



SERVIÇO PÚBLICO FEDERAL
MINISTÉRIO DA EDUCAÇÃO
UNIVERSIDADE FEDERAL DE UBERLÂNDIA
FACULDADE DE ODONTOLOGIA
PROGRAMA DE PÓS-GRADUAÇÃO



Fernanda Pereira Silva

**Avaliação dos parâmetros físicos e biomecânicos de
materiais restauradores de uso direto e indireto**

Tese apresentada ao Programa de Pós-Graduação em Odontologia da Faculdade de Odontologia da Universidade Federal de Uberlândia, como parte dos requisitos para obtenção do título de Doutora em Odontologia na Área de Clínica Odontológica Integrada.

Uberlândia, 2019

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Orientador: Prof. Dr. Murilo de Sousa Menezes

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Uberlândia, 2019



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ATA

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Defesa de: Tese de Doutorado - COPOD

Data: 22/02/2019

Discente: **Fernanda Pereira Silva (11513ODO004)**

Título do Trabalho: **Avaliação dos parâmetros físicos e biomecânicos de materiais restauradores de uso direto e indireto.**

Área de concentração: Clínica Odontológica Integrada.

Linha de pesquisa: Biomecânica aplicada à Odontologia.

Projeto de Pesquisa de vinculação: Biomecânica aplicada à Odontologia.

As **oito horas e trinta minutos** do dia **vinte e dois de fevereiro de 2019** no Anfiteatro Bloco 4T - Campus Umuarama da Universidade Federal de Uberlândia, reuniu-se a Banca Examinadora, designada pelo Colegiado do Programa de Pós-graduação em janeiro de 2019, assim composta: Professores Doutores: Carlos José Soares (UFU); Gisele Rodrigues da Silva (UFU); Marcelo Bighetti Toniollo (UniRV); Hugo Lemes Carlo (UFJF); e orientador(a) do(a) candidato(a) **Murilo de Sousa Menezes (UFU)**.

Iniciando os trabalhos o(a) presidente da mesa Dr. Murilo de Sousa Menezes apresentou a Comissão Examinadora e o candidato(a), agradeceu a presença do público, e concedeu ao Discente a palavra para a exposição do seu trabalho. A duração da apresentação do Discente e o tempo de argüição e resposta foram conforme as normas do Programa.

A seguir o senhor(a) presidente concedeu a palavra, pela ordem sucessivamente, aos (às) examinadore (as), que passaram a argüir o(a) candidato(a). Finalizada a argüição, que se desenvolveu dentro dos termos regimentais, a Banca, em sessão secreta, atribuiu os conceitos finais.

Em face do resultado obtido, a Banca Examinadora considerou o(a) candidato(a) (A)provado(a).

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Nada mais havendo a tratar foram encerrados os trabalhos às 12 horas e 40 minutos. Foi lavrada a presente ata que após lida e achada conforme foi assinada eletronicamente pela Banca Examinadora.



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Documento assinado eletronicamente por **Carlos José Soares, Professor(a) do Magistério Superior**, em 22/02/2019, às 12:49, conforme horário oficial de Brasília, com fundamento no art. 6º, § 1º, do [Decreto nº 8.539, de 8 de outubro de 2015](#).



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Documento assinado eletronicamente por **Hugo Lemes Carlo, Usuário Externo**, em 22/02/2019, às 12:50, conforme horário oficial de Brasília, com fundamento no art. 6º, § 1º, do [Decreto nº 8.539, de 8 de outubro de 2015](#).



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Dados Internacionais de Catalogação na Publicação (CIP)
Sistema de Bibliotecas da UFU, MG, Brasil.

S586a
2019 Silva, Fernanda Pereira, 1979
 Avaliação dos parâmetros físicos e biomecânicos de materiais restauradores de uso direto e indireto [recurso eletrônico] / Fernanda Pereira Silva. - 2019.

Orientador: Murilo de Sousa Menezes.

Tese (Doutorado) - Universidade Federal de Uberlândia, Programa de Pós-Graduação em Odontologia.

Modo de acesso: Internet.

Disponível em: <http://dx.doi.org/10.14393/ufu.te.2019.1218>

Inclui bibliografia.

Inclui ilustrações.

1. Odontologia. 2. Resinas dentárias. 3. Restauração (Odontologia). 4. Cerâmicas dentárias. I. Menezes, Murilo de Sousa, 1979, (Orient.) II. Universidade Federal de Uberlândia. Programa de Pós-Graduação em Odontologia. III. Título.

CDU: 619

Angela Aparecida Vicentini Tzi Tziboy – CRB-6/947

DEDICATÓRIA

**Dedico este trabalho à Deus, aos meus pais Carlos Henrique e Kátia
Cristina e minha irmã Daniella.**

AGRADECIMENTOS ESPECIAIS,

À Deus,

Pela constante presença em minha vida. Obrigada senhor pela saúde, pela família maravilhosa, pela profissão que tanto amo e pela realização deste trabalho que também é a realização de um sonho. Mesmo com momentos difíceis, eu sabia que não seria impossível pois sempre tive seu amparo.

Aos meus pais, Carlos Henrique e Kátia Cristina,

É difícil transmitir em palavras tudo que vocês significam na minha vida. E agora, é um daqueles tantos momentos que eu só tenho a agradecer pelo amor incondicional e dedicação que sempre tiveram comigo. Obrigada pelo apoio e suporte que me deram ao longo da minha trajetória acadêmica, sem vocês essa etapa seria impossível. Amo muito vocês!

À minha irmã Daniella,

Por sempre estar presente em minha vida, pelos conselhos, momentos de confiança e por torcer pelas minhas vitórias. Amo você!

Ao Fernando,

Obrigada por toda carinho, cumplicidade, apoio e companheirismo. Você é uma pessoa muito especial para mim. Agradeço também a sua família que me acolheu tão bem.

Aos meus queridos avós,

Obrigado por todo amor e carinho que sempre tiveram por mim. Amo vocês!

A minha família, aos meus queridos tios e primos,

obrigado por torcerem por minhas conquistas e estar presentes em todas as etapas da minha vida. Amo vocês!

Ao professor Murilo de Sousa Menezes,

Agradeço ao Professor Murilo por todas as oportunidades que me deu durante toda minha vida acadêmica. Obrigada por ter me concedido a chance de trabalhar desde a iniciação científica, quando eu era uma aluna de graduação tímida e que tinha vergonha até mesmo de dirigir a palavra a você. Obrigada por todos os ensinamentos na área da odontologia, por todos os conselhos de vida e por acreditar no meu trabalho. Se hoje me sinto realizada profissionalmente devo muito a você. Serei eternamente grata!

Às amigas Giselle, Marcela e Ana Laura,

Me sinto muito honrada em poder chama-las de amigas. Obrigada por dividir comigo todos os momentos ao longo dessa longa trajetória até finalizar o doutorado, por todos os conselhos, apoio e ajuda. Agradeço à Deus por ter colocado vocês em minha vida!

À amiga Fabiane,

um verdadeiro presente de Deus em minha vida, que tornou mais que uma amiga, uma irmã. Obrigada pelos conselhos, apoio e por ser sempre tão querida!

Aos muitos amigos que fazem parte da minha vida, sintam-se todos lembrados e agradecidos.

Aos amigos da pós-graduação, como foi bom dividir todos esses anos com vocês. Obrigada pela amizade e momentos de descontração. Sempre guardarei com muito carinho cada um de vocês!

À Julia, aluna que co-orientei durante a iniciação científica,

Sou muito grata a você pela parceria e auxílio na realização deste trabalho. Parabéns por toda competência e seriedade! Conte sempre comigo!

À professora Gisele Rodrigues da Silva, algumas atitudes nos faz ser extremamente gratas a algumas pessoas e você Gisele é uma delas. Obrigada por todo auxílio que me deu durante a condução deste trabalho e principalmente o suporte quando estava prestando o concurso em Rio Verde. A você deixo toda minha admiração como docente e ser humano.

Ao Professor Luís Henrique Raposo Araújo, que me auxiliou durante a execução deste trabalho. Obrigada pelo suporte científico e por sempre estar disposto a me auxiliar. Você é um profissional que sempre admirei pela competência, engajamento e seriedade.

Ao professor Carlos José Soares, pelos ensinamentos e auxílio durante toda minha trajetória na pós-graduação. Você tem uma competência admirável!

Aos professores Paulo César Freitas Santos Filho, Paulo Vinícius Soares, Veridiana, Paulo Simamoto e Alfredo,

Muito obrigada por todos os ensinamentos, por estarem sempre dispostos a ajudar, pelo profissionalismo e competência. Me espelho muito em vocês para continuar seguindo a carreira acadêmica.

Aos professores da FOUFU,

Obrigado pelos ensinamentos e por contribuir com a minha formação. Sinto orgulho de ter sido aluna de cada um de vocês.

A Graça e a Brenda,

Obrigada por toda ajuda durante esses anos de pós-graduação.

A Universidade de Rio Verde, aos docentes e alunos, obrigada por terem me acolhido tão bem e pela oportunidade de trabalhar com cada um de vocês. Sinto muito orgulho em fazer parte dessa instituição.

Ao professor Marcelo Bighetti Toniollo,

obrigada por aceitar nosso convite e compor a banca avaliadora deste trabalho. Admiro muito seu trabalho, comprometimento e competência.

Ao professor Hugo Lemes Carlo,

obrigada por aceitar nosso convite e compor a banca avaliadora deste trabalho. É uma honra sua presença.

AGRADECIMENTOS,

À Faculdade de Odontologia da Universidade Federal de Uberlândia (FOUFU)

Ao CPbio (Centro de Pesquisa de Biomecânica, Biomateriais e Biologia Celular da FOUFU), por disponibilizar espaço físico e equipamentos necessários para realização desta pesquisa.

Ao curso de Física da Universidade Federal de Uberlândia, em especial ao professor Adamo e André por disponibilizar equipamentos necessário para realização desta pesquisa e todo auxílio na condução deste trabalho.

Ao Programa de Pós- Graduação,

A CAPES, Coordenação de Aperfeiçoamento de Pessoal de Nível Superior pela concessão da bolsa de doutorado.

Ao CNPq, pelo apoio financeiro.

À empresa FGM, pela doação de materiais.

EPÍGRAFE

“Tudo que um sonho precisa para ser realizado é alguém que acredite que ele possa ser realizado.”

Roberto Shinyashiki

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Resumo

RESUMO

O sucesso e a longevidade de uma restauração de cerâmica ou resina composta estão relacionados com selamento marginal satisfatório, integridade do material e estética favorável. A substituição ainda ocorre com frequência devido a manifestações clínicas indesejáveis como fraturas, alteração de cor e perda de brilho. Assim, é importante avaliar possíveis fatores relacionados a falhas de restaurações indiretas e diretas a fim de prevenir retratamento. Desta forma, os objetivos desta tese foram: **Objetivo 1:** avaliar o efeito de diferentes protocolos de acabamento e polimento nas propriedades mecânicas e topografia de superfície de cerâmica vítrea a base de fluorapatita. **Objetivo 2:** avaliar o efeito do polimento imediato e tardio na microdureza, estabilidade de cor e topografia de superfície de três diferentes tipos de resinas compostas, após imersão em café. **Objetivo 3:** avaliar propriedades mecânicas e físicas de resinas compostas bulk-fill polimerizadas com diferentes unidades fotoativadoras. Os métodos experimentais utilizados nos estudos laboratoriais foram perfilômetria e microscopia de força atômica para caracterizar a rugosidade de superfície, espectrofotômetro associado à fibra óptica para mensurar o brilho, microscopia eletrônica de varredura para avaliação qualitativa da topografia de superfície, resistência à flexão biaxial para avaliar a resistência à flexão, espectrofotômetro para análise de cor, espectroscopia infravermelha transformada de Fourier (FTIR) para avaliar o grau de conversão e teste de indentação para cálculo de dureza Knoop. Após análise estatística dos dados, pôde-se concluir que o protocolo de polimento utilizando sistemas com pontas de polimento de abrasividades sequenciais pode ser uma boa alternativa para o acabamento em consultório após ajustes de superfícies de cerâmica vítrea à base de fluorapatita. O polimento imediato ou após 24 horas diminui a rugosidade de superfície e alteração de cor de diferentes categorias de resinas compostas, além disso provoca o aumento de dureza da restauração. As resinas compostas bulk fill apresentam comportamento mecânico dependente da fonte fotopolimerizadora. O grau de conversão, a microdureza e a resistência a tração diametral de diferentes resinas bulk fill são

influenciados pelas unidades de fotopolimerização utilizadas na prática odontológica.

Palavras- chaves: Resinas compostas, Cerâmica, Polimento Dentário, Dureza.

Abstract

ABSTRACT

The success and longevity of a ceramic restoration or composite resin are related to satisfactory marginal sealing, material integrity and aesthetics. Replacement of restorations as general practitioners in clinical manifestations such as fractures, color change and loss of gloss. Thus, it is important to evaluate possible factors related to failure of indirect and direct restorations in order to prevent retreatment. Thus, the objectives of this study were: **Objective 1:** to evaluate the effect of different finishing-polishing protocols on the mechanical properties and surface topography of pressable fluorapatite glass ceramic. **Objective 2:** to evaluate the effect of immediate and delay polishing on the mechanical properties of hardness, color stability and surface topography of three different types of composite resins after immersion in coffee. **Objective 3:** to evaluate the mechanical and physical properties of different bulk-fill polymerized with different light-curing units. The experimental methods used in the laboratory studies were profilometry and atomic force microscopy to characterize surface roughness, spectrophotometer associated with optical fiber to measure gloss, scanning electron microscopy for qualitative evaluation of surface topography, resistance to biaxial flexural strength to evaluate the flexural strength, spectrophotometer for color analysis, Fourier transform infrared spectrometer (FTIR) to evaluate the degree of conversion and indentation test to calculate Knoop microhardness. After statistical analysis, it can be concluded that the polishing protocol using systems with polishing tips of sequential abrasives can be a good alternative for chairside finishing of adjusted pressable fluorapatite glass ceramic surfaces. Polishing immediately or after 24 hours decreases the surface roughness and color change of different categories of composite resins, in addition causes the increase of hardness of the restoration. The bulk fill resin composites present mechanical behavior depending on the photopolymerizing source. The degree of conversion, microhardness and diametral tensile strength of different bulk fill resins are influenced by the photopolymerization units used in dental practice.

Key-Words: Composite resins, Ceramics, Dental Polishing, Hardness.

Introdução e Referencial teórico

1. Introdução e Referencial Teórico:

A odontologia contemporânea visa a longevidade das reabilitações, evitando procedimentos de substituições precoces das restaurações. Dentre os materiais restauradores mais utilizados atualmente, destacam-se as cerâmicas odontológicas para uso indireto e as resinas composta para uso direto. O sucesso de uma restauração de cerâmica ou resina composta está relacionado com o selamento marginal satisfatório e estética favorável, mimetizando características anatômicas e funcionais dos dentes naturais. No entanto, a substituição ainda ocorre com frequência devido à fatores como fraturas, alteração de cor, perda de brilho, forma anatômica insatisfatória e descoloração marginal (Anusavice, 2012). Portanto, é imprescindível avaliar tais fatores a fim de prevenir o retratamento de reabilitações dentárias.

Nas reabilitações indiretas, as cerâmicas odontológicas permitem mimetizar os dentes naturais e são muito utilizadas devido às suas excelentes propriedades físicas e mecânicas, estética, biocompatibilidade, baixa condutividade térmica e resistência ao desgaste (Ozarlan *et al.*, 2016). Além disso, houve considerável evolução das técnicas e dos materiais adesivos utilizados para fixação destas à estrutura dental, resultando em formação de corpo uniforme, o que amplia assim suas indicações (Fischer *et al.*, 2008).

Diferentes tipos de cerâmica podem ser empregados para confecção de restaurações indiretas. Dentre elas, a cerâmica vítrea de fluorapatita obtida por meio do sistema de pastilhas de prensagem com injeção sobre óxido de zircônio. Nas reabilitações com sistemas cerâmicos, as peças protéticas devem apresentar superfície lisa e polida, assim, são tradicionalmente tratadas em laboratório com aplicação de glaze (Yilmaz *et al.*, 2008). Este tratamento é considerado padrão ouro, pois gera alta lisura de superfície e biocompatibilidade (Fasbinder & Neiva, 2016), que podem melhorar a resistência à flexão e preservar o brilho da restauração (Wang *et al.*, 2011).

Contudo, ajustes das restaurações com pontas diamantadas são realizados com frequência na prática clínica, visando melhor assentamento nos dentes preparados, oclusão adequada e estética. Esse procedimento, remove

a camada de glaze da cerâmica, torna a superfície áspera e rugosa (Aykent *et al.*, 2010), favorece a formação de biofilme dentário (Kim *et al.*, 2017), resultando maior acúmulo de bactérias (Aykent *et al.*, 2010, Dutra *et al.*, 2018) facilitando a descoloração da restauração (Motro *et al.*, 2012). Adicionado a esses fatores também está o aumento do desconforto do paciente e da possibilidade de concentração de tensões nas ranhuras geradas pela ponta diamantada que pode resultar em trincas e estas se propagarem e decorrer em falha prematura destas restaurações (Fischer *et al.*, 2003).

Assim, após os ajustes oclusais e estéticos com pontas diamantadas, as restaurações cerâmicas devem ser submetidas a um protocolo de polimento adicional, uma vez que a lisura da superfície é essencial para o sucesso clínico destas restaurações cerâmicas (Silva *et al.*, 2015). Atualmente, diversos sistemas para polimento de cerâmicas odontológicas estão disponíveis no mercado. Estes sistemas devem resultar em lisura e brilho da superfície semelhante ao da técnica de glazeamento. No entanto, ainda não existe consenso sobre qual material é mais eficaz para realização do polimento da cerâmica durante o atendimento clínico odontológico.

Dentre os materiais restauradores de uso direto, destacam-se as resinas compostas. Vários fatores contribuíram para sua ampla utilização, incluindo estética, propriedades mecânicas satisfatórias e preservação de tecido dental sadio (Ferracane, 2011). Desde a sua introdução na odontologia, um desenvolvimento contínuo na tecnologia das resinas compostas ocorreu e atualmente há diversos tipos disponíveis comercialmente. Estes materiais apresentam diferentes propriedades físicas e mecânicas e são classificados de acordo com a matriz resinosa, com o tamanho, quantidade e distribuição das partículas de carga inorgânica, na sua composição que pode ser microparticulados, híbridos, nanoparticulados (Ferracane, 2011; de Moraes *et al.*, 2009) e bulk-fill, que consiste em uma nova categoria de resinas compostas introduzidas no mercado.

Assim como relatado para as reabilitações realizadas com sistemas cerâmicos, o sucesso clínico e a longevidade das restaurações em compósitos

resinosos também estão relacionados com a aparência estética e integridade das margens da restauração. Estudos relatam que a substituição de restaurações diretas ocorre com frequência devido à fatores como cárie secundária (Demarco *et al.*, 2012), fratura (Demarco *et al.*, 2012), pigmentação, perda de brilho, alteração de forma anatômica e descoloração marginal (Mjor *et al.*, 2000; Opdam *et al.*, 2014; Demarco *et al.*, 2015).

A degradação química que ocorre na cavidade oral pode alterar a longevidade da resina composta, visto que na dieta humana há uma variedade de alimentos e bebidas contendo ácidos e corantes (Bagheri *et al.*, 2005; Topcu *et al.*, 2009). Tais componentes podem promover alterações na topografia de superfície e energia livre superficial (Fucio *et al.*, 2008), diminuição da microdureza e degradação da matriz orgânica (Karaman *et al.*, 2014). Outro aspecto observado frequentemente nas reabilitações diretas é a alteração de cor que ocorre nas resinas compostas (Mjor *et al.*, 2000; Opdam *et al.*, 2014; Demarco *et al.*, 2015). Ela pode ocorrer por absorção e/ou adsorção de corantes durante o período de exposição da restauração a alimentos e bebidas (Bagheri *et al.*, 2005; Topcu *et al.*, 2009). Estudos prévios demonstram que o café, por exemplo, causa mudanças significativas de cor nas resinas compostas após 7 dias de imersão (Barutcgil *et al.*, 2018; Alawjali & Lui, 2013).

O acabamento e polimento é de extrema importância para a qualidade da restauração em resina composta e cerâmica. Quando este procedimento é negligenciado ou inadequado, o aumento na rugosidade de superfície pode resultar em problemas clínicos, como pigmentação (Celik & Ozgunaltay, 2009), inflamação gengival (Aykent *et al.*, 2010), abrasividade e desgaste de dentes antagonistas, desconforto do paciente (Jefferies, 2007) e risco de recidiva de cárie, por se tornar uma área de retenção de placa bacteriana (Alawjali & Lui, 2013). Outra consideração importante é o tempo decorrido após a confecção da restauração e a realização do acabamento e polimento. Na literatura existem controvérsias do momento ideal. Alguns estudos indicam a realização do acabamento e polimento 24 horas após a finalização da restauração e justificam ter efeito benéfico sobre as propriedades físicas e ópticas dos

materiais restauradores (de Moraes *et al.*, 2015), como a redução da microinfiltração (Lopes *et al.*, 2002) e também do risco de falhas prematuras. No entanto, a maioria dos clínicos prossegue com a conclusão desta etapa imediatamente após a confecção da restauração. Além disso, alterações dimensionais ocorrem em diferentes tipos de compósitos e estão associadas com os efeitos das técnicas de polimento na rugosidade superficial, microdureza (Venturini *et al.*, 2006) e estabilidade de cor, uma vez que estas são dependentes do tipo do material (Beltrami *et al.*, 2018). No entanto, por ter sido introduzido recentemente no mercado, há poucas informações na literatura sobre o efeito do acabamento e polimento nos compósitos resinosos bulk-fill e a influência do momento de realização deste procedimento nas suas propriedades.

As resinas *bulk-fill* apresentam variedades de modificações em sua composição para possibilitar maior profundidade de polimerização em associação com menor contração de polimerização que permite assim inserção em único incremento de até 4-5mm, sem que haja interferência negativa na sua cinética de polimerização (Alrahlah *et al.*, 2014; Zorzini *et al.*, 2015). Uma das modificações é a alta translucidez que permite maior passagem de luz pela massa de resina durante a etapa de fotoativação para garantir adequada polimerização em maiores profundidades (Bucuta & Ilie, 2014). Além disso, a incorporação de monômeros resinosos que apresentam alívio de tensões de contração de polimerização, moduladores específicos de polimerização, fotoiniciadores mais reativos e de partículas pré-polimerizadas na composição desses compósitos resinosos resultam em menor contração de polimerização (Moszner *et al.*, 2008; Goracci *et al.*, 2014).

A eficácia da polimerização depende da sensibilização de fotoiniciadores e radicais livres pela unidade fotopolimerizadora utilizada. No mercado existem inúmeros sistemas que variam desde luz halógena de quartzo tungstênio até os diodos emissores de luz de alta intensidade (LED) de várias gerações e diversos comprimentos de onda. Sabe-se que para a resina composta possuir polimerização apropriada, o aparelho fotoativador deve emitir comprimentos de onda que se sobrepõem à absorção do fotoiniciador presente

na sua composição (Santini *et al.*, 2013). Estudos mostram que a conversão adequada dos monômeros presentes nas resina bulk-fill é essencial para determinar o seu desempenho físico e mecânico (Ferracane & Greener 1986; Leprince *et al.*, 2013). A literatura ainda é escassa de estudos que avaliam a influência do espectro de emissão de luz das diferentes unidades fotopolimerizadoras, ao utilizar resinas compostas bulk-fill de múltiplos iniciadores.

Portanto, o presente trabalho é justificado pela necessidade de avaliar fatores que possam interferir na longevidade e necessidade de substituição de restaurações indiretas e diretas. Assim, o desempenho mecânico e topografia de superfície de cerâmica vítrea foram avaliados frente a diferentes sistemas de polimento; a performance física e mecânica de resinas compostas foi avaliada por meio do momento da realização do acabamento e polimento e da análise do material restaurador bulk-fill frente diferentes fontes de ativação.

Objetivos

2. Objetivos:

Objetivo Geral:

Avaliar fatores que possam interferir no desempenho físico e biomecânico de materiais restauradores de uso indireto e direto.

Objetivos Específicos:

Objetivo específico 1:

Capítulo 1- *Surface topography, gloss and flexural strength of pressable ceramic after finishing-polishing protocols.*

Este objetivo específico avaliou o efeito de diferentes protocolos de acabamento e polimento na rugosidade da superfície, brilho, resistência à flexão biaxial e morfologia de cerâmica vítrea.

Objetivo específico 2:

Capítulo 2- *Effect of polishing time on color stability, surface roughness and microhardness of resin composites*

Este objetivo específico avaliou o efeito do polimento imediato e tardio de diferentes categorias de resina composta na alteração de cor, rugosidade superficial e microdureza após imersão em café.

Objetivo específico 3:

Capítulo 3- *Effect of light curing units on Degree of Conversion, microhardness and Mechanical properties of a bulk fill composite*

Este objetivo específico avaliou as propriedades mecânicas e físicas de resinas compostas bulk-fill polimerizadas com diferentes unidades fotoativadoras.

Capítulos

3. Capítulos:

Serão apresentados nesta sessão três artigos, sendo que cada um corresponde a um capítulo.

Capítulo 1

3.1. Capítulo 1: Artigo aceito para publicação pelo periódico Brazilian Dental Journal

Title: Surface topography, gloss and flexural strength of pressable ceramic after finishing-polishing protocols.

Short title: Polishing protocols for pressable ceramic

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SUMMARY

This study evaluated the effect of different finishing-polishing protocols on surface roughness, gloss, biaxial flexural strength and morphology of pressable fluorapatite glass ceramic. Thirty ceramic discs (12x1mm) were produced and divided into five groups (n=5): CT- control (glaze); DA- fine grit diamond bur; DG- DA + new glaze layer; DP- DA + felt disk with fine grit diamond paste; DK- DA+ sequential polishing with silicon abrasive instruments, goat hair brush and cotton wheel. The specimens were analyzed for surface roughness (Ra) under profilometry and atomic force microscopy (AFM). Gloss was measured with spectrophotometry and micromorphology with scanning electron microscopy (SEM). Flexural strength was assessed by biaxial flexural strength test. Data were analyzed using one-way ANOVA and Tukey's post hoc test ($\alpha=0.05$). DK showed the lower surface roughness values and DA presented the higher in the profilometer analysis. No significant differences were observed in the AFM for the CT, DG and DK groups, which presented the lower surface roughness; DA and DP had the higher Ra values. The DA, DP and CT showed the lowest surface gloss values, and the reflectance was significantly different from those observed for DK and DG groups. SEM analysis revealed the smoothest surface for DK group, followed by DG and CT groups; DA and DP groups exhibited variable degrees of surface irregularities. No significant differences were observed among groups for the biaxial flexural strength. The polishing protocol used in DK group can be a good alternative for chairside finishing of adjusted pressable fluorapatite glass ceramic surfaces.

Key-Words: ceramics, roughness, surface, polishing.

INTRODUCTION

Dental ceramics are currently one of the most popular restorative materials because of their natural and esthetic appearance, low heat conduction, good mechanical properties and wear resistance (1). These materials have been extensively used over metallic structures or in all-ceramic restorations like inlays, onlays, veneers and full crowns (2).

Ceramic restorations are commonly fabricated by dental laboratories using different techniques, such as CAD/CAM and press-over. In the heat pressing technique, a final contour wax-up is made on the sintered zirconia framework and invested, burned out, and pressed with fluorapatite pressable ceramic. This technique has the advantages of being faster, with detailed and esthetic reproduction of the wax-up, presenting less distortion, increased marginal accuracy, and being less costly (3, 4). During the restoration fabrication process, a final glaze surface is created in the restoration with an oven firing process, by natural or additive procedures (5). This process results in restorations with smooth and biocompatible surface (6), which can improve the flexural strength and preserves surface gloss (7). However, despite the careful procedures taken by the practitioner and dental technician to produce indirect restorations, it is not uncommon to perform clinical adjustments in the glazed ceramic surface using diamond burs or other instruments for correcting occlusal contacts and/or inadequate contours (8). After performing clinical adjustments, surface roughness of ceramic restorations will predictably increase (8). The rough surfaces can cause biofilm accumulation (8), and may lead to severe wearing of opposite teeth (9). Furthermore rough or irregular ceramic surfaces may cause stress concentration and initiate cracking propagation, resulting in premature failure of the restoration (10).

Hence, all adjusted ceramic restorations should be submitted to a further polishing protocol since surface quality is essential for the clinical success of ceramic restorations (11). Finishing and polishing procedures are important for reducing the roughness of adjusted ceramic surfaces (1, 2), preventing discoloration of roughened areas in order to maintain the natural appearance (1,

7), as the gloss of the ceramic surface is commonly restored (7). Currently, several systems for finishing-polishing of dental ceramics are available in the market. These systems can be effective in producing a regular surface and save working time after small ceramic adjustments, since it is not necessary to return the restorations to the dental laboratory (2).

Thus, the aim of this study was to evaluate the effect of different surface treatments on the surface roughness, gloss and biaxial flexural strength of a pressable fluorapatite glass ceramic. The null hypothesis tested was that the different surface treatments would not influence the mechanical properties, roughness and reflectance of the pressable ceramic as compared to ceramic surfaces obtained with laboratory glaze procedures.

METHODS AND MATERIALS

Specimen preparation

Thirty pressable fluorapatite glass ceramic discs, 12 mm in diameter and 1 mm thick, were fabricated according to the manufacturer's recommendations (IPS e.max ZirPress, Ivoclar Vivadent, Schaan, Liechtenstein). For preparing the specimens, standard disc-shaped acrylic resin patterns (Duralay, Reliance Dental Manufacturing, Chicago, Illinois, USA) were confectioned (n=5). The resin discs were polished with silicon carbide abrasive papers (#180, 320, 600, Norton, Campinas, SP, Brazil) until the correct thickness was obtained. A digital caliper (Mitutoyo, Santo Amaro, SP, Brazil) was used to ensure consistency in the dimensions of the resin patterns.

The specimens in acrylic resin were invested in a phosphate-based investment material (IPS PressVest, Ivoclar Vivadent). Resin patterns were eliminated in a proper furnace and prefabricated pressable fluorapatite glass ceramic ingots were then pressed using the lost-wax technique. The pressing process was carried out in a press furnace (EP3010, Ivoclar Vivadent) according to the manufacturer's instructions. After divesting, specimens were polished with silicon carbide abrasive papers (#600) followed by ultrasonic cleaning in distilled water for 10 min. Sequentially, all specimens were polished

with abrasive rubber discs and submitted to glazing firing using glaze paste (e.max Glaze Paste, Ivoclar Vivadent). This process was performed with Ivoclar preset programming P310 furnace protocol (Ivoclar Vivadent) (initial temperature 403 °C; temperature rise 60°/min; final glazing temperature 770 °C for 90 s; vacuum initiated at 450 °C and released at 769 °C). Ten minutes after opening the furnace, the discs were removed, and bench cooled.

Experimental Groups

The specimens were randomly divided into 5 experimental groups (n=6) according to surface treatments: CT- control group (glaze); DA- surface abrasion with fine grit diamond bur (#4137F, KG Sorensen, Barueri, SP, Brazil); DG- abrasion with fine grit diamond bur (#4137F) followed by application of a new glaze layer; DP: abrasion with fine grit diamond bur (#4137F), followed by polishing with felt disk (DHPro; Paranaguá, PR, Brasil) impregnated with fine-grit diamond paste (Diamond Excel, KG Sorensen); DK- abrasion with fine grit diamond bur (#4137F), followed by sequential polishing with silicon carbide abrasive instruments (coarse, medium, and fine grit), goat hair brush and cotton wheel (DHPro). One trained operator subjected all the specimens from each group to the respective finishing-polishing protocols for 30 s using slow-speed hand piece (Kavo Kerr, Biberach, Germany), under water cooling to simulate clinical procedures. Each polisher was used with the same slow-speed hand piece maintaining uniform and intermittent pressure for all groups. The equipment was set to 9,000 rpm. After the surface treatments, all the specimens were ultra-sonically cleaned in distilled water for 10 min.

Surface Roughness

For analyzing the surface roughness of the specimens, the arithmetic mean (Ra- average surface roughness) was assessed with a digital profilometer (Surftest SJ-410; Mitutoyo Corp, Tokyo, Japan). For measuring the roughness profile value in micrometers (μm), a diamond stylus with 5 μm tip radius was moved across the surface under a constant load of 4 mN with a speed of 0.25 mm/s and a range of 0.8mm. Three traces were recorded for each specimen at three different positions (parallel, perpendicular, and oblique), resulting in nine

tracings per specimen. The mean surface roughness was then calculated for each specimen. An atomic force microscope (AFM, XE-70; Park Systems Inc. Santa Clara, CA, USA) operated in no contact mode, was used to obtain quantitative and qualitative evaluation of the specimens. In no contact mode, the force between the AFM tip and the specimen surface was kept constant by the microscope feedback system, while the specimen surface was scanned beneath the AFM tip and the vertical piezoelectric ceramic movement was recorded. Images with 512 x 512 pixels were acquired with a scan size of 20 μ m x 20 μ m at a scan rate of 1.00 Hz. An NP-type V-shape Si₃N₄ cantilever with a tip radius of approximately 10 nm was used. The AFM obtained a 3-dimensional image of the surface of the specimen. Two different areas were measured in each specimen at different regions, all located in the center of the object. The surface roughness parameters Ra were then calculated in both slow and fast scanning directions using the built-in functions of the Mountains map 3.0.0.1 software. Means and standard deviation of surface roughness were then determined.

Spectrophotometric reflectance

The reflectance values of the specimens were measured using a spectrophotometer (USB4000, Ocean Optics, FL, USA) associated with an optical fiber cable. The specimens were positioned in a device platform and a light beam was focused over the ceramic surface to allow measuring the intensity of reflected light. For each specimen four measurements were conducted. Data were recorded on Origin 8.0 software (Origin 8.0, OriginLab Corporation, Northampton, MA, USA).

Biaxial Flexural strength

The biaxial flexural strength of the discs was checked with a tension-compression device (DL2000, EMIC, São José dos Pinhais, PR, Brazil) at a crosshead-speed of 0.5 mm/min. The specimens were orientated so the finished-polished surfaces were subjected to compressive stressing and tested according to ISO 6872 (12). To support the specimens, 3 hardened steel balls (3.2 mm in diameter) were placed at an angle of 120 degrees relative to each

other. The diameter of the support circle was 10 mm. Each specimen was centrally located on the supports and the loading was applied at the center of the glazed surfaces of the specimens. A flat piston (1.4 ± 0.2 mm in diameter) was used during loading until fracture of the specimens. At this point, the fracture load was recorded.

Scanning Electron Microscopy Analysis

Representative specimens of each group were selected to evaluate the effects of the surface treatments on the micromorphology of the ceramic discs. The specimens were analyzed under scanning electron microscope (SEM, Leica EM SCD050, Leica Microsystems, Wetzlar, Germany) at 15.0 kV, after sputter-coating with a thin film of gold. The SEM photomicrographs were taken with 100x magnification for qualitative analysis of the specimens.

Statistical Analysis

The data were analyzed using the SigmaStat v.3.5 statistical software package (Systat Software Inc., Chicago, IL, USA). The surface roughness, spectrophotometric reflectance and biaxial flexural strength data were individually submitted to one-way analysis of variance followed by Tukey's test ($\alpha = 0.05$), since data presented normal distribution based on the Shapiro–Wilk test ($p > 0.05$).

RESULTS

Surface roughness

The results for the surface roughness are summarized in Table 1. For the profilometry, significant differences were found among the surface roughness of the experimental groups ($P \leq 0.001$). Specimens of DK group presented the lower mean surface roughness values and DA group presented the higher mean surface roughness.

The AFM analysis showed that the groups submitted to the glazing process, CT and DG, and to the sequential finishing-polishing protocol (DK) presented the lower mean surface roughness values ($p \leq 0.001$) (Table I). The

group abraded with fine grit diamond bur only (DA) and abrasion followed by polishing with fine-grit diamond paste (DP) showed the higher mean surface roughness. The AFM images obtained from the specimens corroborate with the measured roughness values (Figure 1A-E), showing the different surface roughness characteristics for all the surface treatments.

Spectrophotometric reflectance

One-way analysis of variance identified significant differences in the gloss verified among the experimental groups ($p < 0.001$). The DA and DP groups presented the lowest gloss values. DK and DG groups showed the highest gloss values. The CT group exhibited intermediary gloss values, not differing statistically from the other experimental groups.

Biaxial flexural strength

One-way ANOVA showed the surface treatments had no significant effect on the biaxial flexural strength of the groups ($p=0.274$), which showed the following mean strength values (MPa): CT (100.6 ± 17.72), DA (101.5 ± 19.34), DG (124.1 ± 17.84), DP (116.2 ± 10.51) and DK (119.6 ± 32.17).

SEM analysis

SEM analysis confirmed the roughness findings, with DG and DK groups exhibiting a morphological pattern similar to that of CT group with a smoother surface (Fig. 2A, 2C and 2E). The DA and DP groups exhibited variable degrees of surface irregularities (Fig. 2B and 2D). The surface irregularities and voids were reduced by sequential polishing with abrasive silicon carbide points (DK), even though, some voids still persisted after using this protocol (Fig. 2E).

DISCUSSION

The null hypothesis was rejected, since the different surface treatments tested affected the physical properties of the pressable fluorapatite glass ceramic evaluated as compared to ceramic surfaces obtained with laboratory glaze procedures.

The wide application of dental ceramics in contemporary restorative dentistry has created numerous concerns for the clinicians regarding the correct protocols for finishing and polishing these restorations when clinical adjustments are needed. As seen, ceramic restorations needs suitable surface treatments procedures to achieve smooth surfaces, avoiding biofilm accumulation, crack propagation (8), decrease in the porcelain strength (10), besides reducing the wear of the opposing natural teeth as well as restorative surfaces (9). The application of a glass ceramic layer by the glazing process is considered the gold standard after performing adjustments in ceramic restorations (6); however, it has some disadvantages, such as, additional time due to the laboratory processing and the impossibility to be made after luting procedures (13).

The surface roughness and gloss of dental ceramics varies according to the methods and materials/instruments used for during laboratory manufacturing. Attempting to reestablish surface smoothness to roughened ceramics after clinical adjustments, several chairside ceramic finishing/polishing systems are available nowadays, although there is still controversy about their effectiveness in literature. The finishing and polishing protocol used for the DK group presented the best performance among all protocols, with similar results to the CT group. On the other hand, as one would expect, the group in which only finishing with fine grit diamond burs was carried out, showed the worst performance among all the experimental groups. This fact is particularly important to aware clinicians when performing chairside adjustments of glazed ceramic restorations before or after luting procedures, since special attention should be given for recovering surface characteristics similar to laboratory glazed or polished restorations.

To fully characterize the surface of pressable ceramic after using different surface treatments, a multimatic approach was employed. A profilometer and atomic force microscope (AFM) were used to measure the roughness of the ceramic surfaces. The SEM and AFM techniques were also employed to qualitatively evaluate the morphology and surface texture of the specimens. In the current study, no contact mode was used in the AFM to

evaluate the surface roughness of ceramics as this approach has had several advantages when compared to other methods (14). The resolution of an optical light sensor is considerably higher than the mechanical pen used in contact scanners (15). Thus, the roughness values obtained using this technique are more precise, especially when evaluating very smooth surfaces such those of dental ceramics after finishing/polishing procedures. In addition, non-contact acquisition mode for assessing roughness avoids surface damage that could be caused by a contact mechanical sensor, what could produce biased results (15). Since several authors found significant differences between the roughness values obtained using these two methodologies (14), direct comparison of the Ra values of the present study with the reports of other studies, as well as the reported threshold, must be conducted with caution.

The assessment of surface roughness with profilometry and AFM was calculated by using the Ra parameter. The results showed that the DK group presented the lower surface roughness. These results are in accordance with previous studies (1, 16, 17) which found similar roughness values between glass ceramics submitted to glaze or sequential polishing with silicon carbide abrasive instruments (2, 14, 18). The DK group specimens have been submitted to sequential finishing/polishing with abrasive silicon carbide points with coarse, medium and fine grit, associated to goat hairbrush and cotton wheel, what may explain the findings of the present study. The abrasives particles of the silicon carbide points are hard enough to remove the irregularities from the ceramic surfaces (16), and final polishing with extremely fine abrasive materials are capable to reduce roughness (18). The CT and DG groups showed satisfactory results in the profilometer analysis as the glaze procedures obliterates the irregularities present at the ceramic surface, improving surface smoothness (7).

The size of abrasive particles has a fundamental role in the resulting topographic characteristics of ceramic materials submitted to finishing/polishing protocols with different instruments. However, in this study, the experimental group submitted to finishing with diamond paste (DP) containing only homogeneous particle size, has not shown satisfactory surface roughness

compared to CT group. This fact, can be explained because the polishing paste cannot promote an efficient polishing when used alone, showing that these materials needs to be associated to other polishing systems for dental ceramics (16). The DA group presented the highest surface roughness, showing that ceramic adjustments performed only with diamond points may cause grooves and gaps on the ceramic surface. These surface irregularities must be polished after the adjustment procedures in order to reduce the risk of crack propagation on the ceramic surface.

The SEM images confirmed the results found in the profilometer and AFM analyses. The DK group showed the most homogeneous and smooth surface, with few bubbles and flaws being detected (16). Groups DG and CT presented similar surface characteristics to DK, with no relevant irregularities, but showing few bubbles due to the glazing process. DA group also showed the worst surface characteristics on the SEM images followed by the DP group, which presented several surfaces irregularities, grooves and gaps. The use of the polishing diamond paste after the adjustment procedures using diamond burs was not effective in regularizing the ceramic surface, since the diamond particles were not sufficient to produce a smooth surface, as shown by the SEM images. As expected, DA group showed the higher irregularities in the surface which were caused by the abrasion of the diamond points following adjustments without subsequent polishing. Again, this finding highlights the importance of performing chairside polishing of ceramic restorations after adjustments using diamond burs (19). Additionally, maybe the use of a polishing paste after the finishing/polishing protocol from DK group could provide a slight improvement in the surface roughness when used, but, it was not effective in removing the irregularities when used isolated, as shown in the SEM images from DP group.

The spectrophotometric reflectance evaluated the surface gloss of the pressable ceramic. The surface gloss represent the amount of specular (mirror-like) reflection from a surface (19, 20). Therefore, gloss is affected by the index of refraction of a material (measure of the ability of a material to change the velocity and direction of incident light upon contact with its surface) and the topography of its surface (20). In this study, the highest surface gloss value

was observed for DK (3364.7 ± 5.893), followed by DG (3360.8 ± 5.336) and CT (3356.8 ± 3455). DK group presented satisfactory gloss because the protocol was finished with goat hairbrush and cotton wheel, what lead to satisfactory brightness. These results support the idea that surface roughness may affect the general appearance of dental ceramics (21), since surface gloss and fluorescence can modify the color composition (hue, chroma and value) of teeth and restorative materials (22). If the object has a smooth surface, the light is reflected in a narrow cone centered about the angle of reflectance. In contrast, an increasingly roughened surface would reflect individual segments of the specular beam at slightly different angles (23), reducing the resulting surface gloss.

No significant differences were verified between the biaxial flexural strength among the experimental groups tested in this study. The intensity of the surface roughness changes generated by the finishing-polishing procedures was probably too small to produce any deleterious effects on the biaxial flexural strength, since the polishing procedures tested were not detrimental to the mechanical properties of the pressable ceramic. Probably the surface irregularities and defects observed in the glaze layer were reduced after finishing and polishing, what consequently caused a numerical increase in the biaxial flexural strength of the ceramic specimens, despite non-significant differences were verified for the groups (17). This result can be explained because the flexural strength of dental ceramics depends more on the intrinsic factors such as microstructural stresses and bulk defects than on surface roughness (24). The quality of ceramic and the sintering protocol used can be responsible for deeper changes in the characteristics of the ceramic, irrespective of the finishing/polishing protocol (3). As the specimens used in this study were produced with a pressed ceramic material, this resulted in more homogeneous ceramic discs, what also may help to explain the results observed in the biaxial flexural strength test. Moreover, leucite containing ceramics have higher strength since the grains within the glass reduce flaws (4). Additionally, the biaxial flexural strength values found for the specimens of

all experimental groups in the present study are within the values reported for pressable fluorapatite glass ceramic in the current literature (4).

The physicommechanical properties of dental ceramics are closely related to the polishing capacity of these materials. After the rupture of the glaze layer by performing adjustments in the restoration, the best option for finishing and polishing ceramics will depend on the type of material used (25). Thus, the protocol for finishing and polishing dental ceramics must be adjusted to each material correctly. Based on the results of this study, it was observed that the polishing protocols had no effect on the biaxial flexural strength of the pressable ceramic tested. However, the sequential finishing-polishing protocol employed with silicon carbide abrasive instruments (coarse, medium, and fine grit), goat hair brush and cotton wheel systems was effective in reducing the surface roughness, producing a smoother surface with higher gloss in comparison with the other protocols used. Therefore, chairside sequential systems can be a good alternative for clinical finishing-polishing of the adjusted surfaces in pressable ceramic.

Based on the results of this study, it may be concluded that: 1) glazed and polished surfaces produced statistically similar biaxial flexural strength for the pressable ceramic used (IPS e.max ZirPress, Ivoclar Vivadent), irrespective of the polishing protocols tested. 2) Performing adjustments without polishing the surfaces affected the surface roughness of the ceramic material; however, when polishing was performed with sequential finishing-polishing protocol (DK) using silicon carbide abrasive instruments (coarse, medium, and fine grit), goat hair brush and cotton wheel, a significant reduction was observed for the surface roughness. 3) Glazing (CT), reglazing (DG) or sequential polishing (DK) are effective for increasing surface gloss, resulting in smoother surfaces. A clinical implication of the present study is that chairside sequential systems can be a good alternative for clinical finishing-polishing of adjusted surfaces for the pressable ceramic tested.

SUMMARY IN PORTUGUESE

O objetivo deste estudo foi avaliar o efeito de diferentes protocolos de acabamento e polimento na rugosidade da superfície, brilho, resistência à flexão biaxial e morfologia de cerâmica prensada. Trinta discos de cerâmica (12x1mm) foram produzidos e divididos em cinco grupos (n = 5): CT- controle (glaze); DA- ponta diamantada de granulação fina; DG: DA + nova camada de glaze; DP: DA + disco de feltro com pasta de diamante de grânulo fino; DK: DA + polimento seqüencial com instrumentos abrasivos de silício, escova de cabra e roda de algodão. Os espécimes foram analisados quanto à rugosidade da superfície (Ra) sob profilometria e microscopia de força atômica (AFM). O brilho foi medido com espectrofotometria e a micromorfologia com microscopia eletrônica de varredura (SEM). A resistência à flexão foi avaliada pelo teste de resistência à flexão biaxial. Os dados foram analisados usando ANOVA um fator e teste post hoc de Tukey ($\alpha = 0,05$). DK mostrou mais baixos valores de rugosidade da superfície e DA apresentou o maior na análise do perfilômetro. Não foram observadas diferenças significativas no AFM para os grupos CT, DG e DK, que apresentaram a menor rugosidade da superfície; DA e DP apresentaram os maiores valores Ra. O DA, DP e CT mostraram os mais baixos valores de brilho superficial, e a reflectância foi significativamente diferente da observada para os grupos DK e DG. A análise de SEM revelou a superfície mais suave para o grupo DK, seguido de grupos DG e CT; Os grupos DA e DP exibiram graus variáveis de irregularidades da superfície. Não foram observadas diferenças significativas entre os grupos quanto à resistência à flexão biaxial. O protocolo de polimento utilizado no grupo DK pode ser uma boa alternativa para o acabamento em consultório de ajustes de superfícies de cerâmica prensada.

ACKNOWLEDGMENTS

This research was partially supported by the Minas Gerais State Agency for Research and Development (FAPEMIG – Grant 2015-0528).

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Table 1. Means values and standard deviation (\pm) for the experimental conditions

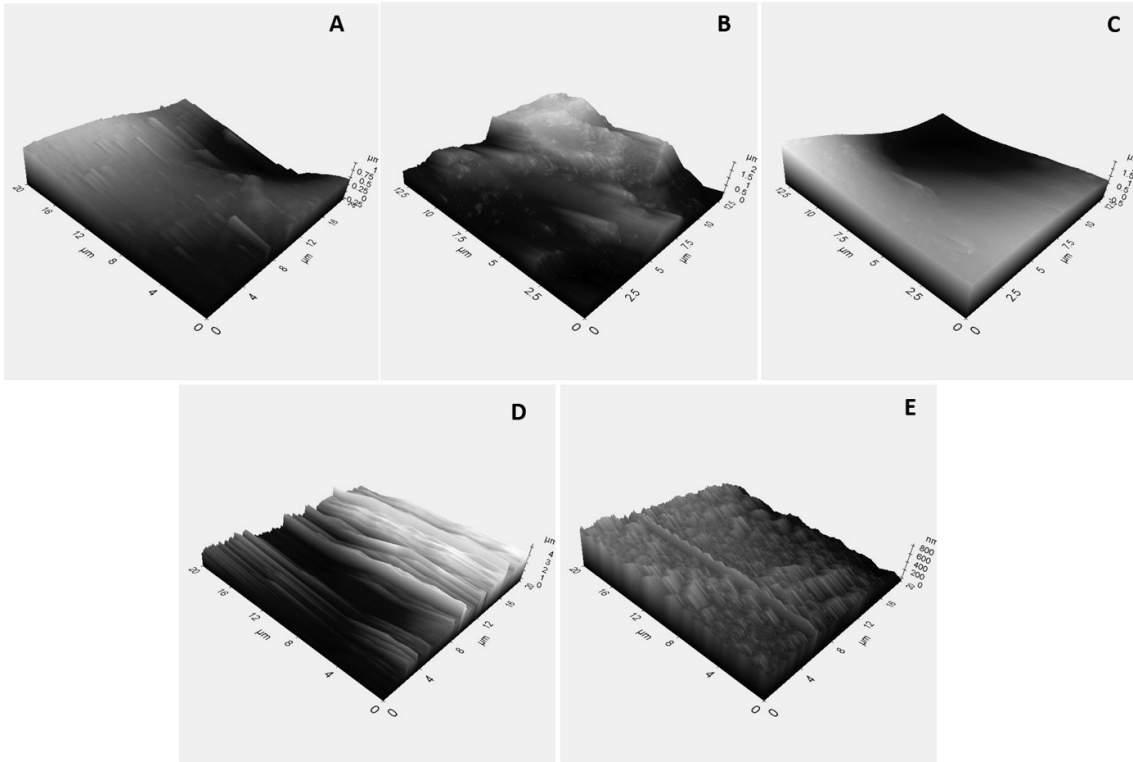
Experimental Group	Surface roughness - Ra (μm)		Gloss values (Gloss Unit- GU)	Biaxial flexural strength (MPa)
	Perfilometry	AFM		
CT	0.83 \pm 0.12 ^b	0.10 \pm 0.05 ^a	3356.8 \pm 3455 ^{ab}	100.6 \pm 17.72 ^a
DA	1.53 \pm 0.13 ^d	0.27 \pm 0.12 ^b	3349.7 \pm 3325 ^b	101.5 \pm 19.34 ^a
DG	0.84 \pm 0.06 ^b	0.12 \pm 0.10 ^a	3360.8 \pm 5336 ^a	124.1 \pm 17.84 ^a
DP	1.17 \pm 0.10 ^c	0.30 \pm 0.14 ^b	3350.7 \pm 4771 ^b	116.2 \pm 10.51 ^a
DK	0.38 \pm 0.51 ^a	0.09 \pm 0.02 ^a	3364.7 \pm 5893 ^a	119.6 \pm 32.17 ^a

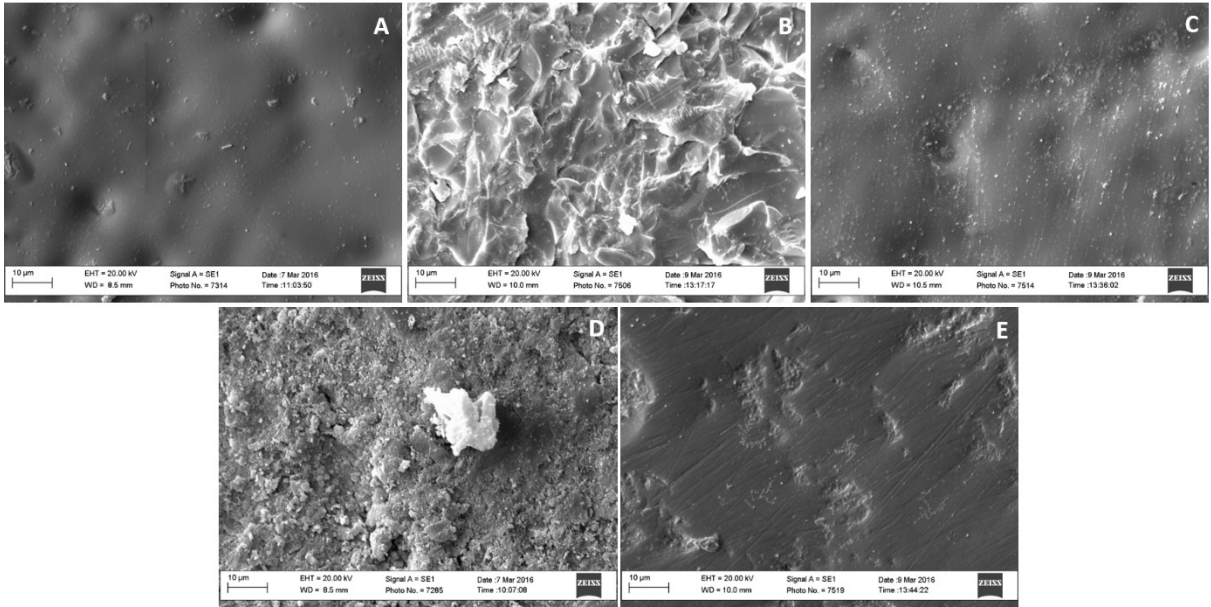
*Distinct lowercase letters indicates significant differences between groups in vertical ($p < 0.05$). Means (95% confidence intervals) for the different tested response variables.

FIGURE CAPTIONS

Figure 1. Representative AFM images of the topographic profiles at the ceramic surfaces of each experimental group: A: CT- control group (glaze); B: DA- fine grit diamond bur; C: DG- fine grit diamond bur + new glaze layer; D: DP- fine grit diamond bur + felt disk with fine grit diamond paste; E: DK- fine grit diamond bur + sequential polishing with silicon abrasive instruments, goat hair brush and cotton wheel.

Figure 2. Superficial morphological pattern (100× magnification): A: CT- control group (glaze); B: DA- fine grit diamond bur; C: DG- fine grit diamond bur + new glaze layer; D: DP- fine grit diamond bur + felt disk with fine grit diamond paste; E: DK- fine grit diamond bur + sequential polishing with silicon abrasive instruments, goat hair brush and cotton wheel.





Capítulo 2

3.2. Capítulo 2: Artigo será submetido para o periódico Journal of Esthetic and Restorative Dentistry.

Title: Effect of polishing time on color stability, surface roughness and microhardness of resin composites

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"The authors do not have any financial interest in the companies whose materials are included in this article."

ABSTRACT

Objectives: This study evaluated the effect of polishing time on color stability, surface roughness and microhardness of three different types of resin composites after immersion in coffee for one and seven days.

Materials and Methods: Ninety discs specimens (6x2 mm) were made from a microhybrid (Filtek Z250), nanofill (Filtek Z350) and bulk fill (Filtek Bulk Fill) resin composites. The specimens were divided into three groups (n=10) according to polishing time: (CT: Control group- no polishing; IP: immediate polished; DP: delay polished, after 24 hours). Color was recorded with a spectrophotometer, surface roughness (Ra) under profilometry and microhardness (KHN) was measured with a Knoop indenter. The data were analyzed with two-way repeated ANOVA and Tukey`s test.

Results: Polishing improved the stain resistance of composite resins. The polishing procedure decreased Ra and increases KNH of all resins composites. Nanofill has the highest microhardness.

Conclusions. The susceptibility of staining, surface roughness and microhardness depend mainly on the composition of the material and the polishing procedures.

Key-Words: Color stability, Composite resins, Surface roughness, polishing.

CLINICAL SIGNIFICANCE:

Polishing time not affect the color change, surface roughness and microhardness of different resin composites after immersion in coffee for seven days. However, polishing is recommended to improve these properties.

Introduction

The use of resin composites has become a routine procedure for the restoration of anterior and posterior teeth (1). Several factors have contributed

to their acceptance, including esthetics, improved mechanical properties and reduction of sound tissue removal (2). A continuous development in composites technology has also occurred in the past decades. Several different composites are available in the market, including: microfilled composites, providing a more polish able surface, but are not as fracture-resistant as the other classes of composites (2, 3); hybrid composites jointing resistance and smooth surface (2, 3) ; composites with nanofillers with excellent polish ability as well as superior polish retention and excellent mechanical properties (3, 4) and newer materials, such as bulk fill composite been subsequently introduced into the dental market.

The bulk fill resin composites are introduced to simplify and shorten the application time, making the easier to use clinical procedure. According to the manufacturers, these materials can be sufficiently cured with light up to 4 mm in a single increment, without influencing the shrinkage of the polymerization, degree of conversion or adaptation of the cavity (5). Various strategies are implemented by different manufacturers for a greater depth of cure and less shrinkage. The addition of stress-relieving monomers, more reactive photoinitiators and pre-polymerized particles result in lower polymerization shrinkage (6, 7). In addition, the increased translucency of these resins allows for greater light transmission and an adequate cure depth (8).

In the oral cavity, alteration in the final color of the restorative material influence the failure or success of an esthetic restoration. Resin composite often become discolored because of intrinsic factors, including the choice of material, the properties of the matrix, the interface between the matrix and the fillers, degree of conversion and water sorption (9, 10). Additionally, extrinsic factors resulting from the contamination of these materials with exogenous sources such as foods, drinks, water, dyes, ultraviolet radiation, heat, smoking, and oral hygiene habits can cause various degrees of discoloration (11, 12). Coffee cause significant color changes in composite resins after 7 days of immersion (12, 13).

Finishing/polishing procedures may also affect the composite surface quality. An increase in surface roughness may create clinical problems such as

staining, plaque retention, gingival irritation, abrasivity and wear kinetics as well as tactile perception, thereby increasing the risk of both caries and periodontal inflammation (4, 9, 13, 14). Surface roughness influences resistance to staining and the natural gloss of the restoration (15).

Another important consideration is the elapsed time after composite placement and timing of the finishing/polishing. Several authors have proposed a 24-hour delay before the completion of finishing procedures, because suggested that might have an effect on the physical properties of the restorative materials, reduced microleakage, and might increase the risk of premature failures (14, 15). However, most clinicians proceed with finishing immediately after the restoration procedure is complete. It is known that dimensional changes occur in different types of composites. A little information is available of the effect of delaying polishing procedures in bulk-fill resin composites.

Thus, the aim of this study was to evaluate the effect of different time of polishing of three composite resin materials. The research null hypothesis was that the polishing time and type of composite resin would not affect the color stability, roughness and microhardness of composites resin.

Methods and materials

Methods and materials

Three different composite resins of shade A1 were selected for this study: microhybrid composite resin (Filtek Z250; 3M ESPE; St. Paul, MN, USA) nanofill composite resins (Filtek Z350 XT; 3M ESPE) and bulk fill (Filtek Bulk Fill; 3M ESPE) (Table 1). Ninety disk-shaped specimens (6 mm in diameter X 2 mm thick) were made. The specimens were produced by packing the composite into a teflon mold. A mylar strip was placed over the surface of the uncured specimen and pressed against it with a glass plate in order to extrude the excess material. The specimens were light cured for the time recommended by the manufacturers (Table 1), using a light-emitting diode light-curing device (Radii-Cal, SDI, Bayswater, Victoria, Australia). The light irradiance was

monitored with an LED radiometer (ECEL, series no. 000165, Ribeirao Preto) before usage and ranged between 653 and 663 mW/cm².

All the specimens were removed from the mold immediately after light curing and the top surface was finished on 600 grit silicon carbide paper on a rotary polisher (Politriz Universal, Arotec, São Paulo, SP, Brasil) for 30 seconds, under running water.

Polishing procedure

For each composite resin, the specimens were then randomly allocated to each of the polishing time: CT: Control group- Mylar strip; IP: specimens were polished immediately after curing; DP: specimens were delayed polished 24 hours after curing and all the specimens were immersed in coffee solution. Each polishing point was used under dry conditions, for 20s, with a low-speed hand piece (KaVo, Joinville, SC, Brazil), rinse and dry with water / air syringe for 10 seconds. The polishing procedure was performed by a single operator, according to the manufacturer's instructions: Jiffy polishers—silicon impregnated rubber discs (three-step system): Step 1 (coarse grit): green disk; Step 2 (medium grit): yellow disk; Step 3 (fine grit): white disk and Step 4: brush silicon carbide-coated for 20 seconds.

Baseline color, surface roughness and microhardness measurements of all specimens were recorded with using methods as described later. The staining agent used for this study was 15 g coffee (Nescafe Classic, Nestle, Switzerland) dissolved in 500 ml of boiling distilled water. Following the initial evaluation, all specimens from each combination of composite resins and polishing time were immersed in the prepared coffee solution. Each group of specimens was stored in a container at 37°C for one day and seven days in an incubator to simulate the temperature of the oral cavity. The coffee solution was freshly prepared and changed every day. The measurements were made after one day and seven days of storage in coffee solution. Before each measurement, the specimens were cleaned using distilled water for 1 min and dried with absorbent paper.

Assessment of color change

Color measurements of all specimens were recorded with the spectrophotometer (Ci64, X-Rite, Pantone, Grand Rapids, Michigan, USA) before and after exposure to the staining agent. The spectrophotometer was calibrated as recommended by the manufacturer at the beginning of the day using a black followed by a white calibrating tile provided by the manufacturer. The CIE (Commission Internationale de L'Eclairage) standard illuminant D65, a standardized CIE illuminant that approximates natural outside daylight was used in this study. Each specimen was removed from the container and dried with an absorbent paper before placing it against the eyelet of the 6 mm diameter aperture. All the measurements were made at the center with each specimen coming in contact against a white background with the eyelet at a radius of 3 mm. Three readings were taken for each specimen at the top surface. The average CIE lab reading was then computed and recorded. Three measurements were recorded for each specimen and the average value obtained. The color change values (ΔE^*) between baseline and different staining intervals (after one day and seven days) were calculated from the mean ΔL^* , Δa^* and Δb^* values for each specimen with using the following formula:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

L^* refers to the lightness coordinate with values ranging from zero (black) to 100 (white) whereas a^* and b^* are chromaticity coordinates in the red-green axis and the yellow-blue axis respectively. Positive a^* values indicate a shift to red and negative values indicate a shift to green. Similarly, positive b^* values indicate the yellow color range and negative values indicate the blue color range.

The whitening indexes (WI) were calculated using the following formula:
 $WI = 0.551 * L - 2.324 * a - 1.1 * b$.

Knoop microhardness

A microhardness tester (FM-7000, FUTURE-TECH CORP., Kawazaki, Japan) with a Knoop diamond indenter was used to apply a static load of 50 g (0.98 N) for 15 s to each composite surface. For each specimen, the averages of five indentations at each depth were used in the statistical analysis.

Surface Roughness

For analyzing the surface roughness (Ra) of the specimens, the arithmetic mean (Ra- average surface roughness) was assessed with a digital profilometer (Surftest SJ-410; Mitutoyo Corp, Tokyo, Japan). For measuring the roughness profile value in micrometers (μm), a diamond stylus with 5 μm tip radius was moved across the surface under a constant load of 4 mN with a speed of 0.25 mm/s and a range of 0.8mm. Three traces were recorded for each specimen at three different positions (parallel, perpendicular, and oblique), resulting in nine tracings per specimen. The mean surface roughness was then calculated for each specimen.

Statistical analysis

Per each composite, data of ΔE , WI and opacity were individually submitted to two-way repeated measure ANOVA. The factors analyzed were “polishing procedure” and “assessment time”, which was defined as repetition. Multiple-comparisons were performed by Tukey’s test. The significance level was set at $\alpha = 0.05$ for all analyses.

Results

Color change

The behavior of each color parameter (L^* , a^* , and b^*) during the experiment is presented in figure 1. Parameter L^* decreased for all resin composites in different polish procedure during immersion in coffee, unlike the Parameter a^* , that increased. Parameter b^* constantly increases from baseline up to 24 hours of immersion, after this period tends to keep constant with slight increase in some groups.

Results of ΔE are presented in figure 2. For Z250, there is not a statistically significant difference ($P = 0.349$) for polishing procedure. There is a statistically significant difference ($P = <0.001$) for assessment time. ΔE was significantly higher after seven days. There is not a statistically significant interaction between polishing procedure and assessment time ($P = 0.066$). For Z350, there is a statistically significant interaction between polishing procedure and assessment time ($P = 0.007$). No-polished CT showed higher ΔE after 24 hours and 7 days. For assessment time, 7 days showed higher ΔE than 24 hours. For Bulk Fill, after one day there is a statistically significant difference ($P = <0.001$) for polishing procedure. DP Polished after 24 hours showed higher ΔE and IP polished immediately lower ΔE . After seven days, there is a statistically significant difference ($P = <0.001$) for polishing procedure. Polished after 24 hours DP and no-polished CT showed higher ΔE . There is not a statistically significant interaction between polishing procedure and assessment time ($P = 0.426$).

To facilitate the visualization of color changes, the values of L^* , a^* , and b^* were converted to an RGB (red, green, and blue) system, and colored cylinders were drawn using the software CorelDraw Graphics Suite X8 (Corel Corporation, Ottawa, ON, Canada) in the RGB model (Figure 3).

The mean values and standard deviations of whitening index and opacity for each resin composite and polishing procedure are summarized in Table 2.

Two Way Repeated Measures ANOVA showed a significant difference of the whitening index (WI) for Z250, Z350 and Bulk Fill. Z250 showed a statistically significant interaction between polishing procedure and different assessment times ($P = 0.049$). The lower values were observed for IP immediately polish. For assessment times, 7 days of immersion had the worst values. For Z350, there is a statistically significant interaction between polishing procedure and different assessment times ($P = <0.001$). WI reduced progressively after staining in coffee (after 24 hours, and 7 days), respectively for all polishing procedures. DP polishing after 24h have the higher WI after staining in coffee. For bulk fill, there is a statistically significant interaction between polishing procedure and different

assessment times ($P = <0.001$). WI was not presented difference after staining in coffee. Polishing after 24h have the worst WI after staining in coffee.

In the opacity results, for Z250, two Way Repeated Measures ANOVA showed that there is not a statistically significant difference ($P = 0.119$) for factor assessment times but, there is a statistically significant difference ($P = <0.001$) for polish procedure. Baseline data had the lowest values. There is not a statistically significant interaction between the factors ($P = 0.052$). For z350, two Way Repeated Measures ANOVA showed that there is not a statistically significant difference ($P = 0.826$) for factor assessment times but, there is a statistically significant difference ($P = <0.001$) for polish procedure. Staining in coffee for 7 days had the Higher values of opacity, and baseline data had the lowest values. There is not a statistically significant interaction between the factors ($P = 0.091$). For Bulk fill, two Way Repeated Measures ANOVA showed that there is a statistically significant interaction between polished procedure and assessment times ($P = 0.002$). CT no-polished and IP immediately polish had the lowest values in baseline data.

Microhardness and Surface roughness

The mean values and standard deviations of Knoop hardness (KNH) and surface roughness parameter Ra (mm) for each resin composite and polishing procedure are summarized in Table 3.

Two-way ANOVA showed a significant difference of the KHN among the polishing procedure ($P = 0.017$) and resin composite ($P = <0.001$). Also, there wasn't a significant interaction between these two independent variables ($P = 0.105$). The hardest surface was observed in z350 and independent of polishing procedure. Bulk-fill showed the lowest values. Two-way ANOVA showed a statistically significant interaction between Polishing and Resin composite ($P = 0.001$) for Ra roughness parameter. The smoothest surface (Ra) was observed in z350 and Definite when they were polished After 24h DP (0.16 mm) and CT no polished (0.17 mm) and for bulk fill when they were polished After 24h DP (0.14 mm).

Discussion

The polishing time procedures should be carried out remains controversial. Despite of studies recommended delayed for 24 hours or more for polishing in order to reduce microleakage because of the hygroscopic expansion of the material (14) and reduce the flow of the composites due to thermal insults of polishing (15), several professionals also does of the finishing and polishing were completed immediately after curing of the restorative materials, since this procedure reduces the number of clinical sessions and the wellness of the patients. The concern about the possible detrimental effects of immediate finishing / polishing procedures on distinct restorative composites are available in this study. The null hypothesis was rejected since immediate or delay polishing showed significantly difference on color stability, microhardness and roughness of composites resins after immersion in coffee solution for one day and seven days compare to no polish.

Even though acceptable survival rates were achieved, the replacement of failing restorations is still a relevant issue. A successful direct composite resin restoration should not only demonstrate high strength and durability, but also should be esthetically pleasing. Staining or discoloration is one of the primary reasons for replacement of composite restorations (16, 17). In the present study the effect of coffee staining on the color stability of bulk-fill and conventional composite resins was evaluated over a period of 7 days. Coffee was used as the storage media because of its frequent consumption in daily life and have contact with resin composite restorations in the oral environment.

A smooth and glossy surface is generally obtained under a Mylar strip without subsequent finishing or polishing (18), but in clinical practice, some functional and esthetics adjustments are necessary in nearly all restorations. In this study, finishing was carried out with standardized 600-grit silicon carbide paper under running water to simulate the texture of a fine diamond bur (19), therefore producing specimens with similar surface characteristics before applying the tested polishing procedures/conditions. The system used for finishing and polishing also should be taken into account. In this study the all

polishing protocols use sequential silicon impregnated rubber discs and brush silicon carbide-coated.

The CIELAB colorimetric system was used to evaluate color differences. The ΔE value presents relative color differences of dental materials or tooth surfaces before and after an intervention. Several studies have reported that the color difference ΔE values ranging from 1 to 3 are perceptible to the naked eye and ΔE values greater than 3.3 are clinically unacceptable (20, 21).

The color stability of the composite resin was influenced by the polishing procedure for nanofill and bulk fill. The polishing protocols showed a significantly lower color change after immersion in coffee solution for one day and seven days without polishing for nanofill. The color stability of a resin composite is related to the discoloration of the resinous material itself in the matrix / matrix interface and fillers (21), depth of polymerization, adsorption / absorption of coloring agents such as coffee (10, 22). In addition, polishing procedures can influence the quality of the composite surface and may therefore be related to the discoloration of resins (23). Roughness surfaces have been shown to retain surface stains more mechanically than smooth surfaces. This finding is in line with several earlier studies which have reported that higher smooth surfaces produce less discoloration of the resins (23).

Microhybrid resin composite was exhibited the smaller color change after staining. This can be justified because although we used same shade A1 for all composite resins, a nanocomposite in enamel shade and bulk fill which may show higher translucency (8, 24, 25), compared to a microhybrid resin-based composite in universal color, and this probably increased the color change values (26). Other factor that can be significant impact on the color stability, is the differences in composites resins and the characteristics of filler particle. Bulk fill resins consist of a variety of fillers, prepolymer shrinkage stress reliever, different photoinitiator system, and light sensitivity system. These differences might influence the staining susceptibility (7-9). However, after 1 or 7-days immersion in coffee solution, all the resin composites tested had perceptible color changes and were clinically unacceptable ($\Delta E \geq 3.3$). When specimens

were submitted to coffee staining, discoloration occurred due to the adsorption and absorption of pigments into the organic phase of resin-based materials (9). Additionally, coffee contains significant amounts of staining agents such as gallic acid, which facilitate staining (27).

The surface roughness of a restoration is important for patient's comfort, esthetics, plaque retention and staining (13, 28). Surface roughness refers to the finer irregularities of the surface texture that usually result from the action of the production process or the material's characteristics (25, 28). This study showed that delay polishing significantly decreased the roughness for nanofill and bulk fill resins compare to no polish. It has been reported that the roughness values above 0.2 mm may increase the plaque accumulation and the risks of secondary caries and periodontal problems (29). According to this study immediately or delay polishing significantly decreased average surface roughness below the 0.2- μm threshold, except for microhybrid resin. Another study reported that a change of surface roughness in the order of 0.3 μm can be detected by the tip of the patient's tongue (30). According results of this study, the experimental group submitted to no polishing showed an average surface roughness above the 0.3- μm threshold, unsatisfactory surface roughness. Nanofill composite resin had smoother surface under all conditions of polishing timings than microhybrid composite. A possible explanation is the difference in the filler particles and matrix composition may influence the performance of dental composites (31). It has been suggested that resin composites with smaller particle sizes obtain a higher gloss and lower surface roughness after finishing and polishing (32).

Hardness is a mechanical property that may determined by the degree of cure of restorative materials (33). This property predicts the wear resistance of a material and the ability to abraded by opposing dental structures or materials. The timing of the polishing procedure might have an effect on the physical properties of the restorative materials, and might increase the risk of premature failures (34). In this experiment, all the evaluations were conducted after one- or seven-days storage in coffee solution for all specimens. In this way, the maturity of the composites was common at the evaluation time (32), and any differences

in hardness could be attributed to the effects of the polishing procedures at both intervals or type of resin composite.

The findings of this research disclosed that, immediate or delayed polishing influenced microhardness of the tested composite resins, significantly increasing these values. The superficial layer is essentially composed by organic matrix, being hence, less dense than the underlying layer. Thus, the removal of this layer by polishing procedures increases the surface resistance (35).

Conclusion:

Under the limitations of this in vitro study, it might be concluded that:

The color change, surface roughness and hardness of the composite resins was affected by polishing procedures, type of composite, and the period of immersion in the staining agent.

Table 1. Types of composite resins and manufacturer information

Composite Resin	Type of Filler	Shade	Organic Matrix	Filler	Light Curing Time (Seconds)
Filtek Z250 (3M ESPE)	Microhybrid (Lot 1608800723)	A1	BisGMA, UDMA, and BisEMA	oxide, zircon/silica (0.01-3.5mm) 61% in vol., 78% in wt	40
Filtek Z350 XT	Nanofill (Lot 1615900296)	A1E	BisGMA, BisEMA, UDMA, TEGDMA	Silica and zirconia (clusters of 0.6-1.4 μ m— individual particle size of 5-20 nm); 59.5% in vol., 73.2% in wt	20
Filtek Bulk Fill (3M ESPE)	Nanofill (Lot 1415820296)	A1	Bis-GMA, Bis-EMA, UDMA, TEGDMA, Procrylat resins	Zirconia/silica, ytterbium trifluoride 42.5%	20

Table 2. Means (standard deviation) of whitening index and opacity measured for each composite in different assessment times.

Composite	Polishing	Whitening index			Opacity (%)		
		Baseline	24h in coffee	7 days in coffee	Baseline	24h in coffee	7 days in coffee
Z250	CT: Absent	28.8 (0.2) ^{ABa}	19.4 (1.6) ^{Ab}	15.7 (2.2) ^{Ac}	86.7 (2.1) ^{Ba}	87.9 (1.0) ^{Aa}	89.9 (1.4) ^{Aa}
	IP: Immediately	25.0 (1.6) ^{Ba}	14.4 (4.0) ^{Bb}	9.5 (6.2) ^{Bc}	84.7 (1.3) ^{Ba}	88.7 (0.8) ^{Aa}	89.7 (0.5) ^{Aa}
	DP: After 24h	30.1 (0.8) ^{Aa}	23.7 (0.9) ^{Ab}	14.1 (2.0) ^{Ac}	83.2 (1.9) ^{Ba}	87.9 (3.2) ^{Aa}	87.7 (1.8) ^{Aa}
Z350	CT: Absent	34.3 (0.8) ^{Aa}	16.9 (3.0) ^{Bb}	11.2 (3.7) ^{Cc}	78.0 (0.7) ^{Ca}	83.5 (1.2) ^{Ba}	87.8 (2.4) ^{Aa}
	IP: Immediately	32.3 (0.7) ^{Aa}	18.9 (1.6) ^{Bb}	14.4 (1.4) ^{Bc}	79.3 (1.7) ^{Ca}	83.5 (1.0) ^{Ba}	87.7 (1.1) ^{Aa}
	DP: After 24h	33.3 (0.7) ^{Aa}	23.8 (1.3) ^{Ab}	18.0 (1.7) ^{Ac}	79.0 (2.5) ^{Ca}	82.2 (2.6) ^{Ba}	87.3 (1.4) ^{Aa}
Bulk-Fill	CT: Absent	33.7 (0.9) ^{Aa}	16.4 (1.5) ^{Aa}	7.9 (0.6) ^{Ba}	74.0 (1.1) ^{Bb}	79.3 (1.7) ^{Aa}	80.8 (2.1) ^{Aa}
	IP: Immediately	30.5 (1.1) ^{Ba}	19.1 (1.9) ^{Aa}	12.2 (2.6) ^{Aa}	74.0 (1.1) ^{Bb}	79.2 (1.6) ^{Aa}	78.3 (2.5) ^{Aa}
	DP: After 24h	33.7 (1.1) ^{Aa}	13.1 (1.8) ^{Ba}	7.7 (3.9) ^{Ba}	78.2 (1.8) ^{Ab}	79.3 (1.1) ^{Aab}	80.9 (2.9) ^{Aa}

For each composite, distinct letters (uppercase comparing column, lower comparing row) indicate statistical difference at Tukey's test ($p < 0.05$).

Table 3. Means (standard deviation) of hardness and surface roughness measured for each composite according to the polishing procedures.

Composite	Hardness (KHN) according to the polishing procedure			Surface roughness- RA (μm) according to the polishing procedure		
	CT Absent	IP Immediately	DP After 24h	CT Absent	IP Immediately	DP After 24h
Z250	60.5 (2.9) Bb	62.2 (3.0) ^{Bab}	67.9 (8.9) ^{Ba}	0.58 (0.15) ^{Aa}	0.20 (0.08) ^{Ab}	0.35 (0.15) ^{Aab}
Z350	81.9 (2.6) Ab	82.4 (10.9) Aab	82.4 (7.7) ^{Aa}	0.17 (0.02) ^{Ba}	0.15 (0.04) ^{Aa}	0.16 (0.02) ^{Ba}
Bulk-Fill	41.3 (5.2) Cb	56.2 (5.5) ^{Cab}	55.5 (10.4) ^{Ca}	0.40 (0.16) ^{Aa}	0.17 (0.03) ^{Ab}	0.14 (0.03) ^{Bb}

For each composite, distinct letters (uppercase comparing column, lower comparing row) indicate statistical difference at Tukey's test ($p < 0.05$).

Figure 1. The behavior of color parameters (L^* , a^* , and b^*) according to composite, polishing procedure and assessment time. Parameter L^* : white to the black axis. (b) Parameter a^* : red to the green axis. (c) Parameter b^* : yellow to the blue axis.

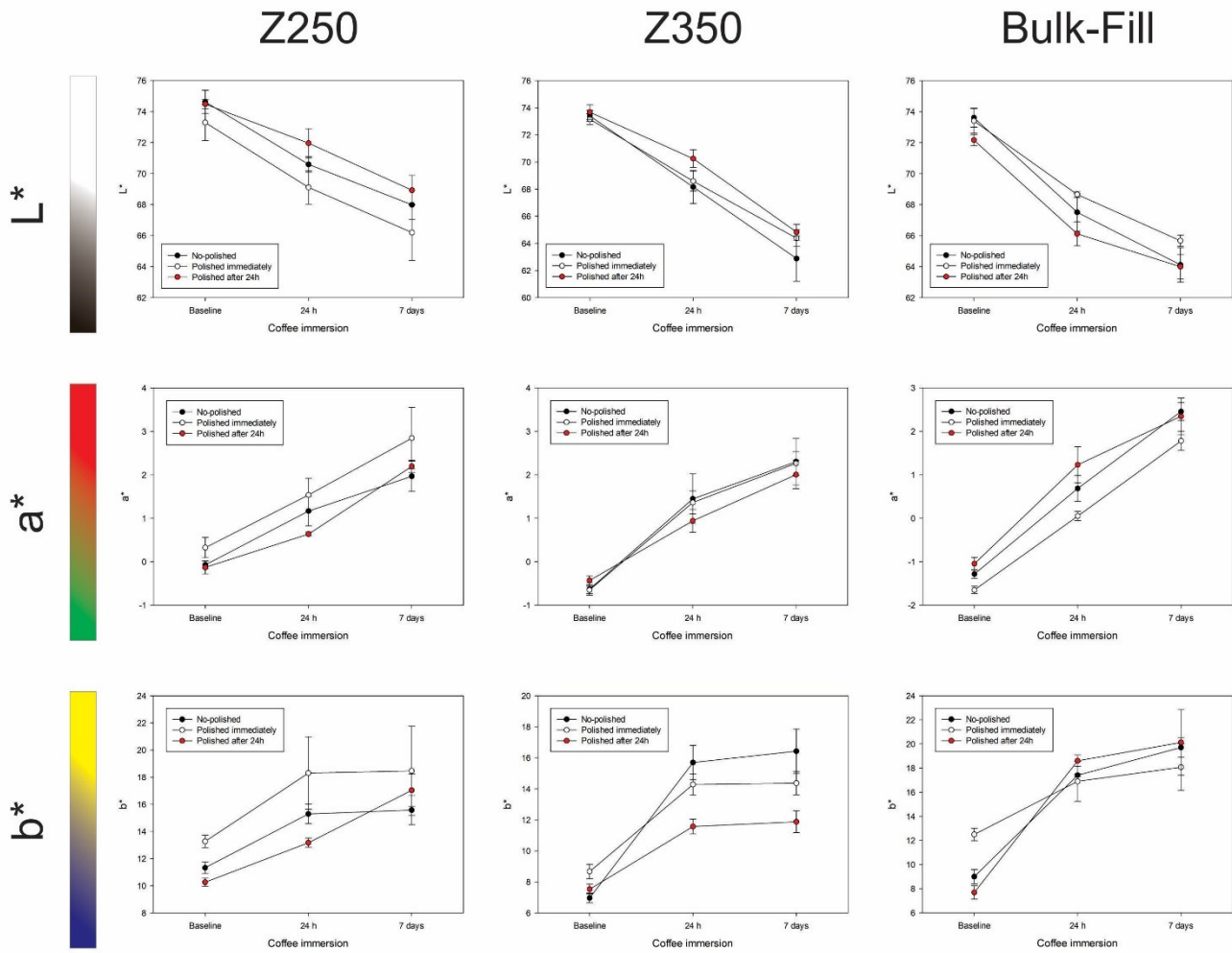


Figure 2. Bar plot presenting the results of ΔE according to composite, polishing procedure and assessment time. For each composite, distinct letters (uppercase comparing polishing procedures, lower comparing assessment times) indicate statistical difference at Tukey's test ($p < 0.05$).

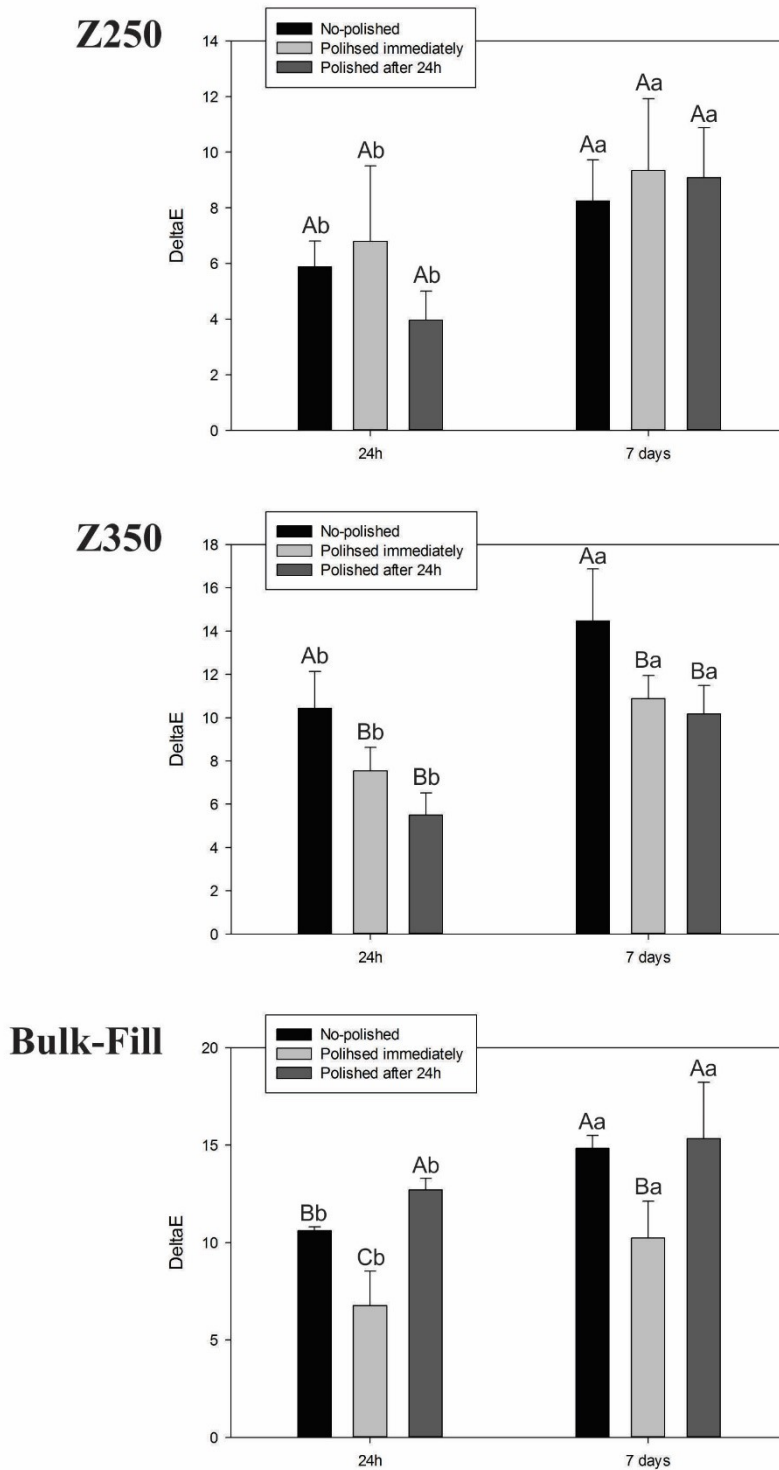
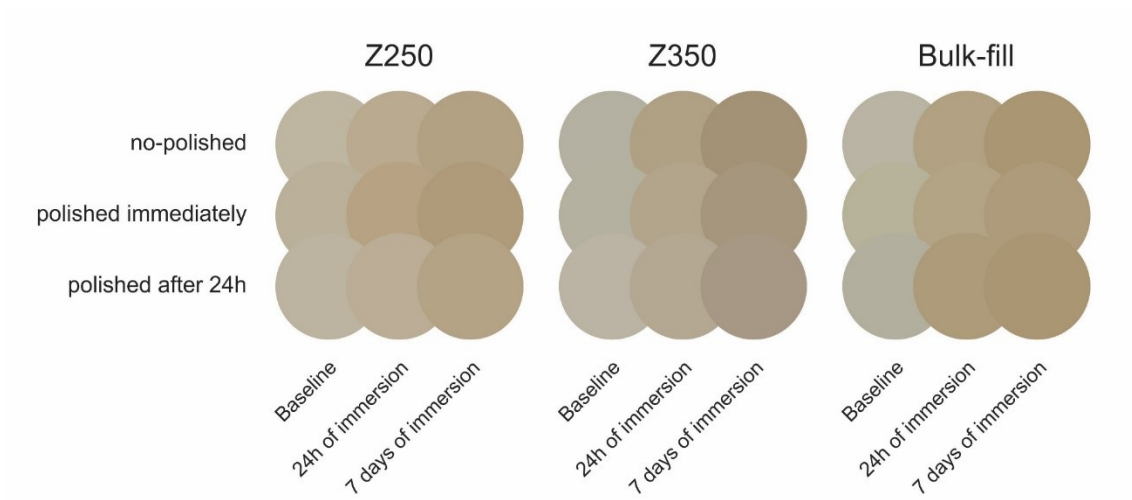


Figure 3. Illustrative cylinder-shaped composite specimens drawn based on data from L*a*b* converted to RGB system demonstrating the color changes of specimens during the experiment.



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Capítulo 3

3.3. Capítulo 3: Artigo será submetido para o periódico Journal of Esthetic and Restorative Dentistry.

Title: Effect of light curing units on Degree of Conversion, microhardness and Mechanical properties of a bulk fill composite

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"The authors do not have any financial interest in the companies whose materials are included in this article."

Abstract

Objectives: Evaluated mechanical and physical properties of bulk-fill composites (RBC) polymerized with different light-curing units (LCUs).

Materials and Methods: Composite discs (2x4 mm thick) were produced with Tetric N-Ceram Bulk-Fill (TNB), Filtek Bulk-Fill (FBF) and Opus Bulk-Fill (OBF), divided into 5 groups (n=5) according to LCUs: polywave LED Bluephase or VALO, monowave LED Radical or Emitter C and quartz–tungsten– halogen Optilux. Degree of conversion (DC) was determined using Fourier transform infrared spectrometer, microhardness (KHN) was measured with a Knoop indenter and mechanical properties with tensile strength diametral (DTS). Data were analyzed using two-way ANOVA and Tukey's post hoc test ($\alpha=0.05$).

Results: Regardless of light-curing, OBF has the highest DC and KHN. Bluephase showed higher DC, with no significant difference for Optilux. For KHN, there was significant difference for factors ($p < 0.05$), as well as for interactions of factors ($p= 0.011$). Regardless of LCU, Opus no presented difference. TNB showed higher KHN for Bluephase and Optilux. FBF showed lower KHN for Radical. Bluephase, Valo and Optilux showed highest DTS and there was no significant difference among the different RBC.

Conclusions: The bulk-fill composites exhibited influenced in DC, KHN and DTS between different curing lights evaluated.

Keywords: composite resins, polymerization, Fourier-transform infrared spectroscopy, hardness, Curing Lights Dental;

CLINICAL SIGNIFICANCE:

The degree of conversion, microhardness and mechanical properties of bulk-fill composites are influenced by light-curing units used in dental practice. Polywave LEDs or quartz-halogen-tungsten are more effective to RBC photopolymerization.

Introduction:

The clinical use of resin-based composite (RBC) has increased substantially in recent years due to increased aesthetic demands of patients, improvements in the formulation and simplification of bonding procedures (1). In an attempt to accelerate the restoration process in posterior restorations, bulk fill composites were introduced into the dental market. This category currently gains popularity due to the simplification and reduction of application time, making the clinical procedure more friendly (2). Unlike conventional RBC that are layered to a maximum thickness of 2 mm (3), bulk fill RBC can be placed up to 4 mm thick with suitable polymerization and low polymerization shrinkage (4) (5).

Polymerization effectiveness of light-activated materials is related by absorbed by photoinitiators and free radicals are formed in the presence of activators. Free radicals subsequently trigger the polymerization reaction. Ideally, during the polymerization reaction all the monomer in the resin composite material would have been converted to polymer (6). The adequate conversion of RBC materials is essential in determining their physical and mechanical performance (7, 8).

Current bulk fill RBC utilize a variety of compositional modifications to achieve greater depth of cure, lower shrinkage to allow such bulk placement, that may also change the polymerization kinetics. The increased translucency of these resins allows greater light transmission and adequate cure depth (9). Moreover, addition of stress relieving monomers, specific polymerization modulators, more reactive photoinitiators, and prepolymerized particles results in less polymerization contraction (10, 11). The main alpha diketone initiators used in bulk fill is camphorquinone (CQ), phenylpropanedione (PPD), acyl phosphine oxide (APO) and germanium-based compounds such as bis-(4-ethoxybenzoyl) diethyl-germane (Ivocerin). Where the polymerization process is usually activated by applying visible light. The activation of these photoinitiators is dependent upon the appropriate dental light curing unit (LCU) irradiating at wavelengths that overlap the absorption of the photoinitiator (12).

Two common types of dental LCUs are quartz-halogen-tungsten (QHT) and high-intensity light-emitting diode (LED). Quartz–tungsten–halogen (QTH) curing unit has been extensively used for a long time. Their wide range of wavelengths (of light between 370 nm and 550 nm) allows for the curing of composites employing CQ, PPD and APO as photoinitiators (12, 13). However, light-emitting diode (LED) is becoming popular in dental practice, because device shows lower degradation over time, do not require cooling fans, consume less energy, have extended lifetimes without significant loss of light intensity and have blue light emission without requiring filter. The output from conventional single peak LED units, is designed to activate the CQ photoinitiator (12, 14). Both first- and second-generation LED lights used only one type of LED (monowave [single-peak] technology) and were unable to cure composites with PPD and APO initiator systems. Some third generation broad-spectrum LED units include additional LED emitters that produce light at these lower wavelengths, which are in the ‘violet’ range, to make these LCUs compatible with a wider range of photoinitiators and avoid wavelength-compatibility issues by deploying polywave (dual/multi-peak) technology (14). The clinicians also need to know the emission spectrum of the light emitted from the LCU, so that they can match the light to the resin composite they are using (15).

Therefore, the objective of this study was to compare the effectiveness of cure of three bulk-fill composites with polywave LEDs, monowave LEDs, and conventional halogen curing lights using degree of conversion (DC), Knoop microhardness (KHN) and diametral tensile strength (DTS) testing. The null hypothesis that no difference in the effectiveness of cure existed between the polywave LED, monowave LED, and QHT curing lights for different bulk-fill composites if the total light energy was kept constant.

Material and Methods:

Three high-viscosity bulk-fill resin-based composites were investigated: Tetric N-Ceram Bulk Fill (TNB) containing the initiators CQ and Ivocerin; Filtek Bulk Fill (FBF) containing the initiators CQ and Opus Bulk Fill (OBF) containing

the initiators CQ and APS (advanced polymerization system). The compositions, lot numbers, and manufacturer information for these products are presented in Table 1.

Composite discs (n=5) of each material were prepared and polymerized with five different curing lights were fabricated. The composites were placed in a single increment into cylindrical silicone mold (Contrast, VOCO, Cuxhaven, Germany); with 2-mm height and 4-mm diameter. A transparent mylar strip was placed on the top of the mold, and a glass slide (1-mm thick) were used to squeeze out the excess of the materials. The composites were then irradiated through the top glass slide using either a polywave LED (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) or (VALO Cordless Ultradent, South Jordan, UT, United States), a monowave LED (Radii cal, SDI, Basywater, Victoria, Australia) or (Emitter C, Schuster, Santa Maria, RS, Brazil) and QHT (Optilux 501, Kerr, Orange, CA, USA). Table 2 provides details of the LCUs used in the study. Each specimen was irradiated once for the length of time recommended by the manufacturer according to LCUs (table 1 and 2). Light emittance was measured daily using a radiometer (Kondortech Equipamentos Odontológicos, São Carlos, SP, Brasil) to ensure consistent irradiance.

Degree of conversion

The DC was analyzed using attenuated total reflectance/ Fourier transform infrared spectroscopy (Vertex 70, BrukerOptik GmbH, Germany). The number of remaining carbon double bonds was determined. The remaining unconverted carbon double bonds were calculated by comparing the percentage of aliphatic C=C (vinyl) (1638 cm^{-1}) and aromatic C=C absorption (1608 cm^{-1}) between cured and uncured specimens. The spectra of the cured and uncured specimens were obtained using 32 times over a range of 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} . The spectra were subtracted from the background spectra using software provided with the FTIR unit. The acquired spectra were expanded and analyzed in the region of interest from 1560 to 1670 cm^{-1} . The degree of conversion was calculated using the standard baseline technique and a comparison of peak area at 1639 cm^{-1} (aliphatic

C=C) and internal standard peak at 1609 cm⁻¹ (aromatic C=C). Then, DC was calculated by the following equation:

$$\text{DC (\%)} = (1 - [\text{Cured aliphatic /aromatic ratio} / \text{Uncured aliphatic=aromatic ratio}]) \times 100.$$

Knoop microhardness

After storage for 24 hours, at 37°C in distilled water, the same specimen locations analyzed using Fourier transform infra-red spectroscopy were also used to determine KHN values. A microhardness tester (Microhardness tester Future-Tech FM-700, Instytut Metalurgii Żelaza, Gliwice, Poland) with a Knoop diamond indenter was used to apply a static load of 100 g (0.98 N) for 10 s to each composite surface. For each specimen, the averages of three indentations at each depth were used in the statistical analysis.

Diametral Tensile Strength

A diametral tensile strength test was performed in the specimens after used for obtaining DC and KHN (n=5) using a mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, Brazil). Specimens were positioned vertically on the testing machine between a stainless-steel flat tip and base; a compressive load was applied vertically on the lateral portion of the cylinder at a crosshead speed of 0.5 mm/min, producing tensile stresses perpendicular to the vertical plane passing through the center of the specimen until failure. After each compressive test, the fracture load (F) was recorded in Newtons (N), and the diametral tensile strength (σ_t) was calculated (MPa) as follows: $\sigma_t = 2F / \pi dh$ where, d is the diameter (4 mm), h the height (2 mm) of specimens, and the constant π is 3.1416.

Statistical analysis

The statistical analysis was performed using the SigmaPlot (Sigma plot Verson 12.0, Systat Software Inc., San Jose, CA, USA). The data were normal and homoskedastic for all experiments. Two-way ANOVA/Tukey's test was performed to evaluate degree of conversion, Knoop microhardness and diametral tensile strength data (factors: Bulk-fill Resin Composites and dental

light curing unit). A 95% level of significance was adopted ($\alpha = 0.05$) for all tests.

Results:

Three bulk fill RBCs and five dental LCU were evaluated to determine the effect on the DC, DTS, KHN. Means and standard deviations for DC are presented in Table 3. In terms of LCUs polymerization ($P = <0.001$), Bluephase showed higher DC, with no significant difference for Optilux. Valo resulted in intermediate values that were not different from the Optilux. Radicall and Emittera showed lower DC. When the restorative materials tested, the OBF and TNB groups had the highest DC values and the FBF group had the lowest ($P = <0.001$). There is not a statistically significant interaction between bulk fill RBC and LCU ($P = 0.070$).

The KHN results are presented in Table 4. ANOVA showed significant difference for factors ($p < 0.05$), as well as for interactions of factors ($p = 0.011$). The LCUs showed no difference for the OPUS composite. TNB showed higher KHN for Bluephase and Optilux compared to Radicall, Valo and Emittera. FBF showed higher KHN for Bluephase, with significant difference only for Radicall. Bulk-fill composite resins not present significant difference when polymerized with Bluephase and Optilux. The TNB composite showed lower KHN for Valo than other RBCs. The OPUS present high KHN for Radicall and Emittera.

The DTS results are presented in Table 5. For DTS analysis, Bluephase showed higher DTS, with no significant difference for Valo and Optilux. Radicall and Emittera showed lower DTS, with no significant difference for Optilux ($P = 0.023$). There is not a statistically significant difference among the different levels of bulk fill RBC ($P = 0.146$). There is not a statistically significant interaction between bulk fill RBC and LCU ($P = 0.301$).

Discussion:

Since their introduction into the dental market, LCUs have been regularly improved by manufacturers to provide better and faster polymerization. The effectiveness of cure of bulk-fill composites with polywave LED, monowave

LED, and conventional halogen curing lights was evaluated. According to the results of the present study, the null hypothesis was rejected as significant differences in effectiveness of cure existed between the different lights and bulk fill resin composites.

Adequate photopolymerization of resin-based composites is a crucial factor for optimization of mechanical properties, biocompatibility and clinical longevity of composite restorations (16, 17). Visible light-activated resins initiate the polymerization process through light absorption by a photoinitiator, which, once activated, reacts as a reducing agent to produce free radicals. From that point on, there is the polymerization of the methacrylic monomers that form a polymeric matrix with cross links (6). Curing efficacy can be assessed by several techniques. FTIR spectroscopy is a direct method used to analyze the chemical bonds of polymers. This technique allows detection of the amount of unreacted C=C in the resin matrix (18), but this property alone is not enough to characterize all the resinous material structure (18, 19). Hardness measurement is an indirect method that has proven to be the best indicator of the extent of polymerization and has been classified as a high level of evidence. It has been reported in many studies that, resin-based filling materials should exhibit a minimum of 80% bottom/top hardness percentage when cured in order to be considered as adequately polymerized (20).

Several parameters can affect the degree of polymerization of bulk fill RBCs involve composition (photoinitiators, type of resin-matrix, filler type, size and loading), viscosity of material, thickness and opacity (21). In the present study, both composites are regular materials with modified monomers and have relatively similar loading % by volume of filler content. The thickness controlled during the experiment, since all specimen have the same dimension (2 x 4 mm). All LCU have the hability to give superior degree of conversion and hardness of OPS. This result can be explained by their photoinitiators APS (APS: Advanced Polymerization System). According to the manufacturer, APS has a small concentration of canphorquinone and an addition of other initiators / co-initiators. This system can potentiate the energy coming from the LCU, because the photoinitiators interact with each other and release more free radicals by

increasing the polymerization capacity. The bulk-fill composite TNB also had a high degree of conversion. This RBC has the photoinitiator Ivocerin, a derivative of dibenzoyl germanium, in addition to the camphorquinone/amine initiator system. Ivocerin is excited by shortwave visible light (380–450 nm) and is a more efficient free-radical generator than camphorquinone, leading to polymerization and monomer conversion. For microhardness, Bluephase showed more efficient for excited Ivocerin than the others LCU. Filteck Bulk Fill Posterior containing the initiator CQ, that have good hardness for Bluephase, Valo, Optilux and Emitter. Hardness testing was done 24 hours after photopolymerization to allow for composite postcure.

Curing efficacy can be affect also by light-curing constraints, include light type, total light energy, intensity, spectral wavelengths, exposure time, curing distance and shape of the different curing tips. This study used laboratory conditions for light-curing, in which no distance remained between the tip of the light source and the restorative material. So, this variable including curing distance and exposure time were controlled during the experiment. The results can be attributed to light type. The results showed that DC and KHN of Bluephase and Optilux was higher than the others LCUs, regardless of bulk fill RBC. The harder surface associated with halogen light could be contributed in part to a thermal effect. Studys reported the heating of composites from halogen lights has been increase hardness (22, 23) . Moreover, LCUs do not merely emit total energy to bulk structure of restorative material for photoinitiator activation. Uniform distribution of emitted energy in all layers of restorative material has been reported as crucially important to produce sufficient numbers of free radicals for adequate polymerization (24).

Time-saving procedures are an ongoing demand for restorative applications. A thirdgeneration LEDs curing units were marketed that claims to reach high irradiances and allow for shorter clinical application times. In this study, Bluephase and Valo were used in hight polywave mode. The results of Valo were significantly lower than Bluephase in all tests. Although high irradiance may be interpreted as a shorter curing time from a total energy density concept, the results showed that DC and KNH decreases with short

exposure time at high radiant exitance. This result supports the concern of Peutzfeldt and Asmussen that have also pointed out that the degree of cure increases with increasing radiant exposure, but decreases with short exposure time at high radiant exitance, despite achieving the same energy density (25). Short exposure time at high radiant exitance may result in the formation of short chains, which contain fewer cross links (26).

According to AG Pereira et al 2016, the battery level of the cordless LED unit affected the battery voltage and light intensity of the equipment in addition to the degree of conversion and mechanical properties of resin composite. Low battery levels affect the battery voltage and consequently influence the light intensity of cordless LED units, also changing some properties of composite resins. In this study, Valo, Radicall and Emittera are cordless LED unit, but before specimen preparations, the maximum number of cycles that could be completed with the fully charged batteries (100%) was determined (27).

This study used laboratory conditions for light-curing. The situation may be different and worse in clinical situations, when the distance between the light source and the polymeric restorative material is increased by limiting factors, such as in the restoration of deep cavities, teeth position and morphology of fissures and cusps, which decreases irradiance and may impair polymerization efficacy. The bulk fill RBCs, usually applied in deep posterior cavities. A recent study suggest that, the operator visibility and access was the worst in the posterior region, this fact affected negatively the irradiance for 22 Contemporary Light Curing Units (28). The intensity and DC decrease with increasing distance, between the curing tip and the material surface to be irradiated (29).

Bulk-fill composite materials are likely to fulfil some requirements, low polymerization shrinkage, ease of use, improved depth of cure and enhanced physical characteristics. The latter is particularly important since bulk-fill composites will represent all of the restoration. According to the present work significant differences ($p < 0.05$) also were observed for considered mechanical properties within the bulk-fill composite category as different LCU. The mechanical properties of the bulk-fill composites are mostly lower when

monowave LEDs were used. This results can be explain because these LCUs were unable to cure some photoinitiator like PPD and APO initiator systems present in RBCs (14).

Conclusions:

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

Degree of conversion and microhardness values are affected by the different LCU and type of bulk fill resin composite. Photopolymerization with polywave LED (Bluephase G2 and VALO) and QHT (Optilux 501) may be more effective for mechanical properties of bulk-fill composites evaluated.

Table 1: Technical Profiles of Bulk-Fill Composites Evaluated

Material	Abbreviation	Shade/ Batch Number	Composition	Filler Loading (% by Volume)	Recommended Curing Time and Light Intensity	Manufacturer
Tetric N- Ceram Bulk Fill	TNB	A2/ Lot	Resin: dimethacrylates Filler: barium glass, ytterbium trifluoride, mixed oxide and copolymers	80%	20 s for ≥ 500 mW/ cm ² or 10 s for \geq 1000 mW/cm ²	Ivoclar Vivadent, Schaan, Liechtenstein
Filtek Bulk Fill Posterior	FBF	A2	Bis-GMA, Bis EMA, UDMA, TEGDMA, Procrylat resins Filler: Zirconia/silica, ytterbium trifluoride	76.5%	40 s	3M ESPE, St Paul, MN, USA
Opus Bulk Fill	OBF	A2	Urethane- dimetacrylic monomers Filler: silicon dioxide (silica)	79%	40 s for ≤ 1000 mW/cm ² or 30 s \geq 1000 mW/cm ²	FGM, Joinvile, SC, Brazil

Table 2: Technical Profile of Curing Lights and Modes Evaluated			
LCU	Type	Irradiance*/Recommended Curing time*	Manufacturer
Bluephase N Polywave	LED 3rd generation Polywave	High mode: ~1200 mW/cm ² 10%, 20 s	Ivoclar Vivadent, Schaan, Liechtenstein
VALO	LED 3rd generation Polywave	High-power mode: ~1400 mW/cm ² 10%, 12 s	Ultradent Products Inc, South Jordan, UT, USA
Optilux 501	QTH	: ~600 mW/cm ²	Kerr, Orange, CA, USA
Radii cal	LED/monowave	: ~600 mW/cm ²	SDI, Basywater, Victoria, Australia
Emitter C	LED/monowave	: ~600 mW/cm ²	Schuster, Santa Maria, RS, Brazil

Material	LCU				
	Bluephase	Valo	Optilux	Radicall	Emitter
OBF	70.9 (4.59)Aa	68.84 (6.18) Ba	68.03 (4.80) ABa	60.42 (2.14) Ca	61.14 (2.50) Ca
TNB	70.49 (4.28) Aa	62.61 (2.67) Ba	67.49 (4.78) ABa	59.82 (2.20) Ca	60.90 (4.34) Ca
FBF	65.96 (3.51) Ab	63.32 (2.23) Bb	62.19 (4.53)ABb	58.35 (6.63) Cb	54.22 (8.76) Cb

Means followed by the same letter (uppercase compares columns, lowercase compares rows) are not statistically different ($p < 0.05$).

Material	LCU				
	Bluephase	Valo	Optilux	Radicall	Emitter
OBF	53.2 (2.19) Aa	51.9 (2.14) Aa	51.9 (2.16) Aa	51.0 (2.86) Aa	51.2 (2.22) Aa
TNB	54.0 (1.91) Aa	47.5 (0.58) Bb	51.4 (1.91) Aa	47.5 (0.84) Bb	47.5 (1.14) Bb
FBF	52.1 (1.84) Aa	51.6 (0.71) ABa	51.3 (1.18) ABa	48.7 (0.56) Bab	49.5 (1.78) ABab

Means followed by the same letter (uppercase compares columns, lowercase compares rows) are not statistically different ($p < 0.05$).

Table 5: Means (standard deviation) diametral tensile Strength (DTS), of composites at different LCU					
Material	LCU				
	Bluephase	Valo	Optilux	Radical	Emitter
OBF	38.35 (4.33) Aa	36.87 (5.74) ABa	32.49 (2.61) ABa	35.82 (5.44) Ba	33.3 (4.35) Ba
TNB	39.70 (6.49) Aa	29.4 (6.6) ABa	34.5 (3.61) ABa	28.78 (5.59) Ba	28.2 (4.92) Ba
FBF	37.67 (2.79) Aa	38.9 (7.49) ABa	32.9 (6.95) ABa	30.25 (4.55) Ba	34.87 (4.64) Ba

Means followed by the same letter (uppercase compares columns, lowercase compares rows) are not statistically different ($p < 0.05$).

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Considerações finais

4 – Considerações finais

Considerando as limitações deste estudo, pode-se concluir:

O protocolo de polimento utilizando sistemas de pontas abrasivas com sequencias decrescentes de granulometria pode ser uma boa alternativa para o polimento das superfícies de cerâmica vítrea à base de fluorapatita, após as etapas clínicas de ajuste.

O polimento imediato ou após 24 horas reduziu a rugosidade de superfície e alterou a cor de diferentes tipos de resina composta, quando armazenadas em café. Além disso, promove o aumento de dureza. No entanto, quando o polimento é negligenciado, as resinas compostas apresentam topografia de superfície mais rugosa e maior alteração de cor.

As resinas bulk fill apresentam comportamento mecânico dependente da unidade fotopolimerizadora. O grau de conversão, a microdureza e a resistência a tração diametral dessas diferentes resinas são influenciados pelas unidades de fotoativação utilizadas na prática odontológica.

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Anexos

6- Carta de aceite e normas dos periódicos:

Fwd: Brazilian Dental Journal - Decision on Manuscript ID BDJ-2018-2101.R2

11-Oct-2018

Dear Dr. Murilo

I am pleased to inform you that your manuscript BDJ-2018-2101.R2 has now been accepted for publication in the Brazilian Dental Journal

As informed in the Letter of Receipt (and agreed by you), all manuscripts accepted for publication undergo a Technical Review, which includes revision of language and scientific writing style, formatting corrections and technical editing to fit the manuscript into Journal's standards, and preparation of galley proofs. Additional modifications to the text, tables and/or images may have to be made at this moment. The Technical Review is part of the review process that precedes publication and the cost is charged to the authors (Technical Review Fee).

Please confirm receipt of this message and your agreement with these conditions by sending an email to bdj@forp.usp.br with copy to our Text & Technical Editor, Dr. Renata P. Ramos at renatapramos@gmail.com.

Thank you for your fine contribution.

On behalf of the Editors of the Brazilian Dental Journal, we look forward to your continued contributions to the Journal.

Sincerely,
Dr. Manoel Neto
Editor-in-Chief, Brazilian Dental Journal
sousanet@forp.usp.br

Capitulo 1- Brazilian Dental Journal

Form and preparation of manuscripts

**BRAZILIAN
DENTAL
JOURNAL**

THE FOLLOWING GUIDELINES MUST BE FOLLOWED CAREFULLY.

General

- The authors must submit the manuscript in Word and in PDF, comprising the title page, text, tables, figure captions and figures (photographs, micrographs, radiographs, schematic drawings, graphs, computer-generated images, etc).
- The manuscript must be typed in Times New Roman 12 font, with 1.5 spacing, 2.5-cm margins at each side. **DO NOT USE** bold letters, watermarks or other resources to make the text visually attractive.
- Pages should be numbered consecutively, starting with the summary.
- Full-length manuscripts are assembled in the following sections:
 - 1) Title Page
 - 2) Summary and Key Words
 - 3) Introduction; Material and Methods; Results; Discussion
 - 4) Summary in Portuguese (an item necessary for Latin American Indexing)

Services that will be provided for non-Brazilian authors by the Journal)

- 5) Acknowledgements (if any)
- 6) References
- 7) Tables
- 8) Figure captions
- 9) Figures

- All titles of sections (Introduction, Material and Methods, etc) must be capitalized in regular font type (not bold).
- Results and Discussion **MUST NOT** be joined in a single section.
- Short Communications and Case Reports should be divided into appropriate sections.
- Products, equipments and materials: the trade name must be followed by the manufacturer's name, city, state and country, within parentheses upon first mention. For further mentions, only the manufacturer's name is required.
- All abbreviations must be explained at first mention.

Title page

- The first page must contain the title of the manuscript, a short title (maximum of 40 characters, to be used as a running head), author(s) name(s) (no more than 6) and their Department(s), School(s) and/or University (s). **DO NOT INCLUDE** the author's titles (DDS, MSc, PhD, etc.) or position (Professor, Graduate student, etc.).
- Provide the name and **complete** address of the corresponding author (inform email, telephone and fax numbers).
- The title page must be uploaded at the website as a separate file (not included in the body of the manuscript).

Manuscript

- The first page of the manuscript must contain: title of the manuscript, short tile with no more than 40 characters, and NO authors' names or identification.

Summary

- The second page should contain a summary of no more than 250 words, stating the aims, methods, results, and any conclusions drawn from the study. Do not use topics and paragraphs and do not cite

- references in the Summary.
- A list of key words (no more than 5) should be included below the summary in lowercase letters, separated by commas.

Introduction

- Summarize the purpose of the study, giving only pertinent references. Do not review existing literature extensively. State clearly the working hypothesis.

Material and Methods

- Material and methods should be presented in sufficient detail to allow confirmation of the observations. **Indicate the statistical methods used, if applicable.**

Results

- Present the results in a logical sequence in the text, tables and figures, emphasizing the important information.
- Do not repeat in the text data contained in the tables and illustrations. The important observations should be emphasized.
- Do not repeat the same data in tables and figures.
- Describe the statistical data in this section.

Discussion

- Summarize the findings without repeating in detail the data given in the Results section.
- Relate your observations to other relevant studies and point out the implications of the findings and their limitations. Cite pertinent studies.
- Present your conclusions at the end of the Discussion, indicating how your study is pertinent and/or its clinical implications. Presentation of the conclusions in topics should be avoided.

Summary in Portuguese (for Brazilian authors only)

- The Summary in Portuguese should be **IDENTICAL** to the English version (Summary). **DO NOT INCLUDE** title and key words in Portuguese.

Acknowledgements

- Financial support by government agencies should be acknowledged. If appropriate, technical assistance or assistance from colleagues may be acknowledged.

References

- References must follow the Journal's style. Authors should refer to a current issue of the BDJ for guidance on reference citation and presentation of the reference list.
- References must be numbered consecutively in the text in order of citation, within parentheses, without space between numbers: (1), (3,5,8), (10-15). **DO NOT USE** superscript numbers.
- For papers with two authors, cite both authors in the text, as follows: Ex: "According to Santos **and** Silva (1)...". If there are more than 3 authors, cite only the first author and add "et al.". Ex: "Pécora et al. (2) reported that..."
- All authors of each paper should be included in the Reference List unless there are 7 or more. In this case, the first 6 authors should be given, followed by "et al.".
- The reference list must be typed at the end of the manuscript in numerical sequence. **No more than 25 references may be cited.**
- Citation of abstracts and books, as well as articles published in non-indexed journals should be avoided, unless absolutely necessary. **Do not cite references in Portuguese.**
- Abbreviations of journal titles should conform to those used in Dental Index. The style and punctuation of references must follow the format illustrated below:

Journal articles

1. Lea SC, Landini G, Walmsley AD. A novel method for the evaluation of powered toothbrush oscillation characteristics. *Am J Dent* 2004;17:307-309.

Book

2. Shafer WG, Hine MK, Levy BM. *A Textbook of Oral Pathology*. 4th ed. Philadelphia: WB Saunders; 1983.

Chapter in a Book

3. Walton RE, Rotstein I. Bleaching discolored teeth: internal and external. In: *Principles and*

Practice of Endodontics. Walton RE (Editor). 2nd ed. Philadelphia: WB Saunders; 1996. p 385-400.

Tables

- Each table with its title must be typed after the text. Tables should be numbered with Arabic numerals. **DO NOT USE** vertical lines, bold letters and capital letters (except the initials).
- The corresponding title should appear at the top of each table.
- Tables must contain all necessary information and be understandable without allusions to the text.

Figures

- **BDJ WILL NOT ACCEPT FIGURES EMBEDDED IN FILES ORIGINATED IN TEXT-EDITING SOFTWARE (WORD OR SIMILAR) OR FIGURES ORIGINATED IN POWER POINT.**
- The digital files of the images should be generated in Photoshop, Corel or any other image-editing software and saved in the CD-ROM. Image files should have TIFF extension and 300 dpi minimum resolution. Only BLACK & WHITE figures are accepted. Save the figures in the CD-ROM.
- Lettering and identifying marks must be clear and sharp, and the critical areas of x-rays and photomicrographs must be demarcated and/or isolated.
- Separate parts of composite figures must be labeled with capital letters (A, B, C, etc). Single figures and composite figures must have minimum width of 8 cm and 16 cm, respectively.
- Figure captions should be numbered with Arabic numerals and typed on a separate page, after the lists of references or after the tables (if any)

Submission of manuscripts

CHECKLIST FOR AUTHORS PRIOR TO SUBMISSION

1. Submission letter;
2. Title page.

3. Manuscript file (text, tables, figure captions).

4. In the manuscript, observe:

- identification of authors only on the title page.
- text typed in Times New Roman 12 font, with 1.5 spacing, 2.5-cm margins at each side.
- tables, figure captions and figures at the end of the manuscript.

5. Digital files of figures, black & white, saved in TIFF format with minimum resolution of 300 dpi.

There are no fees for submission and evaluation of articles.

The Technical Review Fee ranges from R\$450,00 to R\$ 550,00 Reais Brasileiros (for Brazilian authors) or U\$200 to 300 American dollars (for foreign authors) and will be charged to the corresponding author, even if only minor corrections to the manuscript are needed.

Capitulo 2 e 3- Journal of Esthetic and Restorative Dentistry



FORMATTING YOUR SUBMISSION

Manuscript Types Accepted:

Original Research Articles are related to laboratory research or clinical research.

Clinical Technique Articles describe significant achievements and improvements in clinical practice such as comprehensive interdisciplinary dental treatment, introduction of new technology or practical approaches to recognized clinical challenges. They should conform to the highest scientific and clinical practice standards with supporting references where indicated.

Case Reports must represent new or novel approaches to dealing with specific clinical problems. Proper qualifying and/or disclaiming statements should be included if inadequate research is available to validate the techniques being presented.

Review Articles may be submitted independently or invited by the Editor and include systematic literature reviews of topics related to esthetic and restorative dentistry, as well as more general, comprehensive reviews or updates of a given topic.

Abstract

A structured abstract of no more than 200 words must be provided for each article. Footnotes, references, and abbreviations are not used in the abstract.

For original research articles, the abstract should include the following headings and sections: (1) Objective. This section includes a statement of the problem and the purpose of the study, (2) Materials and Methods. This section should include materials, methods and statistical analyses employed in the study. (3) Results. (4) Conclusions.

For clinical technique articles and case reports, the abstract should include the following headings and sections: (1) Objective. This section includes a statement of the problem and a general description of the topic or treatment to be addressed. (2) Clinical Considerations. This section should include a brief description of the clinical materials and techniques employed. (3) Conclusions.

For systematic literature review articles, the abstract should include the following headings and sections: (1) Objective. This section should include a statement of the topic to be reviewed and a description of the search strategy of relevant literature (search terms and databases), (2) Materials and Methods. This section should contain inclusion criteria (language, type of studies i.e. randomized controlled trial or other, duration of studies and chosen endpoints). (3) Results. This section should include evaluation of papers and level of evidence. (4) Conclusions.

For general review articles the abstract should include the following headings and sections: (1) Objective. This section should include a statement of the topic to be reviewed. (2) Overview. This section should include a brief summary of the findings of the review. (3) Conclusions.

In addition to Abstracts, all papers should include the following:

Clinical Significance

In a few sentences, please indicate the clinical importance and implications of the research or clinical technique discussed, and if applicable, its relevance to esthetic dentistry.

Keywords

Add at least five keywords that reflect the primary content of the paper.

All manuscripts should adhere to the formatting guidelines below.

Title Page

The title page must include all authors' full names, academic degrees, and institutional affiliations and locations. If the manuscript was originally presented as part of a meeting or conference, please include the appropriate name, date, and location. Sources of support in the form of grants, equipment, products, and/or drugs must be disclosed. A corresponding author must be designated and full details of the correspondent's address provided: name, address, telephone and fax numbers, and e-mail address. Unless specified otherwise, the corresponding author's address also will be used for reprint requests.

Disclosure Statement and Acknowledgements (on Title Page)

Please provide any information you wish to include acknowledging contributions from individuals such as for statistical support, lab work, etc. It is imperative that you provide a disclosure statement if you have any financial interest in any of the companies whose products or devices are included in the paper. If no financial interest exists, the following statement must be used: "The authors do not have any financial interest in the companies whose materials are included in this article."

References

References should be numbered consecutively in the order in which they are first mentioned in the text, and listed at the end of the text in numeric, not alphabetic, order. Identify references in text, tables, and legends by Arabic numerals in superscript. References cited only in tables or figure legends should be numbered subsequent to the numbering of references cited in the text. Unpublished sources, such as manuscripts in preparation and personal communications, are not acceptable as references. Only sources cited in the text should appear in the reference list. List all authors when four or fewer; when more than four, list the first three and add "et al."

How to Format Citations

Journal Articles:

Donnelly PV, Miller C, Ciardullo T, et al. Occlusion and its role in esthetics. *J Esthet Restor Dent.*, 1996; 8:111-8.

Books:

Hickey JC, Zarb GA. *Boucher's prosthodontic treatment for edentulous patients*. 9th ed. St. Louis (MO): CV Mosby; 1985.

Tables

Type or print out each table with double spacing on a separate page. Ensure that each table is cited in the text, number tables consecutively in the order of their first citation in the text, and provide a brief title for each. Give each column a brief, descriptive heading. No table should contain data that could be included in the text in several sentences.

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Please include on a separate page all figure and/or illustration legends. This page should be clearly marked. Figure legends must be numbered to correspond with the figures and typed or printed on a separate page. Symbols, arrows, or letters used to identify parts of the illustration must be explained clearly in the legend. If a figure has been previously published, the legend must acknowledge the original source.

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Images must be submitted as individual files, in either TIF or EPS format, as indicated below.

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Line drawings should be prepared in Microsoft Word or PowerPoint, or in Adobe Illustrator without embedded images from other sources. Existing line drawings should be scanned at 1,200 dpi at a minimum of 12.5 cm (5 in.) in width and saved as EPS files.

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References in the text and figure legends to teeth illustrated in a figure should be identified by name (eg, upper right central incisor), not by number.

The manuscripts submitted to the journal must be written in appropriate English. It is the author's responsibility to ensure this by either having sufficient English language skills or by obtaining the services of an English-as-second-language expert.

Please note that the term “esthetic” should be used in manuscripts as opposed to the alternative spelling “aesthetic.”

The same general headings and sections should be used in the articles as used in the abstract.

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The Journal of Esthetic and Restorative Dentistry follows current HIPAA guidelines for the protection of patient/subject privacy. If an individual pictured in a digital image or photograph can be identified, his or her permission is required to publish the image. The journal may not collect consent forms under HIPPA guidelines, however authors are expected to be able to present a signed consent form if asked. Authors must have patient authorization for images, or else the image/photo must be altered such that the individual cannot be identified (black bars over eyes, etc).

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