### Abstract

Background: Resin cement and preheated restorative resin composite may be used for luting laminate veneers. Main advantage of resin composite is increased wear resistance, which could lead to better marginal performance in long term.

Setting: This article reports a clinical treatment with feldspar laminate veneers luted to maxillary teeth with preheated resin composite in a private practice. Case was finalized in May, 2009 and followed by 10 years.

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<sup>3</sup> Artigo aceito para publicação no periódico Contemporary Clinical Dentistry (Anexo B).

Results: Excellent clinical service and remarkable long-lasting marginal integrity were observed after 123 months. Scanning electron microscopy analysis showed no wear, gaps, or ditching at the margins. Restorative margins showed smooth transition between ceramic and tooth with no signs of degradation.

Conclusion: Preheated resin composite for luting ceramic laminate veneers may be considered an excellent clinical option.

Keywords: dental veneers; dental porcelain; resin composites; longevity; scanning electron microscopy.

## Introduction

Ceramic laminate veneers are widely used for esthetic restorations. Clinical studies report survival rates above 80 % in up to 20 years of follow up.<sup>[1-3]</sup> In addition to ceramic cracking, chipping and fractures, main reported reasons for failures of ceramic laminate veneers are related to marginal adaptation, integrity, and/or discoloration.<sup>[1-3]</sup> It is known that patient specific risks and variables influence the success of laminate veneers. For instance, smoking and the presence of endodontic treatment have been associated with increased marginal discoloration.<sup>[1,4]</sup> Marginal failures also could be associated with the resin-based luting agent used. A recent prospective trial of laminate veneers up to 11 years reported low rates of marginal failures.<sup>[4]</sup> It is speculated that such a finding is explained by the use of preheated resin composite to lute the laminate veneers, but that was not the focus of the study. The report by Friedman<sup>[5]</sup> is likely the first on the use of restorative resin composite as luting agent, but no preheating was described by the author. Preheating is necessary to reduce viscosity and film thickness,<sup>[6]</sup> which are of particular importance for thin restorations. As compared with resin cements, restorative composites have the advantage of increased filler loading, wear resistance and mechanical strength. Less marginal ditching has also been suggested.<sup>[7]</sup> These characteristics, in the long term, could reflect in less marginal problems and staining. The objective of this article is to report a clinical treatment in which ceramic laminate veneers were luted to maxillary anterior teeth with preheated resin composite and showed excellent clinical service and remarkable marginal integrity after 123 months of follow up.

## Clinical Report

The CARE guideline was used for this report.<sup>[8]</sup> A 28-years old female patient had a complaint about esthetics in her maxillary anterior teeth. The six maxillary anterior teeth had complete or partial resin composite veneers including a diastema closure (Fig. 1A). Restorations had problems of chipping and minor fractures, staining, surface roughness and texture, and loss of surface gloss (Figs. 1B, 1C). The anamnesis appointment took place in May 2009. The patient reported that the treatment had been finalized six months before and asked for longer-lasting restorations. Use of ceramic laminate veneers was proposed for eight maxillary teeth to widen the buccal corridor and because the first premolars had gingival recession. Potential risks were discussed with the patient, who agreed with the treatment. A double impression technique with polyvinylsiloxane – PVS (Panasil Putty and Light, Kettenbach, Eschenburg, Germany) was made for obtaining stone cast models, from which the occlusion was analyzed on articulator and a diagnostic waxing was created. Tooth preparation was carried out with K0082 Magne bur system (Brasseler, Georgetown, GA) over the direct resin composites with little (if any) extension into the underlying enamel. Refining was carried out ultrasonically with diamond tips (T9 and T10; Sonicflex, KaVo, Biberach, Germany). Figure 1D shows the definitive teeth preparations. A double impression with PVS (Panasil) was made. Mockup and provisional restorations were created with acrylic resin (New Outline; Anaxdent, Stuttgart, Germany).

Feldspar laminate veneers (IPS d.SIGN; Ivoclar Vivadent, Schaan, Liechtenstein) with thicknesses between 0.2 and 0.4mm were created by using a layering technique (Figs. 2A, 2B). For luting, the intaglio ceramic surfaces were etched with 9.5 % hydrofluoric acid for 60 seconds (Porcelain Etchant; Bisco, Schaumburg, IL), cleaned with phosphoric acid for 15 seconds (Ultra Etch; Ultradent, South Jordan, UT), silanated (Bis-Silane; Bisco), and filled adhesive from a 3-step system (OptiBond FL; Kerr, Brea, CA) was applied. The operative field was isolated by using a modified rubber dam technique. Enamel was etched with phosphoric acid gel for 30 seconds and the same adhesive used. Compules of resin composite Filtek Z250, shade A1 (3M ESPE, St. Paul, MN) were preheated to 68 °C for 10 minutes (Calset warmer; AdDent, Danbury, CT) and used as luting material. The composite was applied to the veneers with Centrix syringe, restorations were positioned on prepared teeth, and seating hand pressure applied. Excess resin composite was removed and photoactivation was

carried out for 60 seconds with a LED unit (Radii 2; SDI, Bayswater, Australia). Finishing was carried out with scalpel blades and polishing with diamond polishers (D.Fine; Clinician's Choice, New Milford, CT). Figures 3A and 3B show clinical pictures after luting (same day).

After 21 days, occlusion was rechecked and the treatment was finalized. The patient returned for follow up appointments after every 18 to 24 months. The last follow up visit was in June 2019, 123 months the treatment was finalized. Pictures and a PVS impression were made (Elite Putty and Regular; Zhermack, Badia Polesine, Italy). The mold was poured with epoxy resin (Fiberglass, Porto Alegre, Brazil) for observation of the restorations by using scanning electron microscopy – SEM (JSM6610; Jeol, Tokyo, Japan). The biological, esthetic, and mechanical success of the treatment was clinically evident (Figs. 4A, 4B). Figure 5 presents an overlapping between clinical and SEM pictures to show that the restorative margins had no gaps nor signs of deterioration, marginal ditching, wear, or staining 2. SEM images of the laminate veneer bonded to maxillary right central incisor (Figs. 6A, 6B) show the integrity of tooth-composite-ceramic interface after 123 months of clinical service. No wear, gaps, or any signs of degradation were observed at the margins, which showed a smooth transition between substrates. A cone-beam computed tomography image of the same tooth (Fig. 6C) showed excellent adaptation of the laminate veneer; one can also notice the thickness of resin composite layer at the bonded interface. Both patient and dentist were well satisfied with the excellent, long-lasting results. The patient signed an informed consent term to allow reproduction of images.

## Discussion

Reports on the use of preheated resin composite as luting agent for laminate veneers are available, but this is the first with a clinical follow up time longer than 5 years and with a close analysis on marginal integrity. Exceptional long-term biological, esthetic, and mechanical results were observed, notably regarding the absence of any marginal deterioration and maintenance of a smooth ceramic-tooth transition. The same could happen for other restoration types, provided that the restoration allows adequate light transmission for photopolymerization. Benefits of resin composites over resin cements as luting agents include more shades available, lower polymerization shrinkage/stress, and improved mechanical strength.

The main shortcoming usually reported for preheated resin composites is higher film thickness. A recent study showed that selection of resin composite should consider its response to preheating since viscosity, flowability, and even the reinforcing effect provided to thin ceramic structures are material dependent.<sup>[6]</sup> Since that information was not available at the time the present treatment was conducted, perhaps the resin composite used was not the best in terms of response to preheating. That did not preclude an excellent marginal and internal adaptation, and a long-lasting clinical service. One should note that an optimal preheating temperature (68°C) and time (10 minutes) were used, different preheating approaches could lead to distinct results. Maintaining the temperature and gained flowability is a challenge because heat dissipation occurs fast after preheating is ceased. Heating devices also offer the possibility of warming up the ceramic laminate veneers, which could reduce heat dissipation. In addition, up-to-date clinical luting approaches with preheated resin composite include an ultrasonic activation step to further increase flowability and reduce film thickness. Taking all into account and considering the excellent long-term clinical service reported here, preheated resin composite may be considered an excellent clinical option for luting ceramic laminate veneers.

## Conclusion

Preheated resin composite for luting ceramic laminate veneers may be considered an excellent clinical option since no signs of marginal degradation or staining were observed after 10-years of clinical service. The smooth marginal transition between ceramic, luting agent, and tooth and the absence of marginal gaps and ditching indicate that the restorative resin composite was able to withstand the abrasive and surface challenges imposed by the oral environment in the long term.

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Figure 1. A, Patient had complete or partial resin composite veneers in maxillary anterior teeth including diastema closure. Restorations had problems including chipping, fractures, staining, surface roughness and texture, and loss of surface gloss. B, Patient smile with lips and cheeks retracted. C, Maxillary teeth with a black background. D, Low-invasive teeth preparations.

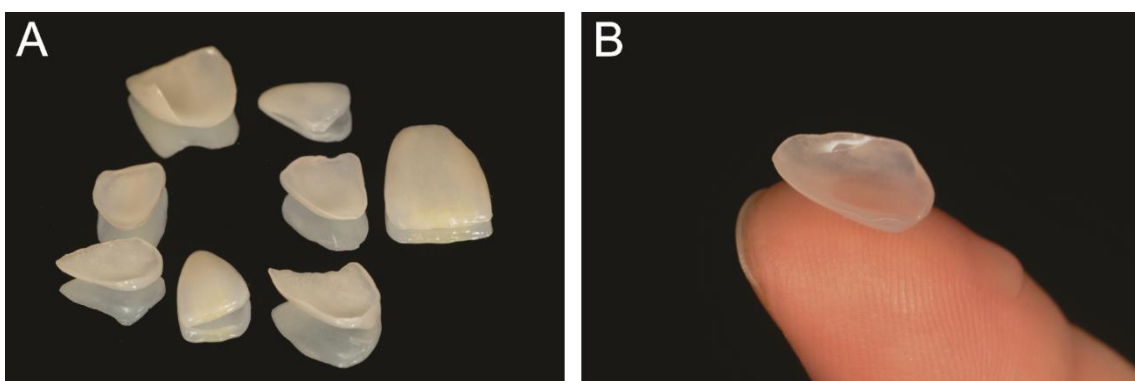


Figure 2. A, Laminate feldspar ceramic veneers (IPS d.SIGN) with thicknesses between 0.2 and 0.4 mm were created by using layering technique. B, Translucent, thin aspect of restoration.

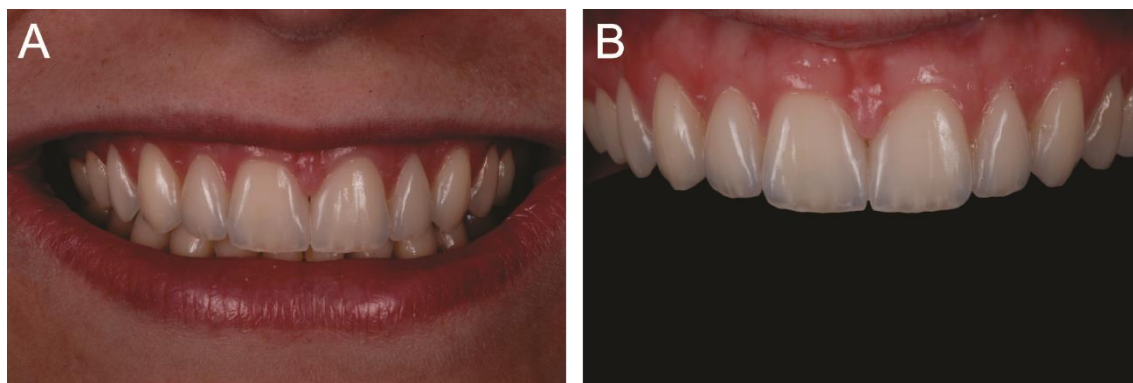


Figure 3. A, Clinical aspect of ceramic laminate veneers after luting to prepared teeth with preheated resin composite (same day of luting). 3. B, Maxillary teeth with black background.

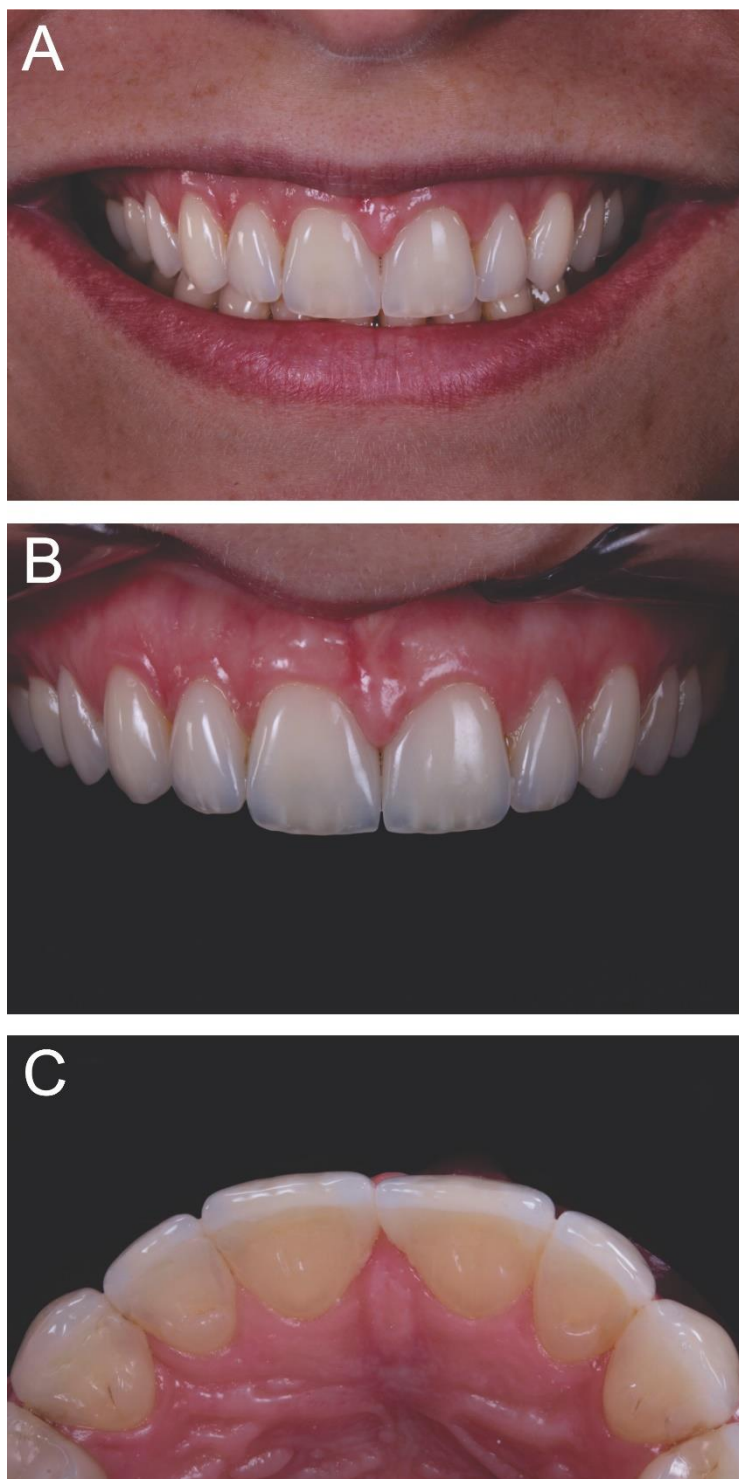


Figure 4. A, Ceramic laminate veneers showed remarkably good clinical performance and aspect after 123 months of clinical service, with no signs of marginal deterioration, marginal ditching, or staining. B, Maxillary teeth with black background. C, Palatal view of maxillary anterior teeth.



Figure 5. Overlapping between clinical and scanning electron microscope images ( $\times 12$ ). Restorative margins showed no gaps nor signs of deterioration, wear, ditching, or staining during 123 months of clinical service.

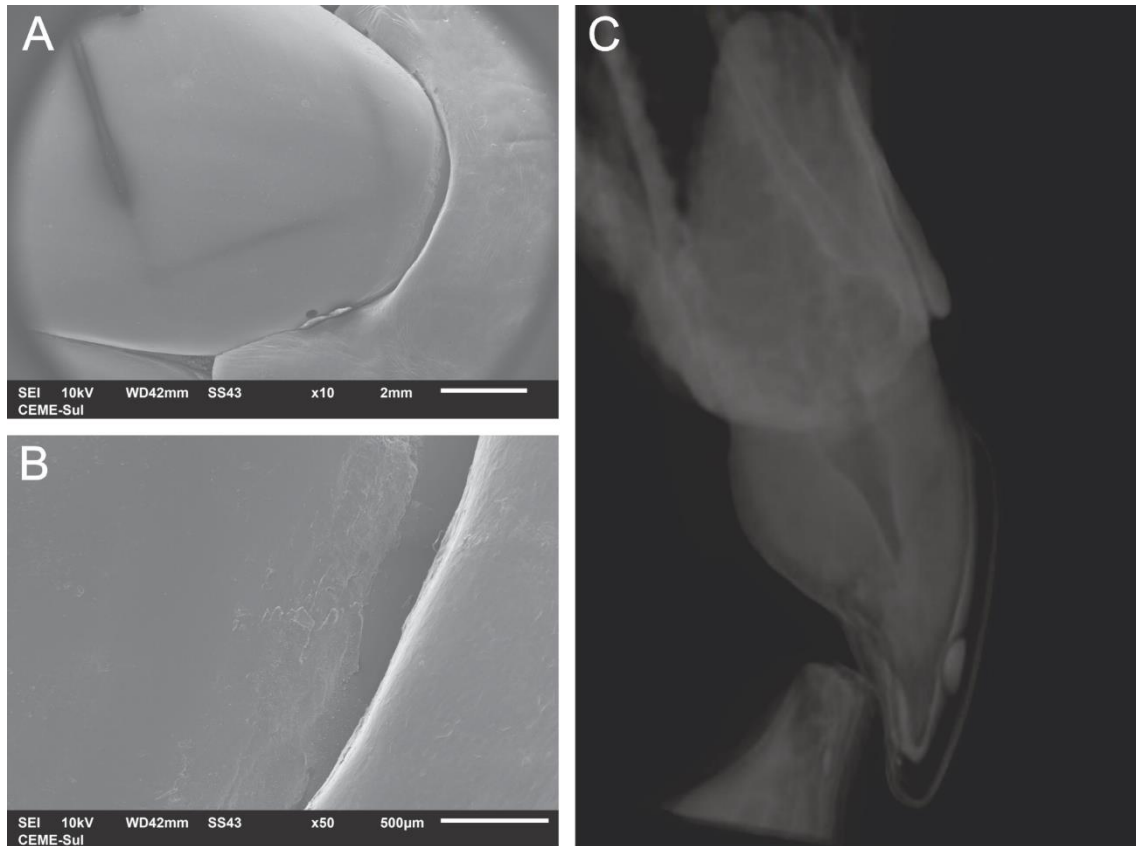


Figure 6. A, Scanning electron microscope images of ceramic veneer bonded to maxillary right central incisor after 123 months of clinical service. B, Tooth-resin composite-ceramic interface had no wear, gaps or any sign of degradation, with smooth transition between substrates. Original magnification A,  $\times 10$ ; B,  $\times 50$ . C, Cone beam computed tomography image of same tooth showing excellent adaptation of ceramic veneer.

## 5 Considerações Finais

Os resultados dos estudos aqui apresentados fornecem uma visão inicial sobre a possibilidade da utilização de resinas compostas de uso convencional como agente cimentante de restauros indiretos. Embora alguns pontos ainda possam ser uma incógnita para decisão de qual o melhor material restaurador pode ser utilizado como material cimentante de restaurações indiretas, o nosso estudo verificou em três fases tanto os materiais mais adequados, como também realizamos a descrição da técnica e o relato de caso clínico de long prazo.

O primeiro artigo deste estudo investigou a viscosidade (reologia), a cinética térmica de 10 resinas compostas restauradoras pré-aquecidas selecionadas e o efeito da energia do ultrassom na espessura do filme, concluindo que resinas compostas restauradoras com formulações distintas reagem de forma diferente ao pré-aquecimento, afetando a viscosidade e a espessura do filme. Isso sugere a necessidade de cuidado na escolha do material. Também identificamos que o tempo ideal de trabalho do compósito pré-aquecido é curto e devemos adequar a sequência de cimentação para aproveitar as altas temperaturas encontradas nos primeiros 15 s. Sobre a aplicação de ultrassom, percebemos que é eficaz na redução da espessura do filme e pode auxiliar resinas compostas restauradoras a obter filmes abaixo de 50  $\mu\text{m}$ . Assim, entendemos que o desempenho geral da técnica de resina composta de pré-aquecida depende da seleção adequada do material e que é aplicável como material cimentante, assim como da técnica de aplicação.

Para isso, no segundo artigo descrevemos as fases clínicas da cimentação com material resinoso restaurador, identificando os principais pontos que diferem da aplicação com cimentos resinosos comumente utilizados para cimentação de restauros indiretos. Espera-se que este relato de técnica possa auxiliar dentistas que estão iniciando o uso da técnica, abordando aspectos relevantes da sequência clínica de aplicação. Já no terceiro artigo, realizamos um estudo clínico de avaliação longitudinal de 11 anos de uma paciente que possui 10 laminados cerâmicos que foram cimentados com material restaurador, avaliamos não somente a estética dos restauros, como também as margens da intersecção entre dente, material cimentante e restauro indireto através de uma análise de superfície com microscopia eletrônica

de varredura. Embora seja um relato de caso, indica que a técnica pode gerar uma linha de cimentação e adaptação marginal resistentes ao desgaste clínico em longo prazo, sendo o relato mais antigo presente na literatura. De forma geral, os estudos apresentados nesta tese abordam aspectos e dúvidas relevantes na aplicação contemporânea de resinas compostas pré-aquecidas como material de cimentação, abrindo espaço para futuros estudos laboratoriais e clínicos para ajudar na consolidação da técnica.



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## **Apêndices**

## **Apêndice A – Nota da Tese**

### **Resina composta restauradora pré-aquecida como agente de cimentação adesiva de restaurações indiretas**

#### ***Preheated restorative resin composite as adhesive luting agent of indirect restorations***

O dia-a-dia de dentistas envolve restaurar dentes de seus pacientes por motivos diversos, como cárie, fraturas e estética. Uma das opções de material para restauração são materiais cerâmicos, conhecidos como porcelanas. Para unir a porcelana aos dentes são usados materiais resinosos fluidos, que podem sofrer desgaste na boca. Este trabalho estudou a possibilidade de trocar esses materiais fluidos por materiais mais resistentes para aprimorar os resultados clínicos. Estes materiais mais resistentes precisam ser aquecidos antes de seu uso para que fiquem fluidos também e possam ser usados na colagem da porcelana. Este trabalho mostrou que esta técnica diferente é uma excelente opção para dentistas, mas que a escolha do material para colagem precisa ser feita com cautela pois nem todos ficam bem fluidos quando aquecidos, o que pode atrapalhar o procedimento.

**Campo da pesquisa:** Odontologia restauradora.

**Candidato:** Rogério Luiz Marcondes, cirurgião-dentista pela Pontifícia Universidade Católica do Paraná (1997).

**Data da defesa e horário:** 01/10/2021 às 14h

**Local:** Ambiente virtual Google Meet.

**Membros da banca:** Prof. Dr. Rafael Ratto de Moraes, Prof. Dr. Gregori Franco Boeira, Prof. Dr. Leandro Augusto Hilgert, Prof. Dr. Tiago Veras Fernandes, Profa. Dra. Giana da Silveira Lima (Suplente) e Profa. Dra. Priscilla Cardoso Lazari (Suplente)

**Orientador:** Prof. Dr. Rafael Ratto de Moraes

**Co-orientador:** Prof. Dr. Marco Aurélio de Carvalho

**Informação de contato:** Rogério Luiz Marcondes, Email: Dr.rogeriomarcondes@gmail.com.

## **Apêndice B – Súmula do currículo do candidato**

### **Súmula do currículo**

Rogério Luiz Marcondes nasceu em 16 de julho de 1973, em Curitiba, Paraná. Coursou o ensino médio no Colégio Dom Bosco, na mesma cidade. Em 1993 ingressou na Faculdade de Odontologia da Pontifícia Universidade Católica do Paraná (PUC-PR), tendo sido graduado como cirurgião-dentista em julho de 1997. Durante o período de graduação, foi monitor das disciplinas Dentística I e II durante 3 semestres. Entre 1999 e 2000, frequentou como estudante convidado o programa de Fellowship em Reabilitação Oral da Universidade Estadual de Ohio, EUA sob orientação do Professor Stephen Rosenstiel, em 2006 finalizou o aperfeiçoamento em próteses sobre implante no Instituto Latino Americano de Pesquisa e Ensino Odontológico, Brasil. Em 2008, concluiu especialização em dentística restauradora pela Força Aérea Brasileira. Em 2013, tornou-se Presidente da Sociedade Brasileira de Odontologia Estética. No ano de 2017, ingressou no doutorado no Programa de Pós-Graduação em Odontologia da Universidade Federal de Pelotas e desenvolveu trabalhos na linha de pesquisa com materiais resinosos aquecidos e utilização de aparatos ultrassônicos com coadjuvantes no assentamentos de restauros indiretos.

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## **Anexos**

## Anexo A – Capa do artigo oriundo do Capítulo 1

DENTAL MATERIALS 36 (2020) 1356–1364

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### Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness

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ARTICLE INFO

**Keywords:**  
Luting  
Temperature  
Flowability  
Ultrasonics  
Resin cement  
Flowable resin composite

ABSTRACT

**Objective.** This study investigated viscosity and thermal kinetics of 10 selected preheated restorative resin composites and the effect of ultrasound energy on film thickness.

**Methods.** A range of different resin composites was tested: Charisma Diamond, IPS Empress Direct, Enamel Plus HRI, Essentia, Estelite Omega, Filtek Z100, Filtek Z350 XT, Gradia, TPH Spectrum and VisCalor. A flowable resin composite (Opallis Flow) and two resin cements (RelyX Veneer, Variolink Esthetic LC) also were tested. Viscosity (Pa s) was measured at 37 °C and 69 °C (preheating temperature) using a rheometer. Film thickness (μm) was measured before and after application of ultrasound energy. Temperature loss within resin composite following preheating (°C/s) was monitored. Data were statistically analyzed ( $\alpha = 0.05$ ).

**Results.** Viscosity at 69 °C was lower than at 37 °C for all materials except the flowable resin composite. Preheating reduced viscosity between 47% and 92% for the restorative resin composites, which were generally more viscous than the flowable materials. Film thickness varied largely among materials. All preheated resin composites had films thicker than 50 μm without ultrasound energy. Application of ultrasound reduced film thickness between 21% and 49%. Linear and nonlinear regressions did not identify any relationship between filler loading, viscosity, and/or film thickness. All materials showed quick temperature reduction following preheating, showing maximum temperature loss rates after approximately 10 s.

**Significance.** Distinct restorative resin composites react differently to preheating, affecting viscosity and film thickness. The overall performance of the preheating technique depends on proper material selection and use of ultrasound energy for reducing film thickness.

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<https://doi.org/10.1016/j.dental.2020.08.004>  
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## Anexo B – Capa do artigo oriundo do Capítulo 3

### Case Report

## Ceramic Laminate Veneers Luted with Preheated Resin Composite: A 10-Year Clinical Report

### Abstract

Resin cement and preheated restorative resin composite may be used for luting laminate veneers. The main advantage of resin composite is increased wear resistance, which could lead to better marginal performance in long term. This article reports a clinical treatment with feldspar laminate veneers luted to the maxillary teeth with preheated resin composite in a private practice. Case was finalized in May 2009 and followed by 10 years. Excellent clinical service and remarkable long-lasting marginal integrity were observed after 123 months. Scanning electron microscopy analysis showed no wear, gaps, or ditching at the margins. Restorative margins showed a smooth transition between ceramic and tooth with no signs of degradation. Preheated resin composite for luting ceramic laminate veneers may be considered an excellent clinical option.

**Keywords:** Dental porcelain, dental veneers, longevity, resin composites, scanning electron microscopy

### Introduction

Ceramic laminate veneers are widely used for esthetic restorations. Clinical studies report survival rates above 80% in up to 20 years of follow-up.<sup>[1-3]</sup> In addition to ceramic cracking, chipping and fractures, the main reported reasons for failures of ceramic laminate veneers are related to marginal adaptation, integrity, and/or discoloration.<sup>[1-3]</sup> It is known that patient-specific risks and variables influence the success of laminate veneers. For instance, smoking and the presence of endodontic treatment have been associated with increased marginal discoloration.<sup>[1,4]</sup> Marginal failures also could be associated with the resin-based luting agent used. A recent prospective trial of laminate veneers up to 11 years reported low rates of marginal failures.<sup>[4]</sup> It is speculated that such a finding is explained by the use of preheated resin composite to lute the laminate veneers, but that was not the focus of the study. The report by Friedman<sup>[5]</sup> is likely the first on the use of restorative resin composite as a luting agent, but no preheating was described by the author. Preheating is necessary to reduce viscosity and film thickness,<sup>[6]</sup>

which are of particular importance for thin restorations. As compared with resin cements, restorative composites have the advantage of increased filler loading, wear resistance, and mechanical strength. Less marginal ditching has also been suggested.<sup>[7]</sup> These characteristics, in the long term, could reflect in less marginal problems and staining. The objective of this article is to report a clinical treatment in which ceramic laminate veneers were luted to the maxillary anterior teeth with preheated resin composite and showed excellent clinical service and remarkable marginal integrity after 123 months of follow up.

### Clinical Report

The CARE guideline was used for this report.<sup>[8]</sup> A 28-year-old female patient had a complaint about esthetics in her maxillary anterior teeth. The six maxillary anterior teeth had complete or partial resin composite veneers including a diastema closure [Figure 1a]. Restorations had problems of chipping and minor fractures, staining, surface roughness and texture, and loss of surface gloss [Figure 1b and c]. The anamnesis appointment took place in May 2009. The patient reported that the treatment had been finalized 6 months

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Submitted : 04-Sep-2020  
Revised : 10-Oct-2020  
Accepted : 25-Oct-2020  
Published : \*\*\*

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Website:  
[www.contempdent.org](http://www.contempdent.org)  
DOI: 10.4103/ood.ood\_788\_20

Quick Response Code:



How to cite this article: Marcondes RL, Lima VP, Isolan CP, Lima GS, Moraes RR. Ceramic laminate veneers luted with preheated resin composite A 10-year clinical report. Contemp Clin Dent 2020;XX:XX-XX.

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