

UNIVERSIDADE DE BRASÍLIA

Faculdade de Ciências da Saúde

Programa de Pós-Graduação em Odontologia



Tese de Doutorado

Influência do pré-aquecimento nas propriedades das resinas compostas

Déborah Lousan do Nascimento Poubel

Brasília, 20 de junho de 2022

Déborah Lousan do Nascimento Poubel

Influência do pré-aquecimento nas propriedades das resinas compostas

Tese apresentada ao Programa de Pós-Graduação em Odontologia da Faculdade de Ciências da Saúde da Universidade de Brasília, como requisito parcial à obtenção do título de doutor em Odontologia.

Orientador: Fernanda Cristina Pimentel Garcia

Brasília, 2022

Déborah Lousan do Nascimento Poubel

Influência do pré-aquecimento nas propriedades das resinas compostas

Tese aprovada, como requisito parcial para obtenção do grau de Doutor em Odontologia, Programa de Pós-Graduação em Odontologia da Faculdade de Ciências da Saúde da Universidade de Brasília.

Data da defesa: 27 de maio de 2022

Banca examinadora:

Dra. Fernanda Cristina Pimentel Garcia (Presidente)

Dr. Paulo Henrique Perlatti D'alpino

Dr. Fabrício Luscino Alves de Castro

Dr. Bruna Genari Degrazia

Dr. Lucas Fernando Tabata

“Quem tem medo de crescer, morre pequeno”.

Dedico não só esse trabalho, mas a minha vida ao meu pai.

AGRADECIMENTOS

Antes de começar, deixo clara a minha justificativa de escolher uma carta para mim mesma em forma de agradecimento. Isso porque, em algum momento no futuro, eu irei folhear esse trabalho novamente e nada mais justo que haja algumas palavras carregadas de lembranças e sentimentos para que eu relembre minha trajetória e todas as pessoas que me acompanharam até aqui.

Déborah,

Eu poderia começar a falar desse projeto que durou 5 anos, mas antes, quero relembrar em poucas palavras o que nos trouxe aqui e como chegamos. Você cresceu em Taguatinga, um bairro periférico de Brasília, juntamente com seus pais e dois irmãos mais velhos. Antes mesmo de entrar na escola, você se atrevia a estar junto quando seus irmãos estavam aprendendo algo e acabou dando uns pulinhos na etapa da alfabetização. Você não só aprendeu a ler e escrever totalmente na escola pública, você fez parte disso durante toda a sua jornada colegial e, acredite, isso não te ensinou só o conhecimento básico, como muitos princípios que você carrega até hoje. Lá pra sexta série, lembro de você afirmar com todas as letras: “vou fazer faculdade na federal” e seus amigos questionaram. Como é que você ia chegar lá vindo de um bairro tão escondido, se levamos em consideração a magnitude do país que vivemos?

De fato, até pelas condições de estudo e vida, você fez faculdade particular e isso não tirou em nada o mérito dessa caminhada. Eu vi você lá estudando, se empenhando, aprendendo, porque não havia outra opção, era preciso aproveitar essa oportunidade de estar em uma graduação, nossa família não pertencia a esse lugar desde sempre. Você se graduou, se pós-graduou, fez diversos cursos e se tornou mestre na Federal que você sonhava. Não dá pra dizer que você não tinha um plano.

A chance do doutorado surgiu e você agarrou isso também. E, por não acreditarmos puramente em meritocracia, aqui, seria impossível não mencionarmos como tanta gente foi agregada a sua vida profissional e pessoal durante todas essas

etapas. A Fernanda, sua então orientadora, te deu uma coisa muito difícil de conseguir, credibilidade e confiança. Seria impossível não sermos extremamente gratas por isso. Não é só um curso, um mestrado, um doutorado, é uma oportunidade, é uma chance de você trilhar um caminho melhor pra você, sua família e sua história. Você conheceu profissionais admiráveis, pesquisadores incríveis, clínicos invejáveis e tudo isso foi pautando a profissional que você é hoje e, mais do que isso, a profissional que você quer ser.

Quando a gente vê a jornada de cinco anos ser simplificada em poucas palavras, parece até que não foi tão difícil assim, mas você sabe o que passamos. E, às vezes, é preciso lembrar as adversidades para que as vitórias sejam devidamente reconhecidas. Logo no primeiro ano do doutorado, você perdeu uma das pessoas mais importantes da sua vida, seu pai. Isso te afetou profundamente e colocou em dúvida todas as suas prioridades. Nesse momento, você entendeu que você não é a sua profissão, você não é um título do seu currículo e isso não é a coisa mais importante do mundo, apesar de você amar o que faz e se esforçar muito para que isso seja uma vertente de orgulho na sua vida. E talvez isso tenha te dado um empurrãozinho para seguir em frente.

Nesse tempo, você trocou de emprego, deu aulas em diferentes níveis, se tornou empresária e passou por muitas provações no ambiente militar, principalmente quando era pra tentar administrar o tempo para poder fazer tudo o que você tinha se proposto. Por isso, precisamos reconhecer aqui o quanto foi difícil conciliar tudo isso, mas você cumpriu as disciplinas, desenhou um projeto que te fazia brilhar os olhos, qualificou e quando achou que tudo estava indo da forma mais redonda possível, nós passamos por uma pandemia. Não tinha como continuar os testes, a educação foi posta em cheque, a humanidade foi questionada, a empatia foi brutalmente encolhida e, ainda assim, foi preciso tentar enxergar a linha de chegada para não parar. Você se reinventou mais uma vez, fez o que era possível, bolou outro plano, teve apoio de mais um tanto de gente para chegar onde está hoje, finalizando esse capítulo. O doutorado te ensinou muita coisa, mas muito mais do que o tecnicismo, ele te ensinou a lidar com as frustrações de depender do apoio e decisão de terceiros, mas principalmente com as suas próprias. Nem sempre nossos planos são seguidos na íntegra e a gente precisou fazer umas anastomoses no caminho que a gente trilhou.

Sua vida profissional por muitos anos foi a parte mais importante da sua vida, era a face que te definia e, também acho que o que estamos colhendo hoje, foi fruto dessa dedicação. Mas, hoje, acima disso, o seu tempo é a coisa mais importante, as pessoas são seu alicerce e o amor é o fundamento para sua vida. Que isso nunca deixe de ser assim e eu tenho certeza que ainda teremos uma caminhada incrível. E, por falar em amor, que rede de apoio incrível você sempre teve. Desde seus pais, irmãos e família que não mediram esforços para que você conseguisse alcançar todos os degraus que vislumbrou, como seus amigos que estiveram com você ouvindo todas as reclamações, tentando achar soluções juntos para os problemas e, na maioria das vezes, rindo muito. E, não seria nem possível mencionar tudo isso, sem reconhecer o parceirão de vida que você tem. Seu marido que muito além de um par romântico, é uma fortaleza que não te deixa desmoronar. Nós não seríamos nada do que somos hoje sem essas pessoas.

No final das contas, o encerramento desse ciclo tem um significado muito importante para você. Não é como se fosse só um capítulo que está se fechando, é como se a gente estivesse acabando um livro inteiro, onde várias histórias paralelas aconteceram, e que dá aquela sensação de despedida, mas também de missão cumprida. Essa carta é como o epílogo, para que você sempre recorra a ela quando precisar se lembrar de que, apesar de todas as dificuldades, você seguiu em frente e sempre muito bem acompanhada. Sua jornada me dá um orgulho danado.

RESUMO

As resinas compostas são materiais restauradores versáteis e amplamente utilizados na odontologia, apresentam excelentes propriedades físicas e mecânicas, abrangendo uma vasta possibilidade estética e funcional. O pré-aquecimento dos compósitos tem sido investigado há alguns anos e tem demonstrado alguns benefícios clínicos e laboratoriais associados à técnica de cimentação. Embora seu uso esteja ganhando espaço, é necessário que o profissional tenha conhecimento sobre os fatores que podem interferir nos resultados esperados, buscando a maior longevidade para os elementos dentais. Com o objetivo de identificar as metodologias de aquecimento empregadas na técnica de cimentação com resinas compostas pré-aquecidas, uma revisão da literatura foi conduzida em plataformas de busca, identificando 304 artigos, dos quais 14 foram incluídos para análise. Além da revisão, um estudo *in vitro* foi executado para determinar o tempo necessário de aquecimento para resinas de diferentes marcas e com formulações distintas, bem como analisar o comportamento viscoelástico de cada resina sob diferentes temperaturas. O trabalho mostrou que o uso de aquecedores que mantenham as resinas sem umidade a uma temperatura entre 54 e 68°C podem ser utilizados. Ao serem submetidas a 60°C (± 4), foram necessários 10 minutos para que as resinas testadas alcançassem a temperatura pré-estabelecida e apresentassem redução de sua viscosidade inicial com valores entre 84 e 94%. No entanto, após a remoção dos materiais da fonte aquecedora, 48% da temperatura foi perdida após 1 minuto e, em apenas 5 minutos, todas as resinas testadas retornaram à temperatura ambiente e viscosidade inicial, limitando consideravelmente o tempo de trabalho. A técnica de pré-aquecimento tem sido vinculada a uma melhor adaptação marginal da restauração indireta devido ao escoamento do material às paredes cavitárias, promovido pela considerável redução de sua viscosidade e espessura adequada de película de cimentação. No entanto, fatores como a composição do material, forma de aquecimento, tempo de trabalho, características da restauração indireta e tempo de fotoativação podem influenciar diretamente na longevidade do procedimento e devem nortear a decisão do cirurgião-dentista quanto à escolha da técnica. Deste modo, estudos que simulem sua aplicação clínica, determinando o tempo de trabalho ideal e melhor protocolo de aplicação, com

enfoque na manutenção e melhora das propriedades dos materiais devem ser conduzidos para que a técnica alcance maior praticidade e eficiência.

ABSTRACT

The composite resins are versatile restorative materials widely used in dentistry, they have excellent physical and mechanical properties, covering a wide range of esthetic and functional possibilities. The preheating of composites has been investigated for some years and has demonstrated some clinical and laboratory benefits associated with the cementation technique. It is necessary that the clinician have knowledge about the factors that can interfere with the results. The research was performed by scanning papers that use preheated resins, and it has been found 304 articles, 14 of which were included for review. In addition to the review, a different *in vitro* study was well determined to determine the time marks required for resins with different formulations, such as studying the viscoelastic behavior under different temperatures. The work showed that the use of heaters can keep a resin free of moisture between 54 and 68°C. Upon reaching 8 minutes at the initial temperature at 60°C (± 4), the values at 80 minutes for resin-1 reduction were presented, the values reaching 8 its initial reduction at 60°C. However, after removing the materials from the heating source, 48% of the temperature was lost in the first minute, and after 5 minutes all the resins returned to the minute and minute temperature of the initial temperature, limiting themselves to considering the working time. The increase of the temperature improves a better marginal adaptation and better reduction of the thickness of the cement film. However, the composition of the material, the characteristics of the technique, and the time necessary for the procedure can directly influence the technique. In this way, new research can determine the ideal working time and simulation protocol, focusing on the application of materials and improving the technical properties, greater practicality, and efficiency.

SUMÁRIO

1	INTRODUÇÃO, REVISÃO DA LITERATURA E OBJETIVOS	12
1.1	INTRODUÇÃO	12
1.2	REVISÃO DA LITERATURA	14
1.2.1	Resinas Compostas.....	14
1.2.2	Influência do aquecimento nas resinas compostas.....	16
1.3	OBJETIVOS	18
1.3.1	Objetivo Geral.....	18
1.3.2	Objetivos Específicos	19
2	COMPOSITE RESIN PREHEATING TECHNIQUES FOR CEMENTATION OF INDIRECT RESTORATIONS	20
3	EFFECT OF PREHEATING ON THE VISCOSITY OF COMPOSITE RESINS ..	42
4	DISCUSSÃO GERAL E CONCLUSÕES	57
4.1	DISCUSSÃO GERAL	57
4.2	CONCLUSÕES	64
5	REFERÊNCIAS.....	65
6	PRESS RELEASE	71
7	APÊNDICES.....	72

1 INTRODUÇÃO, REVISÃO DA LITERATURA E OBJETIVOS

1.1 INTRODUÇÃO

As resinas compostas são consideradas materiais restauradores definitivos com ampla utilização na odontologia restauradora. Esses materiais são constituídos por uma matriz polimérica, à base de dimetacrilatos, partículas minerais de natureza inorgânica (quartzo, vidro ou sílica), agente de união, além de diluentes e outras partículas que conferem propriedades físicas e mecânicas adequadas e modulam a reação de polimerização, tornando-as um material versátil na odontologia adesiva. (1-4) Inicialmente, sua indicação estava restrita a procedimentos restauradores diretos, no entanto, sua utilização passou a ser investigada em procedimentos semidiretos e, até mesmo, indiretos, independente da região. (1) As resinas podem apresentar formulações distintas, variando em relação aos seus componentes, tamanhos de partículas, consistência de manipulação, características de polimento, valores de resistência, dentre outros aspectos. (1, 4, 5)

Esses materiais sofreram mudanças ao longo do tempo, principalmente no desenvolvimento de sistemas que sejam capazes de reduzir a contração de polimerização e melhorar a biocompatibilidade com o remanescente dentário. (1) Dependendo da formulação, podem variar entre si em relação à viscosidade, sendo possível distingui-las em regulares e fluidas, estas permitindo uma melhor adaptação pelo escoamento da resina no substrato dentário, principalmente em regiões pequenas ou de difícil acesso. (1, 6) Embora apresentem propriedades mecânicas e estéticas satisfatórias, as resinas compostas possuem limitações que não devem ser negligenciadas, como a contração de polimerização, resistência às tensões excêntricas oriundas da mastigação, grau de conversão, adaptabilidade, escoamento, manchamento, entre outros. (7)

O aquecimento de resinas compostas regulares tem sido investigado há anos. (8-11) Inicialmente sua proposta era aumentar a taxa de conversão submetendo restaurações extra orais de resina ao aquecimento tardio, após a fotopolimerização,

como uma etapa complementar. No entanto, seu uso passou a ser empregado como uma fase prévia à restauração com o objetivo de alterar a viscosidade do material, dessa forma, a técnica passou a ser aplicada sob resinas utilizadas para restaurar diretamente cavidades classe II. (10, 12) Quando utilizadas com esse objetivo, pode ser observada uma menor formação de *microgaps* nas regiões cervicais, devido ao maior escoamento, facilidade de inserção e adaptação às áreas mais críticas do preparo, (13, 14) demonstrando uma redução da infiltração marginal em relação à resina em temperatura ambiente. (14) Além disso, a técnica poderia ser capaz de superar as desvantagens apresentadas pelas resinas fluidas, uma vez que esses materiais são tipicamente produzidos a partir da redução do percentual da carga inorgânica (1, 7), o que pode acarretar em prejuízo das propriedades mecânicas e aumentar a possibilidade de falha, interferindo na longevidade das restaurações e limitando sua indicação. (1, 5, 6)

Devido sua redução da viscosidade frente ao aquecimento, as resinas compostas regulares passaram a ser cogitadas como agentes de cimentação. Os cimentos resinosos são comumente considerados o material de escolha para cimentações de restaurações, apresentando boas características de escoamento, espessura de película e adaptação. (15) No entanto, esses materiais podem ser menos resistentes à degradação marginal pela exposição da linha de cimentação em meio oral e menor resistência à fratura, quando comparados às resinas compostas regulares. (15, 16) Devido sua maior quantidade de partículas inorgânicas, apresentam vantagens mecânicas também em relação aos cimentos resinosos. (11, 17) Por esse motivo, protocolos alternativos têm sido sugeridos para combinar e melhorar ainda mais as propriedades das resinas compostas, como, por exemplo, o aquecimento desses materiais previamente ao procedimento cimentação. Isso porque são consideradas um material pseudoplástico, sendo possível alterar suas características de viscosidade, tornando-as mais fluidas ao submetê-las a temperaturas elevadas. (18) Dessa maneira, essa condição poderia melhorar a capacidade de adaptação, além de promover um aumento no grau de conversão de monômeros em polímeros, somando-se às vantagens já existentes desses materiais. (1, 5) Com resultados laboratoriais positivos e controle de casos por até 10 anos, o uso de resinas aquecidas poderia ser considerado uma excelente opção, onde aspectos biológicos excepcionais, estéticos

e mecânicos são observados, como aumento da resistência à sorção, melhor performance marginal a longo prazo, menor estresse de polimerização, além de maior disponibilidade de cores para mimetizar o elemento dentário. (12, 19, 20)

1.2 REVISÃO DA LITERATURA

1.2.1 Resinas Compostas

Atualmente, os compósitos odontológicos são constituídos basicamente por um monômero multifuncional capaz de formar uma rede de ligações cruzadas, se unindo a partículas inorgânicas revestidas por silano (1, 5, 7, 21, 22). Diluentes e outras moléculas como iniciadores, estabilizadores e pigmentos também estão presentes em suas formulações e, embora diferentes sistemas de polimerização tenham sido desenvolvidos, as resinas compostas atuais são consideradas como fotoativáveis. (18) A partir da ativação de iniciadores presentes por um determinado comprimento de luz visível e o consumo dos inibidores, os radicais livres dos monômeros ficam disponíveis para reagir com outras moléculas, formando um radical de cadeia que poderá continuar reagindo com outros monômeros consecutivamente até formar uma cadeia polimérica, sendo esse processo conhecido como polimerização. (1, 5, 7, 21, 22)

As resinas compostas podem ser moduladas a partir da disposição e quantidade dos componentes presentes em sua composição, por exemplo, o Bis-GMA, comumente encontrado, é um monômero resinoso de alto peso molecular e alta viscosidade, caracterizado por ser uma molécula de cadeia longa e rígida. (1-4, 18) Quanto maior for a extensão da molécula monomérica, menor será a contração de polimerização, entretanto, se o Bis-GMA estiver em alta concentração, maior será a quantidade de monômeros residuais resultantes da polimerização. (1, 5, 7, 21, 22) Resinas com alto teor de Bis-GMA são mais consistentes à manipulação e, para driblar

isso, podem ser incorporados diluentes, como o UDMA, monômero de peso molecular próximo ao Bis-GMA que apresenta baixa viscosidade e, por ser altamente flexível, produz menor contração de polimerização. (1, 5, 7, 21, 22) Por sua vez, o comonômero e diluente TEGDMA também é flexível e consegue se dobrar e reagir mais tardiamente, fazendo ligações inter e intramoleculares. (1, 5, 7, 21, 22) Se um diluente for incorporado em excesso com objetivo de facilitar a mobilidade dos monômeros e conseqüentemente a manipulação da resina, a contração de polimerização será maior. (18) A distância entre dois monômeros é de 0,3 a 0,4 nanômetros (nm), determinada pela distância de Van Der Waals, porém ao serem fotoativados, é estabelecida uma união covalente com distância de 0,15nm entre os monômeros, aumentando a densidade do material e originando uma contração volumétrica de aproximadamente 1,5 a 3% nas resinas compostas. (23)

A velocidade em que a reação de polimerização acontece também representa um aspecto importante, uma vez que, conforme a reação química continua, as cadeias poliméricas vão formando estruturas emaranhadas, tornando-as rígidas e fazendo com que as taxas de conversão monômero-polímero não atinjam sua totalidade, devido à rápida formação de um sólido densamente reticulado, provocando um aumento da viscosidade em toda a matriz orgânica e limitando a mobilidade dos monômeros ainda disponíveis. (1, 5, 7, 21, 22) Isso impede que os radicais continuem reagindo e suas ligações duplas aconteçam, resultando em uma grande diminuição na taxa de conversão. (18, 22) A conversão monomérica de uma resina composta comercial atinge valores em torno de 45 a 75%, dependendo da composição dos monômeros e das partículas de carga, tipo de fotoiniciador e do protocolo de fotoativação. (22) Uma porção significativa de monômeros não reagidos, ou seja, não convertidos ficam disponíveis na massa resinosa, afetando as propriedades mecânicas do material, em geral. Quanto menor o grau de conversão, pior será a dureza superficial, a resistência à flexão, o módulo de elasticidade, a tenacidade à fratura, a resistência à tração e ao desgaste. (9) Ademais, os monômeros residuais podem ser facilmente extraídos para a saliva num processo de lixiviação, além de estimular o crescimento bacteriano ao redor pela rugosidade superficial, aumentar a sorção e o manchamento, e contar com um considerável potencial alergênico. (1)

Com o objetivo de analisar e modular essas desvantagens, o aquecimento das resinas compostas foi proposto há aproximadamente 16 anos e tem ganhado enfoque principalmente nos últimos anos, com base nos resultados laboratoriais favoráveis, como o aumento do grau de conversão, menor sorção e degradação marginal. (5, 8, 10, 22, 24-26).

1.2.2 Influência do aquecimento nas resinas compostas

Inicialmente o uso do calor era realizado de forma complementar ao processo de polimerização em restaurações extraorais, ou seja, após a fotoativação e conversão total possível dos monômeros. Estudos que avaliaram a relação entre o aquecimento tardio e o grau de conversão de materiais resinosos como resinas e cimentos são capazes de mostrar o aumento significativo desses valores, permitindo uma conversão final aumentada. (21, 22, 27-30)

Seguindo essa mesma lógica, diversos estudos foram conduzidos a fim de avaliar a influência do aquecimento de resinas compostas e cimentos resinosos previamente à fotopolimerização nesses mesmos aspectos. (21, 22, 27-29) Ao serem comparados à temperatura ambiente (22°C), o aquecimento prévio a 60°C foi capaz de provocar um aumento do grau de conversão em 38% na base de restaurações de 2 milímetros (mm) de profundidade e de até 36% quando se avaliou a superfície. (5, 8, 22) A influência térmica, nesse caso, exige menos energia para quebrar as ligações duplas de carbono (C=C) e permite que a fotoativação seja realizada por tempos menores, além de favorecer a propagação da reação por um período mais longo antes do ponto de geleificação. (5, 8, 22). Por sua vez, resinas compostas armazenadas em refrigeradores devem ser trazidas à temperatura ambiente, pois sua taxa de conversão pode chegar somente a 10%, demonstrando mais uma vez a influência da temperatura em relação às propriedades desses materiais. (8, 9)

Outros tópicos também têm sido investigados, como resistência flexural, resistência de união, contração volumétrica, alteração de cor, selamento marginal, propensão à microinfiltração e espessura de película. (11) Aquecendo a resina

composta entre 55-60°C (Celsius), ocorre uma maior mobilidade das moléculas, afetando conseqüentemente sua viscosidade, resultando em um maior escoamento dos compósitos pré-aquecidos, o que torna possível uma melhor adaptação às paredes cavitárias. (9-11) Quando utilizada para cimentar restaurações, isso pode ser traduzido em uma espessura de película mais delgada em relação às resinas em temperatura ambiente, devido sua fluidez. (8) Apesar disso, não há consenso sobre a influência quantitativa nesta espessura (18), assim como também não há concordância sobre qual seria a espessura ideal para essas situações clínicas (10), embora haja um encorajamento por parte dos autores ao sugerirem que a linha de cimentação, independente do material, não ultrapasse 120µm (micrômetros). (10, 19, 31)

Por sua vez, cimentações com resinas compostas aquecidas têm ganhado enfoque e apresentam adaptação marginal superior com uma menor contração de polimerização, (32) menor sorção de água e maior resistência à solubilidade oral. (20, 33) Em referência aos cimentos resinosos e avaliadas sob microscopia eletrônica de varredura, as resinas pré-aquecidas também apresentam valores de adaptação e selamento maiores. (13, 14, 34) Ainda que o aquecimento das resinas não seja capaz de garantir por si só uma adaptação adequada da restauração indireta, é possível afirmar que há uma influência na fluidez e manipulação desses materiais. (13, 14, 34) E sob essa ótica, é de extrema importância avaliar a forma do preparo, bem como a adaptação da restauração antes da cimentação, pois esses fatores também têm impacto direto na espessura da película e adaptação da restauração como um todo. Além disso, a indicação e espessura da restauração devem ser respeitadas para que haja passagem de luz suficiente e conseqüente grau de conversão adequado. (13)

Os procedimentos que utilizam o pré-aquecimento das resinas compostas podem ser considerados simples, seguros e, no geral, uma técnica benéfica. (9) Entretanto, apesar de apresentar resultados promissores, a proporção de cada um dos monômeros e as diferenças nas formulações resinosas são relevantes às propriedades do compósito e, por isso, cada resina pode reagir de maneiras distintas frente ao aquecimento. (10, 14, 18)

Adicionalmente, existem outros critérios muito importantes a serem considerados quando essa técnica de aquecimento prévio é adotada como, por exemplo, a preservação do tecido pulpar a qualquer injúria. Ao serem aquecidos, os compósitos podem oferecer risco à polpa caso sejam inseridos nas cavidades em temperaturas muito altas (24, 35), uma vez que um pequeno aumento de 5,5°C da temperatura intrapulpar poderia acarretar na perda da vitalidade de dentes hígidos. (24, 35) Embora isso possa ser decisivo para o protocolo clínico, as resinas aquecidas ao serem removidas do dispositivo de aquecimento, sofrem uma queda exponencial da temperatura, além de haver a equilíbrio térmico também por parte do remanescente dentário, sendo assim, o aquecimento da resina a 54-60°C não é capaz de causar uma agressão pulpar. (24)

Diferentes métodos de aquecimento, utilizando equipamentos variados, têm sido reportados na literatura, abrangendo variações amplas de temperatura (23°C a 68°C) e do tempo necessário para o aquecimento dos compósitos (5 a 60 minutos). (36) Esses fatores podem influenciar diretamente no sucesso da técnica e, por isso, devem ser determinados. Para se alcançar bons resultados, são necessários conhecimento e treinamento para se combinar as vantagens conseguidas dentro de uma aplicação clínica correta e atenciosa quantos aos detalhes que permeiam a técnica. (9)

1.3 OBJETIVOS

1.3.1 Objetivo Geral

Determinar os parâmetros relacionados à técnica de pré-aquecimento das resinas compostas com enfoque na cimentação de restaurações.

1.3.2 Objetivos Específicos

A tese foi dividida em duas etapas, resultando em dois artigos independentes que seguem os seguintes objetivos: (1) Avaliar quais equipamentos têm sido utilizados para este fim, quanto tempo de aquecimento, quais temperaturas empregadas, bem como entender as influências que o pré-aquecimento gera sobre as propriedades das resinas compostas. (2) Avaliar *in vitro* e quantificar a interferência do aquecimento de diferentes resinas compostas em relação à sua viscosidade.

Os artigos estão dispostos em capítulos a seguir e constam no apêndice deste trabalho em seu formato original de artigo conforme as normas das revistas em que foram submetidos.

2 COMPOSITE RESIN PREHEATING TECHNIQUES FOR CEMENTATION OF INDIRECT RESTORATIONS

ABSTRACT

Purpose: Resin-based materials have been preheated by using different techniques and commercial devices. However, a consensus on the clinical protocol for cementing with preheated composite resins is lacking. The aim of this scoping review was to identify the different methods used for heating composite resins as used for cementing indirect adhesive restorations and to determine benefits and limitations. **Study selection:** A search was performed on PubMed/MEDLINE, Embase, Cochrane, Web of Science, Scopus, Livivo, and the nonpeer-reviewed literature database. Studies on preheating composite resins for cementing indirect restorations were included, with no restrictions on the type of study, year of publication, or language. The following data were extracted: preheating technique, device used for preheating, preset temperature, and warming time. **Results:** In total, 304 studies were identified. After removing duplicates, 270 articles were selected, and 14 articles were included in the final evaluation. Half of the included studies reported similar preheating techniques using the Calset device for composite resins. The temperatures of 54 °C and 68 °C were most frequently reported, with a mean warming time of 5 minutes. **Conclusions:** Preheating composite resins for the cementation of indirect restorations reduces viscosity, but the material must be used promptly after removal from the device.

Practical Implications

Different methodologies for preheating composite resins have been reported and used in clinical dental practice. To achieve good results and guide the clinician on use, the techniques for heating composite resins for cementation need to be standardized. Keeping the material warm until the restorative procedure, the thickness of the indirect restoration, and the composition of the composite resins can directly affect the outcome of the procedure.

KEYWORDS: composite resin, preheating, dental materials, cementation

1. INTRODUCTION

Following the concept of adhesion as described by Buonocore et al. in 1955 [1], the basic concepts of dentistry have changed, with a focus on the most conservative intervention. Among the restorative treatments available, direct composite resin restorations are indicated for teeth with minor loss of structure, while indirect restorations are indicated for teeth with significant functional, structural, and/or esthetic deficiencies [2]. The indirect restorative treatment, which includes laminates, onlays/inlays, and crowns using ceramic, metal, or laboratory-fabricated composite resin as materials, allows for better mechanical properties and marginal adaptation when compared with directly placed composite resins, avoiding polymerization shrinkage and improving wear resistance [3]. Ceramic restorations are resistant to fatigue, with low thermal conductivity and satisfactory biocompatibility [3]. Moreover, indirect restorations which are cemented to the prepared teeth using luting cements have better marginal adaptation [4,5]. Of the various materials available for cementation, resin cements, available in light-, chemically, or dual-polymerized forms, are currently preferred because of their hardness, low solubility in oral fluid, and micro-mechanical bonding to enamel and dentin [2]. Given the different indirect restorative cements, other materials, including different types of composite resins, have been studied for this purpose. Light-polymerized composite resins have advantages over dual-polymerized resin cements that include stain resistance, color stability, and mechanical wear resistance because of increased inorganic filler loading [6,7]. The high inorganic filler content directly influences the viscosity of the composite resin, making it less fluid and leading to a thicker, undesirable, cementation line at the adhesive interface [8].

As an alternative to reduced viscosity luting agents, the preheating of these materials has been suggested [8-15]. By increasing the temperature from 54 °C to 70 °C, the degree of conversion of the resin becomes similar to that of dual-polymerizing resin cement, the consistency of flow improves, and a thinner cementation line becomes possible [8,10,14,16,17]. Different preheating techniques, devices, temperatures, heating durations, and transport methods have been reported [8,11,13].

A systematic review of the heating and preheating of dental restorative materials (composite resins and glass ionomer cements) has recently been published [16].

Although the authors concluded that the preheating technique can improve the physical and mechanical properties of these materials, clinical studies to confirm the advantages of this technique in improving restoration performance are lacking [16]. Many studies have demonstrated the performance of preheating different materials [8,10,14,16], but there remains a lack of evidence that preheating of restorative materials improves the quality and durability of indirect restorations.

Thus, this study aimed to review the influence of the heating protocols for and methods of preheating composite resins used as the luting agent for indirect adhesive restorations.

2. MATERIAL AND METHODS

2.1 Selection criteria and search methods

This scoping review was performed according to The Joanna Briggs Institute (JBI) Reviewers Manual 2015 - Methodology for JBI Scoping Reviews [18] and Preferred Reporting Items for Systematic reviews and Meta-Analyses extension for Scoping Reviews (PRISMA-ScR) Checklist [19]. It was registered at the Open Science Framework (<https://osf.io>) under the number [DOI:10.17605/OSF.IO/GXMQE](https://doi.org/10.17605/OSF.IO/GXMQE).

The studies were selected according to the eligibility criteria based on the PCC strategy [20] as follows: population (P), composite resins; concept (C), preheating techniques; and context (C), cementation of indirect restorations. There were no restrictions on language, date or type of studies. The exclusion criteria were studies that investigated resin cements, preheating associated with restorative techniques other than cementation of indirect restorations, or studies that analyzed only composite resin properties.

Studies were screened using a search strategy adapted for the following electronic databases: PubMed (MEDLINE), Embase, Cochrane, Scopus, Web of Science, and Livivo [Table 1]. The search strategy was developed by using the MeSH terms and associated terms. Hand searches were performed on the reference lists to identify additional studies. In addition, non-peer-reviewed studies (OpenGrey, Proquest, and Google Scholar) were searched by screening the titles and abstracts. The first 100 hits were selected (filtered by 'relevance') on Google Scholar. A specialist

opinion was also consulted. Duplicate studies were excluded by using the EndNote Web [21] and Rayyan [22] software programs. The search was conducted on July 21, 2020. A new search was conducted on January 4, 2021, and additional studies were included.

2.2 Data collection

A Kappa test (K) was applied to measure the calibration between the first and second reviewers. After analyzing 10% of all included studies, a 0.81 value of Kappa was obtained. The study selection followed three steps. First, two investigators screened the titles of the studies that appeared to meet the inclusion criteria. In the second phase, the same reviewers independently read the abstracts of potentially relevant articles. Finally, they independently read the full text of the selected articles and excluded those that did not meet the inclusion criteria. Disagreements at any of the three stages were resolved by discussion and mutual agreement among the reviewers. If no consensus was reached, a third author was consulted to reach the final decision.

2.3 Data analyses

Data extraction was performed by the first investigator and followed the mean characteristics of the study: author(s), year of publication, objective, conclusion, primary and secondary testing methods, number of specimens, materials used, preheating device, temperature tested, mean preheating duration, and all details relating to the technique. The second author examined all the retrieved information for the analysis. In an attempt to retrieve missing information, the corresponding author of the studies was contacted when important data were not described in the studies. Information was obtained from two of the four authors contacted.

3. RESULTS

3.1 Description of studies

Based on the search strategy, 304 studies were identified, including the first 100 studies detected on Google Scholar. After the removal of duplicates, 270 studies were analyzed by title and abstract. The inclusion process resulted in 14 studies [11,13,23-34] in the second phase. Of these, 12 were *in vitro* research studies [11,13,23-27, 30-

34] and two were clinical case reports [28,29]. The flow chart was adapted from PRISMA [20] and is illustrated in Figure 1. The timeline of the publications in the English language and from six countries, namely, Brazil [13,27-30], Chile [28], the United States [23,24,26,31-34], Italy [11], the Czech Republic [25], and Thailand [29] from 2009–2018 is shown in Table 2.

3.2 Preheating device

Ten studies used the Calset oven (AdDent Inc.) device to preheat composite resin [11,23,24,26,29-34]. The other preheating devices described were the Digital wax pot (SJK) [13], ENA heat (Micerium S.p.A) [25], and Wax Heater Pot 4 (manufacturer not mentioned) [28]. Only one study used an incubator (manufacturer not mentioned) for preheating, but its specifications were not provided [27].

3.3 Temperatures and preheating times

The temperatures used to preheat composite resins were 54 °C [11,23,29,32,34], 55 °C [25], 58 °C [28], 60 °C [27], 64 °C [13], and 68 °C [24,30,33]. The reported range was from 54 °C to 68 °C. The temperatures of 54 °C and 68 °C were most frequently reported. Two studies did not report the preheating temperature [26,31]. A warming time of 5 min was specified in eight studies [13,23,24,26,28,31,32,34] and of 30, 15, and 60 min in three other studies [25,27,29]. Three studies did not report the time used for heating composite resins [11,30,33].

3.4 Preheating methods and mean required time of the clinical procedure

Only one study reported details relating to the glass container in which the resin was placed when heated in the device [27]. Almeida et al. [27] reported removing the increment from the oven and immediately applying it to the ceramic. However, the transport time was not specified. Goulart et al. [13] stated that the time taken for the material to be removed from the heating device until the assessed property was measured was less than 30 s. Other studies did not report this aspect.

3.5 Composite resins tested and light-polymerizing units

Seven studies preheated the micro-hybrid composite resin Filtek Z100 (3M ESPE) [23,24,26,30,32-34]. Other micro-hybrid composite resins used were Gradia Direct posterior (GC) [31] and Venus1 (Kulzer) [11,13]. Four articles preheated

composite resins with nano-hybrid particles, including Filtek Z350 XT (3M ESPE) [27], Tetric N-Ceram (Ivoclar Vivadent) [28], Z250 XT (3M ESPE) [13], Miris 2 (Coltene-Whaledent) [29], and other composite resins such as Enamel Plus Hri (Micerium) [25]. Goulart et al. [13] compared the use of composite resins at room temperature ($\pm 23^{\circ}\text{C}$) for cementing indirect restorations. Acquaviva et al. [11] conducted a study comparing composite resin at room temperature, heated composite resin, and resin cement. Another five studies focused on the preheating of composite resin and resin cement [13,24,27] for cementing indirect restorations. Two clinical case reports [28,29] and seven in vitro studies [23,24,26,30-34] used heated composite resin. In no study were details provided regarding the amount of preheated composite resins used for cementing indirect restorations.

The following brands of light-polymerization unit were used: halogen lamp Swiss Master Light1 (EMS, Neun, Switzerland), FlashLight (Dental Discus), Optilight Max (Gnatus), Den-Mat (Allegro) and Valo (Ultradent). The light-polymerization power was between 400 mW/cm^2 and 1200 mW/cm^2 , and the duration ranged from 40 s to 120 s.

3.6 Property testing

From the included in vitro studies, different properties of composite resins were evaluated, including the degree of conversion [11], color stability [27], microtensile bond strength-adhesive interfaces [13], fatigue resistance [23,26,30-34], vertical seating [24], and vertical marginal discrepancy [25].

4. DISCUSSION

Indirect adhesive restorations can be cemented with preheated composite resins yielding reduced viscosity materials that provide a clinically acceptable cement film thickness and have better mechanical properties than those of conventional cements [8-11,14]. Despite clinical and laboratory evidence suggesting the advantages of preheating composite resins [8-11,14], their protocol for use as a cementation agent for indirect restorations has not yet been fully elucidated. Advantages reported in studies on preheating resin materials include an increased degree of conversion [10], improved marginal adaptation of restorations because of reduced viscosity [35], and decreased polymerization contraction [36]. However, the methodologies used in the

preheating of composite resins, their mechanical properties, and their performance as luting cements for indirect restorations must be analyzed.

The composite resin is typically preheated in a device that is programmed to reach a certain temperature that should be confirmed for accuracy and monitored and controlled during storage in the heater [37]. The time taken to dispense the preheated composite resin is approximately 50% in 2 min [37], as it cools rapidly when removed from the heating device. Thus, the material should be placed, adapted, the restoration seated, and light-polymerized rapidly. When the high temperature is maintained, monomer conversion will be greater than at room temperature (± 23 °C) [11].

Seven of the included articles reported similar preheating techniques with a commercially available device (Calset, AdDent Inc.) that the manufacturer claims preheats and stores composite resins at temperatures of 54 °C, 60 °C, or 64 °C until they are ready for use. Composite resin syringes can be heated and the resin can then be directly injected onto the restoration or prepared tooth, reducing the clinical time [8,37,38]. Despite what was specified by the manufacturer, Daronch et al. [37], who used Calset (Calset, Addent Inc.) in their study, reported that the maximum temperature reached was 48.3 °C and 54.7 °C when the preset temperature of the device was set at 54 °C and 60 °C, respectively. The equipment (ENA heat, Micerium) used for heating composite resins in the study by Mounajjed et al. [25] was preset at temperatures from 39 °C to 55 °C. A temperature of 55 °C was recommended by the manufacturer for heating composite resins for cementation and had six spaces for heating syringes of composite resin.

Previous studies evaluated the use of this heating equipment [39-41], but Goulart et al. [13] and Olivares et al. [28] used wax pot heaters in their studies (Digital wax pot, SJK and Wax Heater Pot 4, Denshine) because these devices allowed setting the temperature according to clinical needs. The preheating of composite resins in a wax pot heater has also been described in another study where the heating time of the composite resins was around 2 to 3 min. The authors concluded that the wax pot was a straightforward, rapid, and economical option [42]. Almeida et al. [27] used an incubator oven to heat composite resins at 60 °C for 30 min. However, they did not provide manufacturer specifications for the equipment. However, a preheating

technique in a bacteriological oven (model 502, Fanem) at a temperature of 54 °C has been described [43].

Composite resins can be heated in different dry heating devices, as long as the temperature is controlled and remains stable until clinical use. The use of a specifically marketed device such as Calset (AdDent Inc.) facilitates the technique and ensures standardization of the preheating process. The use of parallel heaters requires the preset temperature of the device to be measured and checked until the required temperature is reached.

The temperatures for preheating composite resins described in the studies ranged from 54 °C to 68 °C. The authors are unaware of a published study that evaluated the differences between preheating at 54 °C and 68 °C [16], and this range has been considered ideal for improving the working properties of the material. Daronch et al. [10] evaluated the degree of conversion at temperatures ranging from 3 °C to 60 °C and reported that, at the highest temperature, a greater degree of conversion was reached and that, even if the composite resin reached temperatures between 40 °C and 30 °C, the degree of conversion increased to over 22°C. Daronch et al. [37] used the Calset device (AdDent Inc.) and reported a drop in the degree of conversion after a certain temperature because of degradation of the photoinitiator. In monomers such as bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) or ethoxylated bisphenol A dimethacrylate (Bis-EMA), the volatility limit of dimethacrylate monomers used in resin formulations occurs close to 90°C, a temperature that could damage some composite resin components and harm pulpal tissue. However, 90 °C is above the maximum temperature allowed by the heating device. In addition, because of incomplete polymerization, unreacted monomers may leach into the saliva, promoting undesirable consequences, and the loss of plasticizers may decrease mechanical strength, dimensional stability, and color change and allow bacterial growth. Unreacted monomers can also cause allergic and sensitivity reactions [44].

The heating time for the composite resin inside the heater is also an important evaluation parameter, with the average time for the device to reach both tested preset temperatures (54°C or 60°C) being 11 min [37]. Therefore, in addition to ensuring that the heating device can maintain a controlled and predefined temperature, the temperature should be reached in a predictable time. A drop in the temperature of the

composite resin was reported between its removal from the heating device and the mouth, estimated to be 50% after 2 min and 90% after 5 min when heated to 60 °C and removed from the device, indicating the need for calibration during all processes. In addition, heated composite resins have been reported to provide better results than composite resins at room temperature [10,16,37]. Composite resins with different compositions can take different times to reach a stable temperature, and some low-molecular-weight components of the photo-initiator system can be volatilized with prolonged heating [10,37]. Therefore, different heating methods have been used for *in vitro* studies and for clinical techniques. Lopes et al. [16] reported that some studies used the reasonable clinical time of approximately 15 min.

The temperature must be controlled to avoid causing pulpal damage, but increasing the composite resin temperature to between 54 °C and 60 °C does not significantly change the intrapulpal temperature [45]. Lopes et al. [16] noted that dentin thickness acts as a thermal barrier, preventing harmful stimuli and protecting the dental pulp.

According to manufacturers, the Calset (AdDent Inc.) and ENA heat (Micerium) devices are designed to attach a syringe, acting as a container. The wax pot heaters (Digital wax pot, SJK and wax heater pot 4, Denshine) can directly heat the composite resin inside of the syringe. Comparing the two preheating methods, Daronch et al. [37] concluded that the composite resins already assembled in the application syringe showed a significantly higher maximum temperature ($36.6 \text{ °C} \pm 2.2 \text{ °C}$) than the composite resins that were heated without a container ($33.6 \text{ °C} \pm 0.5 \text{ °C}$). Thus, the composite compule preloaded into a delivery syringe was more efficient. Higher temperatures were attained with this method as opposed to preheating the compule separately [16].

The way the composite resins is arranged and placed in the preheating device affects its clinical application, as the working time should be minimum owing to the decrease in temperature after its removal from the heater [10,37]. Once the composite resin is attached to a syringe or loaded into the indirect restoration to be cemented, the dentist can simply remove and apply it to the teeth, without concerns about assembling the application system, thus reducing the working time, and maintaining the temperature as high as possible [37].

The viscosity of composite resins is linked to factors in their composition such as the organic matrix and amount and size of inorganic fillers [46]. Analyzing the particle size, materials with smaller particles appear more fluid when compared with those that contain bigger particles. Regarding the amount of filler, the higher the filler load, the higher the viscosity [46]. The amount and type of monomer can also cause an increase in viscosity, as monomers such as Bis-GMA and urethane dimethacrylate are quite viscous, whereas Bis-EMA and triethylene glycol dimethacrylate are more fluid [46,47]. Among the studies included, Goulart et al. [13] compared the use of two preheated composite resins, a micro-hybrid (Venus 1, Kulzer) and a nano-hybrid (Z250 XT, 3M ESPE) with the same preheating protocol and mechanical test. The nano-hybrid resin, as evaluated by SEM after cementation, formed a thicker film than the micro-hybrid resin.

The degree of conversion can be increased by preheating, decreasing the light-polymerization time and maintaining a degree of conversion similar to or even better than when the composite resins are irradiated for longer at 22 °C [10,11]. Preheating the resins to 60 °C increased the conversion of monomers by increasing molecular mobility. Compounds with higher conversion have greater crosslinking, reducing the free space of the polymers and improving their mechanical properties [10]. A 5-s light-polymerization time with a composite resin preheated to 57 °C resulted in a higher conversion rate than that observed after a 40-s exposure at 22 °C [10]. According to Acquaviva et al. [11], the thickness of the onlays affected the degree of conversion of both composite resins and dual-polymerizing cements, and an excellent degree of conversion can be achieved by preheating the light-polymerizing composite resins. No ideal light-polymerizing time or intensity have yet been determined. Thus, purely light-polymerizing cements or resins must be used with care, as there must be enough light to pass through the materials for adequate conversion of the monomers. If an indirect restoration is thinner than 2 mm, the light passage should be adequate [48].

A consensus on the limits for a clinically acceptable film thickness is lacking. Marcondes et al. [49] stated that composite resins, being restorative materials, are designed to provide intraoral resistance. Therefore, an increased cementation thickness, even if it exceeds the value defined by ISO 4049 [50], should be clinically acceptable. Composite resins are designed for color stability and abrasion resistance,

as shown in laboratory and clinical studies [10,11,37,48,49]. In general, when composite resins are preheated, viscosity is reduced and adaptation to cavity walls improved [49].

The presence of amines in chemically polymerized resins, including dual-resin cements, may eventually result in the staining of indirect restorations, and, therefore, they should be avoided for cementation of translucent or thin restorations [7]. Almeida et al. [27] reported that dual-resin cements showed greater color variation than preheated composite resins, light-polymerized resin cements, and flowable composite resins. Goulart et al. [13] reported that preheating composite resins did not increase the cementation bond strength of indirect restorations, even though increased mechanical properties have been reported because of increased conversion [8,12]. The results could be explained by the loss of temperature of the material during the bonding procedure and not reaching an adequate degree of conversion. In contrast, the conclusion was that the material can still be used to reduce its viscosity and improve the fit of the restoration [13]. The use of different luting agents provides many alternatives for cementation, which also can produce varying results in vertical discrepancy of the definitive restoration [51]. Mounajjed et al. [25] compared the vertical marginal discrepancy of pressed crowns of lithium disilicate by using different cementing agents. The preheated composite resin Enamel Plus Hri (Micerium) obtained higher values of marginal discrepancies than a cement flow resin (Harvard Premium Flow) and the dual-resin cement, RelyX Ultimate (3M ESPE). The authors stated that the methodology used may have affected the results of the study. For example, using a specimen at low temperature, which could have reduced the fluidity of the resin, and the difficulty of standardizing the seating pressure at different viscosities. Magne et al. [24] investigated the vertical displacement of composite resin inlays, onlays, and computer-aided design and computer-aided manufactured overlays. The preheated composite resin used in cementation resulted in the better seating of inlays, onlays, and overlays than the dual-polymerizing resin cement [24]. Magne et al. [23,24,32-34], Oderich et al. [26], Schlichting et al. [30], and Soares et al. [31] evaluated the fatigue strength of different restorative materials. However, preheated composite resins were not evaluated; only the use of unheated composite

resins was reported. Olivares et al. [28] and Rickman et al. [29] did not evaluate properties in their clinical case studies.

This scoping review observed a large variation in the use of preheating techniques on composite resins used for cementation of indirect restorations. No consensus was found regarding the recommended preheating devices, heating durations, or temperatures for this clinical procedure. Some aspects can be considered relevant when considering preheating techniques for composite resins: **(1)** All heating devices demonstrated effectiveness in heating composite resins used for the cementation of indirect restorations. **(2)** The ideal heating device must be free of moisture and calibrated to reach a predetermined temperature (between 54 °C and 68 °C) on heating and must maintain stability at the predetermined temperature after heating. **(3)** The preheated material must be used as soon as possible after being removed from the device, as the temperature of the composite resin will decrease quickly. **(4)** Preheating the material directly in the prosthetic restoration or the dispensing syringe reduces clinical time. **(5)** The indirect restoration must be less than 2 mm thick if a light-polymerizing luting cement or composite resin is to be used. **(6)** The composition of the composite resins directly affects the viscosity reached after preheating. Therefore, materials indicated for this purpose or that show increased fluidity when heated should be used.

Based on the results of the included studies, more research is needed on preheating techniques for composite resins used for the cementation of indirect restorations; in particular, longitudinal clinical evaluations are needed. Furthermore, studies correlating the composition of composite resins and their behavior when heated are required.

REFERENCES

- [1] Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955; 34:849–53.
- [2] Powers JM, Sakaguchi RL. *Craig's Restorative Dental Materials*. 12th ed. Philadelphia, PA: Elsevier Publishing 2006:479–511.
- [3] Mei ML, Chen YM, Li H, Chu CH. Influence of the indirect restoration design on the fracture resistance: a finite element study. *Biomed Eng Online* 2016; 15:3.
- [4] Guess PC, Vagkopoulou T, Zhang Y, Wolkewitz M, Strub J R. Marginal and internal fit of heat pressed versus CAD/CAM fabricated all-ceramic onlays after exposure to thermo-mechanical fatigue. *J Dent* 2014; 42:199–209.
- [5] Silveira ACP, Chaves SB, Hilgert LA, Ribeiro AP. Marginal and internal fit of CAD-CAM fabricated composite resin and ceramic crowns scanned by 2 intraoral cameras. *J Prosthet Dent* 2016; 117:386–92.
- [6] Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002; 87:642-9.
- [7] Smith DS, Vandewall KS, Whisler G. Color stability of composite resin cements. *Gen Dent* 2011; 59:390–4.
- [8] Lucey S, Lynch C D, Ray N J, Burke F M, Hannigan A. Effect of pre-heating on the viscosity and microhardness of a resin composite. *J Oral Rehabil* 2010; 37:278–82.
- [9] Blalock JS, Holmes RG, Rueggeberg FA. Effect of temperature on unpolymerized resin film thickness. *J Prosthet Dent* 2006; 96:424–32.
- [10] Daronch M, Rueggeberg FA, Goes MF. Monomer conversion of pre-heated composite. *J Dent Res* 2005; 84:663–7.
- [11] Acquaviva PA, Cerutti F, Adami G, et al. Degree of conversion of three composite materials employed in the adhesive cementation of indirect

- restorations: a micro-Raman analysis. *J Dent* 2009; 37:610–15.
- [12] Daronch M, Rueggeberg FA, Goes MF, Giudice R. Polymerization kinetics of Pre-heated Composite. *J Dent Res* 2006; 85:38–43.
- [13] Goulart M, Borges Veleda B, Damin D, Bovi Ambrosano GM, et al. Preheated composite resin used as a luting agent for indirect restorations: effects on bond strength and resin-dentin interfaces. *Int J Esthet Dent* 2018; 13:86–97.
- [14] Fróes-salgado NR, Silva LM, Kawano Y, Francci C, Reis A, Loguercio AD. Composite pre-heating: effects on marginal adaptation, degree of conversion and mechanical properties. *Dent Mater* 2010; 26:908–14.
- [15] Deb S, Di Silvio L, Mackler HE, Millar BJ. Pre-warming of dental composites. *Dent Mater* 2011; 27:51–9.
- [16] Lopes LCP, Terada RSS, Tsuzuki FM, Giannini M, Hirata R. Heating and preheating of dental restorative materials-a systematic review. *Clin Oral Investig* 2020; 24:4225–35.
- [17] Trujillo M, Newman SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. *Dent Mater* 2004; 20:766–77.
- [18] Peters MDJ, Godfrey C, Mclnerney P, Munn Z, Tricco AC, Khalil, H. Chapter 11: Scoping Reviews (2020 version). In: Aromataris E, Munn Z (Editors). *JBIM Manual for Evidence Synthesis*, JBI, 2020. Available from <https://synthesismanual.jbi.global>. <https://doi.org/10.46658/JBIMES-20-12>.
- [19] Aromataris E, Munn Z (Editors). *JBIM Manual for Evidence Synthesis*. JBI, 2020. Available from <https://synthesismanual.jbi.global>. <https://doi.org/10.46658/JBIMES-20-01>.
- [20] Moher D, Liberati A, Tetzlaff J, Altman DG. Preferred reporting items for systematic reviews and meta-analyses: The PRISMA statement. *BMJ* 2009; 339:2535.
- [21] EndNote Web, Clarivate Analytics, USA. 2017. Available at: <http://www.endnote.com/>. Accessed on January 30, 2018.

- [22] Ouzzani M, Hammady H, Fedorowicz Z, et al. Rayyan—a web and mobile app for systematic reviews. *Syst Rev* 2016; 5:210.
- [23] Magne P, Oderich E, Boff LL, Cardoso AC, Belser UC. Fatigue resistance and failure mode of CAD/CAM composite resin implant abutments restored with type III composite resin and porcelain veneers. *Clin Oral Implants Res* 2011; 22:1275–81.
- [24] Magne P, Razaghy M, Carvalho MA, Soares LM. Luting of inlays, onlays, and overlays with preheated restorative composite resin does not prevent seating accuracy. *Int J Esthet Dent* 2018; 13:318–32.
- [25] Mounajjed R, Salinas TJ, Ingr T, Azar B. Effect of different resin luting cements on the marginal fit of lithium disilicate pressed crowns. *J Prosthet Dent* 2017; 119:975–80.
- [26] Oderich E, Boff LL, Cardoso AC, Magne P. Fatigue resistance and failure mode of adhesively restored custom implant zirconia abutments. *Clin oral implants res* 2012; 23:1360–8.
- [27] Almeida JR, Schmitt GU, Kaizer MR, Boscato N, Moraes RR. Resin-based luting agents and color stability of bonded ceramic veneers. *J Prosthet Dent* 2015; 114:272–7.
- [28] Olivares JM, Hidalgo A, Pavez JP, Benadof D, Iribarra R. Functional and esthetic restorative treatment with preheated resins in a patient with ectodermic dysplasia: A clinical report. *J Prosthet Dent* 2018; 119:526–9.
- [29] Rickman LJ, Padipatvuthikul P, Chee B. Clinical applications of preheated hybrid resin composite. *Br Dent J* 2011; 21:63–7.
- [30] Schlichting LH, Maia HP, Baratieri LN, Magne P. Novel-design ultra-thin CAD/CAM composite resin and ceramic occlusal veneers for the treatment of severe dental erosion. *J Prosthet Dent* 2011; 105:217–26.
- [31] Soares LM, Razaghy M, Magne P. Optimization of large MOD restorations: Composite resin inlays vs. short fiber-reinforced direct restorations. *Dent Mater* 2018; 34:587–97.

- [32] Magne P, Knezevic A. Simulated fatigue resistance of composite resin versus porcelain CAD/CAM overlay restorations on endodontically treated molars. *Quintessence Int* 2009; 40:125–33.
- [33] Magne P, Schlichting LH, Maia HP, Baratieri LN. In vitro fatigue resistance of CAD/CAM composite resin and ceramic posterior occlusal veneers. *J Prosthet Dent* 2010; 104:149–57.
- [34] Magne P, Knezevic A. Influence of overlay restorative materials and load cusps on the fatigue resistance of endodontically treated molars. *Quintessence Int* 2009; 40:729–37.
- [35] Baroudi K, Mahmoud S. Improving Composite Resin Performance Through Decreasing its Viscosity by Different Methods. *Open Dent J* 2015; 9:235–42.
- [36] Didron P, Ellakwa A, Swain M. Effect of Preheat Temperatures on Mechanical Properties and Polymerization Contraction Stress of Dental Composites. *MSA* 2013; 4:374–85.
- [37] Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent* 2006; 18:34051.
- [38] Friedman J. Heating assembly for preheating dental materials. US patent 6236020. 2001.
- [39] D’Amario M, De Angelis F, Vadini M, Marchili N, Mummolo S, D’Arcangelo C. Influence of a repeated preheating procedure on mechanical properties of three resin composites. *Oper Dent* 2015; 40:181–9.
- [40] Theodoridis M, Dionysopoulos D, Koliniotou-Koumpia E, Dionysopoulos P, Gerasimou P. Effect of preheating and shade on surface microhardness of silorane-based composites. *J Investig Clin Dent* 2017; 8:1–6.
- [41] Dionysopoulos D, Papadopoulos C, Koliniotou-Koumpia E. Effect of temperature, curing time, and filler composition on surface microhardness of composite resins. *J Conserv Dent* 2015; 18:114–8.
- [42] Arora V, Arora P, Shamrani A, Fahmi MK. A New, simple and innovative technique for Pre-heating/Pre-warming of Dental Composite resins in thermal

- assisted light polymerization technique. *J Dent Oral Biol* 2017; 2:1–7.
- [43] Theobaldo JD, Aguiar FH, Pini NI, Lima DA, Liporoni PC, Catelan A. Effect of preheating and light-curing unit on physicochemical properties of a bulk fill composite. *Clin Cosmet Investig Dent* 2017; 9:39–43.
- [44] Bouschlicher MR, Rueggeberg FA. Effect of ramped light intensity on polymerization force and conversion in a photoactivated composite. *J Esthet Dent* 2000; 12:328–39.
- [45] Daronch M, Rueggeberg FA, Hall G, De Goes MF. Effect of composite temperature on in vitro intrapulpal temperature rise. *Dent Mater* 2007; 23:1283–8.
- [46] Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. *Dent Mater* 2006; 22:515–26.
- [47] Gonçalves F, Kawano Y, Pfeifer C, et al. Influence of BisGMA, TEGDMA, and BisEMA contents on viscosity, conversion, and flexural strength of experimental resins and composites. *Eur J Oral Sci* 2009; 117:442–6.
- [48] Pacheco RR, Carvalho AO, André CB, Ayres APA, et al. Effect of indirect restorative material and thickness on light transmission at different wavelengths. *J Prosthodont Res* 2019; 63:232–8.
- [49] Marcondes RL, Lima VP, Barbon FJ, Isolan CP, Carvalho MA, Salvador MV, Lima AF, Moraes RR. Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness. *Dent Mater* 2020; 36:1356–63.
- [50] International Standard ISO 4049. Dentistry – polymer based restorative materials. Fifth ed; 2019.
- [51] Ng J, Ruse D, Wyatt C. A comparison of the marginal fit of crowns fabricated with digital and conventional methods. *J Prosthet Dent* 2014; 112:555–60.

FIGURE LEGEND

Figure 1 – Description of included records in the Scoping Review

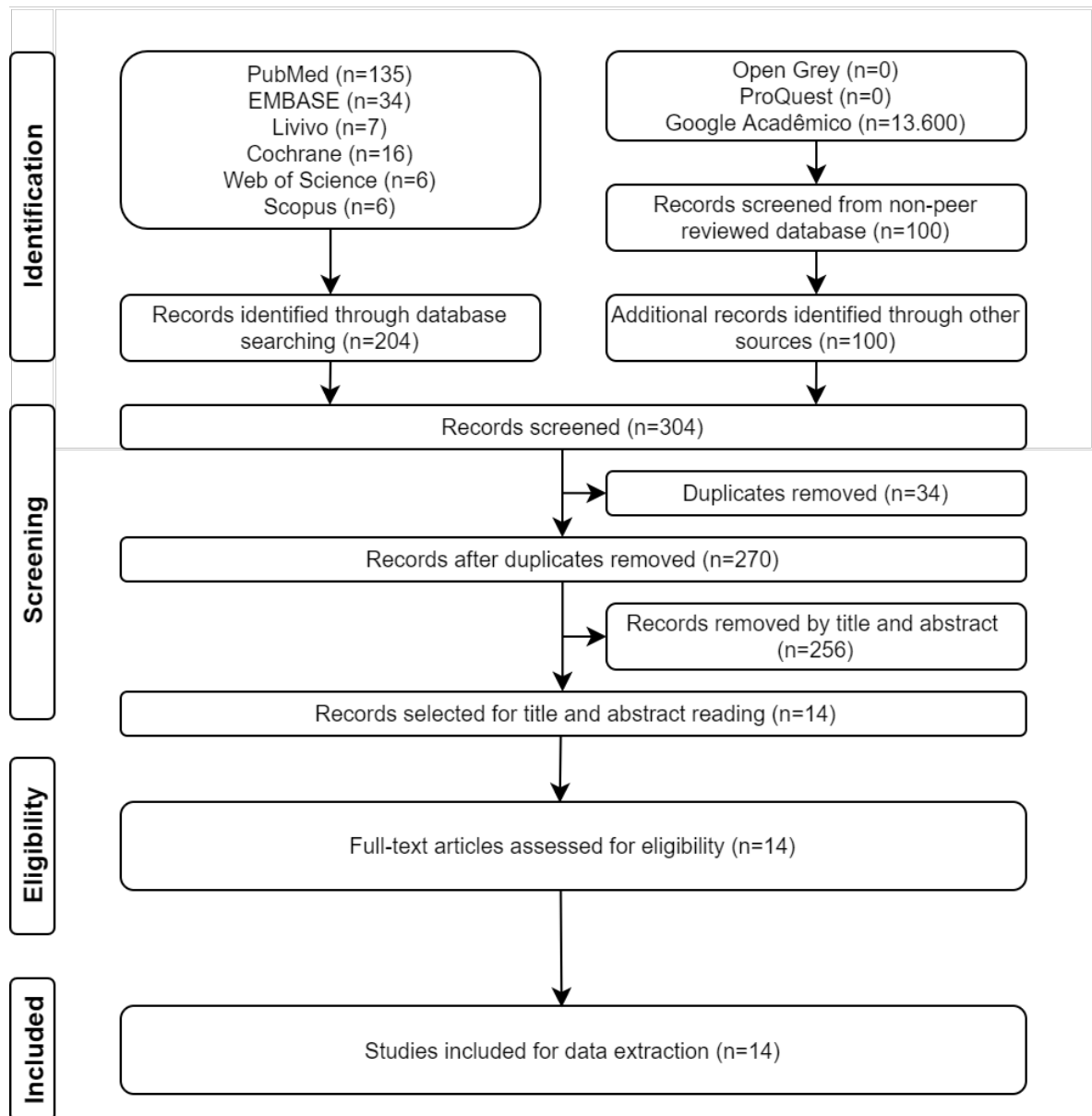


Table 1 – Electronic database and search strategy (PubMed)

((("composite resins"[MeSH Terms] OR "composite resin"[All Fields] OR "composite dental resin"[MeSH Terms] OR "composite dental resin"[All Fields] OR "composite dental resin"[All Fields] OR "bisphenol a-glycidyl methacrylate"[MeSH Terms] OR "bisphenol a-glycidyl methacrylate"[All Fields] OR "composite properties"[All Fields] OR "composite dental material"[All Fields] OR "composite dental restorative"[All Fields] OR "composite dental restoratives"[All Fields] OR "composite dental restorative material"[All Fields] OR "composite dental restorative materials"[All Fields] OR "composite dental filling"[All Fields] OR "composite dental filling material"[All Fields] OR "composite dental filling materials"[All Fields] OR "Methacrylate, Bisphenol A-Glycidyl"[All Fields] OR "Bis(Phenol A-Glycidyl Methacrylate)"[All Fields] OR "Bis-GMA"[All Fields] OR "Bis GMA"[All Fields] OR "Bisphenol A-Glycidyl Methacrylate Homopolymer"[All Fields] OR "Bisphenol A Glycidyl Methacrylate Homopolymer"[All Fields] OR "Bis(Phenol A-Glycidyl Methacrylate), Homopolymer"[All Fields] OR "Poly(Bis-GMA)" [All Fields] OR "Bis-GMA Resin"[All Fields] OR "Bis GMA Resin"[All Fields] OR "Bis-GMA Resins"[All Fields] OR "Resin, Bis-GMA"[All Fields] OR "Resins, Bis-GMA"[All Fields] OR "Bisphenol A-Glycidyl Methacrylate Polymer"[All Fields] OR "Bisphenol A Glycidyl Methacrylate Polymer"[All Fields] OR "2-Propenoic acid, 2-methyl-, (1-methylethylidene)bis(4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)) ester, homopolymer"[All Fields] OR "Bis-GMA Polymer"[All Fields] OR "Bis GMA Polymer"[All Fields] OR "Bis-GMA Polymers"[All Fields] OR "Polymer, Bis-GMA"[All Fields] OR "Polymers, Bis-GMA"[All Fields]) AND ("preheat"[All Fields] OR "preheated"[All Fields] OR "preheating"[All Fields] OR "hot temperature"[MeSH Terms] OR "hot temperature"[All Fields]) AND ("cementation"[MeSH Terms] OR "cementations"[MeSH Terms] OR "cementation"[All Fields] OR "cementations"[All Fields] OR "dental cement"[All Fields] OR "dental cements"[MeSH Terms] OR "dental cements"[All Fields] OR "luting agent"[All Fields] OR "luting agents"[All Fields] OR "cementation agents"[All Fields] OR "cementation agent"[All Fields] OR "Cement, Dental"[All Fields] AND "Permanent dental restoration"[MeSH Terms] OR "Permanent dental restorations"[All Fields] OR "Restorations, Permanent Dental"[All Fields] OR "Dental Restoration, Permanent"[All Fields] OR "Restoration, Permanent Dental"[All Fields] OR "Dental Restorations, Permanent"[All Fields] OR "Dental Permanent Fillings"[All Fields] OR "Filling, Permanent Dental"[All Fields] OR "Permanent Dental Fillings"[All Fields] OR "Permanent Fillings, Dental"[All Fields] OR "Permanent Filling, Dental"[All Fields] OR "Dental Filling, Permanent"[All Fields] OR "Dental Permanent Filling"[All Fields] OR "Filling, Dental Permanent"[All Fields] OR "Filling, Permanent Dental"[All Fields] OR "Permanent Dental Filling"[All Fields] OR "Fillings, Dental Permanent"[All Fields] OR "Dental Fillings, Permanent"[All Fields]))))

Table 2. Main characteristics of the included studies.

N°	Author, year, country	Total "n" of specimens	Heated composite resin trademark, classification, color and volume	Preheating device	Temperature, warm-up time, means of transport and transport time	Valued property and assessment device	Light curing trademark, light curing time and characteristics	Valued property control group (non-preheated)
1	Acquaviva et al., 2009, Italy	180; 5 preheated	Venus1 (Kulzer); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; N/A; N/A; N/A	Degree of conversion; Spectrometer micro-raman dilor (HR LabRam)	Halogen lamp Swiss Master Light1 (EMS); 40 s, 60 s, 120 s; 1200 mW/cm ² ; 800 mW/cm ² ; 400 mW/cm ²	Calibra1 (Dentsply), dual-cured resin cement; Variolink1 II (Ivoclar-Vivadent), dual-cured resin cement; Venus1 (Kulzer), micro-hybrid composite resin
2	Almeida et al., 2015, Brazil	40; 10 preheated	Filtek Z350 XT (3M/Espe); nano-hybrid composite resin; A1; N/A	Incubator (N/A)	60°C; 30 min; glass container; "Immediately"	Color stability; Spectrophotometer (Easysshade, Vita Zahnfabrik)	FlashLight (Discus Dental); 1 min; 800 mW/cm ²	RelyX ARC (3M/Espe), dual-cured resin cement; RelyX Veneer (3M/Espe), lightpolymerizing cement; Filtek Z350 Flow (3M/Espe), flowable
3	Goulart et al., 2018, Brazil	50; 50 preheated	Venus1 (Kulzer); micro-hybrid composite resin; A2; N/A and Z250 XT (3M/Espe); nano-hybrid	Digital wax pot (SJK)	64°C; 5 min; N/A; "reduced to 30 s"	Microtensile bond strenght and adhesive interfaces; Stereomicroscope (EMZ, Meji Techno)	Optilight Max (Gnatus); 40 s; 900 mW/cm ²	Venus1 (Kulzer), micro-hybrid composite resin color A2; Z250 XT (3M), micro-hybrid composite resin color A2; RelyX ARC (3M), dual-cured resin
4	Magne et al., 2009, United States	30; 30 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; 5 min; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	N/A; 60 s; N/A	None
5	Magne et al., 2010, United States	30; 30 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	68°C; N/A; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Allegro (Den-Mat); 60 s; 1000 mW/cm ²	None

6	Magne et al., 2009, United States	30; 30 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; 5 min; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Allegro (DenMat); 60 s; N/A	None
7	Magne et al., 2011, United States	28; 28 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; 5 min; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Valo (Ultradent); 60 s; 1000 mW/cm ²	None
8	Magne et al., 2018, United States	60; 30 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	68°C; 5 min; N/A; N/A	Vertical seating; Acumen III (MTS Systems)	Valo (Ultradent); 60 s; N/A	RelyX Ultimate cement (3M), dual-cured resin cement
9	Mounajjed et al., 2017, Czech Republic	18; 6 preheated	Enamel Plus Hri (Micerium S.p.A); nano-hybrid composite resin; N/A; N/A	Heater ENA heat (Micerium S.p.A)	55°C; 1 hour; N/A; N/A	Vertical marginal discrepancy; Microscopy at x200 magnification with special image analysis software (Keyence)	Valo (Ultradent); 60 s; N/A	Harvard PremiumFlow cement (GmbH), nano-hybrid composite resin; RelyX Ultimate cement (3M/Espe), dual-cured resin cement
10	Oderich et al., 2011, United States	60; 60 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	N/A; 5 min; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Valo (Ultradent); 60 s; N/A	None
11	Olivares et al., 2011, Chile	10; 10 preheated	Tetric N-ceram (Ivoclar Vivadent AG); nano-hybrid composite resin; A2; N/A	Wax Heater Pot 4 (Denshine)	58°C; 5 min; N/A; N/A	None	N/A; 60 s; N/A	None
12	Rickman et al., 2011, Thailand	7; 7 preheated	Miris 2 (Coltene-Whaledent); nano-hybrid composite resin; A2; N/A	Calset (AdDent Inc.)	54°C; 15 min; N/A; N/A	None	N/A; N/A; N/A	None
13	Schlichting et al., 2011, Brazil	40; 40 preheated	Filtek Z100 (3M/Espe); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	68°C; N/A; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Allegro (DenMat); 60 s; 1000 mW/cm ²	None

14	Soares et al., 2018, United States	45; 30 preheated	Gradia Direct posterior (GC); micro-hybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	N/A; 5 min; N/A; N/A	Fatigue resistance; Closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Valo (Ultradent); 60 s; 1000 mW/cm ²	None
----	------------------------------------	------------------	--	----------------------	----------------------	---	---	------

*N/A – not available // None – not applicable

3 EFFECT OF PREHEATING ON THE VISCOSITY OF COMPOSITE RESINS

ABSTRACT

Aim To determine the effects of time and temperature on the viscosity of preheated composite resins. *Methods* Eleven composite resins were heated to 60°C, and temperature analyses were performed at intervals of 1 minute until they had cooled to 25°C. The permanent oscillatory shear test was performed at 25°C, 35°C, 50°C, and 60°C for three composite resins under a shear rate of 1s⁻¹. One- and two-way ANOVA were used for the analysis ($\alpha=0.05$). *Results* There was no significant interaction between the composite resin and time ($p=0.9304$), and only the main effect time was significantly different ($p<0.0001$). A difference was observed between T0 and T6 ($p<0.001$), but not after T7. The increase in temperature resulted in a viscosity reduction ($p<0.05$). At 25°C, Beautifil II presented higher viscosity. Palfique LX5 showed a significant viscosity reduction with increasing temperature compared with the others ($p<0.05$). For Beautifil II and Z100, there was no difference at temperatures of 50°C and 60°C, while for Palfique LX5, no statistical difference was observed at 35°C, 50°C, and 60°C. *Conclusions* Ten minutes of preheating were sufficient to reach a temperature of 60°C, reducing viscosity by at least 84%. However, 5 min after removal, the composite resin cooled to room temperature.

Clinical Significance

Preheating composite resin has potential benefits. To determine how this approach will work in clinical practice, it is important to define the protocols of this technique and understand its limitations.

Keywords: composite resin, dental materials, preheating, viscosity

INTRODUCTION

Adhesive dentistry is a major component of contemporary practice that is used in virtually all clinical situations, including direct and indirect restorations. When indirect restorations are cemented, both the dental substrate and the chosen restorative material must be considered, and resin cements are currently the materials of choice for providing a bond between ceramics or resins and tooth structure.^{1,2} Resin cements have low solubility in oral fluids and favorable mechanical resistance.³ Despite the many available cementing agents, other materials have been used for this purpose, including light-polymerized composite resins.⁴

Composite resins, because of their high inorganic filler content, have greater resistance to wear compared with resin cement⁵, as well as greater stain and mechanical resistance.⁶ However, the higher inorganic filler content of composite resins leads to a higher viscosity than that of a resin cement.⁶ Heating composite resins has been used as a strategy to decrease their viscosity, reducing the cement line at the adhesive interface.^{7,8} When heated to between 54°C and 70°C, the degree of conversion of the resin is similar to that of a dual-polymerizing resin cement.^{4,7,8} In addition, preheating this material can reduce polymerization shrinkage^{7,8} and reduce photopolymerization time by approximately 75%, resulting in a similar or better conversion than when the composite resin is light polymerized at room temperature for the manufacturer's recommendation time.^{7,8} However, when the composite resin is removed from the heater at 60°C, there is significant and immediate cooling.^{7,8}

Composite resins have different compositions and particle content, which leads to different degrees of fluidity after heating.^{5,9,10} Although rapid heat loss can avoid pulpal damage, any delay before restoration cementation will increase viscosity, leading to a thicker cement line, preventing complete seating, and adversely affecting restoration longevity.^{5,9-11} Determining the time that a composite resin takes to reach the heating temperature and then cool down to room temperature is important to define the working time and to ensure that the heated composite resin technique is optimized.

Thus, the objectives of the present study were to determine the time required for different resins to reach a stable heating temperature, the cooling time required to return to room temperature, and the influence of different heating temperatures on viscosity. The research hypothesis was that time and temperature would affect the viscosity of preheated composite resins.

MATERIALS AND METHODS

Time-Temperature Analysis

This study analyzed the effects of time and temperature on different composite resins (Tab. 1), shade A2, and on their viscosity under heating with precise temperature control based on rheometer tests at the Department of Pharmaceutical Sciences (LabMac – ULEG, UnB, Brazil). The names and brands of the composite resins were hidden during testing, and all experimental tests were conducted blind and by the same researcher.

Table 1 – Characteristics and formulation of composite resins tested

Materials	Type	Manufacturer	Formulation	
			Resin phase	Filler wt% (vol%)
Beautifil II	Nanohybrid	Shofu	Bis-GMA, TEGDMA	83.3 (68.6)
Charisma Classic	Micro-hybrid	Kulzer	Bis-GMA	(61)
Charisma Diamond	Nanohybrid	Kulzer	Bis-GMA, UDMA, TEGDMA, TCD-DI-HEA	(64)
Filtek Z100	Micro-hybrid	3M ESPE	Bis-GMA, TEGDMA	84,5 (66)
Filtek Z250	Micro-hybrid	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA e TEGDMA	81,8 (67,8)
Filtek Z350XT	Nanofill	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA	78.50 (63.3)

Forma	Nanohybrid	Ultradent	Bis-GMA, TEGDMA, Bis-EMA, UDMA	
Llis	Micro-hybrid	FGM	Bis-GMA, Bis-EMA, TEGDMA	77,5 (56)
Opallis	Microfill	FGM	Bis-GMA, Bis-EMA, TEGDMA	80 (72)
Palfique LX5	Supernanofill	Tukuyama	Bis-GMA, TEGDMA	82 (71)
Vittra	Nanofill	FGM	Bis-GMA, Bis-EMA, UDMA, TEGDMA	82 (60)

Bis-GMA, bisphenol-A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; TCD-DI-HEA, Bis-(acryloyloxymethyl) tricyclodecane; Bis-EMA, bisphenol-A ethoxylated dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate

A portable device with digital temperature control was used to heat the composite resins. The temperature reported on the display was the same as the actual temperature inside the device. This was confirmed after five tests at different times and on different days with the thermal sensor (Simpla TA21, AKSO, São Leopoldo, RS, Brazil). The heating protocol was determined for this digital oven as follows: After turning on the device, the temperature was set to 60°C; this was followed by waiting for 40 min so that the device could stably reach the preset temperature. A variation of $\pm 4^{\circ}\text{C}$ was observed.

To determine the time required for the resins to reach this preheating temperature, each composite resin was tested individually at 60°C ($\pm 4^{\circ}\text{C}$). The resins were kept in a refrigerator at 5 °C until the tests were performed when they were removed from the refrigerator and kept at room temperature (approximately 25°C) for 20 min until their temperature stabilized at the laboratory temperature.¹² As soon as their temperature was stable, the resins were individually weighed, and a predetermined volume (0.6 mL) was dispensed as a Ø1-cm specimen onto a glass plate and heated. The thermal sensor was positioned close to the composite resin specimens. All tests were conducted in triplicate (n=3), and the means and standard

deviations of the results were determined, thus obtaining the time required for each resin to reach the predetermined temperature.

As soon as the resin reached the heating temperature, the glass plate was removed, and the specimen temperature read with an infrared thermometer (Simpla TI38, 8:1, AKSO, São Leopoldo, RS, Brazil) with a detection range of -5°C to 380°C. The thermometer was fixed at a distance of 8 cm from the Ø1-cm composite resin specimen on the glass plate. The temperature was analyzed at controlled intervals of 1 min until the resin returned to room temperature (25°C) as follows: T0, immediately after removal of the heat device; T1 1 minute; T2, 2 minutes; T3, 3 minutes; T4, 4 minutes; T5, 5 minutes; T6, 6 minutes; T7, 7 minutes; T8, 8 minutes; T9, 9 minutes; and T10, 10 minutes after removal. The data were analyzed with the statistical software program Stata SE 15.1 (StataCorp, College Station, TX, USA). For the analysis, a two-way analysis of variance (ANOVA), with temperature as the dependent variable and resin-based composite and time as the two independent variables, as well as a post hoc Tukey test, were used ($\alpha=0.05$).

Viscosity Analysis

After the first phase, 11 resins of different brands and characteristics were tested. The three heated resins with the highest fluidity were selected for viscosity testing to determine the rheological properties with the MCR 301 rheometer (Anton Paar Physica). All measurements were performed in a temperature-controlled room (25°C), and the instrument was isolated from external vibrations on a rigid support table.

A permanent oscillatory shear test was performed at 25°C, 35°C, 50°C, and 60°C for the Z100 (3M ESPE, SP, SP, Brazil), Beautifil II (Shofu, SP, SP, Brazil), and Palfique LX5 (Tokuyama, Via Chizzalung, Sandrigo, Italy) composite resins under 1s-1 shear rate with a rotating rheometer. The composite resins were removed from their packaging with a spatula and placed in a semicircular mold to standardize the 0.6-mL volume. The test material was dispensed on the lower plate of the rheometer,

positioned with a 0.05-mm gap between the plates, and heated with the rheometer. The viscosity (Pa·s) was measured until the designated temperature was reached.

To perform the ramp analysis, the composite resins were coupled to the device at room temperature, heated to 60°C for 10 min, remained stable for 5 min more, and returned to room temperature (25°C) in 5 min, with the values defined in the time-temperature analysis. The measurements were performed at 25°C, 35°C, 50°C, and 60°C. Each composite resin was tested four times. Data were analyzed using the statistical software program Stata SE 15.1 (StataCorp). One-way ANOVA and post hoc Tukey tests were used to compare the viscosity at each temperature ($\alpha=0.05$).

RESULTS

The original data were entered into the Figshare repository (DOI: 10.6084/m9.figshare.17708510).

Eleven different composite resins were evaluated at 11 time periods (0-10 minutes after the removal of the heat device). As presented in Table 2, the two-way ANOVA found no statistically significant interaction between the composite resin and time ($F=0.77$, $p=0.9304$), and only the main effect time was significantly different ($p<0.0001$).

Table 2 – Two-way ANOVA of dependent variable as temperature and the two independent variables (resin-based composite and time)

Source	Partial SS†	DF†	MS†	F†	p-value
Model	50863.029	120	423.85857	386.65	<0.0001
Composite	10.360285	10	1.0360285	0.95	0.4925
Time	50767.952	10	5076.7952	4631.16	<0.0001*

Composite	84.716705	100	.84716705	0.77	0.9304
#Time					

Total	51128.316	362	141.23844		
-------	-----------	-----	-----------	--	--

† SS: Sum-of-Squares; DF: Degrees of Freedom; MS: Mean Square; F: F ratio

* Indicates significant difference (p<0.05)

Regarding the main effect time, a difference was observed between T0, T1, T2, T3, T4, T5, and T6 (Tukey, p<0.001). However, this difference was not observed after 6 min. T6 was not statistically different from T7 (p=0.39); T7 was not statistically different from T8 (p=1.0), T8 was not statistically different from T9 (p=1.0), and T9 was not statistically different from T10 (p=1.0). All experimental composite resins returned to the initial temperature (25 °C) within 5 minutes of removal from the heating device (Fig.1).

The composite resins showed a rapid loss of temperature, losing 100% of the temperature acquired after 5 minutes, that is, returning to the ambient temperature (25°C). An average of -48% of the temperature was recorded after 1 minute, -74% after 2 minutes, -87% after 3 minutes, and -95% after 4 minutes.

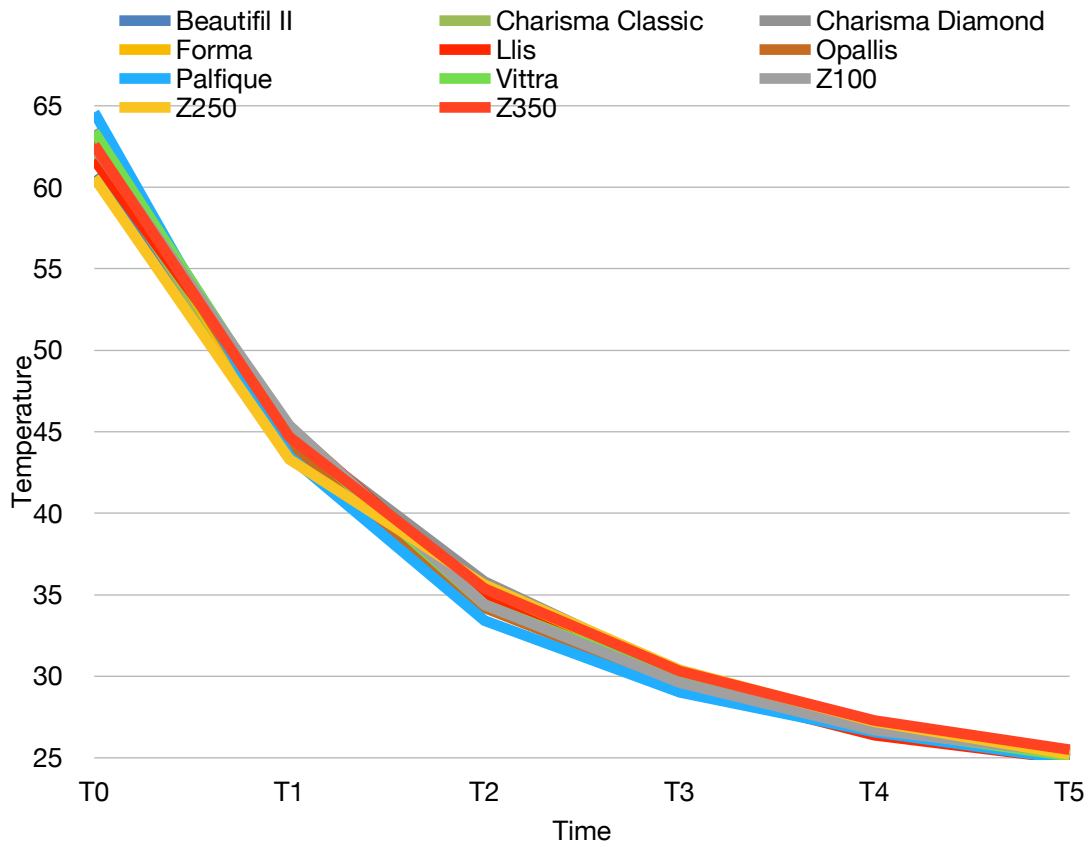


Fig. 1 – Cooling analysis of preheated composite resins according to time (T0-T5) in minutes after removing the heater.

The results for viscosity at 25°C, 35°C, 50°C, and 60°C are shown in Table 3. For the three composite resins analyzed, the increased temperature resulted in a significantly reduced viscosity ($p < 0.05$). At the initial temperature (25°C), Beautifil II (Shofu) had a significantly higher viscosity than Palfique LX5 (Tokuyama) and Z100 (3M ESPE). Palfique LX5 (3M ESPE) started with the lowest viscosity, and, with increasing temperature, showed a significant viscosity reduction compared with the other two composite resins ($p < 0.05$). For both Beautifil II (Shofu) and Z100 (3M ESPE), there was no difference in the viscosity at the temperatures of 50°C and 60°C, while for Palfique LX5 (Tokuyama), no statistical difference was observed at 35°C, 50°C, and 60°C. The hypothesis that time and temperature would influence viscosity properties was accepted.

Table 3 – Mean and SD† of viscosity experimental test (Pa S‡)

Temperature	25 °C	35 °C	50 °C	60 °C
Beautifil II	8442.5 (1361) ^{a,A}	4592.5 (454) ^{a,B}	931 (187) ^{a,C}	452.5 (112) ^{a,C}
Palfique LX5	3012.5 (493) ^{b,A}	576 (58) ^{b,B}	272.75 (11) ^{b,B}	206.5 (13) ^{b,B}
Z100	4595 (442) ^{b,A}	2035 (103) ^{c,B}	1115.5 (118) ^{a,C}	690 (61) ^{c,C}

† SD: Standard Deviation

‡ Pa S: Pascal-second, unit of dynamic viscosity

^{a,b,c} Different lower-case letters identify statistical difference between groups in the same columns; ^{A,B,C} Different upper-case letters identify statistical difference between groups in the same row.

DISCUSSION

Composite resins are direct restorative materials, although they can also be used as cementing agents for indirect restorations. The indication for these materials can vary according to their composition and physical and chemical properties, and additional protocols have been suggested to improve the characteristics of the composite resins, including warming. As the use of preheated resins in dentistry has become popular, the criteria for this technique require clarification.

The composite resins can be preheated in different dry-heating devices. The temperatures for preheating composite resins described in the literature range from 54°C to 68°C, with warm-up times of 5 to 60 minutes.^{5,7-13} Daronch et al.¹² (2006) used a device (Calset, Addent Inc.) in their study and reported that the maximum temperatures reached were 48.3°C and 54.7°C when the preset temperature of the device was set at 54°C and 60°C, respectively.¹² The digital heater used in the present study had been previously tested to avoid possible errors. After repeating the experiments on different days, the temperatures appearing on the digital display and inside the device were confirmed to be identical. Considering this, the experimental resins were subjected to heating at 60°C, with possible variations of $\pm 4^\circ\text{C}$. A device

developed specifically for this purpose facilitates this technique clinically and ensures the standardization of the preheating process, but similar heaters can also be used, provided that the temperature is controlled and remains stable until clinical use.

A wide range of heating times have been specified in the literature, varying from 5 to 60 minutes.^{5,7-13} Eleven composite resins were tested with respect to time-temperature, and, although the materials had different compositions and characteristics, they behaved similarly in terms of heating and cooling, and a statistical difference was not found. The hypothesis tested that time and temperature would influence viscosity properties was accepted. A 10-minute heating time at 60°C (\pm 4°C) was sufficient for all the tested composite resins to reach the desired temperature, which seems a reasonable time for clinical application.

To implement this technique, the speed at which the temperature loss of each resin occurs needs to be established. According to Marcondes et al.¹⁰ (2020), composite resins preheated to 68°C showed temperature losses ranging between 45% and 61% 15 sec after being removed from the heater, an 84% loss of temperature in 30 sec, and a 96% loss in 60 sec, suggesting that clinicians have a working time of between 10 and 15 sec when seating a restoration with preheated composite resin.¹⁰ This can also be seen in this study in part because the composite resins showed a considerable loss of temperature. However, this loss occurred over a longer time period (-48%, 1 minute; -74%, 2 minutes; -87%, 3 minutes; -95%, 4 minutes; and -99%, 5 minutes), thus allowing additional working time and preserving the benefits of viscosity. Other *in vitro* and clinical studies are needed to determine an ideal working time based on these findings by considering all the criteria and sequences for the procedures. Pulpal damage has been a concern with the use of heated composite resins in dental procedures. One reason to consider the procedure safe and unlikely to damage the pulp is the rapid drop in temperature. Daronch et al.¹³ (2007) reported that intrapulpal temperature values may not change significantly because the preheated resins are not as hot as expected when the restoration is delivered.¹³ The luting procedure using preheated resins is limited to restorations of less than 2 mm thick because sufficient light needs to pass through to ensure complete polymerization.^{4,5} Even if the technique is indicated for thin restorations, there would

still be a considerable amount of excess, further reducing the chances of any pulpal damage.^{5,12,13}

Regarding viscoelastic properties, restorative composite resins with different formulations can react differently to preheating.^{10,14,15} The characteristics of the inorganic particles may also influence flowability, affecting the thickness of the film when used as a cement.¹⁰ Preheating to 69°C has been reported to reduce the viscosity of restorative resin composites by 47% to 92% compared with that at 37°C.¹⁰ Therefore, thermal conductivity must also be considered in the choice of material in addition to its handling characteristics^{15,16}, including filler content, particle type, shape, size, nature of the particle surface, and spatial arrangement of the filler within the composite resin¹⁰, because only the relationship between viscosity and the filler content is negative.^{10,17} Although there is a relationship between viscosity and temperature, no change was noted in the mechanical properties of the composite resins at the same loading of particles of different sizes.¹⁸

The viscosity of composite resins, however, is affected by the resin matrix formulation, the interlocking between the filler particles, and the interfacial interaction between the filler particles and resin matrix. An inverse relationship was observed between viscosity and polymerization shrinkage, depending primarily on the monomer types and ratio of the resin matrix¹⁶, and the filler volume has been reported to be more important than the filler weight.¹⁵ Regarding the filler size, composite resins with a small filler size were more viscous than those with a larger filler size, even though the filler content was the same because the number of filler particles was higher for identical filler volumes. As a result, the increased surface area led to a greatly increased interaction between the resin matrix and filler particles and between filler particles.¹⁵ The viscosity of the composite resin with a round filler seems to be much lower than that with an irregular filler because of the lower friction at similar filler sizes.¹⁵ Diluents such as triethyleneglycol-dimethacrylate and urethane dimethacrylate have lower molecular weights and lower viscosities than bisphenol A-glycidyl methacrylate(18, 37).^{15,16} Therefore, their low molecular weights and resultant high numbers of double bonds per unit of weight create a high degree of cross-linking and a relatively high amount of shrinkage.¹⁶ However, manufacturers do not always disclose all the formulation details.

Resins that showed greater fluidity for handling after being heated to 60°C were selected for the analysis of viscosity. These were the nanohybrid composite Beautifil II (Shofu), micro-hybrid Filtek Z100 (3M ESPE), and supernanofill Palfique (Tokuyama). Increased temperature led to a significant viscosity reduction, ranging between 84% and 94%. However, it is expected that the preheated resin composite resins will not show viscosity values in the range of the flowable composite or resin cements.¹⁰ For both Beautifil II (Shofu) and Z100 (3M ESPE), there was no difference in the viscosity from temperatures 50°C to 60°C, while for Palfique LX5 (Tokuyama), no statistical difference between 35°C, 50°C, and 60°C was observed. Among them, Palfique LX5 (Tokuyama) showed the lowest viscosity when heated. Although there is no difference between 50°C and 60°C in the viscosity analysis for the three resins, adopting higher temperatures will extend the working time, since the rapid loss of temperature is a limiting factor of the technique. The overall performance of the preheated composite resin technique depends on proper material selection and correct procedure because the change in viscosity upon preheating alone is not an appropriate parameter for selecting a restorative composite resin for luting purposes.^{10,19} The filler content of the resins does not interfere directly with the thickness of the film¹⁵, and Marcondes et al.¹⁰ (2020) reported that some composite resins that showed the highest reduction in viscosity were not always able to yield the thinnest films.¹⁰

Based on the results of this *in vitro* study, heating composite resins influences their viscosity, consistent with previous studies.^{14-16,20} Ahn et al.²⁰ (2015) speculated that preheating increased the mobility of the unpolymerized monomer and allowed a better distribution of the filler particles, becoming more fluid when it was manipulated. Even though preheating alters the structure of composite resins, it seems to be beneficial because this improvement in the internal structure persists, even after cooling.²⁰

Limitations of this study included that only 11 composite resins (shade A2) were investigated. Even though the preheating of composite resins affects their viscosity, future studies should be conducted on how different monomers and filler features might influence the reaction of composite resin to preheating. A working time from 30 seconds to 1 minute may preserve their benefits and allows for better manipulation of composite resins during the luting procedure, but it seems not to be reproducible in the

clinical scenario. In addition, clinical and laboratory studies should be developed to simulate the clinical procedure and determine the protocol for composite preheating.

CONCLUSIONS

The preheating of composite resins affects their viscosity; however, the following aspects are relevant to this technique. The time of 10 minutes at ≥ 60 °C seems to be adequate to heat composite resins, reducing their viscosity by at least 84%. All tested resins lost 100% of the temperature gained during heating after 5 minutes.

REFERENCES

1. Martin MP. Material and clinical considerations for full-coverage indirect restorations. *Compend Contin Educ Dent* 2012;33(6):2-5.
2. Ferrari M, Dagostin A, Fabianelli A. Marginal integrity of ceramic inlays luted with a self-curing resin system. *Dent Mater* 2003;19(4):270-276.
3. Fabianelli A, Goracci C, Bertelli E, Davidson CL, Ferrari M. A clinical trial of Empress II porcelain inlays luted to vital teeth with a dual-curing adhesive system and a self-curing resin cement. *J Adhes Dent* 2006;8(6):427-431.
4. Vargas MA, Bergeron C, Diaz-Arnold A. Cementing all-ceramic restorations: recommendations for success. *J Am Dent Assoc* 2011;142(2):20-24.
5. Acquaviva PA, Cerutti F, Adami G, Gagliani M, Ferrari M, Gherlone E, et al. Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: A micro-Raman analysis. *J Dent* 2009;37(8):610-615.

6. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002;87(6):642-649.
7. Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. *J Dent Res* 2006;85(1):38-43.
8. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. *J Dent Res* 2005;84(7):663-667.
9. Froes-Salgado NR, Silva LM, Kawano Y, Francci C, Reis A, Loguercio AD. Composite pre-heating: effects on marginal adaptation, degree of conversion and mechanical properties. *Dent Mater* 2010;26(9):908-914.
10. Marcondes RL, Lima VP, Barbon FJ, Isolan CP, Carvalho MA, Salvador MV, et al. Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness. *Dent Mater* 2020;36(10):1356-1364.
11. Magne P, Razaghy M, Carvalho MA, Soares LM. Luting of inlays, onlays, and overlays with preheated restorative composite resin does not prevent seating accuracy. *Int J Esthet Dent* 2018;13(3):318-332.
12. Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent* 2006;18(6):340-350.
13. Daronch M, Rueggeberg FA, Hall G, De Goes MF. Effect of composite temperature on in vitro intrapulpal temperature rise. *Dent Mater* 2007;23(10):1283-1288.
14. Al-Ahdal K, Silikas N, Watts DC. Rheological properties of resin composites according to variations in composition and temperature. *Dent Mater* 2014;30(5):517-524.
15. Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. *Dent Mater* 2006;22(6):515-526.

16. Ellakwa A, Cho N, Lee IB. The effect of resin matrix composition on the polymerization shrinkage and rheological properties of experimental dental composites. *Dent Mater* 2007;23(10):1229-1235.
17. Deb S, Di Silvio L, Mackler HE, Millar BJ. Pre-warming of dental composites. *Dent Mater* 2011;27(4):51-59.
18. Habib E, Wang R, Zhu XX. Correlation of resin viscosity and monomer conversion to filler particle size in dental composites. *Dent Mater* 2018;34(10):1501-1508.
19. Gugelmin BP, Miguel LCM, Baratto Filho F, Cunha LFD, Correr GM, Gonzaga CC. Color Stability of Ceramic Veneers Luted With Resin Cements and Pre- Heated Composites: 12 Months Follow-Up. *Braz Dent J* 2020;31(1):69-77.
20. Ahn KH, Lim S, Kum KY, Chang SW. Effect of preheating on the viscoelastic properties of dental composite under different deformation conditions. *Dent Mater J* 2015;34(5):702-706.

DATA AVAILABILITY

Datasets related to this article can be found at DOI: 10.6084/m9.figshare.17708510, hosted at Figshare repository.

4 DISCUSSÃO GERAL E CONCLUSÕES

4.1 DISCUSSÃO GERAL

O pré-aquecimento das resinas compostas para cimentação de restaurações indiretas pode ser considerado uma técnica viável do ponto de vista clínico e biológico, no entanto, há uma discrepância na literatura quanto aos parâmetros que a norteiam, uma vez que há no mercado diversas marcas e formulações resinosas que podem interferir diretamente sobre o resultado final esperado. Os detalhes metodológicos, como tempo de aquecimento, dispositivo de escolha, a forma de aplicação, entre outros, também são considerados decisivos. (38) A partir de uma revisão ampla da literatura acerca desse pré-tratamento utilizando calor, é possível observar que o dispositivo utilizado para aquecer pode variar entre os testes, porém o mais comumente relatado é o Calset (AdDent Inc.). (27, 39, 40) Esse dispositivo permite aquecer os materiais e manter a temperatura constante a 37°C, 54°C e 68°C, além de disponibilizar diversas opções de armazenamento (seringas, compules, bisnagas, diretamente sobre a restauração). (Fig. 1)



Figura 1 – Calset (AdDent Inc., Cosmedent), dispositivo de aquecimento com suas formas de aplicação. Imagem retirada diretamente do site do fabricante, disponível no link <https://cosmedent.com.br/produto/calset/>

Outras fontes de calor foram relatadas na literatura, nas quais se fazem determinantes o aquecimento a seco e a estabilidade da temperatura do dispositivo para que o material atinja o resultado esperado. No estudo laboratorial realizado neste trabalho, foi utilizado um dispositivo genérico de aquecimento a seco, digital e, para garantir que a temperatura pré-programada no display fosse mesma do interior da câmara de aquecimento, foi realizada quintuplicata de conferência a partir de um termômetro digital com capacidade de aferição de -5°C e 380°C (Simpla TA21, AKSO, São Leopoldo, RS, Brasil), em dias diferentes. Após observação de estabilidade e checagem da temperatura, observou-se que quando a temperatura selecionada era de 60°C , havia uma variação no termômetro com valores aproximados a 60°C (± 4), o que seria compatível com os equipamentos utilizados e relatados na literatura. (38) O estudo de Daronch et. al., utilizando o Calset, verificou que a temperatura máxima alcançada foi de $48,3^{\circ}\text{C}$ e $54,7^{\circ}\text{C}$, quando selecionadas 54°C e 60°C , respectivamente. (8) Esse achado, bem como o respaldo de outros estudos científicos que utilizaram outros equipamentos com o mesmo fim, como aquecedores de cera, incubadoras e fornos, tornam a técnica ainda mais viável clinicamente, não sendo obrigatório o uso de aquecedores específicos. (8, 38, 41, 42)

Outro ponto a ser observado é se há discrepância do tempo necessário para que diferentes resinas atinjam a mesma temperatura. No teste laboratorial conduzido, onze espécimes de diferentes resinas com 1 cm de diâmetro e volume de 0,6mL foram testados em triplicatas, observando-se que o tempo de 10 minutos sob aquecimento a 60°C (± 4) é suficiente para que todos os compósitos testados alcancem a temperatura pré-definida, não havendo diferença significativa entre eles. Isso está de acordo com os achados da literatura, onde após revisão sistemática criteriosa, o aquecimento por um tempo de 15 minutos, pareceu ser razoável para que as resinas alcancem e estabilizem a temperatura (9, 38) e essa variação de tempo pode estar diretamente relacionada ao volume do material a ser aquecido. Contudo, ao serem removidas da fonte de aquecimento e armazenadas em temperatura ambiente (25°C), as resinas tiveram uma queda térmica exponencial, retornando aos valores iniciais em apenas 5 minutos, o que afetaria diretamente o tempo de trabalho do clínico. Em consonância a isso, o método como a resina será aquecida também pode exercer influência na temperatura alcançada. Comparando dois protocolos distintos, Daronch

et. al. observaram que quando aquecida diretamente nas seringas de aplicação, a temperatura real da resina é mais alta do que nos outros métodos. (8) Levando em consideração esses aspectos, pode-se justificar o uso das resinas diretamente em dispositivos de inserção (seringas ou compules) no caso de restaurações diretas e, no caso de cimentações, diretamente sob a restauração indireta. (8-10, 38) Dessa forma, o clínico conseguiria uma maior facilidade e velocidade de aplicação durante o procedimento.

O aumento da temperatura diminui a viscosidade do sistema, por permitir uma maior mobilidade molecular dos grupos reativos presentes na matriz resinosa, resultando em uma polimerização adicional e uma possível interferência na taxa de conversão. (14) Também ocorre um aumento do volume-livre, ou seja, do espaço entre as cadeias moleculares que não se acomodaram com perfeição, aumentando a mobilidade de alguns radicais aprisionados na rede polimérica. (22) Embora as resinas se tornem mais fluidas ao serem submetidas à fonte de calor, por apresentarem diferentes formulações e componentes, pode haver diferença dessa influência entre as marcas e tipos de resinas, ainda que com a mesma classificação. (18) Isso porque quanto maior a % de volume de carga em sua composição, maior será viscosidade. (18) Entretanto, no estudo *in vitro* realizado neste trabalho, foram testadas três resinas que se comportaram de maneira similar sob elevadas temperaturas, tendo sua viscosidade reduzida em 84-95% quando comparadas à temperatura ambiente. Essa característica se faz muito importante para procedimentos de cimentação, uma vez que pode torná-los mais práticos, além de combinar algumas vantagens conseguidas com a técnica, como por exemplo, melhor adaptação marginal e escoamento.

Baseado nos resultados de Darabi et. al., o pré-aquecimento de resinas compostas regulares diminui as falhas marginais, tanto em esmalte quanto em dentina, quando comparadas às restaurações realizadas utilizando compósitos em temperatura ambiente. (43) Isso devido a maior capacidade de escoamento, permitindo uma melhor e mais rápida adaptação às paredes cavitárias, (43) além do adequado assentamento da restauração com uma menor exposição da linha de cimentação em meio oral. (10) Quando comparadas com as resinas fluidas, as resinas convencionais aquecidas apresentam menor fluidez, todavia é possível observar uma

maior quantidade de carga inorgânica, bem como propriedades mecânicas superiores. (1-4, 6) Os aspectos reológicos e sua influência nas propriedades mecânicas não só afetam a viscosidade e característica de manipulação, mas também afetam sua durabilidade. (6) As resinas que foram testadas quanto à viscosidade nesse trabalho *in vitro* e que apresentaram uma importante e similar redução da viscosidade inicial podem ser consideradas mais maleáveis em detrimento de outras opções disponíveis no mercado, mesmo em temperatura ambiente, tendendo a reagir seguindo a mesma lógica quando aquecidas, (10, 19) o que poderia direcionar a escolha da resina composta utilizada na referida técnica.

As resinas compostas submetidas ao calor podem apresentar redução entre 4 e 77% da espessura de película, dependendo do material, porém somente essa alteração não seria um parâmetro suficiente para a escolha da resina composta indicada para o procedimento, sobretudo em casos de cimentação, pois a espessura de película conseguida não é diretamente proporcional à redução da viscosidade. (10) Não é possível afirmar que há uma correlação linear entre a concentração das partículas inorgânicas, a redução da viscosidade e a espessura da película, isso porque não só a concentração da carga afetaria essa propriedade, mas também o tipo de partícula, tamanho, volume, natureza da partícula e o arranjo espacial entre elas. (10, 19, 44, 45) O inadequado escoamento do material e assentamento de restaurações delgadas, como laminados cerâmicos, poderia provocar a fratura da restauração pela pressão exercida durante o posicionamento da restauração. (19) A consequência de uma espessura maior exposta ao meio oral é adaptação marginal pobre e microinfiltração a longo prazo, além de uma maior interferência estética pela instabilidade de cor que as resinas podem apresentar a longo prazo. (11, 27) A linha formada pela resina composta aquecida é 4,5 vezes mais espessa do que linhas conseguidas com cimentos resinosos, utilizando o mesmo sistema adesivo. (17) Dessa forma, uma possível compensação clínica seria a associação entre aquecimento e o uso de vibração ultrassônica, promovendo ainda mais a redução da espessura de película com valores abaixo de 50µm, sendo recomendado após os três minutos iniciais, onde a restauração já teria sido posicionada, bem como os excessos mais grosseiros removidos. (10, 12, 19, 45) Apesar disso, a adição de outra etapa

clínica necessária vai de encontro com o conceito de simplificação dos sistemas atuais.

Embora as resinas, de maneira geral, fiquem mais fluidas com o aquecimento, nem todas são indicadas para a técnica. (19) Resinas como Z350XT (3M ESPE), TPH Spectrum (Dentsply), Charisma Diamond (Kulzer), Essentia (GC) não parecem ser a melhor alternativa em casos de cimentação por apresentarem uma espessura de película mais espessa das que outras resinas testadas, como a Gradia, IPS Empress Direct e a Z100. (10, 38, 46) Outros fatores também devem ser levados em consideração para pautar a escolha do material, como estabilidade de cor, resistência à abrasão, resistência à solubilidade, microdureza, capacidade de polimerização, entre outras propriedades físicas e mecânicas. (8, 10, 19, 32, 47) De acordo com os resultados encontrados no trabalho laboratorial executado, as resinas que tinham em sua composição apenas moléculas de Bis-GMA e TEGDMA apresentaram maior facilidade de manipulação do que resinas com outros componentes adicionais, no entanto, a concentração de cada partícula presente na matriz resinosa poderia afetar diretamente esse aspecto e essa informação não é fornecida pelas empresas.

Apesar de alguns estudos confirmarem a interferência positiva no grau de conversão, (28, 29, 48) somente essa associação isolada não deve ser usada como critério, pois devem ser levadas em consideração as características da rede polimérica, ou seja, embora um polímero tenha grau de conversão similar, a densidade das ligações cruzadas pode ser diferente, afetando a estabilidade das resinas compostas. (4, 22) Além disso, a espessura das restaurações indiretas pode afetar diretamente no grau de conversão monomérico por constituir uma barreira física criada entre o fotoativador e o agente cimentante, em outras palavras, a espessura dessas restaurações, assim como a opacidade delas podem interferir nos resultados, sendo observadas que espessuras maiores que 1,0mm provocam uma queda considerável no grau de conversão, independente do material de escolha utilizado para cimentação. (19, 27, 29, 46) Outros autores não observaram essa vantagem em relação ao grau de conversão, apesar de sugerirem o uso da resina composta aquecida em função dos seus benefícios clínicos, como melhor adaptação marginal e menor taxa de degradação, isso porque a matriz polimérica absorve menos solvente e perde menos componentes para o meio oral, diminuindo essa taxa. (19, 26, 27, 49)

Com o objetivo de analisar os resultados apresentados pelas resinas compostas aquecidas utilizadas como agente cimentante, os cimentos resinosos são usados como controle na maioria dos artigos. Sob essa ótica, no que diz respeito à resistência de união, as resinas aquecidas não foram significativamente diferentes quando comparado aos cimentos resinosos, garantindo, no mínimo, a mesma efetividade (13, 34) Além disso, o aquecimento gera maior resistência à fratura do que restaurações cimentadas com cimentos resinosos. (17) Em contrapartida, visando o sucesso clínico, a interposição da restauração é fator decisivo, pois resinas compostas não apresentam dois tipos de curas de polimerização, sendo obrigatório seu uso somente em casos onde haja passagem adequada de luz. (6)

Muitos fatores exercem influência na cinética de fotopolimerização, como a concentração de fotoiniciador, a taxa de produção de radicais, a quantidade ou concentração de monômero, a razão entre as taxas de propagação e terminação, a mudança na viscosidade do sistema, a densidade de potência do aparelho fotoativador e o tempo de exposição de luz, a espessura do material, a diferença nos índices de refração das partículas de carga e da matriz resinosa, o tipo de partícula, seu tamanho, distribuição da massa resinosa, a opacidade da resina e a sua cor. (5) Além do mais, o pré-aquecimento das resinas compostas acelera a reação de polimerização, podendo estar associado a um maior estresse de polimerização. (28) Apesar disso, a fotopolimerização conduzida no modo “*soft start*” é capaz de compensar essa tensão, sem reduzir o grau de conversão. (48, 50) Por ser inerente ao processo de endurecimento do material, a contração de polimerização é inevitável e essas forças de contração geradas internamente no material podem ser transmitidas, em parte, à interface adesiva dente-restauração, comprometendo a integridade marginal e apresentando, como consequência, a formação de fendas marginais que podem levar à microinfiltração da restauração e à sensibilidade pós-operatória. (23) É nessa situação específica que os valores da tensão de contração gerada pela resina composta passam a ter maior importância do que os valores de contração volumétrica. (23) A tensão gerada pela contração volumétrica da matriz resinosa é diretamente dependente do volume total do material inserido na cavidade, ou seja, no caso de restaurações diretas utilizando resinas aquecidas, ainda assim é recomendado a inserção de forma incremental, evitando a deflexão das paredes

envolvidas no preparo ao mesmo tempo. (5, 8, 22) Para cimentações, o uso das resinas aquecidas já se dá em volume reduzido, uma vez que a quantidade de material utilizado acaba sendo limitada pela espessura entre a restauração a ser cimentada e o remanescente.

Outra preocupação por parte do clínico é a possibilidade de uma injúria pulpar ocasionada pelo sobreaquecimento do remanescente ao entrar em contato com o material aquecido. No entanto, esse risco não é considerado maior do que algumas etapas já executadas e que também geram o aquecimento, como o preparo cavitário e a fotoativação. (5, 8, 28) O aumento dessa temperatura depende da intensidade do fotopolimerizador, tempo de exposição da luz, distância entre a ponta do fotopolimerizador e a superfície da resina composta, composição e cor do compósito, espessura do material e da dentina remanescente. (5, 8, 28) A espessura da dentina entre a parede pulpar ou axial e a polpa dental parece ser um dos fatores mais importantes na determinação da proteção pulpar, pois a dentina age como uma barreira de proteção contra estímulos lesivos, promovendo um equilíbrio térmico. (9) Em soma disso, em casos de cimentação, o selamento dentinário imediato e seu reforço e recobrimento com resina fluida têm sido indicados após o preparo cavitário e antes da etapa de moldagem. (51) Com isso, quando a cimentação ocorrer, a dentina estará devidamente protegida contra estímulos externos, favorecendo ainda mais essa possível proteção contra o aquecimento gerado. (51) Em paralelo a isso, o tempo entre a remoção do material da fonte aquecedora até sua inserção promove uma queda exponencial da temperatura, corroborando com os achados de que a técnica é segura do ponto de vista biológico. (9, 10)

Apesar de estar vinculada a algumas vantagens clínicas e laboratoriais, o uso das resinas compostas aquecidas não deve ser empregado de modo indiscriminado. (11) Sobretudo porque o clínico deve trabalhar de maneira rápida, dispensando o material, adaptando, removendo os excessos e esculpindo, quando necessário, dentro de um tempo limitado. Dessa maneira, protocolos de aplicação devem ser estabelecidos e testados tanto em aspectos laboratoriais, mas principalmente visando sua eficiência e praticidade, para que seja de fácil acesso e reprodução. Um tempo de trabalho extremamente reduzido inviabiliza a técnica do ponto de vista clínico, por isso, estudos com enfoque nessa aplicação devem ser realizados, testando

metodologias de aplicação, cronometrando o tempo das etapas e buscando encontrar formas mais práticas para que o procedimento seja executado seguindo o rigor científico, porém com eficiência e eficácia clínica.

4.2 CONCLUSÕES

Baseado nos resultados dos trabalhos realizados (*scoping review* e estudo *in vitro*), é possível avaliar que o pré-aquecimento das resinas compostas é um método que interfere na sua viscosidade e favorece sua característica de manipulação e inserção do material, por torná-la mais fluida. Os aquecedores utilizados para esse fim, precisam ter ajuste e controle de temperatura, bem como manter a temperatura interna estável para que a resina alcance os valores desejados. O aquecimento deve ser realizado por, pelo menos, 10 minutos a 60°C (± 4) e o tempo de trabalho deve ser respeitado com rigor, visto que após 5 minutos todas as resinas retornaram à temperatura ambiente e, conseqüentemente, à sua viscosidade inicial.

5 REFERÊNCIAS

1. Ferracane JL. Resin composite--state of the art. *Dent Mater.* 2011;27(1):29-38.
2. Bowen RL. Compatibility of various materials with oral tissues. I: The components in composite restorations. *J Dent Res.* 1979;58(5):1493-503.
3. Lutz F, Phillips RW. A classification and evaluation of composite resin systems. *Journal of Prosthetic Dentistry.* 1983;50(4):480-8.
4. Peutzfeldt A. Resin composites in dentistry: the monomer systems. *Eur J Oral Sci.* 1997;105(2):97-116.
5. Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. *J Dent Res.* 2006;85(1):38-43.
6. Zhang X, Zhang Q, Meng X, Ye Y, Feng D, Xue J, et al. Rheological and Mechanical Properties of Resin-Based Materials Applied in Dental Restorations. *Polymers (Basel).* 2021;13(17).
7. Heintze SD, Ilie N, Hickel R, Reis A, Loguercio A, Rousson V. Laboratory mechanical parameters of composite resins and their relation to fractures and wear in clinical trials-A systematic review. *Dent Mater.* 2017;33(3):e101-e14.
8. Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *Journal of esthetic and restorative dentistry : official publication of the American Academy of Esthetic Dentistry [et al].* 2006;18(6):340-50.

9. Lopes LCP, Terada RSS, Tsuzuki FM, Giannini M, Hirata R. Heating and preheating of dental restorative materials-a systematic review. *Clin Oral Investig.* 24. Germany 2020. p. 4225-35.
10. Marcondes RL, Lima VP, Barbon FJ, Isolan CP, Carvalho MA, Salvador MV, et al. Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness. *Dent Mater.* 2020;36(10):1356-64.
11. Barbon FJ, Isolan CP, Soares LD, Bona AD, de Oliveira da Rosa WL, Boscato N. A systematic review and meta-analysis on using preheated resin composites as luting agents for indirect restorations. *Clin Oral Investig.* 2022.
12. 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.* 12. India: Copyright: (c) 2021 Contemporary Clinical Dentistry.; 2021. p. 313-6.
13. Urcuyo Alvarado MS, Escobar Garcia DM, Pozos Guillen AJ, Flores Arriaga JC, Romo Ramirez GF, Ortiz Magdaleno M. Evaluation of the Bond Strength and Marginal Seal of Indirect Restorations of Composites Bonded with Preheating Resin. *Eur J Dent.* 2020;14(4):644-50.
14. Wagner WC, Aksu MN, Neme AM, Linger JB, Pink FE, Walker S. Effect of pre-heating resin composite on restoration microleakage. *Oper Dent.* 2008;33(1):72-8.
15. Coelho NF, Barbon FJ, Machado RG, Boscato N, Moraes RR. Response of composite resins to preheating and the resulting strengthening of luted feldspar ceramic. *Dent Mater.* 2019;35(10):1430-8.
16. Kameyama A, Bonroy K, Elsen C, Luhrs AK, Suyama Y, Peumans M, et al. Luting of CAD/CAM ceramic inlays: direct composite versus dual-cure luting cement. *Biomed Mater Eng.* 2015;25(3):279-88.

17. Barbon FJ, Moraes RR, Isolan CP, Spazzin AO, Boscato N. Influence of inorganic filler content of resin luting agents and use of adhesive on the performance of bonded ceramic. *J Prosthet Dent.* 2019;122(6):566.e1-.e11.
18. Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. *Dent Mater.* 2006;22(6):515-26.
19. Gugelmin BP, Miguel LCM, Baratto Filho F, Cunha LFD, Correr GM, Gonzaga CC. Color Stability of Ceramic Veneers Luted With Resin Cements and Pre- Heated Composites: 12 Months Follow-Up. *Braz Dent J.* 2020;31(1):69-77.
20. Castro FL, Campos BB, Bruno KF, Reges RV. Temperature and curing time affect composite sorption and solubility. *J Appl Oral Sci.* 2013;21(2):157-62.
21. Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Degree of conversion and mechanical properties of a BisGMA:TEGDMA composite as a function of the applied radiant exposure. *J Biomed Mater Res B Appl Biomater.* 2008;84(2):503-9.
22. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. *J Dent Res.* 2005;84(7):663-7.
23. Rueggeberg FA, Daronch M, Browning WD, MF DEG. In vivo temperature measurement: tooth preparation and restoration with preheated resin composite. *J Esthet Restor Dent.* 2010;22(5):314-22.
24. Daronch M, Rueggeberg FA, Hall G, De Goes MF. Effect of composite temperature on in vitro intrapulpal temperature rise. *Dent Mater.* 2007;23(10):1283-8.
25. Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Effect of temperature on composite polymerization stress and degree of conversion. *Dent Mater.* 2014;30(6):613-8.

26. da Silva JC, Reges RV, Rege ICC, Cruz CAS, Vaz LG, Estrela C, et al. Pre-heating mitigates composite degradation. *Journal of Applied Oral Science*. 2015;23(6):571-9.
27. Acquaviva PA, Cerutti F, Adami G, Gagliani M, Ferrari M, Gherlone E, et al. Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: A micro-Raman analysis. *Journal of Dentistry*. 2009;37(8):610-5.
28. Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Effect of temperature on composite polymerization stress and degree of conversion. *Dental Materials*. 2014;30(6):613-8.
29. Mundim FM, Garcia LDFR, Cruvinel DR, Lima FA, Bachmann L, Pires-De-Souza FDCP. Color stability, opacity and degree of conversion of pre-heated composites. *Journal of Dentistry*. 2011;39(SUPPL. 1):e25-e9.
30. Ayub KV, Santos GC, Jr., Rizkalla AS, Bohay R, Pegoraro LF, Rubo JH, et al. Effect of preheating on microhardness and viscosity of 4 resin composites. *J Can Dent Assoc*. 2014;80:e12.
31. Goujat A, Abouelleil H, Colon P, Jeannin C, Pradelle N, Seux D, et al. Marginal and internal fit of CAD-CAM inlay/onlay restorations: A systematic review of in vitro studies. *J Prosthet Dent*. 2019;121(4):590-7.e3.
32. Froes-Salgado NR, Silva LM, Kawano Y, Francci C, Reis A, Loguercio AD. Composite pre-heating: effects on marginal adaptation, degree of conversion and mechanical properties. *Dent Mater*. 2010;26(9):908-14.
33. Lima MO, Catelan A, Marchi GM, Lima DA, Martins LR, Aguiar FH. Influence of pre-heating and ceramic thickness on physical properties of luting agents. *J Appl Biomater Funct Mater*. 2018;16(4):252-9.

34. Goulart M, Borges Veleda B, Damin D, Bovi Ambrosano GM, Coelho de Souza FH, Erhardt MCG. Preheated composite resin used as a luting agent for indirect restorations: effects on bond strength and resin-dentin interfaces. *Int J Esthet Dent*. 2018;13(1):86-97.
35. Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent*. 2006;18(6):340-50; discussion 51.
36. Lousan do Nascimento Poubel D, Ghanem Zanon AE, Franco Almeida JC, Vicente Melo de Lucas Rezende L, Pimentel Garcia FC. Composite Resin Preheating Techniques for Cementation of Indirect Restorations. *International Journal of Biomaterials*. 2022;2022:5935668.
37. Ellakwa A, Cho N, Lee IB. The effect of resin matrix composition on the polymerization shrinkage and rheological properties of experimental dental composites. *Dent Mater*. 2007;23(10):1229-35.
38. Poubel D, Zanon AE, Almeida JC, Rezende L, Garcia FC. Composite resin preheating techniques for cementation of indirect restorations. *International Journal of Biomaterials*; 2022.
39. Magne P, Knezevic A. Influence of overlay restorative materials and load cusps on the fatigue resistance of endodontically treated molars. *Quintessence international (Berlin, Germany : 1985)*. 2009;40(9):729-37.
40. Magne P, Knezevic A. Simulated fatigue resistance of composite resin versus porcelain CAD/CAM overlay restorations on endodontically treated molars. *Quintessence International*. 2009;40(2).

41. Olivares JM, Hidalgo A, Pavez JP, Benadof D, Iribarra R. Functional and esthetic restorative treatment with preheated resins in a patient with ectodermic dysplasia: A clinical report. *J Prosthet Dent.* 2018;119(4):526-9.
42. Rickman LJ, Padipatvuthikul P, Chee B. Clinical applications of preheated hybrid resin composite. *British dental journal.* 2011;211(2):63-7.
43. Darabi F, Tayefeh-Davalloo R, Tavangar SM, Naser-Alavi F, Boorboo-Shirazi M. The effect of composite resin preheating on marginal adaptation of class II restorations. *J Clin Exp Dent.* 2020;12(7):e682-e7.
44. Deb S, Di Silvio L, Mackler HE, Millar BJ. Pre-warming of dental composites. *Dental materials.* 2011;27(4):e51-e9.
45. Falacho RI, Marques JA, Palma PJ, Roseiro L, Caramelo F, Ramos JC, et al. Luting indirect restorations with resin cements versus composite resins: Effects of preheating and ultrasound energy on film thickness. *J Esthet Restor Dent.* 2021.
46. Tomaselli LdO, Oliveira DCRSd, Favarão J, Silva AFd, Pires-de-Souza FdCP, Geraldeli S, et al. Influence of Pre-Heating Regular Resin Composites and Flowable Composites on Luting Ceramic Veneers with Different Thicknesses. *Braz dent j.* 2019;30(5):459-66.
47. Elkaffass AA, Eltoukhy RI, Elnegoly SAE, Mahmoud SH. Influence of preheating on mechanical and surface properties of nanofilled resin composites. *J Clin Exp Dent.* 2020;12(5):e494-500.
48. Taubock TT, Tarle Z, Marovic D, Attin T. Pre-heating of high-viscosity bulk-fill resin composites: effects on shrinkage force and monomer conversion. *J Dent.* 2015;43(11):1358-64.

49. Lohbauer U, Zinelis S, Rahiotis C, Petschelt A, Eliades G. The effect of resin composite pre-heating on monomer conversion and polymerization shrinkage. *Dental Materials*. 2009;25(4):514-9.

50. El-Korashy DI. Post-gel Shrinkage Strain and Degree of Conversion of Preheated Resin Composite Cured Using Different Regimens. *Operative Dentistry*. 2010;35(2):172-9.

51. de Carvalho MA, Lazari-Carvalho PC, Polonial IF, de Souza JB, Magne P. Significance of immediate dentin sealing and flowable resin coating reinforcement for unfilled/lightly filled adhesive systems. *J Esthet Restor Dent*. 2021;33(1):88-98.

6 PRESS RELEASE

A odontologia restauradora, muito mais do que simplesmente devolver uma estrutura perdida, deve priorizar o elemento dentário. Dessa forma, os tratamentos propostos devem ser baseados na longevidade não só do procedimento executado, mas na manutenção do remanescente de forma saudável e funcional pelo maior tempo possível. Para que isso seja alcançado, diversas técnicas restauradoras têm sido constantemente investigadas e atualizadas, como, por exemplo, o uso de materiais já consolidados nos meios científico e clínico, porém com aplicações distintas do habitual. As resinas compostas, por exemplo, são consideradas excelentes materiais restauradores que, além de serem indicadas para procedimentos diretos, também têm sido empregadas na confecção de restaurações semidiretas. Ademais, seu uso tem sido extrapolado para cimentações de restaurações protéticas, desde que submetidas ao aquecimento prévio, alcançando benefícios em relação a sua viscosidade e outras propriedades.

Por meio deste trabalho, foi possível extenuar a literatura acerca dos aspectos relacionados ao pré-aquecimento das resinas compostas utilizadas para cimentação, demonstrando respaldo científico para que técnica seja empregada, porém apontando detalhes imprescindíveis para o sucesso e longevidade do procedimento.






7 APÊNDICES

Hindawi
International Journal of Biomaterials
Volume 2022, Article ID 5935668, 10 pages
<https://doi.org/10.1155/2022/5935668>



Review Article

Composite Resin Preheating Techniques for Cementation of Indirect Restorations

**Déborah Lousan do Nascimento Poubel , Ana Elisa Ghanem Zanon ,
Júlio César Franco Almeida , Liliana Vicente Melo de Lucas Rezende ,
and Fernanda Cristina Pimentel Garcia **

Department of Dentistry, School of Health Sciences, University of Brasilia (UnB), Federal District, DF, Brazil

Correspondence should be addressed to Fernanda Cristina Pimentel Garcia; garciafc@unb.br

Received 28 December 2021; Accepted 18 February 2022; Published 23 March 2022

Academic Editor: Wen-Cheng Chen

Copyright © 2022 Déborah Lousan do Nascimento Poubel et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Purpose. Resin-based materials have been preheated by using different techniques and commercial devices. However, a consensus on the clinical protocol for cementing with preheated composite resins is lacking. The aim of this scoping review was to identify the different methods used for heating composite resins as used for cementing indirect adhesive restorations and to determine the benefits and limitations. *Study Selection.* A search was performed on PubMed/MEDLINE, Embase, Cochrane, Web of Science, Scopus, LIVIVO, and the nonpeer-reviewed literature database. Studies on preheating composite resins for cementing indirect restorations were included, with no restrictions on the type of study, year of publication, or language. The following data were extracted: preheating technique, the device used for preheating, preset temperature, and warming time. *Results.* In total, 304 studies were identified. After removing duplicates, 270 articles were selected, and 14 articles were included in the final evaluation. Half of the included studies reported similar preheating techniques using the Calset device for composite resins. The temperatures of 54°C and 68°C were most frequently reported, with a mean warming time of 5 minutes. *Conclusions.* Preheating composite resins for the cementation of indirect restorations reduces viscosity, but the material must be used promptly after removal from the device. *Practical Implications.* Different methodologies for preheating composite resins have been reported and used in clinical dental practice. To achieve good results and guide the clinician on use, the techniques for heating composite resins for cementation need to be standardized. Keeping the material warm until the restorative procedure, the thickness of the indirect restoration, and the composition of the composite resins can directly affect the outcome of the procedure.

1. Introduction

Following the concept of adhesion, as described by Buonocore in 1955 [1], the basic concepts of dentistry have changed, with a focus on the most conservative intervention. Among the restorative treatments available, direct composite resin restorations are indicated for teeth with minor loss of structure, while indirect restorations are indicated for teeth with significant functional, structural, and/or esthetic deficiencies [2]. The indirect restorative treatment, which includes laminates, onlays/inlays, and crowns using ceramic, metal, or laboratory-fabricated composite resin as materials, allows for better mechanical properties and marginal

adaptation when compared with directly placed composite resins, avoiding polymerization shrinkage and improving wear resistance [3]. Ceramic restorations are resistant to fatigue, with low thermal conductivity and satisfactory biocompatibility [3]. Moreover, indirect restorations which are cemented to the prepared teeth using luting types of cement have better marginal adaptation [4, 5]. Of the various materials available for cementation, resin types of cement, available in light-, chemically, or dual-polymerized forms, are currently preferred because of their hardness, low solubility in oral fluid, and micromechanical bonding to enamel and dentin [2]. Given the different indirect restorative types of cement, other materials, including different

types of composite resins, have been studied for this purpose. Light-polymerized composite resins have advantages over dual-polymerized resin types of cement that include stain resistance, color stability, and mechanical wear resistance because of increased inorganic filler loading [6, 7]. The high inorganic filler content directly influences the viscosity of the composite resin, making it less fluid and leading to a thicker, undesirable, cementation line at the adhesive interface [8].

As an alternative to reduced viscosity luting agents, the preheating of composite resins has been suggested [8–15]. By increasing the temperature from 54°C to 70°C, the degree of conversion of the resin becomes similar to that of dual-polymerizing resin cement, the consistency of flow improves, and a thinner cementation line becomes possible [8, 10, 14, 16, 17]. Different preheating techniques, devices, temperatures, heating durations, and transport methods have been reported [8, 11, 13].

A systematic review of the heating and preheating of dental restorative materials (composite resins and glass ionomer types of cement) has recently been published [16]. Although the authors concluded that the preheating technique can improve the physical and mechanical properties of these materials, clinical studies to confirm the advantages of this technique in improving restoration performance are lacking [16]. Many studies have demonstrated the performance of preheating different materials [8, 10, 14, 16], but there remains a lack of evidence that preheating of restorative materials improves the quality and durability of indirect restorations.

Thus, this study aimed to review the influence of the heating protocols for and methods of preheating composite resins used as the luting agent for indirect adhesive restorations.

2. Materials and Methods

2.1. Selection Criteria and Search Methods. This scoping review was performed according to The Joanna Briggs Institute (JBI) Reviewers Manual 2015-Methodology for JBI Scoping Reviews [18] and Preferred Reporting Items for Systematic reviews and Meta-Analyses extension for Scoping Reviews (PRISMA-ScR) Checklist [19]. It was registered at the Open Science Framework (<https://osf.io>) under the number DOI: 10.17605/OSF.IO/GXMQE.

The studies were selected according to the eligibility criteria based on the PCC strategy [20] as follows: population (P), composite resins; concept (C), preheating techniques; and context (C), cementation of indirect restorations. There were no restrictions on language, date, or type of studies. The exclusion criteria were studies that investigated resin types of cement, preheating associated with restorative techniques other than cementation of indirect restorations, or studies that analyzed only composite resin properties.

Studies were screened using a search strategy adapted for the following electronic databases: PubMed

(MEDLINE), Embase, Cochrane, Scopus, Web of Science, and LIVIVO (Table 1). The search strategy was developed by using the MeSH terms and associated terms. Hand searches were performed on the reference lists to identify additional studies. In addition, non-peer-reviewed studies (OpenGrey, Proquest, and Google Scholar) were searched by screening the titles and abstracts. The first 100 hits were selected (filtered by ‘relevance’) on Google Scholar. A specialist opinion was also consulted. Duplicate studies were excluded by using the EndNote Web [21] and Rayyan [22] software programs. The search was conducted on July 21, 2020. A new search was conducted on January 4, 2021, and additional studies were included.

2.2. Data Collection. A Kappa test (K) was applied to measure the calibration between the first and second reviewers. After analyzing 10% of all included studies, a 0.81 value of Kappa was obtained. The study selection followed three steps. First, two investigators screened the titles of the studies that appeared to meet the inclusion criteria. In the second phase, the same reviewers independently read the abstracts of potentially relevant articles. Finally, they independently read the full text of the selected articles and excluded those that did not meet the inclusion criteria. Disagreements at any of the three stages were resolved by discussion and mutual agreement among the reviewers. If no consensus was reached, a third author was consulted to reach the final decision.

2.3. Data Analyses. Data extraction was performed by the first investigator and followed the mean characteristics of the study: author(s), year of publication, objective, conclusion, primary and secondary testing methods, number of specimens, materials used, preheating device, temperature tested, mean preheating duration, and all details relating to the technique. The second author examined all the retrieved information for the analysis. In an attempt to retrieve missing information, the corresponding author of the studies was contacted when important data were not described in the studies. Information was obtained from two of the four authors contacted.

3. Results

3.1. Description of Studies. Based on the search strategy, 304 studies were identified, including the first 100 studies detected on Google Scholar. After the removal of duplicates, 270 studies were analyzed by title and abstract. The inclusion process resulted in 14 studies [11, 13, 23–34] in the second phase. Of these, 12 were in vitro research studies [11, 13, 23–27, 30–34] and two were clinical case reports [28, 29]. The flow chart was adapted from PRISMA [20] and is illustrated in Figure 1. The timeline of the publications in the English language and from six countries, namely, Brazil [13, 27–30], Chile [28], the United States [23, 24, 26, 31–34], Italy [11], the Czech Republic [25], and Thailand [29] from 2009 to 2018 is shown in Table 2 [35].

TABLE 1: Electronic database and search strategy (PubMed).

((“Composite resins”[MeSH terms] OR “composite resin”[All fields] OR “composite dental resin”[MeSH terms] OR “composite dental resin”[All fields] OR “composite dental resin”[All fields] OR “bisphenol a-glycidyl methacrylate”[MeSH terms] OR “bisphenol a-glycidyl methacrylate”[All fields] OR “composite properties”[All fields] OR “composite dental material”[All fields] OR “composite dental restorative”[All fields] OR “composite dental restoratives”[All fields] OR “composite dental restorative material”[All fields] OR “composite dental restorative materials”[All fields] OR “composite dental filling”[All fields] OR “composite dental filling material”[All fields] OR “composite dental filling materials”[All fields] OR “methacrylate, bisphenol A-Glycidyl”[All fields] OR “Bis(Phenol A-Glycidyl Methacrylate)”[All fields] OR “Bis-GMA”[All fields] OR “bis-GMA”[All fields] OR “bisphenol A-Glycidyl methacrylate Homopolymer”[All fields] OR “bisphenol a-glycidyl methacrylate Homopolymer”[All fields] OR “Bis(Phenol A-Glycidyl methacrylate), Homopolymer”[All fields] OR “Poly(Bis-GMA)” [all fields] OR “Bis-GMA Resin”[All fields] OR “bis-GMA Resin”[All fields] OR “Bis-GMA Resins”[All fields] OR “resin, Bis-GMA”[All fields] OR “resins, Bis-GMA”[All fields] OR “bisphenol A-Glycidyl methacrylate Polymer”[All fields] OR “bisphenol a glycidyl methacrylate Polymer”[All fields] OR “2-propenoic acid, 2-methyl-, (1-methylethylidene) bis(4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)) ester, homopolymer”[All fields] OR “Bis-GMA Polymer”[All fields] OR “bis-GMA Polymer”[All fields] OR “Bis-GMA Polymers”[All fields] OR “polymer, Bis-GMA”[All fields] OR “polymers, Bis-GMA”[All fields]) AND (“preheat”[All fields] OR “preheated”[All fields] OR “preheating”[All fields] OR “hot temperature”[MeSH terms] OR “hot temperature”[All fields]) AND (“cementation”[MeSH terms] OR “cementations”[MeSH terms] OR “cementation”[All fields] OR “cementations”[All fields] OR “dental cement”[All fields] OR “dental cements”[MeSH terms] OR “dental cements”[All fields] OR “luting agent”[All fields] OR “luting agents”[All fields] OR “cementation agents”[All fields] OR “cementation agent”[All fields] OR “cement, Dental”[All fields] AND “permanent dental restoration”[MeSH terms] OR “permanent dental restorations”[All fields] OR “restorations, permanent Dental”[All fields] OR “dental restoration, Permanent”[All fields] OR “restoration, permanent Dental”[All fields] OR “dental restorations, Permanent”[All fields] OR “dental permanent Fillings”[All fields] OR “filling, permanent Dental”[All fields] OR “permanent dental Fillings”[All fields] OR “permanent fillings, Dental”[All fields] OR “permanent filling, Dental”[All fields] OR “dental filling, Permanent”[All fields] OR “dental permanent Filling”[All fields] OR “filling, dental Permanent”[All fields] OR “filling, permanent Dental”[All fields] OR “permanent dental Filling”[All fields] OR “fillings, dental Permanent”[All fields] OR “dental fillings, Permanent”[All fields]))

3.2. Preheating Device. Ten studies used the Calset oven (AdDent Inc.) device to preheat composite resin [11, 23, 24, 26, 29–34]. The other preheating devices described were the Digital wax pot (SJK) [13], ENA heat (Micerium S.p.A) [25], and Wax Heater Pot 4 (manufacturer not mentioned) [28]. Only one study used an incubator (manufacturer not mentioned) for preheating, but its specifications were not provided [27].

3.3. Temperatures and Preheating times. The temperatures used to preheat composite resins were 54°C [11, 23, 29, 32, 34], 55°C [25], 58°C [28], 60°C [27], 64°C [13], and 68°C [24, 30, 33]. The reported range was from 54°C to 68°C. The temperatures of 54°C and 68°C were most frequently reported. Two studies did not report the preheating temperature [26, 31]. A warming time of 5 min was specified in eight studies [13, 23, 24, 26, 28, 31, 32, 34] and of 30, 15, and 60 min in three other studies [25, 27, 29]. Three studies did not report the time used for heating composite resins [11, 30, 33].

3.4. Preheating Methods and Mean Required Time of the Clinical Procedure. Only one study reported details related to the glass container in which the resin was placed when heated in the device [27]. Almeida et al. [27] reported removing the increment from the oven and immediately applying it to the ceramic. However, the transport time was not specified. Goulart et al. [13] stated that the time taken for the material to be removed from the heating device until the assessed property was measured was less than 30 s. Other studies did not report this aspect.

3.5. Composite Resins Tested and Light-Polymerizing Units. Seven studies preheated the microhybrid composite resin Filtek Z100 (3M ESPE) [23, 24, 26, 30, 32–34]. Other microhybrid composite resins used were Gradia Direct posterior (GC) [31] and Venus1 (Kulzer) [11, 13]. Four articles preheated composite resins with nanohybrid particles, including Filtek Z350 XT (3M ESPE) [27], Tetric N-Ceram (Ivoclar-Vivadent) [28], Z250 XT (3M ESPE) [13], Miris 2 (Coltene-Wahledent) [29], and other composite resins such as Enamel Plus Hri (Micerium) [25]. Goulart et al. [13] compared the use of composite resins at room temperature ($\pm 23^\circ\text{C}$) for cementing indirect restorations. Acquaviva et al. [11] conducted a study comparing composite resin at room temperature, heated composite resin, and resin cement. Another five studies focused on the preheating of composite resin and resin cement [13, 24, 27] for cementing indirect restorations. Two clinical case reports [28, 29] and seven in vitro studies [23, 24, 26, 30–34] used heated composite resin. In no study were details provided regarding the amount of preheated composite resins used for cementing indirect restorations.

The following brands of the light-polymerization unit were used: halogen lamp Swiss Master Light1 (EMS, Neun, Switzerland), FlashLight (Dental Discus), Optilight Max (Gnatus), Den-Mat (Allegro), and Valo (Ultradent). The light-polymerization power was between 400 mW/cm² and 1200 mW/cm², and the duration ranged from 40 s to 120 s.

3.6. Property Testing. From the included in vitro studies, different properties of composite resins were evaluated, including the degree of conversion [11], color stability [27], microtensile bond strength-adhesive interfaces [13], fatigue resistance [23, 26, 30–34], vertical seating [24], and vertical marginal discrepancy [25].

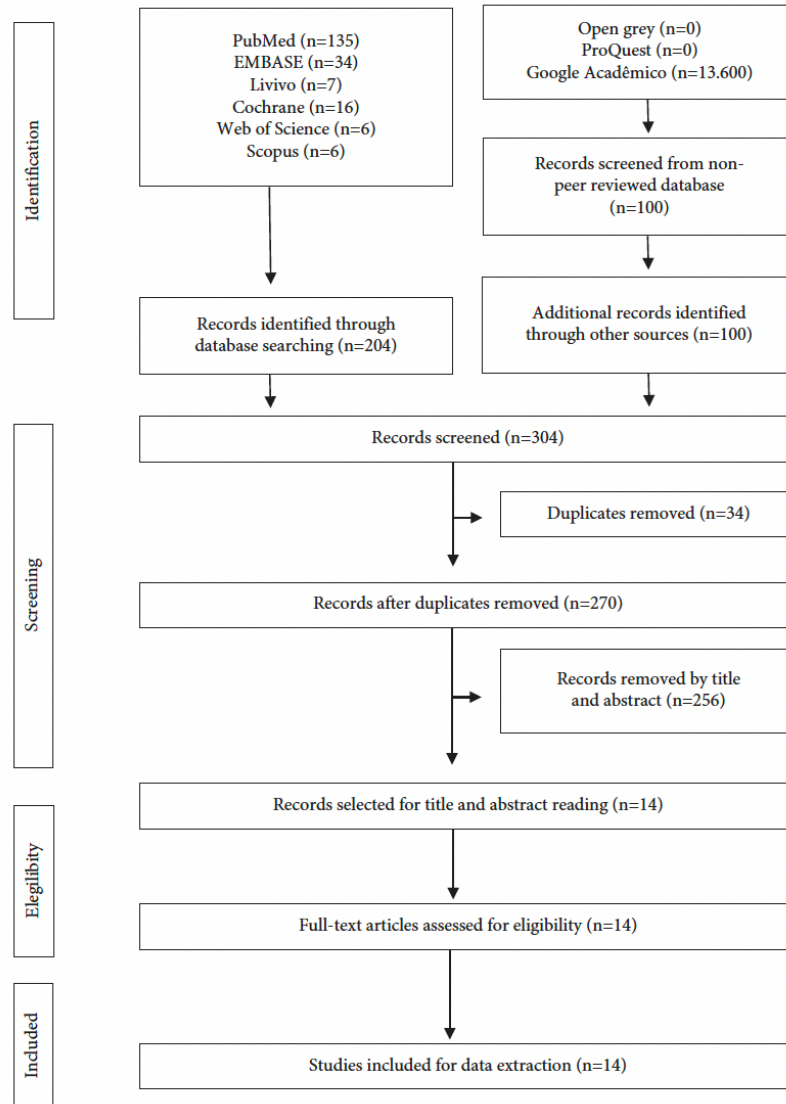


FIGURE 1: Description of included records in the scoping review.

4. Discussion

Indirect adhesive restorations can be cemented with preheated composite resins yielding reduced viscosity materials that provide a clinically acceptable cement film thickness and have better mechanical properties than those of conventional types of cement [8–11, 14]. Despite clinical and laboratory evidence suggesting the advantages of preheating composite resins [8–11, 14], their protocol for use as a cementation agent for indirect restorations has not yet been fully elucidated. Advantages reported in studies on preheating resin materials include an increased degree of conversion [10], improved marginal adaptation of restorations because of reduced viscosity [36], and decreased

polymerization contraction [37]. However, the methodologies used in the preheating of composite resins, their mechanical properties, and their performance as luting types of cement for indirect restorations must be analyzed.

The composite resin is typically preheated in a device that is programmed to reach a certain temperature that should be confirmed for accuracy and monitored and controlled during storage in the heater [38]. The temperature of the preheated composite resin cools rapidly when removed from the heating device, approximately 50% in 2 min [38]. Thus, the material should be placed, adapted, the restoration seated, and light-polymerized rapidly. When the high temperature is maintained, monomer conversion will be greater than at room temperature ($\pm 23^{\circ}\text{C}$) [11].

TABLE 2: Main characteristics of the included studies.

N	Author, year, country	Total "n" of specimens	Heated composite resin trademark, classification, color, and volume	Preheating device	Temperature, warm-up time, means of transport, and transport time	Valued property and assessment device	Light curing trademark, light curing time, and characteristics	Valued property control group (non-preheated)
1	Acquaviva et al., 2009, Italy [11]	180; 5 preheated	Venus1 (kulzer); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; N/A; N/A; N/A	Degree of conversion; spectrometer micro-Raman dilor (HR LabRam)	Halogen lamp swiss master Light1 (EMS); 40 s, 60 s, 120 s; 1200 mW/cm ² ; 800 mW/cm ² ; 400 mW/cm ²	Calibra1 (dentsply), dual-cured resin cement; Variolink1 II (ivoclar-vivadent), dual-cured resin cement; Venus1 (kulzer), microhybrid composite resin RelyX ARC (3 M/Espe), dual-cured resin cement; RelyX veneer (3 M/Espe), light-polymerizing cement; Filtek Z350 flow (3 M/Espe), flowable Venus1 (Kulzer), microhybrid composite resin color A2; Z250 XT (3M), microhybrid composite resin color A2; RelyX ARC (3M), dual-cured resin
2	Almeida et al., 2015, Brazil [27]	40; 10 preheated	Filtek Z350 XT (3 M/Espe); nanohybrid composite resin; A1; N/A	Incubator (N/A)	60°C; 30 min; glass container; "immediately"	Color stability; spectrophotometer (easyshade, vita zahnfabrik)	FlashLight (discuss dental); 1 min; 800 mW/cm ²	
3	Goulart et al., 2018, Brazil [13]	50; 50 preheated	Venus1 (kulzer); microhybrid composite resin; A2; N/A and Z250 XT (3 M/Espe); nanohybrid	Digital wax pot (SJK)	64°C; 5 min; N/A; "reduced to 30 s"	Microtensile bond strength and adhesive interfaces; stereomicroscope (EMZ, Meji Techno)	Optilight max (gnatus); 40 s; 900 mW/cm ²	
4	Magne et al., 2009, United States [32]	30; 30 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; 5 min; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (Mini Bionix II, MTS Systems)	N/A; 60 s; N/A	None
5	Magne et al., 2009, United States [34]	30; 30 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; 5 min; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Allegro (denmat); 60 s; N/A	None
6	Magne et al., 2010, United States [33]	30; 30 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	68°C; N/A; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (mini bionix II, MTS systems)	Allegro (denmat); 60 s; 1000 mW/cm ²	None

TABLE 2: Continued.

N	Author, year, country	Total “n” of specimens	Heated composite resin trademark, classification, color, and volume	Preheating device	Temperature, warm-up time, means of transport, and transport time	Valued property and assessment device	Light curing trademark, light curing time, and characteristics	Valued property control group (non-preheated)
7	Magne et al., 2011, United States [23]	28; 28 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	54°C; 5 min; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Valo (ultradent); 60 s; 1000 mW/cm ²	None
8	Magne et al., 2018, United States [24]	60; 30 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	68°C; 5 min; N/A; N/A	Vertical seating; acumen III (MTS systems)	Valo (ultradent); 60 s; N/A	RelyX ultimate cement (3M), dual-cured resin cement
9	Mounajjed et al., 2017, Czech Republic [25]	18; 6 preheated	Enamel plus HRi (Micerium S.p.A); nanohybrid composite resin; N/A; N/A	Heater ENA heat (micerium S.p.A)	55°C; 1 hour; N/A; N/A	Vertical marginal discrepancy; microscopy at x200 magnification with special image analysis software (Keyence)	Valo (ultradent); 60 s; N/A	Harvard PremiumFlow cement (GmbH), nanohybrid composite resin; RelyX ultimate cement (3 M/Espe), dual-cured resin cement
10	Oderich et al., 2011, United States [26]	60; 60 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	N/A; 5 min; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Valo (ultradent); 60 s; N/A	None
11	Olivares et al., 2011, Chile [28]	10; 10 preheated	Tetric N-ceram (Ivoclar-vivadent AG); nanohybrid composite resin; A2; N/A	Wax Heater Pot 4 (Denshine)	58°C; 5 min; N/A; N/A	None	N/A; 60 s; N/A	None
12	Rickman et al., 2011, Thailand [29]	7; 7 preheated	Miris 2 (coltene-whaledent); nanohybrid composite resin; A2; N/A	Calset (AdDent Inc.)	54°C; 15 min; N/A; N/A	None	N/A; N/A; N/A	None
13	Schlichting et al., 2011, Brazil [30]	40; 40 preheated	Filtek Z100 (3 M/Espe); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	68°C; N/A; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Allegro (denmat); 60 s; 1000 mW/cm ²	None
14	Soares et al., 2018, United States [31]	45; 30 preheated	Gradia direct posterior (GC); microhybrid composite resin; N/A; N/A	Calset (AdDent Inc.)	N/A; 5 min; N/A; N/A	Fatigue resistance; closed-loop servohydraulics (Mini Bionix II, MTS Systems)	Valo (ultradent); 60 s; 1000 mW/cm ²	None

Table 2 is reproduced from “Técnicas de aquecimento de resinas compostas para cimentação de restaurações indiretas: Scoping review” © 2022 by Zanon AEG, Poubel DLN, and Garcia FCP under CC BY 4.0 (<http://creativecommons.org/licenses/by/4.0/>) [35]. N/A: not available; none: not applicable.

Seven of the included articles reported similar preheating techniques with a commercially available device (Calset, AdDent Inc.) that the manufacturer claims preheats and stores composite resins at temperatures of 54°C, 60°C, or 64°C until they are ready for use. Composite resin syringes can be heated and the resin can then be directly injected onto the restoration or prepared tooth, reducing the clinical time [8, 38, 39]. Despite what was specified by the manufacturer, Daronch et al. [38], who used Calset (Addent Inc.) in their study, reported that the maximum temperature reached was 48.3°C and 54.7°C when the preset temperature of the device was set at 54°C and 60°C, respectively. The equipment (ENA heat, Micerium) used for heating composite resins in the study by Mounajjed et al. [25] was preset at temperatures from 39°C to 55°C. A temperature of 55°C was recommended by the manufacturer for heating composite resins for cementation and had six spaces for heating syringes of composite resin.

Previous studies evaluated the use of this heating equipment [40–42], but Goulart et al. [13] and Olivares et al. [28] used wax pot heaters in their studies (Digital wax pot, SJK and Wax Heater Pot 4, Denshine) because these devices allowed setting the temperature according to clinical needs. The preheating of composite resins in a wax pot heater has also been described in another study where the heating time of the composite resins was around 2 to 3 min [11]. The authors concluded that the wax pot was a straightforward, rapid, and economical option [43]. Almeida et al. [27] used an incubator oven to heat composite resins at 60°C for 30 min. However, they did not provide manufacturer specifications for the equipment. However, a preheating technique in a bacteriological oven (model 502, Fanem) at a temperature of 54°C has been described [44].

Composite resins can be heated in different dry heating devices, as long as the temperature is controlled and remains stable until clinical use. The use of a specifically marketed device such as Calset (AdDent Inc.) facilitates the technique and ensures standardization of the preheating process. The use of parallel heaters requires the preset temperature of the device to be measured and checked until the required temperature is reached.

The temperatures for preheating composite resins described in the studies ranged from 54°C to 68°C, and this range has been considered ideal for improving the working properties of the material [11, 13, 23–27, 30–34]. Daronch et al. [10] evaluated the degree of conversion at temperatures ranging from 3°C to 60°C and reported that, at the highest temperature, a greater degree of conversion was reached. Daronch et al. [38] used the Calset device (AdDent Inc.) and reported a drop in the degree of conversion after a certain temperature because of degradation of the photoinitiator. In monomers such as bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) or ethoxylated bisphenol A dimethacrylate (Bis-EMA), the volatility limit of dimethacrylate monomers used in resin formulations occurs close to 90°C, a temperature that could damage some composite resin components and harm pulpal tissue. However, 90°C is above the maximum temperature allowed by the heating device. In addition, because of incomplete polymerization, unreacted monomers may leach into the saliva, promoting undesirable

consequences, and the loss of plasticizers may decrease mechanical strength, dimensional stability, and color change and allow bacterial growth. Unreacted monomers can also cause allergic and sensitivity reactions [45].

The heating time for the composite resin inside the heater is also an important evaluation parameter, with the average time for the device to reach both tested preset temperatures (54°C or 60°C) being 11 min [38]. Therefore, in addition to ensuring that the heating device can maintain a controlled and predefined temperature, the temperature should be reached in a predictable time. A drop in the temperature of the composite resin was reported between its removal from the heating device and the mouth, estimated to be 50% after 2 min and 90% after 5 min when heated to 60°C and removed from the device, indicating the need for calibration during all processes. In addition, heated composite resins have been reported to provide better results than composite resins at room temperature [10, 16, 38]. Composite resins with different compositions can take different times to reach a stable temperature, and some low-molecular-weight components of the photoinitiator system can be volatilized with prolonged heating [10, 38]. Therefore, different heating methods have been used for *in vitro* studies and for clinical techniques. Lopes et al. [16] reported that some studies used a reasonable clinical time of approximately 15 min.

The temperature must be controlled to avoid causing pulpal damage, but increasing the composite resin temperature to between 54°C and 60°C does not significantly change the intrapulpal temperature [46]. Lopes et al. [16] noted that dentin thickness acts as a thermal barrier, preventing harmful stimuli and protecting the dental pulp.

According to manufacturers, the Calset (AdDent Inc.) and ENA heat (Micerium) devices are designed to attach a syringe, acting as a container. The wax pot heaters (Digital wax pot, SJK and Wax Heater Pot 4, Denshine) can directly heat the composite resin inside of the syringe. Comparing the two preheating methods, Daronch et al. [38] concluded that the composite resins already assembled in the application syringe showed a significantly higher maximum temperature ($36.6^{\circ}\text{C} \pm 2.2^{\circ}\text{C}$) than the composite resins that were heated without a container ($33.6^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$). Thus, the composite compule preloaded into a delivery syringe was more efficient. Higher temperatures were attained with this method as opposed to preheating the compule separately [16].

The way the composite resins are arranged and placed in the preheating device affects its clinical application, as the working time should be minimum owing to the decrease in temperature after its removal from the heater [10, 38]. Once the composite resin is attached to a syringe or loaded into the indirect restoration to be cemented, the dentist can simply remove and apply it to the teeth, without concerns about assembling the application system, thus reducing the working time, and maintaining the temperature as high as possible [38].

The viscosity of composite resins is linked to factors in their composition such as the organic matrix and amount and size of inorganic fillers [47]. Analyzing the particle size,

materials with smaller particles appear more fluid when compared with those that contain bigger particles. Regarding the amount of filler, the higher the filler load, the higher the viscosity [46]. The amount and type of monomer can also cause an increase in viscosity, as monomers such as Bis-GMA and urethane dimethacrylate are quite viscous, whereas Bis-EMA and triethylene glycol dimethacrylate are more fluid [47, 48]. Among the studies included, Goulart et al. [13] compared the use of two preheated composite resins, a microhybrid (Venus 1, Kulzer) and a nanohybrid (Z250 XT, 3M ESPE) with the same preheating protocol and mechanical test. The nanohybrid resin, as evaluated by SEM after cementation, formed a thicker film than the microhybrid resin.

The degree of conversion can be increased by preheating, decreasing the light-polymerization time, and maintaining a degree of conversion similar to or even better than when the composite resins are irradiated for longer at 22°C [10, 11]. Preheating the resins to 60°C increased the conversion of monomers by increasing molecular mobility. Compounds with higher conversion have greater crosslinking, reducing the free space of the polymers and improving their mechanical properties [10]. A 5 s light-polymerization time with a composite resin preheated to 57°C resulted in a higher conversion rate than that observed after a 40 s exposure at 22°C [10]. According to Acquaviva et al. [11], the thickness of the onlays affected the degree of conversion of both composite resins and dual-polymerizing types of cement, and an excellent degree of conversion can be achieved by preheating the light-polymerizing composite resins. No ideal light-polymerizing time or intensity has yet been determined. Thus, purely light-polymerizing types of cement or resins must be used with care, as there must be enough light to pass through the materials for adequate conversion of the monomers. If an indirect restoration is thinner than 2 mm, the light passage should be adequate [49].

A consensus on the limits for a clinically acceptable film thickness is lacking. Marcondes et al. [50] stated that composite resins, being restorative materials, are designed to provide intraoral resistance. Therefore, an increased cementation thickness, even if it exceeds the value defined by ISO 4049 [51], should be clinically acceptable. Composite resins are designed for color stability and abrasion resistance, as shown in laboratory and clinical studies [10, 11, 38, 49, 50]. In general, when composite resins are preheated, viscosity is reduced and adaptation to cavity walls is improved [50].

The presence of amines in chemically polymerized resins, including dual-resin types of cement, may eventually result in the staining of indirect restorations, and, therefore, they should be avoided for cementation of translucent or thin restorations [7]. Almeida et al. [27] reported that dual-resin types of cement showed greater color variation than preheated composite resins, light-polymerized resin types of cement, and flowable composite resins. Goulart et al. [13] reported that preheating composite resins did not increase the cementation bond strength of indirect restorations, even though increased mechanical properties have been reported because of

increased conversion [8, 12]. The results could be explained by the loss of temperature of the material during the bonding procedure and not reaching an adequate degree of conversion. In contrast, the conclusion was that the material can still be used to reduce its viscosity and improve the fit of the restoration [13]. The use of different luting agents provides many alternatives for cementation, which also can produce varying results in a vertical discrepancy of the definitive restoration [52]. Mounajjed et al. [25] compared the vertical marginal discrepancy of pressed crowns of lithium disilicate by using different cementing agents. The preheated composite resin Enamel Plus HRi (Micerium) obtained higher values of marginal discrepancies than a cement flow resin (Harvard Premium Flow) and the dual-resin cement, RelyX Ultimate (3M ESPE). The authors stated that the methodology used may have affected the results of the study, for example, using a specimen at low temperature, which could have reduced the fluidity of the resin, and the difficulty of standardizing the seating pressure at different viscosities. Magne et al. [24] investigated the vertical displacement of composite resin inlays, onlays, and computer-aided design and computer-aided manufactured overlays. The preheated composite resin used in cementation resulted in the better seating of inlays, onlays, and overlays than the dual-polymerizing resin cement [24].

5. Conclusion

This scoping review observed a large variation in the use of preheating techniques on composite resins used for cementation of indirect restorations. No consensus was found regarding the recommended preheating devices, heating durations, or temperatures for this clinical procedure. Some aspects can be considered relevant when considering preheating techniques for composite resins. (1) All heating devices demonstrated effectiveness in heating composite resins used for the cementation of indirect restorations. (2) The ideal heating device must be free of moisture and calibrated to reach a predetermined temperature (between 54°C and 68°C) on heating and must maintain stability at the predetermined temperature after heating. (3) The preheated material must be used as soon as possible after being removed from the device, as the temperature of the composite resin will decrease quickly. (4) Preheating the material directly in the prosthetic restoration or the dispensing syringe reduces clinical time. (5) The indirect restoration must be less than 2 mm thick if a light-polymerizing luting cement or composite resins is to be used. (6) The composition of the composite resins directly affects the viscosity reached after preheating. Therefore, materials indicated for this purpose or that show increased fluidity when heated should be used.

Based on the results of the included studies, more research is needed on preheating techniques for composite resins used for the cementation of indirect restorations; in particular, longitudinal clinical evaluations are needed.

Furthermore, studies correlating the composition of composite resins and their behavior when heated are required.

Data Availability

The data used to support the study are available from the corresponding author upon request.

Disclosure

The research was performed as part of the employment of the author Déborah Lousan do Nascimento Poubel.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Authors' Contributions

Déborah Lousan do Nascimento Poubel conceptualized the study, was responsible for design, search, and selection, collected the data, analyzed the data, and prepared the manuscript. Ana Elisa Ghanem Zanon was responsible for search and selection, collected the data, analysed the data, and prepared the manuscript. Júlio César Franco Almeida analysed the data. Liliana Vicente Melo de Lucas Rezende conceptualized the study and designed and analysed the data. Fernanda Cristina Pimentel Garcia conceptualized the study and designed and analysed the data. All authors reviewed the manuscript.

Acknowledgments

This work was supported by the University of Brasilia (DPI/DPG).

References

- [1] M. G. Buonocore, "A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces," *Journal of Dental Research*, vol. 34, no. 6, pp. 849–853, 1955.
- [2] J. M. Powers and R. L. Sakaguchi, *Craig's Restorative Dental Materials* pp. 479–511, Elsevier Publishing, Philadelphia, PA, 12th edition, 2006.
- [3] M. L. Mei, Y. M. Chen, H. Li, and C. H. Chu, "Influence of the indirect restoration design on the fracture resistance: a finite element study," *Biomedical Engineering Online*, vol. 15, no. 1, p. 3, 2016.
- [4] P. C. Guess, T. Vagkopoulou, Y. Zhang, M. Wolkewitz, and J. R. Strub, "Marginal and internal fit of heat pressed versus CAD/CAM fabricated all-ceramic onlays after exposure to thermo-mechanical fatigue," *Journal of Dentistry*, vol. 42, no. 2, pp. 199–209, 2014.
- [5] A. C. P. Silveira, S. B. Chaves, L. A. Hilgert, and A. P. Ribeiro, "Marginal and internal fit of CAD-CAM fabricated composite resin and ceramic crowns scanned by 2 intraoral cameras," *The Journal of Prosthetic Dentistry*, vol. 117, pp. 386–392, 2016.
- [6] K.-H. Kim, J. L. Ong, and O. Okuno, "The effect of filler loading and morphology on the mechanical properties of contemporary composites," *The Journal of Prosthetic Dentistry*, vol. 87, no. 6, pp. 642–649, 2002.
- [7] D. S. Smith, K. S. Vandewalle, and G. Whisler, "Color stability of composite resin cements," *General Dentistry*, vol. 59, pp. 390–394, 2011.
- [8] S. Lucey, C. D. Lynch, N. J. Ray, F. M. Burke, and A. Hannigan, "Effect of pre-heating on the viscosity and microhardness of a resin composite," *Journal of Oral Rehabilitation*, vol. 37, no. 4, pp. 278–282, 2010.
- [9] J. S. Blalock, R. G. Holmes, and F. A. Rueggeberg, "Effect of temperature on unpolymerized composite resin film thickness," *The Journal of Prosthetic Dentistry*, vol. 96, no. 6, pp. 424–432, 2006.
- [10] M. Daronch, F. A. Rueggeberg, and M. F. De Goes, "Monomer conversion of pre-heated composite," *Journal of Dental Research*, vol. 84, no. 7, pp. 663–667, 2005.
- [11] P. A. Acquaviva, F. Cerutti, G. Adami et al., "Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: a micro-Raman analysis," *Journal of Dentistry*, vol. 37, no. 8, pp. 610–615, 2009.
- [12] M. Daronch, F. A. Rueggeberg, M. F. De Goes, and R. Giudici, "Polymerization kinetics of pre-heated composite," *Journal of Dental Research*, vol. 85, no. 1, pp. 38–43, 2006.
- [13] M. Goulart, B. Borges Veleda, D. Damin, G. M. Bovi Ambrosano, F. H. Coelho de Souza, and M. C. G. Erhardt, "Preheated composite resin used as a luting agent for indirect restorations: effects on bond strength and resin-dentin interfaces," *The International Journal of Esthetic Dentistry*, vol. 13, pp. 86–97, 2018.
- [14] N. R. Fróes-Salgado, L. M. Silva, Y. Kawano, C. Francci, A. Reis, and A. D. Loguercio, "Composite pre-heating: effects on marginal adaptation, degree of conversion and mechanical properties," *Dental Materials*, vol. 26, pp. 908–914, 2010.
- [15] S. Deb, L. Di Silvio, H. E. Mackler, and B. J. Millar, "Pre-warming of dental composites," *Dental Materials: Official Publication of the Academy of Dental Materials*, vol. 27, pp. e51–9, 2011.
- [16] L. C. P. Lopes, R. S. S. Terada, F. M. Tsuzuki, M. Giannini, and R. Hirata, "Heating and preheating of dental restorative materials—a systematic review," *Clinical Oral Investigations*, vol. 24, no. 12, pp. 4225–4235, 2020.
- [17] M. Trujillo, S. M. Newman, and J. W. Stansbury, "Use of near-IR to monitor the influence of external heating on dental composite photopolymerization," *Dental Materials*, vol. 20, no. 8, pp. 766–777, 2004.
- [18] M. D. J. Peters, C. Godfrey, P. McInerney, Z. Munn, A. C. Tricco, and H. Khalil, "Chapter 11: scoping reviews (2020 version)," in *JBI Manual for Evidence Synthesis*, E. Aromataris and Z. Munn, Eds., JBI, Adelaide, South Australia. <https://synthesismanual.jbi.global>, 2020.
- [19] E. Aromataris and Z. Munn, *JBI Manual for Evidence Synthesis*, JBI, Adelaide, South Australia. 2020, <https://synthesismanual.jbi.global>.
- [20] D. Moher, A. Liberati, J. Tetzlaff, and D. G. Altman, "Preferred reporting items for systematic reviews and meta-analyses: the PRISMA statement," *BMJ*, vol. 339, Article ID b2535, 2009.
- [21] End Note Web, "Clarivate analytics, USA," 2017, <http://www.endnote.com/>.
- [22] M. Ouzzani, H. Hammady, Z. Fedorowicz, and A. Elmagarmid, "Rayyan—a web and mobile app for systematic reviews," *Systematic Reviews*, vol. 5, no. 1, p. 210, 2016.
- [23] P. Magne, E. Oderich, L. L. Boff, A. C. Cardoso, and U. C. Belser, "Fatigue resistance and failure mode of CAD/CAM composite resin implant abutments restored with type

- III composite resin and porcelain veneers," *Clinical Oral Implants Research*, vol. 22, no. 11, pp. 1275–1281, 2011.
- [24] P. Magne, M. Razaghy, M. A. Carvalho, and L. M. Soares, "Luting of inlays, onlays, and overlays with preheated restorative composite resin does not prevent seating accuracy," *International Journal of Esthetic Dentistry*, vol. 13, pp. 318–332, 2018.
- [25] R. Mounajjed, T. J. Salinas, T. Ingr, and B. Azar, "Effect of different resin luting cements on the marginal fit of lithium disilicate pressed crowns," *The Journal of Prosthetic Dentistry*, vol. 119, pp. 975–980, 2017.
- [26] E. Oderich, L. L. Boff, A. C. Cardoso, and P. Magne, "Fatigue resistance and failure mode of adhesively restored custom implant zirconia abutments," *Clinical Oral Implants Research*, vol. 23, no. 12, pp. 1360–1368, 2012.
- [27] J. R. Almeida, G. U. Schmitt, M. R. Kaizer, N. Boscato, and R. R. Moraes, "Resin-based luting agents and color stability of bonded ceramic veneers," *The Journal of Prosthetic Dentistry*, vol. 114, no. 2, pp. 272–277, 2015.
- [28] J. M. Olivares, A. Hidalgo, J. P. Pavez, D. Benadof, and R. Irribarra, "Functional and esthetic restorative treatment with preheated resins in a patient with ectodermic dysplasia: a clinical report," *The Journal of Prosthetic Dentistry*, vol. 119, no. 4, pp. 526–529, 2018.
- [29] L. J. Rickman, P. Padipatvuthikul, and B. Chee, "Clinical applications of preheated hybrid resin composite," *British Dental Journal*, vol. 211, no. 2, pp. 63–67, 2011.
- [30] L. H. Schlichting, H. P. Maia, L. N. Baratieri, and P. Magne, "Novel-design ultra-thin CAD/CAM composite resin and ceramic occlusal veneers for the treatment of severe dental erosion," *The Journal of Prosthetic Dentistry*, vol. 105, no. 4, pp. 217–226, 2011.
- [31] L. M. Soares, M. Razaghy, and P. Magne, "Optimization of large MOD restorations: composite resin inlays vs. short fiber-reinforced direct restorations," *Dental Materials*, vol. 34, no. 4, pp. 587–597, 2018.
- [32] P. Magne and A. Knezevic, "Simulated fatigue resistance of composite resin versus porcelain CAD/CAM overlay restorations on endodontically treated molars," *Quintessence International*, vol. 40, pp. 125–133, 2009.
- [33] P. Magne, L. H. Schlichting, H. P. Maia, and L. N. Baratieri, "In vitro fatigue resistance of CAD/CAM composite resin and ceramic posterior occlusal veneers," *The Journal of Prosthetic Dentistry*, vol. 104, no. 3, pp. 149–157, 2010.
- [34] P. Magne and A. Knezevic, "Influence of overlay restorative materials and load cusps on the fatigue resistance of endodontically treated molars," *Quintessence International*, vol. 40, pp. 729–737, 2009.
- [35] A. E. G. Zanon, *Técnicas de Aquecimento De Resinas Compostas Para Cimentação De Restaurações Indiretas: Scoping Review, Trabalho de Conclusão de Curso – Departamento de Odontologia da Faculdade de Ciências da Saúde da Universidade de Brasília, Brasília, Brasil*, 2020.
- [36] K. Baroudi and S. Mahmoud, "Improving composite resin performance through decreasing its viscosity by different methods," *The Open Dentistry Journal*, vol. 9, no. 1, pp. 235–242, 2015.
- [37] P. P. Didron, A. Ellakwa, and M. V. Swain, "Effect of preheat temperatures on mechanical properties and polymerization contraction stress of dental composites," *Materials Sciences and Applications*, vol. 04, no. 06, pp. 374–385, 2013.
- [38] M. Daronch, F. A. Rueggeberg, L. Moss, and M. F. De Goes, "Clinically relevant issues related to preheating composites," *Journal of Esthetic and Restorative Dentistry*, vol. 18, pp. 340–351, 2006.
- [39] J. Friedman, "Heating assembly for preheating dental materials," US Patent 6236020, 2001, <https://patentimages.storage.googleapis.com/78/37/24/1703ca99c7976e/US6320162.pdf>.
- [40] M. D'Amario, F. De Angelis, M. Vadini, N. Marchili, S. Mummolo, and C. D'Arcangelo, "Influence of a repeated preheating procedure on mechanical properties of three resin composites," *Operative Dentistry*, vol. 40, pp. 181–189, 2015.
- [41] M. Theodoridis, D. Dionysopoulos, E. Koliniotou-Koumpia, P. Dionysopoulos, and P. Gerasimou, "Effect of preheating and shade on surface microhardness of silorane-based composites," *Journal of investigative and clinical dentistry*, vol. 8, pp. 1–6, 2017.
- [42] D. Dionysopoulos, C. Papadopoulos, and E. Koliniotou-Koumpia, "Effect of temperature, curing time, and filler composition on surface microhardness of composite resins," *Journal of Conservative Dentistry*, vol. 18, no. 2, pp. 114–118, 2015.
- [43] V. Arora, P. Arora, A. Shamrani, and M. K. Fahmi, "A New, simple and innovative technique for Pre-heating/Pre-warming of Dental Composite resins in thermal assisted light polymerization technique," *Journal of Dentistry and Oral Biology*, vol. 2, pp. 1–7, 2017.
- [44] J. Dias Theobaldo, F. H. Baggio Aguiar, N. Pini, D. Alves Nunes Leite Lima, P. C. Suzy Liporoni, and A. Catelan, "Effect of preheating and light-curing unit on physicochemical properties of a bulk fill composite," *Clinical, Cosmetic and Investigational Dentistry*, vol. 9, pp. 39–43, 2017.
- [45] M. R. Bouschlicher and F. A. Rueggeberg, "Effect of ramped light intensity on polymerization force and conversion in a photoactivated composite," *Journal of Esthetic and Restorative Dentistry*, vol. 12, no. 6, pp. 328–339, 2000.
- [46] M. Daronch, F. Rueggeberg, G. Hall, and M. Degoes, "Effect of composite temperature on in vitro intrapulpal temperature rise," *Dental Materials*, vol. 23, no. 10, pp. 1283–1288, 2007.
- [47] J.-H. Lee, C.-M. Um, and I.-B. Lee, "Rheological properties of resin composites according to variations in monomer and filler composition," *Dental Materials*, vol. 22, no. 6, pp. 515–526, 2006.
- [48] F. Gonçalves, Y. Kawano, C. Pfeifer, J. W. Stansbury, and R. R. Braga, "Influence of BisGMA, TEGDMA, and BisEMA contents on viscosity, conversion, and flexural strength of experimental resins and composites," *European Journal of Oral Sciences*, vol. 117, pp. 442–446, 2009.
- [49] R. R. Pacheco, A. O. Carvalho, C. B. André et al., "Effect of indirect restorative material and thickness on light transmission at different wavelengths," *Journal of Prosthodontic Research*, vol. 63, no. 2, pp. 232–238, 2019.
- [50] R. L. Marcondes, V. P. Lima, F. J. Barbon et al., "Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness," *Dental Materials*, vol. 36, no. 10, pp. 1356–1364, 2020.
- [51] ISO, *Dentistry-Polymer Based Restorative Materials*, International Standard ISO 4049, Geneva, 5th edition, 2019.
- [52] J. Ng, D. Ruse, and C. Wyatt, "A comparison of the marginal fit of crowns fabricated with digital and conventional methods," *The Journal of Prosthetic Dentistry*, vol. 112, no. 3, pp. 555–560, 2014.

Effect of preheating on the viscosity of composite resins

ABSTRACT

Aim To determine the effects of time and temperature on the viscosity of preheated composite resins. *Methods* Eleven composite resins were heated to 60°C, and temperature analyses were performed at intervals of 1 minute until they had cooled to 25°C. The permanent oscillatory shear test was performed at 25°C, 35°C, 50°C, and 60°C for three composite resins under a shear rate of 1s⁻¹. One- and two-way ANOVA were used for the analysis ($\alpha=0.05$). *Results* There was no significant interaction between the composite resin and time ($p=0.9304$), and only the main effect time was significantly different ($p<0.0001$). A difference was observed between T0 and T6 ($p<0.001$), but not after T7. The increase in temperature resulted in a viscosity reduction ($p<0.05$). At 25°C, Beautifil II presented higher viscosity. Palfique LX5 showed a significant viscosity reduction with increasing temperature compared with the others ($p<0.05$). For Beautifil II and Z100, there was no difference at temperatures of 50°C and 60°C, while for Palfique LX5, no statistical difference was observed at 35°C, 50°C, and 60°C. *Conclusions* Ten minutes of preheating were sufficient to reach a temperature of 60°C, reducing viscosity by at least 84%. However, 5 min after removal, the composite resin cooled to room temperature.

Clinical Significance

Preheating composite resin has potential benefits. To determine how this approach will work in clinical practice, it is important to define the protocols of this technique and understand its limitations.

Keywords: composite resin, dental materials, preheating, viscosity

INTRODUCTION

Adhesive dentistry is a major component of contemporary practice that is used in virtually all clinical situations, including direct and indirect restorations. When indirect restorations are cemented, both the dental substrate and the chosen restorative material must be considered, and resin cements are currently the materials of choice for providing a bond between ceramics or resins and tooth structure.^{1,2} Resin cements have low solubility in oral fluids and favorable mechanical resistance.³ Despite the many available cementing agents, other materials have been used for this purpose, including light-polymerized composite resins.⁴

Composite resins, because of their high inorganic filler content, have greater resistance to wear compared with resin cement⁵, as well as greater stain and mechanical resistance.⁶ However, the higher inorganic filler content of composite resins leads to a higher viscosity than that of a resin cement.⁶ Heating composite resins has been used as a strategy to decrease their viscosity, reducing the cement line at the adhesive interface.^{7,8} When heated to between 54°C and 70°C, the degree of conversion of the resin is similar to that of a dual-polymerizing resin cement.^{4,7,8} In addition, preheating this material can reduce polymerization shrinkage^{7,8} and reduce photopolymerization time by approximately 75%, resulting in a similar or better conversion than when the composite resin is light polymerized at room temperature for the manufacturer's recommendation time.^{7,8} However, when the composite resin is removed from the heater at 60°C, there is significant and immediate cooling.^{7,8}

Composite resins have different compositions and particle content, which leads to different degrees of fluidity after heating.^{5,9,10} Although rapid heat loss can avoid pulpal damage, any delay before restoration cementation will increase viscosity, leading to a thicker cement line, preventing complete seating, and adversely affecting restoration longevity.^{5,9-11} Determining the time that a composite resin takes to reach the heating temperature and then cool down to room temperature is important to define the working time and to ensure that the heated composite resin technique is optimized. Thus, the objectives of the

present study were to determine the time required for different resins to reach a stable heating temperature, the cooling time required to return to room temperature, and the influence of different heating temperatures on viscosity. The research hypothesis was that time and temperature would affect the viscosity of preheated composite resins.

MATERIALS AND METHODS

Time-Temperature Analysis

This study analyzed the effects of time and temperature on different composite resins (Tab. 1), shade A2, and on their viscosity under heating with precise temperature control based on rheometer tests at the Department of Pharmaceutical Sciences (LabMac – ULEG, UnB, Brazil). The names and brands of the composite resins were hidden during testing, and all experimental tests were conducted blind and by the same researcher.

Table 1 – Characteristics and formulation of composite resins tested

Materials	Type	Manufacturer	Formulation	
			Resin phase	Filler wt% (vol%)
Beautiful II	Nanohybrid	Shofu	Bis-GMA, TEGDMA	83.3 (68.6)
Charisma Classic	Micro-hybrid	Kulzer	Bis-GMA	(61)
Charisma Diamond	Nanohybrid	Kulzer	Bis-GMA, UDMA, TEGDMA, TCD-DI-HEA	(64)
Filtek Z100	Micro-hybrid	3M ESPE	Bis-GMA, TEGDMA	84,5 (66)
Filtek Z250	Micro-hybrid	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA e TEGDMA	81,8 (67,8)
Filtek Z350XT	Nanofill	3M ESPE	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA	78.50 (63.3)
Forma	Nanohybrid	Ultradent	Bis-GMA, TEGDMA, Bis-EMA, UDMA	

Llis	Micro-hybrid	FGM	Bis-GMA, TEGDMA	Bis-EMA, 77,5 (56)
Opallis	Microfill	FGM	Bis-GMA, TEGDMA	Bis-EMA, 80 (72)
Palfique LX5	Supernanofill	Tokuyama	Bis-GMA, TEGDMA	82 (71)
Vittra	Nanofill	FGM	Bis-GMA, Bis-EMA, UDMA, TEGDMA	82 (60)
Bis-GMA, bisphenol-A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; TCD-DI-HEA, Bis-(acryloyloxymethyl) tricyclodecane; Bis-EMA, bisphenol-A ethoxylated dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate				

A portable device with digital temperature control was used to heat the composite resins. The temperature reported on the display was the same as the actual temperature inside the device. This was confirmed after five tests at different times and on different days with the thermal sensor (Simpla TA21, AKSO, São Leopoldo, RS, Brazil). The heating protocol was determined for this digital oven as follows: After turning on the device, the temperature was set to 60°C; this was followed by waiting for 40 min so that the device could stably reach the preset temperature. A variation of $\pm 4^\circ\text{C}$ was observed.

To determine the time required for the resins to reach this preheating temperature, each composite resin was tested individually at 60°C ($\pm 4^\circ\text{C}$). The resins were kept in a refrigerator at 5 °C until the tests were performed when they were removed from the refrigerator and kept at room temperature (approximately 25°C) for 20 min until their temperature stabilized at the laboratory temperature.¹² As soon as their temperature was stable, the resins were individually weighed, and a predetermined volume (0.6 mL) was dispensed as a Ø1-cm specimen onto a glass plate and heated. The thermal sensor was positioned close to the composite resin specimens. All tests were conducted in triplicate (n=3), and the means and standard deviations of the results were determined, thus obtaining the time required for each resin to reach the predetermined temperature.

As soon as the resin reached the heating temperature, the glass plate was removed, and the specimen temperature read with an infrared thermometer (Simpla TI38, 8:1, AKSO, São Leopoldo, RS, Brazil) with a detection range of -5°C to 380°C. The thermometer was fixed at a distance of 8 cm from the Ø1-cm composite resin specimen on the glass plate. The temperature was analyzed at controlled intervals of 1 min until the resin returned to room temperature (25°C) as follows: T0, immediately after removal of the heat device; T1 1 minute; T2, 2 minutes; T3, 3 minutes; T4, 4 minutes; T5, 5 minutes; T6, 6 minutes; T7, 7 minutes; T8, 8 minutes; T9, 9 minutes; and T10, 10 minutes after removal. The data were analyzed with the statistical software program Stata SE 15.1 (StataCorp, College Station, TX, USA). For the analysis, a two-way analysis of variance (ANOVA), with temperature as the dependent variable and resin-based composite and time as the two independent variables, as well as a post hoc Tukey test, were used ($\alpha=0.05$).

Viscosity Analysis

After the first phase, 11 resins of different brands and characteristics were tested. The three heated resins with the highest fluidity were selected for viscosity testing to determine the rheological properties with the MCR 301 rheometer (Anton Paar Physica). All measurements were performed in a temperature-controlled room (25°C), and the instrument was isolated from external vibrations on a rigid support table.

A permanent oscillatory shear test was performed at 25°C, 35°C, 50°C, and 60°C for the Z100 (3M ESPE, SP, SP, Brazil), Beautifil II (Shofu, SP, SP, Brazil), and Palfique LX5 (Tokuyama, Via Chizzalung, Sandrigo, Italy) composite resins under 1s⁻¹ shear rate with a rotating rheometer. The composite resins were removed from their packaging with a spatula and placed in a semicircular mold to standardize the 0.6-mL volume. The test material was dispensed on the lower plate of the rheometer, positioned with a 0.05-mm gap between the plates, and heated with the rheometer. The viscosity (Pa·s) was measured until the designated temperature was reached.

To perform the ramp analysis, the composite resins were coupled to the device at room temperature, heated to 60°C for 10 min, remained stable for 5 min more, and returned to room temperature (25°C) in 5 min, with the values defined in the time-temperature analysis. The measurements were performed at 25°C, 35°C, 50°C, and 60°C. Each composite resin was tested four times. Data were analyzed using the statistical software program Stata SE 15.1 (StataCorp). One-way ANOVA and post hoc Tukey tests were used to compare the viscosity at each temperature ($\alpha=0.05$).

RESULTS

The original data were entered into the Figshare repository (DOI: 10.6084/m9.figshare.17708510).

Eleven different composite resins were evaluated at 11 time periods (0-10 minutes after the removal of the heat device). As presented in Table 2, the two-way ANOVA found no statistically significant interaction between the composite resin and time ($F=0.77$, $p=0.9304$), and only the main effect time was significantly different ($p<0.0001$).

Table 2 – Two-way ANOVA of dependent variable as temperature and the two independent variables (resin-based composite and time)

Source	Partial SS†	DF†	MS†	F†	p-value
Model	50863.029	120	423.85857	386.65	<0.0001
Composite	10.360285	10	1.0360285	0.95	0.4925
Time	50767.952	10	5076.7952	4631.16	<0.0001*
Composite #Time	84.716705	100	.84716705	0.77	0.9304
Total	51128.316	362	141.23844		

† SS: Sum-of-Squares; DF: Degrees of Freedom; MS: Mean Square; F: F ratio

* Indicates significant difference ($p < 0.05$)

Regarding the main effect time, a difference was observed between T0, T1, T2, T3, T4, T5, and T6 (Tukey, $p < 0.001$). However, this difference was not observed after 6 min. T6 was not statistically different from T7 ($p = 0.39$); T7 was not statistically different from T8 ($p = 1.0$), T8 was not statistically different from T9 ($p = 1.0$), and T9 was not statistically different from T10 ($p = 1.0$). All experimental composite resins returned to the initial temperature (25 °C) within 5 minutes of removal from the heating device (Fig.1).

The composite resins showed a rapid loss of temperature, losing 100% of the temperature acquired after 5 minutes, that is, returning to the ambient temperature (25°C). An average of -48% of the temperature was recorded after 1 minute, -74% after 2 minutes, -87% after 3 minutes, and -95% after 4 minutes.

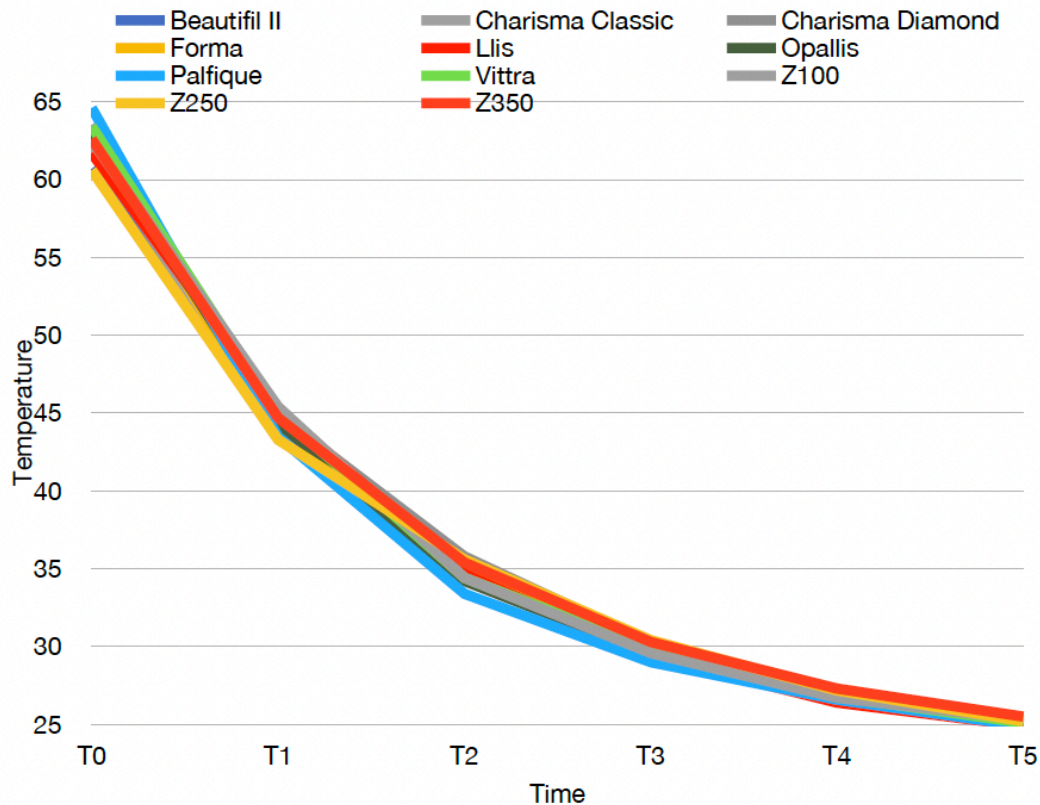


Fig. 1 – Cooling analysis of preheated composite resins according to time (T0-T5) in minutes after removing the heater.

The results for viscosity at 25°C, 35°C, 50°C, and 60°C are shown in Table 3. For the three composite resins analyzed, the increased temperature resulted in a significantly reduced viscosity ($p < 0.05$). At the initial temperature (25°C), Beautifil II (Shofu) had a significantly higher viscosity than Palfique LX5 (Tokuyama) and Z100 (3M ESPE). Palfique LX5 (3M ESPE) started with the lowest viscosity, and, with increasing temperature, showed a significant viscosity reduction compared with the other two composite resins ($p < 0.05$). For both Beautifil II (Shofu) and Z100 (3M ESPE), there was no difference in the viscosity at the temperatures of 50°C and 60°C, while for Palfique LX5 (Tokuyama), no statistical difference was observed at 35°C, 50°C, and 60°C. The hypothesis that time and temperature would influence viscosity properties was accepted.

Table 3 – Mean and SD† of viscosity experimental test (Pa S‡)

Temperature	25 °C	35 °C	50 °C	60 °C
Beautifil II	8442.5 (1361) ^{a,A}	4592.5 (454) ^{a,B}	931 (187) ^{a,C}	452.5 (112) ^{a,C}
Palfique LX5	3012.5 (493) ^{b,A}	576 (58) ^{b,B}	272.75 (11) ^{b,B}	206.5 (13) ^{b,B}
Z100	4595 (442) ^{b,A}	2035 (103) ^{c,B}	1115.5 (118) ^{a,C}	690 (61) ^{c,C}

† SD: Standard Deviation

‡ Pa S: Pascal-second, unit of dynamic viscosity

^{a,b,c} Different lower-case letters identify statistical difference between groups in the same columns; ^{A,B,C} Different upper-case letters identify statistical difference between groups in the same row.

DISCUSSION

Composite resins are direct restorative materials, although they can also be used as cementing agents for indirect restorations. The indication for these materials can vary according to their composition and physical and chemical

properties, and additional protocols have been suggested to improve the characteristics of the composite resins, including warming. As the use of preheated resins in dentistry has become popular, the criteria for this technique require clarification.

The composite resins can be preheated in different dry-heating devices. The temperatures for preheating composite resins described in the literature range from 54°C to 68°C, with warm-up times of 5 to 60 minutes.^{5,7-13} Daronch et al.¹² (2006) used a device (Calset, Addent Inc.) in their study and reported that the maximum temperatures reached were 48.3°C and 54.7°C when the preset temperature of the device was set at 54°C and 60°C, respectively.¹² The digital heater used in the present study had been previously tested to avoid possible errors. After repeating the experiments on different days, the temperatures appearing on the digital display and inside the device were confirmed to be identical. Considering this, the experimental resins were subjected to heating at 60°C, with possible variations of $\pm 4^\circ\text{C}$. A device developed specifically for this purpose facilitates this technique clinically and ensures the standardization of the preheating process, but similar heaters can also be used, provided that the temperature is controlled and remains stable until clinical use.

A wide range of heating times have been specified in the literature, varying from 5 to 60 minutes.^{5,7-13} Eleven composite resins were tested with respect to time-temperature, and, although the materials had different compositions and characteristics, they behaved similarly in terms of heating and cooling, and a statistical difference was not found. The hypothesis tested that time and temperature would influence viscosity properties was accepted. A 10-minute heating time at 60°C ($\pm 4^\circ\text{C}$) was sufficient for all the tested composite resins to reach the desired temperature, which seems a reasonable time for clinical application.

To implement this technique, the speed at which the temperature loss of each resin occurs needs to be established. According to Marcondes et al.¹⁰ (2020), composite resins preheated to 68°C showed temperature losses ranging between 45% and 61% 15 sec after being removed from the heater, an 84% loss of temperature in 30 sec, and a 96% loss in 60 sec, suggesting that clinicians

have a working time of between 10 and 15 sec when seating a restoration with preheated composite resin.¹⁰ This can also be seen in this study in part because the composite resins showed a considerable loss of temperature. However, this loss occurred over a longer time period (-48%, 1 minute; -74%, 2 minutes; -87%, 3 minutes; -95%, 4 minutes; and -99%, 5 minutes), thus allowing additional working time and preserving the benefits of viscosity. Other *in vitro* and clinical studies are needed to determine an ideal working time based on these findings by considering all the criteria and sequences for the procedures. Pulpal damage has been a concern with the use of heated composite resins in dental procedures. One reason to consider the procedure safe and unlikely to damage the pulp is the rapid drop in temperature. Daronch et al.¹³ (2007) reported that intrapulpal temperature values may not change significantly because the preheated resins are not as hot as expected when the restoration is delivered.¹³ The luting procedure using preheated resins is limited to restorations of less than 2 mm thick because sufficient light needs to pass through to ensure complete polymerization.^{4,5} Even if the technique is indicated for thin restorations, there would still be a considerable amount of excess, further reducing the chances of any pulpal damage.^{5,12,13}

Regarding viscoelastic properties, restorative composite resins with different formulations can react differently to preheating.^{10,14,15} The characteristics of the inorganic particles may also influence flowability, affecting the thickness of the film when used as a cement.¹⁰ Preheating to 69°C has been reported to reduce the viscosity of restorative resin composites by 47% to 92% compared with that at 37°C.¹⁰ Therefore, thermal conductivity must also be considered in the choice of material in addition to its handling characteristics^{15,16}, including filler content, particle type, shape, size, nature of the particle surface, and spatial arrangement of the filler within the composite resin¹⁰, because only the relationship between viscosity and the filler content is negative.^{10,17} Although there is a relationship between viscosity and temperature, no change was noted in the mechanical properties of the composite resins at the same loading of particles of different sizes.¹⁸

The viscosity of composite resins, however, is affected by the resin matrix formulation, the interlocking between the filler particles, and the interfacial interaction between the filler particles and resin matrix. An inverse relationship was observed between viscosity and polymerization shrinkage, depending primarily on the monomer types and ratio of the resin matrix¹⁶, and the filler volume has been reported to be more important than the filler weight.¹⁵ Regarding the filler size, composite resins with a small filler size were more viscous than those with a larger filler size, even though the filler content was the same because the number of filler particles was higher for identical filler volumes. As a result, the increased surface area led to a greatly increased interaction between the resin matrix and filler particles and between filler particles.¹⁵ The viscosity of the composite resin with a round filler seems to be much lower than that with an irregular filler because of the lower friction at similar filler sizes.¹⁵ Diluents such as triethyleneglycol-dimethacrylate and urethane dimethacrylate have lower molecular weights and lower viscosities than bisphenol A-glycidyl methacrylate.^{15,16} Therefore, their low molecular weights and resultant high numbers of double bonds per unit of weight create a high degree of cross-linking and a relatively high amount of shrinkage.¹⁶ However, manufacturers do not always disclose all the formulation details.

Resins that showed greater fluidity for handling after being heated to 60°C were selected for the analysis of viscosity. These were the nanohybrid composite Beautifil II (Shofu), micro-hybrid Filtek Z100 (3M ESPE), and supernanofill Palfique (Tokuyama). Increased temperature led to a significant viscosity reduction, ranging between 84% and 94%. However, it is expected that the preheated resin composite resins will not show viscosity values in the range of the flowable composite or resin cements.¹⁰ For both Beautifil II (Shofu) and Z100 (3M ESPE), there was no difference in the viscosity from temperatures 50°C to 60°C, while for Palfique LX5 (Tokuyama), no statistical difference between 35°C, 50°C, and 60°C was observed. Among them, Palfique LX5 (Tokuyama) showed the lowest viscosity when heated. Although there is no difference between 50°C and 60°C in the viscosity analysis for the three resins, adopting higher temperatures will extend the working time, since the rapid loss of temperature is

a limiting factor of the technique. The overall performance of the preheated composite resin technique depends on proper material selection and correct procedure because the change in viscosity upon preheating alone is not an appropriate parameter for selecting a restorative composite resin for luting purposes.^{10,19} The filler content of the resins does not interfere directly with the thickness of the film¹⁵, and Marcondes et al.¹⁰ (2020) reported that some composite resins that showed the highest reduction in viscosity were not always able to yield the thinnest films.¹⁰

Based on the results of this *in vitro* study, heating composite resins influences their viscosity, consistent with previous studies.^{14-16,20} Ahn et al.²⁰ (2015) speculated that preheating increased the mobility of the unpolymerized monomer and allowed a better distribution of the filler particles, becoming more fluid when it was manipulated. Even though preheating alters the structure of composite resins, it seems to be beneficial because this improvement in the internal structure persists, even after cooling.²⁰

Limitations of this study included that only 11 composite resins (shade A2) were investigated. Even though the preheating of composite resins affects their viscosity, future studies should be conducted on how different monomers and filler features might influence the reaction of composite resin to preheating. A working time from 30 seconds to 1 minute may preserve their benefits and allows for better manipulation of composite resins during the luting procedure, but it seems not to be reproducible in the clinical scenario. In addition, clinical and laboratory studies should be developed to simulate the clinical procedure and determine the protocol for composite preheating.

CONCLUSIONS

The preheating of composite resins affects their viscosity; however, the following aspects are relevant to this technique. The time of 10 minutes at ≥ 60 °C seems to be adequate to heat composite resins, reducing their viscosity by at least 84%. All tested resins lost 100% of the temperature gained during heating after 5 minutes.

REFERENCES

1. Martin MP. Material and clinical considerations for full-coverage indirect restorations. *Compend Contin Educ Dent* 2012;33(6):2-5.
2. Ferrari M, Dagostin A, Fabianelli A. Marginal integrity of ceramic inlays luted with a self-curing resin system. *Dent Mater* 2003;19(4):270-276.
3. Fabianelli A, Goracci C, Bertelli E, Davidson CL, Ferrari M. A clinical trial of Empress II porcelain inlays luted to vital teeth with a dual-curing adhesive system and a self-curing resin cement. *J Adhes Dent* 2006;8(6):427-431.
4. Vargas MA, Bergeron C, Diaz-Arnold A. Cementing all-ceramic restorations: recommendations for success. *J Am Dent Assoc* 2011;142(2):20-24.
5. Acquaviva PA, Cerutti F, Adami G, Gagliani M, Ferrari M, Gherlone E, et al. Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: A micro-Raman analysis. *J Dent* 2009;37(8):610-615.
6. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002;87(6):642-649.
7. Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. *J Dent Res* 2006;85(1):38-43.
8. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. *J Dent Res* 2005;84(7):663-667.
9. Froes-Salgado NR, Silva LM, Kawano Y, Francci C, Reis A, Loguercio AD. Composite pre-heating: effects on marginal adaptation, degree of conversion and mechanical properties. *Dent Mater* 2010;26(9):908-914.
10. Marcondes RL, Lima VP, Barbon FJ, Isolan CP, Carvalho MA, Salvador MV, et al. Viscosity and thermal kinetics of 10 preheated restorative resin composites and effect of ultrasound energy on film thickness. *Dent Mater* 2020;36(10):1356-1364.

11. Magne P, Razaghy M, Carvalho MA, Soares LM. Luting of inlays, onlays, and overlays with preheated restorative composite resin does not prevent seating accuracy. *Int J Esthet Dent* 2018;13(3):318-332.
12. Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent* 2006;18(6):340-350.
13. Daronch M, Rueggeberg FA, Hall G, De Goes MF. Effect of composite temperature on in vitro intrapulpal temperature rise. *Dent Mater* 2007;23(10):1283-1288.
14. Al-Ahdal K, Silikas N, Watts DC. Rheological properties of resin composites according to variations in composition and temperature. *Dent Mater* 2014;30(5):517-524.
15. Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. *Dent Mater* 2006;22(6):515-526.
16. Ellakwa A, Cho N, Lee IB. The effect of resin matrix composition on the polymerization shrinkage and rheological properties of experimental dental composites. *Dent Mater* 2007;23(10):1229-1235.
17. Deb S, Di Silvio L, Mackler HE, Millar BJ. Pre-warming of dental composites. *Dent Mater* 2011;27(4):51-59.
18. Habib E, Wang R, Zhu XX. Correlation of resin viscosity and monomer conversion to filler particle size in dental composites. *Dent Mater* 2018;34(10):1501-1508.
19. Gugelmin BP, Miguel LCM, Baratto Filho F, Cunha LFD, Correr GM, Gonzaga CC. Color Stability of Ceramic Veneers Luted With Resin Cements and Pre-Heated Composites: 12 Months Follow-Up. *Braz Dent J* 2020;31(1):69-77.
20. Ahn KH, Lim S, Kum KY, Chang SW. Effect of preheating on the viscoelastic properties of dental composite under different deformation conditions. *Dent Mater J* 2015;34(5):702-706.

DATA AVAILABILITY

Datasets related to this article can be found at DOI: [10.6084/m9.figshare.17708510](https://doi.org/10.6084/m9.figshare.17708510), hosted at Figshare repository.