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JORGE RODRIGO SOTO MONTERO

AVALIAÇÃO DAS PROPRIEDADES MECÂNICAS, FISICAS E OPTICAS DE RESINAS PROVISÓRIAS INDICADAS PARA USO EM IMPRESSORA 3D

EVALUATION OF THE MECHANICAL, PHYSICAL AND OPTICAL PROPERTIES OF 3D-PRINTED TEMPORARY RESTORATIVE RESINS.

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EVALUATION OF THE MECHANICAL, PHYSICAL AND OPTICAL PROPERTIES OF 3D-PRINTED INTERIM RESTORATIVE RESINS.

Tese apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Doutor em Clínica Odontológica na área de Dentística.

Thesis presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Doctor in Clinical Dentistry, in Operative Dentistry area.

Orientador: Prof. Dr. Marcelo Giannini.

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RESUMO

Esta tese foi dividida em dois estudos: 1- avaliação da influência de diferentes tempos de pós-cura na mudança de cor (ΔE_{00}), resistência à flexão (RF), módulo de flexão (MF) e microdureza (MD) em profundidade de quatro resinas para impressão 3D (R3D), 2- mensuração da resistência de união por microtração (μTBS) de dois cimentos resinosos a três R3D e uma de resina CAD-CAM fresada, indicadas para restaurações fixas provisórias. Amostras para ΔE_{00}, RF, MF e MD foram preparadas de acordo com os requerimentos do experimento e avaliadas em cinco tempos de pós-cura (0-, 5-, 10-, 15 e 20 minutos). A MD foi medida transversalmente em blocos de 5 x 5 x 5 mm (n = 10, para cada tempo). Blocos idênticos foram preparados para avaliação da μTBS (n = 8). Metade das amostras foi jateada com partículas de óxido de alumínio e a outra metade ficou sem tratamento. As superfícies tratadas igualmente de dois blocos foram cimentadas com os cimentos resinosos propostos. Após 24 horas, os blocos cimentados foram seccionados para obtenção de espécimes em formato de palitos com secção transversal de 1 x 1 mm. Metade dos palitos foi testada imediatamente e a outra metade foi termociclada (5.000 ciclos, 30s de imersão) antes da avaliação da μTBS. Os resultados da cor foram analisados pela ANOVA de medidas repetidas de um fator (mudança de cor), enquanto a RF e MF foram analisados pela ANOVA de 2 fatores (fatores: Material*Tempo pós-cura). MD foi analisada individualmente para cada material pela ANOVA de 2 fatores (fatores: Profundidade*Tempo pós-cura). μTBS foi analisada por modelo linear generalizado de quatro vias (Material*Jateamento*Cimento*Tempo de avaliação). Os resultados mostram que o tempo de pós-cura influenciou significativamente no ΔE_{00}, RF, MF e MD dos materiais avaliados. Algumas R3D apresentaram valores de ΔE_{00} acima do limite de aceitabilidade após 5 ou 10 minutos de pós-cura. A RF e o MF da maioria dos materiais estabilizaram após 5 minutos de pós-cura. A pós-cura melhorou a MD dos materiais testados, e exposições à luz por mais tempo estiveram associados a maiores valores de MD em profundidade nas amostras. Em relação à μTBS, a resina fresada apresentou a menor resistência de união, independentemente do tipo de cimento, jateamento ou termociclagem. A μTBS dos cimentos resinosos à R3D foi superior a 20 MPa para todas as condições avaliadas. O jateamento melhorou significativamente a μTBS da resina fresada, especialmente após termociclagem, mas não melhorou a μTBS das R3D. Conclui-se que é necessário ajustar o tempo pós-cura em R3D para melhorar as propriedades mecânicas, sem comprometer a cor. Em geral, 5 a 10 minutos de pós-cura produziram propriedades mecânicas adequadas, sem afetar a aceitabilidade na cor da restauração, porém os resultados são dependentes do material. Além disso, as diferenças composicionais e o método de fabricação de materiais das resinas indiretas podem afetar a resistência de união. O jateamento não trouxe benefício para as R3D, embora seja crucial para a cimentação adesiva de resinas fresadas.

Palavras-chave: Desenho assistido por computador; Biomecânica.

ABSTRACT

The purposes of this study were: First, to evaluate the influence of different times of post-curing on the color change (ΔE_{00}), flexural strength (FS), flexural modulus (FM) and microhardness (MH) at depth of four 3D printed (3DP) resins. Then, the microtensile bond strength (µTBS) of two resin cements to three 3DP resins and one milled CAD-CAM resin material, indicated for provisional fixed restorations was measured. Specimens for ΔE_{00}, FS, FM and MH were prepared using the different materials according to the experimental requirements and evaluated under five different post-curing conditions (0-, 5-, 10-, 15, and 20 minutes of post-curing). MH was measured transversally on 5 x 5 x 5 mm blocks (n = 10, for each post-curing time). Identical blocks were prepared for µTBS evaluation (n= 8 per group). Half the specimens were sandblasted with aluminum oxide abrasive particles and the other half was left untreated. The treated surfaces of two blocks were bonded with the evaluated resin cements. After 24 hours, the bonded blocks were sectioned into 1 x 1 mm cross-section sticks. Half of the obtained beams were tested immediately, and the other half was thermocycled (5,000 cycles, 30s dwell-time) before µTBS evaluation. Color results were analyzed by one-way repeated measures ANOVA (factor: color change). FS and FM were analyzed by 2-way ANOVA (factors: Material*post-curing time). MH was analyzed individually for each material by 2-way ANOVA (factors: depth*post-curing time). µTBS was analyzed by four-way Generalized Linear Model (material*sandblasting*cement*aging). The results show that the time of post-curing significantly influenced the ΔE_{00}, FS, FM and MH of the evaluated materials. Some of the 3DP materials presented ΔE_{00} values above the acceptability threshold after 5 or 10 minutes of post-curing. The FS and FM of most materials stabilized after 5 minutes of post-curing. The post-curing process improved the MH of the tested materials, and longer exposure periods were associated to higher MH values at depth. Regarding µTBS, the milled resin exhibited the lowest bond strength, regardless of the cement type, sandblasting or thermocycling. The µTBS of resin cements to 3DP resins was above 20 MPa for all the evaluated cements, surface treatments and evaluation times. Sandblasting significantly improved the µTBS of the milled resin to both cements, especially after thermal aging, but did not improve the µTBS of the 3DP resins. It is concluded that a fine adjustment of the post-curing time is crucial to produce adequate mechanical properties in 3DP resins, while minimizing the color alterations on the restorations. In general, 5 to 10 minutes of post-curing will produce adequate mechanical properties, without affecting the acceptability in the color of the restoration, however, the results are material dependent. Also, differences in the composition and manufacturing method of indirect resin materials can affect their bond strength. Sandblasting is not recommended for 3DP, although is crucial for adhesive cementation of milled temporary resins.

Keywords: Biomechanics; Computer-aided design.

SUMARIO

1. INTRODUÇÃO

Os sistemas automatizados de desenho e fabricação auxiliado por computador (CAD/CAM) são aplicados em diferentes áreas do conhecimento como método de prototipagem rápida para acelerar o processo de desenho de peças específicas e facilitar a manufatura. (Skorulska et al., 2021) Estes processos foram adaptados e incorporados na Odontologia Restauradora, para fabricação de diferentes tipos de restaurações indiretas partir de materiais metálicos,(Bae et al., 2020; Methani et al., 2020) cerâmicos (Almarza et al., 2020; Della Bona et al., 2021) ou resinosos (A. Kessler et al., 2020; Revilla-León et al., 2019). Os sistemas CAD/CAM odontológicos para fabricação de restaurações em resina, podem ser classificados de acordo com o método de produção, sendo divididos principalmente em subtrativos ou aditivos.(Sulaiman, 2020) Tradicionalmente, os métodos CAD/CAM consistiam principalmente na fresagem subtrativa da restauração planejada, a partir de um bloco pré-polimerizado ou pré-sinterizado do material restaurador.(Başaran et al., 2013; Çagri Ural et al., 2010; da Silva et al., 2017; Huettig et al., 2016) A fabricação subtrativa foi introduzida na Odontologia em 1977 com o desenvolvimento do sistema CEREC,(Uzun, 2008) e apesar da importância dessa tecnologia, uma das principais desvantagens dos métodos CAD/CAM subtrativos é que a maior parte do material é desperdiçado como resultado do processo de fresagem.(da Silva et al., 2017; Della Bona et al., 2021; A. Kessler et al., 2020)

Os recentes avanços na Odontologia resultaram na introdução de uma nova estratégia de manufatura CAD/CAM, que consiste na construção aditiva, também conhecida como “impressão 3D”, ou “prototipagem rápida”.(A. Kessler et al., 2020) Os primeiros experimentos na área da impressão 3D iniciaram nos anos 80, e a primeira aplicação de patente ocorreu em 1986(A. Kessler et al., 2020). Nesta categoria de sistemas CAD/CAM, o objeto é construído incrementalmente com base num desenho tridimensional, mediante a aplicação de camadas restritas ao contorno da forma desejada, reduzindo assim drasticamente a quantidade de material desperdiçado.(Della Bona et al., 2021; A. Kessler et al., 2020) Contudo, a introdução das técnicas de impressão 3D na Odontologia ocorreu muito depois, sendo que uma revisão sobre o “estado da arte” das resinas indiretas fabricadas por tecnologia CAD/CAM do ano de 2016, nem sequer incluiu a impressão 3D como uma técnica de fabricação de restaurações indiretas de resina.(Mainjot et al., 2016) Apesar disso, a impressão 3D tem significado um grande desenvolvimento para o processamento de polímeros, principalmente a partir das técnicas de estereolitografia (SLA) e processamento digital de luz (DLP).(Jockusch & Özcan, 2020)

O método de SLA é o mais antigo e também o mais frequentemente usado para impressão 3D de resinas em Odontologia.(A. Kessler et al., 2020) A técnica de SLA pode ser subdividida de acordo com o tipo de fonte de luz e a movimento do feixe no reservatório de resina. No da técnica SLA tradicional, uma fina camada da resina é exposta a um laser que faz uma varredura da camada, ativando a reação de polimerização. Depois desse processo, o laser escaneia a primeira camada, e a plataforma de construção desce uma distância correspondente à espessura da camada desejada e um rolo aplica uma nova camada de resina não curada. O ciclo é repetido para cada camada até que a construção do objeto é completada.(Fuh et al., 1999; A. Kessler et al., 2020; Revilla-León et al., 2019) Por outro lado, a técnica DLP, derivada da SLA, utiliza uma fonte de luz, que pode ser tipo láser ou diodos emissores de luz (LED), que por meio de espelhos fazem uma projeção de toda a camada na tela da impressora, que fica em íntimo contato com o fundo transparente do reservatório resina liquida.(A. Kessler et al., 2020; Nestler et al., 2021; Osman et al., 2017; Revilla-León et al., 2019)

A tecnologia DLP oferece vantagens sobre a SLA tradicional como a rapidez, alta definição e um menor custo.(Alsandi et al., 2021; Lin et al., 2020) Isso porque cada camada pode ser polimerizada com uma única exposição à luz, projetada na tela com o contorno necessário, ao invés de fazer escaneamentos após a impressão das camadas, portanto, um alto número de objetos ou contornos complexos não afetam o tempo de exposição de cada camada. (A. Kessler et al., 2020) As mencionadas vantagens, fizeram com que a tecnologia DLP fosse bem aceita e incorporada para uso inclusive no consultório odontológico.(Alsandi et al., 2019) Apesar das diferenças, o processo de impressão de resinas 3D com técnicas SLA e DLP pode ser dividido de maneira geral em três passos: 1- Exposição à luz; 2- Movimento da plataforma; e 3- Preenchimento do espaço com resina. Estes 3 passos estão interrelacionados, e permitem solidificar a camada pela ativação dos fotoiniciadores pela exposição à luz,(A. Kessler et al., 2020) liberar o espaço necessário para a seguinte camada, e o escoamento da resina no espaço liberado para cobrir a camada previamente polimerizada e continuar com a polimerização de camada subsequente.(Jockusch & Özcan, 2020; A. Kessler et al., 2020)

No entanto, o rápido desenvolvimento tecnológico gerou falta de informações científicas, que muitas vezes foram resolvidas de maneira empírica pelos usuários, sem contar com as evidências para validar ou melhorar os processos, afetando a transmissão dos conhecimentos e o melhor aproveitamento desta tecnologia. (Söderberg, 2013) No caso da impressão 3D, apesar das tentativas de padronização, existem evidentes diferenças entre a literatura e o modo como estas tecnologias são usadas.(Della Bona et al., 2021) Os primeiros estudos de impressão por técnica SLA para materiais restauradores em Odontologia só surgiram nos últimos 5 anos,(Della Bona et al., 2021) porém, o aumento no interesse pelo uso da impressão 3D fez com que recentemente aumentassem exponencialmente os estudos avaliando propriedades mecânicas e físicas das resinas, principalmente estudando a resistência a tração, (Alsandi et al., 2021) flexural (Keßler et al., 2021; D. Kim et al., 2020; Lin et al., 2020; Park et al., 2020) e compressiva destes materiais (Nawal Alharbi et al., 2016), assim como outras propriedades como microdureza,(Grzebieluch et al., 2021; Revilla-león et al., 2020; Simoneti et al., 2020) grau de conversão, (D. Kim et al., 2020; Mayer, Reymus, et al., 2021; Perea-Lowery et al., 2021), estabilidade da cor, (D. Kim et al., 2020; Revilla-León, Umorin, et al., 2020) e precisão das impressões. (Choi et al., 2019; Della Bona et al., 2021; J. Kim & Lee, 2020; Nestler et al., 2021; Osman et al., 2017)

Contudo, apesar do aumento em pesquisas relativas ao uso de restaurações de resina impressas, ainda existem preocupações relativas aos processos de desenho e manufatura, já que múltiplos fatores como a espessura das camadas, (Tahayeri et al., 2018) a angulação em que são impressas as restaurações, (Osman et al., 2017; Revilla-León, Jordan, et al., 2020) o tempo e substancias usadas para lavar as restaurações(Mayer, Reymus, et al., 2021; Mayer, Stawarczyk, et al., 2021) e os protocolos pós-cura(Aati et al., 2021; D. Kim et al., 2020; Reymus & Stawarczyk, 2020a) ainda precisam ser melhor avaliados. No entanto, a aplicação clínica destes materiais vem aumentando e as indicações incluem diversas áreas da Odontologia como a Dentística,(Della Bona et al., 2021; A. Kessler et al., 2020) Cirurgia Oral,(Andreas Kessler et al., 2020) Prótese Total,(N Alharbi et al., 2021; Prpić et al., 2020) Parcial (Jockusch & Özcan, 2020) e Fixa,(Mayer, Reymus, et al., 2021; Park et al., 2020; Reymus et al., 2020) Implantodontia(Jockusch & Özcan, 2020; J. Kim & Lee, 2020; Methani et al., 2020) e Ortodontia.(McCarty et al., 2020; Zhang et al., 2019)

Com relação ao uso destes materiais na Odontologia restauradora, especificamente como material para restaurações indiretas provisórias, é necessário fazer uma exaustiva avaliação dos métodos de fabricação e os melhores protocolos de uso destes materiais, para estabelecer processos eficientes e que finalmente se traduzam em uma aplicabilidade clínica previsível. Este estudo utilizou uma impressora de resina de tecnologia DLP, e uma câmera de pós cura de tecnologia LED para fabricar diferentes tipos de espécimes que permitiram avaliar o efeito que diferentes tempos de pós-cura tem na alteração de cor, na resistência e módulo flexural e na microdureza interna de diferentes resinas para fabricação de restaurações provisórias processadas com impressora 3D. Foi feita também uma caracterização da luz emitida pela câmera de pós-cura para avaliar a homogeneidade da energia fornecida às amostras durante o processo de polimerização e o efeito que este fator pode ter nas propriedades avaliadas.

Além disso, com o objetivo de avaliar a aplicabilidade clínica destes materiais em restaurações provisórias fixas de logo prazo, foi avaliado o efeito do jateamento com óxido de alumínio em conjunto com diferentes cimentos resinosos na resistência de união por microtração de diferentes resinas restauradoras para impressora 3D, após 24 horas de armazenamento em água ou 5000 ciclos de termociclagem. As resinas impressas foram comparadas com uma resina de restauração provisória prepolimerizada para uso em sistemas de fabricação por usinagem, considerado o padrão de referência em sistemas de processamento digital em Odontologia.

2. ARTIGOS

2.1 Artigo 1: Color alterations, flexural strength, and microhardness of 3D printed resins for fixed provisional restoration using different post-curing times

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Abstract

Objectives. To evaluate the effect of post-curing times on the color change, flexural strength (FS), modulus (FM) and microhardness at different depths of four 3D printed resins.

Materials and Methods: A characterization of the light emitted by 3D-resin post-curing unit (Wash and Cure 2.0, Anycubic) was performed. The tested 3D printed resins were Cosmos Temp3D (COS), SmartPrint BioTemp (SM) Resilab3D Temp (RES) and Prizma3D BioProv (PRI) were evaluated under five different post-curing conditions (no post-curing or 5-, 10-, 15, and 20 minutes of post-curing). For color change analysis, 10 mm diameter x 1 mm thick discs (n=7) were printed, and the luminosity, color and translucency were measured before post-curing, and after repeatedly after cycles of 5 minutes of post-curing until a total of 20 minutes was reached for ΔE_{00} [CIED2000 (1:1:1)] calculation. For FS and FM, 25 x 2 x 2 mm (n = 10, for each post-curing time) 3D printed bars were subjected to a 3-point bend test. Knoop microhardness (KHN) was measured transversally on 5 x 5 x 5 mm blocks (n = 10, for each post-curing time). Color results were analyzed by one-way repeated measures ANOVA (factor: color change). FS and FM were analyzed by two-way ANOVA (factors: Material*Post-Curing Time). KHN was analyzed individually for each material by two-way ANOVA (factors: Depth*Post-Curing Time).

Results. The post-curing time significantly influenced the $\Delta E_{00,}$ FS, FM and KHN of all the evaluated materials. COS and SMA presented ΔE_{00} values above the acceptability threshold after 5 and 10 minutes of post-curing, respectively. The FS of RES reached a plateau after 5 minutes of post-curing, and for PRI and SMA, the FS stabilized after 10 minutes of post-curing. The post-curing process improved the KHN of the tested materials, and longer exposure periods were associated to higher KHN values at all the evaluated depths.

Significance: A fine adjustment of the post-curing time is crucial to produce adequate mechanical properties in 3D-printed restorative resins, while minimizing the color alterations on the restorations. For the evaluated resins, 5 to 10 minutes of post-curing will result in adequate mechanical properties, without affecting the acceptability in the color of the material. However, the results are material-dependent, and evaluation of each specific resin is advised.

Introduction

Computer aided design (CAD) and manufacturing (CAM) have revolutionized clinical workflow of dental practices. These technologies are divided into subtractive or milling, and additive, also known as 3D-printing.[1,2] Additive manufacturing has experienced advances that made this technology a useful tool to solve clinical needs in dental specialties such as oral surgery,[3,4] orthodontics,[5] and fixed[6,7] and removable prosthodontics.[6,8] Regarding 3D-printing of dental resins, the development of digital light processing (DLP) technologies using light-emitting diode (LED) screens,[2,9,10] over the most traditional, laser-based stereolithography apparatus (SLA),[2] helped to readily incorporate resin 3D-printing into dental clinics. However, there is few scientifical information on the best printing process and post-curing techniques required to obtain restorations with adequate mechanical properties and esthetics from uncured 3D-printed resins.[2,6]

Some concerns on the biocompatibility of these materials,[11–13] their esthetic[7,14] and mechanical[15] performance have been reported. Regarding the esthetic performance, it has been reported that 3D-printed resins present an unacceptably high variability of shades compared to a reference pattern,[14] and also exhibit poor color stability after storage in water,[7] or in cases of extended exposure to violet and ultraviolet light from the post-curing unit (PCU),[13,16] which could ultimately affect the acceptability of restorations by the patients.[17–19] Also, the findings from direct light-cured resins indicate that factors such as the chemical composition,[11,20] filler type,[2,21,22] photoinitiators[23] and pigments[24,25] of the material, and other related to design and manufacturing steps like layer thickness,[2] specimen angulation,[20,26,27] as well as the washing[28,29] and post curing,[12,16,27,30] protocols could affect the quality of 3D-printed restorations.

There are extensive reports of reduced polymerization at depth for direct light-cured resins,[31–33] and it has been proven that “hardening” of the material does not imply that an adequate degree of conversion has been reached.[31,34–37] For those reasons, extended light-curing times have been proposed to ensure proper polymerization of resin composites,[34,37–39] because an adequate polymerization of the most superficial regions, does not ensure proper curing of the deeper regions.[31,33,40] In the case of DLP 3D-printers, an initial hardening of the resin occurs in the resin vat during the manufacturing process, by the violet light emitted from the printer screen; however, a post-curing step is required to promote the complementary polymerization of the objects and enhance the mechanical properties of the material. Despite previous studies reporting similar optical[14] and mechanical properties between 3D-printed restorative resins and conventional acrylic and bis-acrylic resins[41,42] there is doubt regarding the extent of the polymerization produced by the violet light emitted by the PCU, and the potential benefits or prejudice from extended post-curing times.[4,12,15]

Also, considering that studies reporting the radiant emittance, irradiance, and emission spectrum[40,43–45] of the PCUs used for restorative 3D-printed resins are scarce or non-existent, a thorough characterization of the post-curing light is required to understand the effects that prolonged exposure will produce on 3D-printed resins. Therefore, the purposes of this study were to evaluate the effect of post-curing times on the color change, the flexural strength, modulus and, microhardness at different depths of a variety of 3D printed resins. The following null hypotheses were tested (1) different times of post-curing would not produce changes on the color of the 3D-priting resins; (2) regardless of the material, flexural strength and modulus would not change with different times of post-curing; (3) the time of post-curing would not affect significantly the KHN at depth of the tested materials.

Materials and Methods

Tested Materials and experimental design.

Four different resins, indicated for temporary fixed restorations and designed for manufacturing in digital light processing (DLP) 3D-printer were selected for the study: Cosmos Temp 3D (COS, Yller Biomateriais S.A., Pelotas, RS, Brazil); Smart Print Bio Temp (SMA, MM Tech Projetos Tecnológicos Ltda, São Carlos, SP, Brazil); Resilab 3D Temp (RES, Wilcos do Brasil Ltda, Petrópolis, RJ, Brazil); and Prizma 3D Bio Prov (PRI, Makertech Labs, Tatuí, SP, Brazil). Specifications about the composition, lot number, and shade of the tested products are presented in Table 1. A schematic flowchart of the specimen processing and experimental process is presented in Figure 1.

All specimens were designed using an open-source CAD software (MatterControl v.2.20.1.10422, MatterHackers, CA, USA) and exported to a printer slicer software (Chitubox 64, Chitu Systems, GD, China). The supports were added, and the specimens were sliced using the manufacturer indicated parameters for exposure and off time. Layer height was set to 50 µm at 0° angulation for all the materials and experiments. Specimens for all the selected materials were manufactured using the same root standard tessellation language (STL) files to ensure equal specimen characteristics. After printing, the specimens were washed with 99.5% p.a. isopropyl alcohol (Labsynth, Diadema, SP, Brazil) under agitation for 10 minutes to remove uncured monomers remaining on the surface.[28] For all materials, a group of specimens that were not exposed to post-curing was used as Control. Four different post-curing times were evaluated: 5-, 10-, 15-, and 20 minutes of exposure to violet light in a PCU (Wash and Cure 2.0, Anycubic Technology Co., Shenzen, China).

Characterization of curing light emitted by the printer and curing chamber.

A commercially available DLP, liquid-crystal display (LCD), resin 3D-printer (Photon, Anycubic Technology Co., Shenzen, China), and a light emitting diode (LED) PCU (Wash and Cure 2.0, Anycubic Technology Co., Shenzen, China) were used for all printing and post-curing procedures. Information on the spectral radiant power and radiant emittance of the PCU was obtained using a spectrophotometer (MSC15W, SN 37560; Gigahertz-Optik, Amesbury, MA, USA) coupled to software (MSC15 MEASUREMENT SOFTWARE v.2019.1.0; Gigahertz-Optik, Amesbury, MA, USA), located in front of one LED of the PCU, at 0 mm distance. To calculate the irradiance three measurements of the radiant power of the PCU were made, and an opaque, black cardboard blocking shield with circular 9 mm diameter aperture was placed over the spectrophotometer sensor.

Also, three records of the real-time irradiance received by the spectrometer during 1 minute of exposure on the PCU were obtained using the forementioned spectrophotometer and software. The spectrophotometer was placed on the rotatory base of the PCU, with the sensor located one border of the platform and set to continuously record the irradiance radiant power reaching the sensor during the rotation of the rotatory base. Real time records of the irradiance were obtained with the sensor of the spectrometer in both upward and downward position.

Evaluation of color change

To evaluate the effect of post-curing time on the color and translucency on the resins, seven discs (10 mm diameter, 1 mm thick) were printed with each material. The coordinates of luminosity (L*) and color (a* and b*) of the printed discs prior to post-curing were measured using a commercial spectrophotometer (VITA Easyshade® Advance V, Vita Zahnfabrik, Bad Säckingen, Germany), calibrated according to the manufacturer indications, and fixed with the tip perpendicular to the surface of the samples. The readings were made in a light-controlled box (D65 lightbox GTI MiniMatcher, Gti Graphic Technology, Newburgh, NY, USA) with the samples over a white background, by an experienced operator blinded to the group being tested. Then, the discs were post-cured by a different operator for 5 minutes and the color measurement process was repeated. The post-curing and color measurement procedures were repeated until a post-curing time of 20 minutes was reached. The color difference of the resins (ΔE) was calculated using the CIEDE2000 system[46–48]

To calculate the translucency parameter (TP_{00}), the L*, a*, and b* coordinates values were recorded over a black and white background and entered into the CIEDE2000 (1:1:1) color difference formula.[46–48] Thus, values of translucency difference (ΔT_{00}) were obtained subtracting the baseline values from those obtained after 5-, 10-, 15-, and 20 minutes of post-curing. In the case of discontinuities due to mean hue computation and hue-difference computation when using the CIEDE2000 formula to calculate ΔE_{00} and ΔT_{00}, the criteria discussed and characterized by Sharma et al were considered.[49]

Flexural Strength and Modulus

Evaluation of FS and FM was performed following the standard evaluation norm (ISO 4049) for dental resin composites.[50] For flexural strength and modulus of elasticity measurement, sixty, 25 x 2 x 2 mm specimens[50] were printed for each material at 0 mm angulation. The specimens were divided in 5 groups (n = 12) according to the corresponding post curing time (0-, 5-, 10-, 15-, and 20 minutes). Prior to post-curing, the specimens were finished using a 1200 grit abrasive paper to remove residual flanges after removal of the supports. Then, the specimens were post-cured and stored in water for 24 h at a temperature of 37ºC.[50] The bars were positioned in a 3-point-bending test device (fin distance 20 mm) of a universal testing machine (Model 4411, Instron, Canton, MA, USA) and loaded until fracture with a crosshead speed of 1.0 mm/min.

Knoop Microhardness

Fifty 5 x 5 x 5 mm cubes were printed with each of the evaluated resins and randomly divided in 5 groups (n = 10) corresponding to each of the evaluated post-curing times. The cubes were placed on the post-curing chamber with the face where the fabrication supports were inserted facing up, and post-cured according to the corresponding time. After the post curing process, the face of the cubes that was facing up was painted using a water-resistant marker. The cubes were cross-sectioned using a diamond blade (Isomet Diamond Wafering Blade, no. 11–4244, Buehler Ltd., Lake Buff, IL) with water-cooling. One half of each cube was polished using a sequence of silicon carbide abrasive papers (grits no. 1000,1200, and 2000, Norton Abrasivos, Vinhedo, SP, Brazil) and felt disks containing 1 µm diamond paste (Buehler Ltd.). Specimens were ultra-sonicated (Thornton USC 1400, Unique Group, Indaiatuba, SP, Brazil) in distilled water for 10 min to remove debris and the cross-sectional KHN of the specimens was measured at different depths from the upwards facing surface (50 µm, and 1, 2, 3, 4 and 4.95 mm).

A microhardness tester (Future-Tech FM Corp, Tokyo, Japan; coupled to software FM-ARS 9000, Future-Tech FM Corp) applied a static load of 20 g (0.196 N) for 5 s at each depth. Triplicate hardness indentations were made at each location, and the mean of each location was taken as a single value for the specimen at each depth. To evaluate the polymerization at depth of the specimens, a ratio of the transversal KHN at each depth compared to the highest recorded hardness (D/H ratio) was calculated for all the materials and evaluation times, using the following formula:

$$\text{D/T ratio} = \frac{\text{Highest KHN}}{\text{Mean KHN at each measured depth}} \times 100$$

A previously established parameter for the analysis of the depth of cure of resin-based materials using a KHN ratio of 80% was defined as acceptability threshold for polymerization at each depth.[36]

Statistical Analyses

Data for each of the performed tests was organized in a spreadsheet software (Excel 2016, Microsoft Corporation, Redmond, WA, USA). Spectral output and radiant emittance of the PCU at different wavelength ranges were compared using One-way ANOVA (pre-set $\alpha = 0.05$). For the color change analysis, ΔE data was analyzed by repeated-measures ANOVA (Inter-subject factor: Material; intra-subject factor: post-curing time) and Tukey *post-hoc* ($\alpha = 0.05$). The same analyses were applied for the individual color coordinates to calculate ΔL, Δa and Δb. Also, to evaluate the change in translucency produced by the time of post-curing, the differences between the measurements obtained on the white and black backgrounds were calculated and subjected to repeated measures ANOVA (Inter-subject factor: Material; intra-subject factor: post-curing time) and Tukey *post-hoc* ($\alpha = 0.05$).

Data of FS and FM was subjected to two-way ANOVA and Tukey *post-hoc* ($\alpha = 0.05$) for factors "Material" and "Post-curing time". Microhardness data was analyzed individually for each material by two-way ANOVA (factors: "Depth"*"Post-curing time") and Tukey *post-hoc* ($\alpha = 0.05$) for multiple comparisons. All the statistical analyses were made with a commercially available statistics software (Minitab v.17 for Windows, Minitab LLC, State College, PA, USA)

Results

Light characterization

Information on the spectral power output and radiant emittance of each LED on the PCU is presented in Table 2. The results of the emission spectrum and the real time irradiance of the PCU during the curing cycle are presented in Figure. 2. The spectral emission for the PCU ranges from 390 to 410 nm, with a maximal peak at 401 nm that corresponds to violet light. Real time measurement of the irradiance during the curing cycle shows notorious oscillations on the irradiance depending on the location of the measuring device during the rotation of the base of the curing chamber, ranging from 10 mW/cm² at the position nearest to the LEDs (5 cm distance), to around 4 mW/cm² at the further distance (15 cm distance) with the sensor in the upwards position. When the records were obtained in a downwards position, the recorded irradiance was noticeably reduced, ranging from approximately 6 mW/cm² when the sensor was closest to the LEDs, to 0.5 mW/cm² at the furthest position.

Color change

The detailed results for the initial values of L*, a*, and b* color coordinates, as well as the ΔL, Δa and Δb produced by each time of post-curing are presented in Table 3. Results for ΔE_{00} are presented in Figure 3A. Representative images of the color changes observed at each post-curing time are presented in Figure 3B. For ΔE_{00}, the time ($p<0.0001$) and the interaction between "Material" and "Post curing time" ($p < 0.0001$) significantly influenced the results. Analysis of ΔL and Δb showed that the "Post curing time" ($p < 0.0001$) and the interaction between "Material" and "Post curing time" ($p < 0.0001$) significantly influenced the results. Also, for Δa the time ($p = 0.013$) and interaction "Material*Post curing time" ($p < 0.001$) were significant.

Detailed observation of the L* parameter shows that the observed values were very high, ranging from 99.3 to 100, and the extension of post-curing produced a decrease in the luminosity of the samples. Regarding the a* and b* coordinates, for COS and SMA there was an increase in both values with just 5 minutes of post curing, meaning that the color of these materials changed towards a more green and yellow shade. On the other hand, both RES and PRI presented negative alterations on b*, meaning that the materials became bluer. Also, for these resins the alterations in a* and b* parameters were smaller than those observed in COS and SMA.

The results for ΔT_{00} are presented in Table 4. The ANOVA showed that for ΔT_{00} the time ($p = 0.023$) and the interaction "Material*Post curing time" ($p<0.001$) were significant. The ΔT_{00} alterations were significantly higher for COS and SMA, and both materials had a ΔT_{00} above 1 after 5 and 10 minutes of post-curing respectively. On the other hand, for RES, ΔT_{00} presented negative values below 1 at all evaluated times, and for PRI, this pattern was observed after 10 minutes of post-curing.

Flexural Strength and Modulus

Mean FS and FM values for the evaluated materials are presented in Table 5. Statistical analyses indicated that both "Material" ($p < 0.0001$), "Post curing time" ($p < 0.0001$) and their interaction ($p < 0.0001$) significantly influenced the results. Identically, the statistical analysis of FM showed that the "Material" ($p < 0.0001$), "Post curing time" ($p < 0.0001$) and their interaction ($p < 0.0001$) influenced the FM. For COS, the FS and FM increased significantly when the time of post-curing was extended. Also, after 15 and 20 minutes of post-curing, COS showed the highest FS and FM of all the evaluated materials. For SMA and PRI, the FS reached a plateau after 5 minutes of post-curing, and for RES, the FS stabilized after 10 minutes of light exposure. Interestingly, for SMA, RES and PRI, the FM remained unchanged after 5 minutes of post-curing.

Knoop Microhardness

Mean KHN values for the evaluated materials as a function of the time of post-curing are presented in Table 6. Statistical analyses indicated that the time of post-curing ($p < 0.001$), the depth ($p < 0.001$) as well as the double interaction between factors ($p < 0.001$) significantly influenced KHN results for COS, RES and PRI. For SMA, the time of post-curing ($p < 0.001$), and the depth ($p < 0.001$) were significant, although the interaction between factors was not ($p = 0.330$). In general, there was a trend towards increased KHN values when the time of exposure was extended. Also, for all the evaluated materials the highest KHN values were recorded at the superficial measurement (50 µm depth) decreased on the 2-, 3- and 4- mm deep measurements, and tended to increase at the 5 mm measurement.

Analysis of the D/H ratio showed that COS had a D/T ratio below 80% at the 1-, 2-, 3- and 4- mm measurements, for all the evaluated post-curing times, except for the 1 mm measurement after 5 minutes of post-curing. Interestingly, the 5 mm measurement showed a D/T ratio above 80% for all the evaluated post-curing times. On the contrary, for SMA, RES and PRI, the D/T ratio was over 80% at all the evaluated depths, regardless of the post-curing time.

Discussion

The results of this study demonstrate that the time of post curing can significantly affect the optical and mechanical properties of 3D-printed resins for temporary fixed restorations. A fine tuning of the time of exposure is required for each material to obtain an adequate equilibrium between esthetics and mechanical resistance in 3D printed restorations. Hence, the first null hypothesis was rejected, because the time of post curing was associated to changes in the color of all the evaluated materials. Despite the statistically significant differences, analysis of the L* parameter shows that only COS and SMA showed measurable alterations, and even in the worst measured scenarios, the reduction was very low (2.2% for COS and 0.2% for SMA). Also, for RES and PRI there were no alterations on this parameter. Hence, despite any statistically significant alteration on the luminosity associated with post-curing, the observed alterations would hardly be of clinical relevance. Previous studies report L* values for acrylic and 3D-printed resins ranging from 79 to 82 and from 72 to 83 respectively,[14,20] which might indicate that the evaluated 3D-printed resins exhibit similar or better luminosity than other resinous materials used for temporary fixed restorations.

In general, the changes in a* and b* could explain the significantly relevant ΔE_{00} values observed for COS and SMA,[14] after 10 and 15 minutes of post-curing respectively. On the other hand, despite the statistically significant ΔE_{00}, neither RES nor PRI showed color alterations above the threshold of 4.6 reported for temporary 3D printed resins, and both resins showed negative alterations on b*, meaning that the materials became bluer, which could compensate for any noticeable yellowing of the restoration by producing an enhanced whiteness perception. [52,53] Also, the alterations in a* and b* parameters were smaller than those observed in COS and SMA.

Analysis of the translucency showed a coincidence with the ΔE_{00} findings, because the alterations were significantly higher for COS and SMA, and both materials had a ΔT_{00} above the perceptibility threshold (PT: 0.62) after 5 and 10 minutes of post-curing respectively, although none of them surpassed the ΔT_{00} acceptability threshold of 2.62 for dental restorative materials.[18] Coincidentally, ΔT_{00} for RES was below the PT at all the evaluated times, and for PRI, ΔT_{00} was above the PT only after 20 minutes of post-curing. A recent study evaluated the color alterations on 3D-printed resins after different post-curing times; however, only the ΔE_{00} values were reported, and changes in each specific coordinate and on the translucency were not addressed.[12] Interestingly, their results reported that the evaluated materials presented a darkening on the yellow colors, and intensification of the reddish colors. However, the evaluated post-curing times were excessive and unrealistic (going from 15 to 120 minutes), and there is imprecise information about the spectral range and radiant emittance of the post-curing unit, and the irradiance received by the samples.[38]

It must be considered that findings of color alteration are material dependent. There are several proposed acceptability thresholds for color alteration on dental materials; however, most of them were obtained using ceramics[17] or composite resins[18,19] that present different optical and surface properties than 3D-printed resins for provisional restorations or fail to explain how the evaluation criteria were established.[12] Hence, despite the methodological limitations, the acceptability parameters established in a previous study using similar materials, evaluation conditions, and measurement tool were selected to maintain comparability between the results.[14]

Regarding the mechanical properties of the evaluated resins, different times of post-curing resulted in differences on the FS of COS and RES, and on the FM of COS; hence, the second null hypothesis was rejected. Also, all the evaluated resins showed significant differences compared to the Control that did not receive any post-curing. The differences between the Controls and those subjected to post-curing confirm that adequate post-curing is required to ensure that the material reaches the expected rigidity and strength to resist masticatory forces during occlusion function. Also, the results for SMA, RES, and PRI are in line with a previous study showing that there is no significant difference in FS between 10 and 20 min of post-curing for 3D-printed indicated for denture bases.[8] Recent studies found that a 3D-printed resins for interim fixed prosthesis exhibited higher FS than acrylic[41] and bis-acrylic resins,[7,41] suggesting that the use of additively manufactured, restorations could be a good alternative from a mechanical point of view.

Coincidentally, the third null hypothesis was also rejected, because the time of post-curing significantly influenced the KHN of the tested materials. As expected, the results showed that post-curing is a crucial procedure that increases the hardness of 3D-printed resins.[15] Post-curing times of 5 minutes for SMA, and 15 minutes for COS, RES and PRI produced KHN values similar or better to those reported in other studies for acrylic,[11] bis-acrylic,[7] and different brands of 3D-printed resins for provisional fixed restorations.[11] Interestingly, COS was the material the showed a greater decrease on the KHN towards the middle of the specimen. This could be explained because titanium dioxide has a strong absorption of light in the range of 200 to 400 nm, which could reduce the amount of photons available to activate the photoinitiators at depth.[2,24] A deeper analysis of the KHN results based on the material composition was not possible because for the novel 3D-printed resins, manufacturers keep the formulation of their products under heavy secret, hence reducing the possibility to establish common patterns regarding monomeric, photoinitiator and particle composition.

Analysis of the D/H ratio showed that for all the materials, at most evaluated times, the ratio decreased as the depth approached the middle of the sample and started to increase again towards the bottom margin of the cubic-shaped samples. Photopolymerization at depth is influenced by material-dependent factors such as the photoinitiators,[25,31] the size, refraction index and load of the fillers,[21] as well as characteristics of the curing equipment such as power,[43] area of emission[40] and wavelength of the curing light.[44,45] In this study, the characteristics of the PCU, such as the wavelength of the emitted light, and the irradiance at different regions of the PCU may explain the heterogeneity on the KHN results.[15] Characterization of the light emitted by the post-curing unit showed an emission peak at 401 nm, corresponding to violet light. The limited penetration of lower wavelength violet light into resinous materials has been reported extensively[31,45] and might explain the differences between the shallow top measurement, where a greater irradiance reaches the material, and the measurements obtained at the middle and bottom of the specimen, that received a reduced number of violet photons to activate the photoinitiators on the material. Also, the observed heterogeneity on the irradiance measured at the platform of the PCU also influenced the KHN, when measured on the different inner and external parts of the samples.

The effect of polymerization on mechanical properties such as KHN and FS has been extensively reported for conventional, direct, light-cured resins.[22,23,36,37,39] However, evidence is unclear for 3D printed resins, and reduced polymerization at the deeper or internal regions of the material, may affect the rigidity, strength, and resistance to masticatory forces of provisional restorations,[15,27] inducing a loss of adaptation and marginal sealing. This study evaluated the effects of depth on the KHN, finding that in general the center of the sample present lower hardness than the outer layers. However, the evaluation of depth on the FS and FM of the evaluated resin was not possible because of methodological limitations such as the size and shape of the specimen for the 3-point bending test.[33] Although increased exposure times on the post-curing step might compensate for a poor depth of cure, factors such as the induced color change may limit the possibility of extended post-curing time. Hence, a fine tuning of the post-curing time is required for 3D-printed resins, in order to obtain adequate mechanical properties, while minimizing the color alterations on the printed restorations. For the evaluated resins, 5 to 10 minutes of post-curing will result in adequate mechanical properties, with an acceptable alteration on the color of the material.

Finally, even though full coverage, indirect restorations such as crowns would hardly exceed a 2 mm thickness in a realistic clinical scenario, the observed KHN pattern could be of importance for other type of thick, bulky restorations such as onlays and pontics in fixed partial dentures, where the core of the restoration would present inferior mechanical properties than the areas directly exposed to light on the PCU. Also, attention must be given to occlusal adjustments in 3D-printed temporary restorations, because the hardest, superficial layer may be removed, exposing softer resin, with lower mechanical resistance and more prone to wear against occlusal loads.

Conclusion

Based on the findings of this study, the following conclusions were reached:

1- The post-curing process causes color changes on 3D-printed resins. In general, longer times of exposure will produce greater color alterations.

2- The FS and FM of 3D-printed resins improve with as little as 5 minutes of post curing. Also, for most materials, the FS and FM do not change with post-curing times longer than 10 minutes.

3- Application of post-curing improved the KHN of the tested materials, and longer exposure periods were associated to higher KHN values at all the evaluated depths.

References

1. Son K, Lee JH, Lee KB. Comparison of intaglio surface trueness of interim dental crowns fabricated with sla 3d printing, dlp 3d printing, and milling technologies. Healthc. 2021;9(8).

2. Kessler A, Hickel R, Reymus M. 3D printing in dentistry-state of the art. Oper Dent. 2020;45(1):30–40.

3. Van Assche N, Van Steenberghe D, Quirynen M, Jacobs R. Accuracy assessment of computer-assisted flapless implant placement in partial edentulism. J Clin Periodontol. 2010;37(4):398–403.

4. Kessler A, Reichl FX, Folwaczny M, Högg C. Monomer release from surgical guide resins manufactured with different 3D printing devices. Dent Mater. 2020;36(11):1486–92.

5. McCarty MC, Chen SJ, English JD, Kasper F. Effect of print orientation and duration of ultraviolet curing on the dimensional accuracy of a 3-dimensionally printed orthodontic clear aligner design. Am J Orthod Dentofac Orthop. 2020;158(6):889–97. https://doi.org/10.1016/j.ajodo.2020.03.023

6. Della Bona A, Cantelli V, Britto VT, Collares KF, Stansbury JW. 3D printing restorative materials using a stereolithographic technique: a systematic review. Dent Mater. 2021;37(2):336–50. https://doi.org/10.1016/j.dental.2020.11.030

7. Scotti CK, Velo MM de AC, Rizzante FAP, Nascimento TR de L, Mondelli RFL, Bombonatti JFS. Physical and surface properties of a 3D-printed composite resin for a digital workflow. J Prosthet Dent. 2020;124(5):614.e1-614.e5. https://doi.org/10.1016/j.prosdent.2020.03.029

8. Aati S, Akram Z, Shrestha B, Patel J, Shih B, Shearston K, et al. Effect of post-curing light exposure time on the physico–mechanical properties and cytotoxicity of 3D-printed denture base material. Dent Mater. 2021; 1–11. https://doi.org/10.1016/j.dental.2021.10.011

9. Osman R, Alharbi N, Wismeijer D. Build Angle: Does It Influence the Accuracy of 3D-Printed Dental Restorations Using Digital Light-Processing Technology? Int J Prosthodont. 2017;30(2):182–8.

10. Nestler N, Wesemann C, Spies BC, Beuer F, Bumann A. Dimensional accuracy of extrusion- and photopolymerization-based 3D printers: In vitro study comparing printed casts. J Prosthet Dent. 2021;125(1):103–10. https://doi.org/10.1016/j.prosdent.2019.11.011

11. Revilla-león M, Morillo JA, Att W, Özcan M. Chemical Composition, Knoop Hardness, Surface Roughness, and Adhesion Aspects of Additively Manufactured Dental Interim Materials. J Prosthodont. 2020;0:1–8.

12. Kim D, Shim JSJS, Lee D, Shin SH, Nam NE, Park KH, et al. Effects of post-curing time on the mechanical and color properties of three-dimensional printed crown and bridge materials. Polymers (Basel). 2020;12(11):1–20.

13. Lin CH, Lin YM, Lai YL, Lee SY. Mechanical properties, accuracy, and cytotoxicity of UV-polymerized 3D printing resins composed of Bis-EMA, UDMA, and TEGDMA. J Prosthet Dent. 2020;123(2):349–54. https://doi.org/10.1016/j.prosdent.2019.05.002

14. Revilla-León M, Umorin M, Özcan M, Piedra-Cascón W. Color dimensions of additive manufactured interim restorative dental material. J Prosthet Dent. 2020;123(5):754–60. https://doi.org/10.1016/j.prosdent.2019.06.001

15. Reymus M, Stawarczyk B. Influence of Different Postpolymerization Strategies and Artificial Aging on Hardness of 3D-Printed Resin Materials: An In Vitro Study. Int J Prosthodont. 2020;33(6):634–40.

16. Reymus M, Stawarczyk B. In vitro study on the influence of postpolymerization and aging on the Martens parameters of 3D-printed occlusal devices. J Prosthet Dent. 2020;125(5):817–23. https://doi.org/10.1016/j.prosdent.2019.12.026

17. Paravina RD, Ghinea R, Herrera LJ, Bona AD, Igiel C, Linninger M, et al. Color difference thresholds in dentistry. J Esthet Restor Dent. 2015;27(S1):S1–9.

18. Salas M, Lucena C, Herrera LJ, Yebra A, Della Bona A, Pérez MM. Translucency thresholds for dental materials. Dent Mater. 2018;34(8):1168–74. https://doi.org/10.1016/j.dental.2018.05.001

19. Rocha R, Fagundes T, Caneppele T, Bresciani E. Perceptibility and Acceptability of Surface Gloss Variations in Dentistry. Oper Dent. 2019;

20. Revilla-León M, Meyers MJ, Zandinejad A, Özcan M. A review on chemical composition, mechanical properties, and manufacturing work flow of additively manufactured current polymers for interim dental restorations. J Esthet Restor Dent. 2019;31(1):51–7.

21. Pfeifer CS. Polymer-Based Direct Filling Materials. Dent Clin North Am. 2017;61:733–50.

22. Fronza BM, Ayres APA, Pacheco RR, Rueggeberg FA, Dias CTS, Giannini M. Characterization of inorganic filler content, mechanical properties, and light transmission of bulk-fill resin composites. Oper Dent. 2017;42(4):445–55.

23. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LES, Martin AAA, et al. Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites. Dent Mater [Internet]. 2015;31(12):1542–51. Available from: http://dx.doi.org/10.1016/j.dental.2015.10.001

24. Kang X, Liu S, Dai Z, He Y, Song X, Tan Z. Titanium dioxide: From engineering to applications. Vol. 9, Catalysts. 2019.

25. Albuquerque PPAC, Moreira ADL, Moraes RR, Cavalcante LM, Schneider LFJ. Color stability, conversion, water sorption and solubility of dental composites formulated with different photoinitiator systems. J Dent. 2013;41(SUPPL. 3):e67–72. http://dx.doi.org/10.1016/j.jdent.2012.11.020

26. Revilla-León M, Jordan D, Methani MM, Piedra-Cascón W, Özcan M, Zandinejad A. Influence of printing angulation on the surface roughness of additive manufactured clear silicone indices: An in vitro study. J Prosthet Dent. 2020;1–7. https://doi.org/10.1016/j.prosdent.2020.02.008

27. Reymus M, Fabritius R, Keßler A, Hickel R, Edelhoff D, Stawarczyk B. Fracture load of 3D-printed fixed dental prostheses compared with milled and conventionally fabricated ones: the impact of resin material, build direction, post-curing, and artificial aging—an in vitro study. Clin Oral Investig. 2020;24(2):701–10.

28. Mayer J, Reymus M, Wiedenmann F, Edelhoff D, Hickel R, Stawarczyk B. Temporary 3D printed fixed dental prosthesis materials: Impact of post printing cleaning methods on degree of conversion as well as surface and mechanical properties. Int J Prosthodont. 2021;1–29.

29. Mayer J, Stawarczyk B, Vogt K, Hickel R, Edelhoff D, Reymus M. Influence of cleaning methods after 3D printing on two-body wear and fracture load of resin-based temporary crown and bridge material. Clin Oral Investig. 2021;

30. Perea-Lowery L, Gibreel M, Vallittu PK, Lassila L. Evaluation of the mechanical properties and degree of conversion of 3D printed splint material. J Mech Behav Biomed Mater. 2021;115(November 2020):104254. https://doi.org/10.1016/j.jmbbm.2020.104254

31. Soto-Montero J, Nima G, Rueggeberg F, Dias C, Giannini M. Influence of Multiple Peak Light-emitting-diode Curing Unit Beam Homogenization Tips on Microhardness of Resin Composites. Oper Dent. 2020 Dec 3;45(3):327–38. http://www.jopdentonline.org/doi/10.2341/19-027-L

32. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the “depth of cure” of dimethacrylate-based dental composites. Dent Mater. 2012;28(5):512–20. http://dx.doi.org/10.1016/j.dental.2011.12.004

33. Mendonça BC de, Soto-Montero J, Castro EF De, Pecorari VGA, Rueggeberg FA, Giannini M. Flexural strength and microhardness of bulk-fill restorative materials. J Esthet Restor Dent. 2021;33(4):628–35.

34. Romano B, Soto-Montero J, Rueggeberg F, Giannini M. Effects of extending duration of exposure to curing light and different measurement methods on depth-of- cure analyses of conventional and bulk-fill composites. Eur J Oral Sci. 2020;128(4):336–44.

35. Price RB, Rueggeberg F, Harlow J, Sullivan B. Effect of mold type, diameter, and uncured composite removal method on depth of cure. Clin Oral Investig. 2016;20(8):1699–707. http://dx.doi.org/10.1007/s00784-015-1672-4

36. Bouschlicher MR, Rueggeberg F, Wilson BM. Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. Oper Dent. 2004;29(6):698–704. http://www.ncbi.nlm.nih.gov/pubmed/15646227

37. Mendonça BC de, Soto-Montero JR, de Castro EF, Kury M, Cavalli V, Rueggeberg FA, et al. Effect of extended light activation and increment thickness on physical properties of conventional and bulk - filled resin - based composites. Clin Oral Investig. 2021;(0123456789).

38. Price RBT. Light Curing in Dentistry. Dent Clin North Am. 2017;61(4):751–78. http://dx.doi.org/10.1016/j.cden.2017.06.008

39. Rueggeberg FA, Cole MA, Looney SW, Vickers A, Swift EJ. Comparison of manufacturer-recommended exposure durations with those determined using biaxial flexure strength and scraped composite thickness among a variety of light-curing units: Masters of esthetic dentistry. J Esthet Restor Dent. 2009;21(1):43–61.

40. Shimokawa CAK, Lacalle M, Giannini M, Braga RR, Price RB, Turbino ML, et al. Effect of light curing units on the polymerization of bulk fill resin-based composites. Dent Mater. 2018;34(8):1211–21. https://doi.org/10.1016/j.dental.2018.05.002

41. Simoneti DM, Pereira-Cenci T, dos Santos MBF. Comparison of material properties and biofilm formation in interim single crowns obtained by 3D printing and conventional methods. J Prosthet Dent. 2020;1–5. https://doi.org/10.1016/j.prosdent.2020.06.026

42. Tahayeri A, Morgan MC, Fugolin AP, Bompolaki D, Athirasala A, Pfeifer CS, et al. 3D printed versus conventionally cured provisional crown and bridge dental materials. Dent Mater. 2018;34(2):192–200. http://dx.doi.org/10.1016/j.dental.2017.10.003

43. Shimokawa CAK, Turbino ML, Harlow JE, Price HL, Price RB. Light output from six battery operated dental curing lights. Mater Sci Eng C. 2016;69:1036–42. http://dx.doi.org/10.1016/j.msec.2016.07.033

44. Issa Y, Watts DC, Boyd D, Price RB. Effect of curing light emission spectrum on the nanohardness and elastic modulus of two bulk-fill resin composites. Dent Mater. 2016;32(4):535–50. http://dx.doi.org/10.1016/j.dental.2015.12.017

45. Harlow JE, Rueggeberg F, Labrie D, Sullivan B, Price RB. Transmission of violet and blue light through conventional (layered) and bulk cured resin-based composites. J Dent [Internet]. 2016;53:44–50. Available from: http://dx.doi.org/10.1016/j.jdent.2016.06.007

46. Luo MR, Cui G, Rigg B. The development of the CIE 2000 colour-difference formula: CIEDE2000. Color Res Appl. 2001;26(5):340–50.

47. CIE Technical Report: Improvement to industrial color difference equation. CIE pub no. Vienna, Austria: CIE Central Bureau; 2001.

48. CIE Technical Report: Colorimetry. In: CIE Pub No 153. Vienna, Austria: CIE Central Bureau; 2004.

49. Sharma G, Wu W, Dalal EN. The CIEDE2000 color-difference formula: Implementation notes, supplementary test data, and mathematical observations. Color Res Appl. 2005;30(1):21–30.

50. International Standardization Organization. Dentistry — Polymer-based restorative materials (ISO 4049:2009). Geneva; 2009.

52. Joiner A, Philpotts CJ, Alonso C, Ashcroft AT, Sygrove NJ. A novel optical approach to achieving tooth whitening. J Dent. 2008;36(SUPPL. 1):8–14.

53. Dantas AAR, Bortolatto JF, Roncolato Á, Merchan H, Floros MC, Kuga MC, et al. Can a bleaching toothpaste containing blue Covarine demonstrate the same bleaching as conventional techniques? An in vitro, randomized and blinded study. J Appl Oral Sci. 2015;23(6):609–13.

Figures

Figure 1. Flowchart summarizing the experimental design, selected materials, times of post-curing, and evaluated properties.

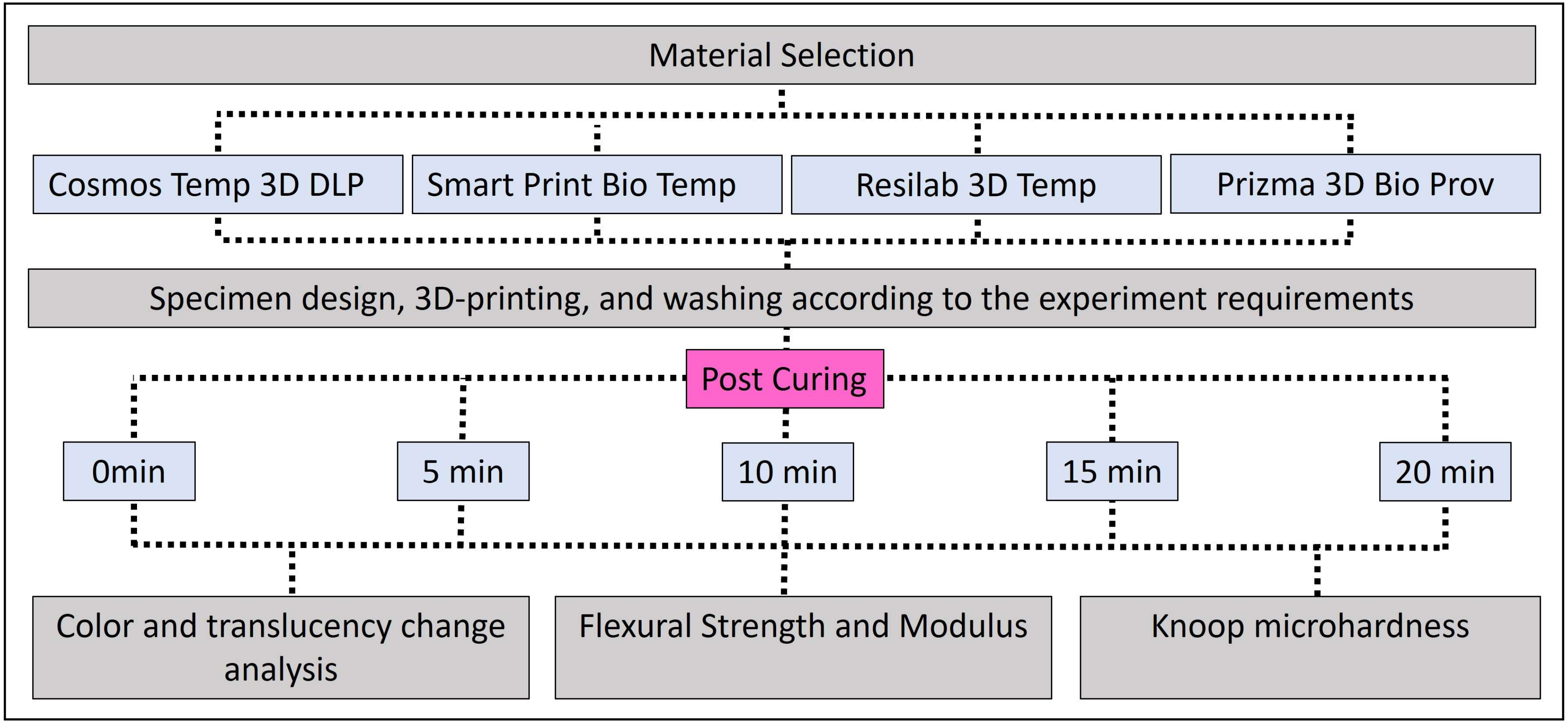

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Figure 2. A- Spectral irradiance (mW/nm/cm2) and emission peak of the used post-curing unit; B- Real-time irradiance of the post-curing unit measured for one minute. The solid line represents the record obtained with the sensor on the upward position. The dotted line shows the recorded irradiance when the sensor was place in a downward position. Green sections on the lines indicate an irradiance ranging from 100 to 80% of the maximal measured irradiance. Yellow segments indicate that the irradiance ranges from tween 80 to 60% of the maximal values. Red segments show irradiance values below 60% of the maximal irradiance measured during the 1-minute cycle.

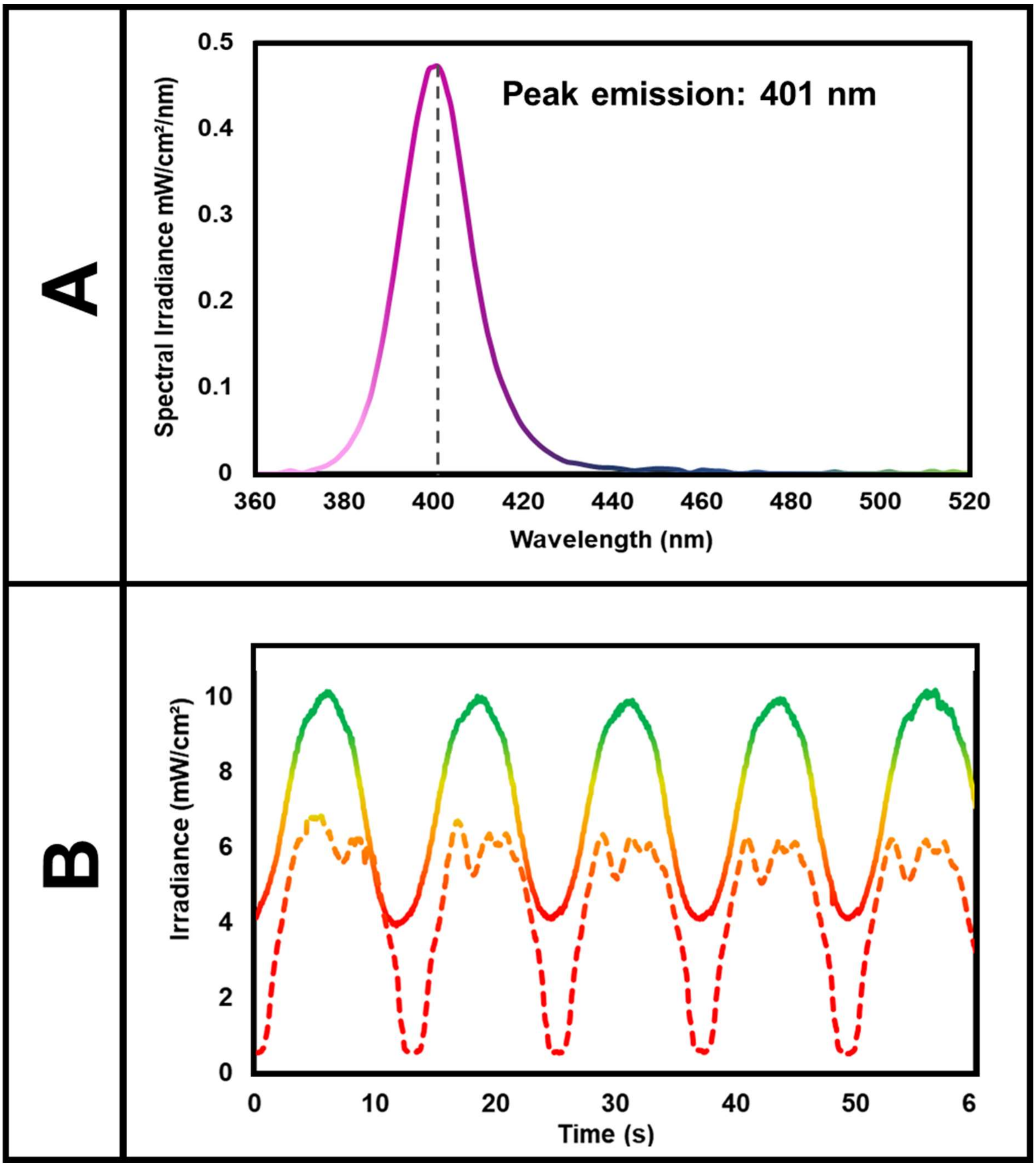

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Figure 3. A- Mean color change (ΔE_{00}) values, according to post-curing time of the 3D-printed resins, compared to the Control (no post-curing). Noticeable changes were observed for COS after 10 minutes of post-curing, and for SMA after 15 minutes of post-curing, according to the parameters established by Revilla Leon et al, 2020.[14] Different letters indicate significant differences. Upper-case letters compare different materials for the same post-curing time. Lower-case letters compare different times of post-curing for the same material. B- Representative images of the color changes observed for the evaluated resins on each post-curing time

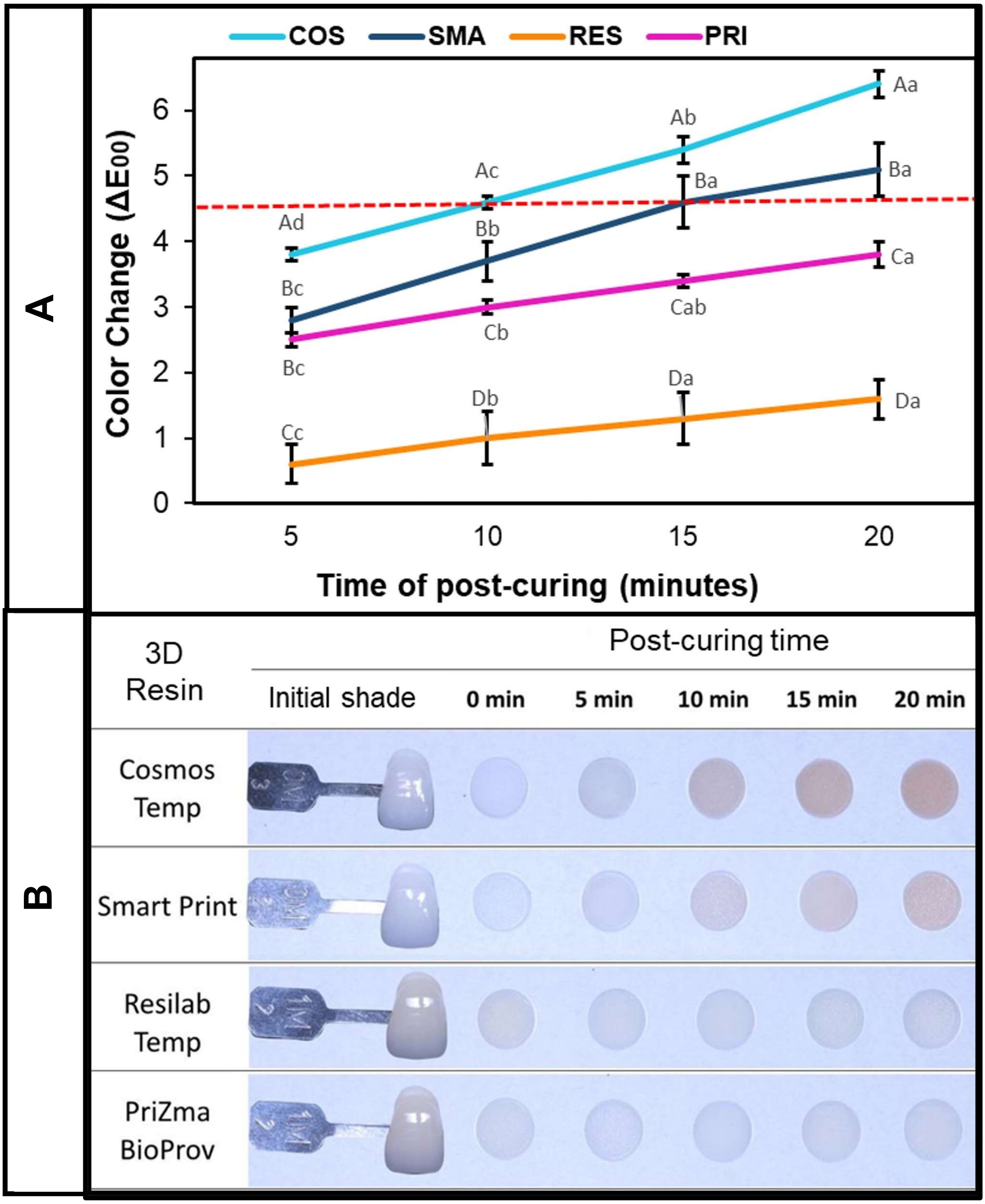

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Figure 4. Depth to top hardness ratio of the tested 3D-printed resins using different exposure durations. The dotted red line represents the acceptable threshold of 80% D/H ratio.

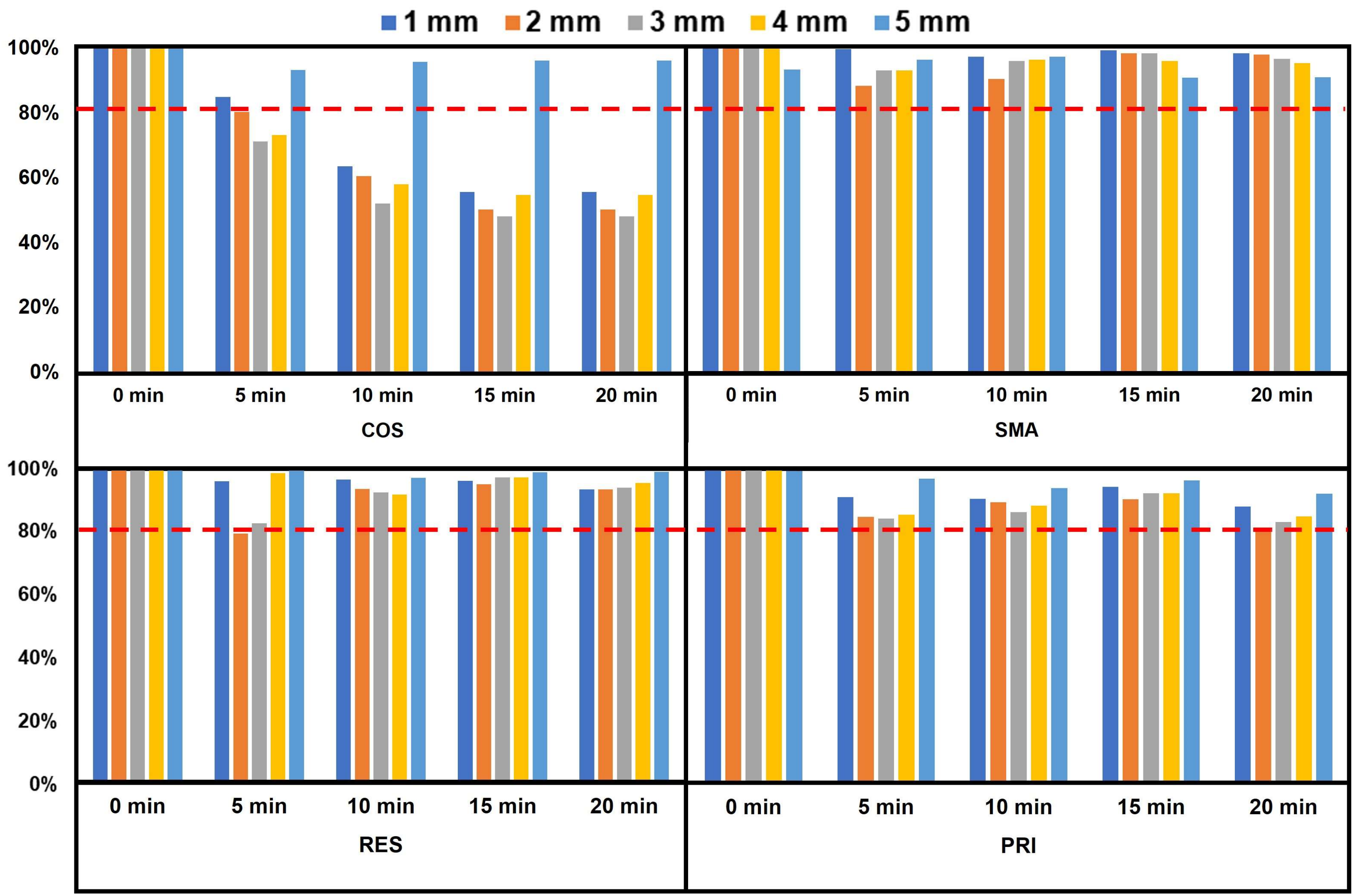

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Table 1. Brand names, compositions, exposure time in seconds (s), shades and lot numbers of tested composites.

Composite (Abbreviations)	Composition	Shade	Lot number
Cosmos Universal Temp 3D (COS)	Methacrylate oligomers, diphenyl-2,4,6-trimethylbenzoyl phosphine oxide, titanium dioxide, carbon black.	A1	00008288
Smart Print Bio Temp (SMA)	Methacrylic ester monomers, stabilizer, fillers, pigments, photoinitiators, accelerators.	B1	PTPB1010/20
Resilab 3D Temp (RES)	Information is not available	A1	1417
Prizma 3D Bio Prov (PRI)	Methacrylic acid esters, acrylic oligomers, acrylic monomers, pigments, proprietary photoinitiator.	A1	1410

Table 2. Wavelength Range, Power Output, and Radiant Emittance of each one light emitting diode of the Post-curing Unit, recorded at 0 mm distance.

Wavelength range (nm)	Power Output (mW)		Radiant Emittance (mW/cm^2)	
	Mean	SD	Mean	SD
360-385	6.2	0.7	9.7	1.1
385-425	207.4	8.7	326.1	9.4
425-515	4.5	0.4	7.1	0.7

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Table 3. Mean ± SD color change, divided by coordinates, of the evaluated resins after different times of post-curing.

Material	L_0	ΔL_{5min}	ΔL_{10min}	ΔL_{15min}	ΔL_{20min}	a_0	Δa_{5min}	Δa_{10min}	Δa_{15min}	Δa_{20min}	b_0	Δb_{5min}	Δb_{10min}	Δb_{15min}	Δb_{20min}
COS	99.3 ±1.3	-0.6±0.3Ab	-0.9±0.3Ab	-1.4±0.4Bb	-2.2±0.3Cb	0.5 ±0.1	2.5±0.2Aa	2.4±0.1 Aa	2.2±0.1Aa	2.1±0.1Aa	17.2 ±0.3	5.4±0.2Da	7.9±0.3Ca	10.1±0.4Ba	12.6±0.5Aa
SMA	100.0 ±0.0	0.0±0.0Aa	0.0±0.1Aa	-0.1±0.2Aa	-0.2±0.3Aa	-4.8 ±0.5	0.7±0.4Ab	0.9±0.4 Ab	1.2±0.4Aab	1.4±0.4Aab	7.8 ±0.6	3.5±0.2Cb	4.6±0.3Bb	5.8±0.3Ab	6.6±0.3Ab
RES	100.0 ±0.0	0.0±0.0Aa	0.0±0.0Aa	0.0± 0.0Aa	0.0±0.0Aa	1.1 ±1.1	-0.1±0.0Ab	-0.1±0.1 Abc	0.1±0.1Ab	0.6±0.1Ab	26.1±0.5	-1.5±0.2Ac	-2.2±0.2ABc	-3.0±0.3BCc	-3.4±0.3Cc
PRI	100.0 ±0.0	0.0±0.0Aa	0.0±0.0Aa	0.0±0.0Aa	0.0±0.0Aa	1.3 ±0.2	1.0±1.1Ab	-0.05±1.8 Bc	-1.5±1.4Bc	-2.2±0.4Cc	29.7±0.7	-4.2±0.7Ad	-5.0±0.7Bd	-5.5±0.7Bd	-6.4±0.7Cd

L_0, a_0, and b_0 are presented for reference only, and indicate the initial baseline value of each coordinate prior to post-curing.

Within a demarcated quadrant, similar letters indicate no significant differences. Upper-case letters compare different post-curing times, within the same parameter and material (Horizontal). Lower-case letters compare different materials for the same post-curing time and parameter (Vertical) ($p<0.05$).

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Table 4: Mean ± SD ΔT_{00} of the evaluated resins, after different times of post-curing. Initial translucency values are shown for reference

Material	**T_0**	ΔT_{5min}	ΔT_{10min}	ΔT_{15min}	ΔT_{20min}
Cosmos 3D Temp	9.5 ± 0.9	1.1 ± 0.4 Aa	1.1 ± 0.2 Aa	1.0 ± 0.3 Aa	0.7 ± 0.4 Aa
Smart Print Bio Prov 3D	13.1 ± 0.7	0.6 ± 0.2 Aa	1.4 ± 1.4 Aa	1.2 ± 0.2 Aa	1.0 ± 0.1 Aa
Resilab Temp 3D	11.1 ± 0.3	-0.3 ± 0.1 Ab	-0.3 ± 0.2 Ab	-0.3 ± 0.2 Ab	-0.2 ± 0.2 Ab
PriZma 3D	11.0 ± 0.5	0.3 ± 0.6 Aab	-0.1 ± 0.7 ABb	-0.5 ± 0.6 ABb	-0.9 ± 0.3 Bb

*Similar letters indicate no significant differences. Upper-case letters compare times, within the same material (Horizontal). Lower-case letters compare different materials for the same post-curing time (Vertical) ($p<0.05$). Initial translucency values are shown for reference in the demarcated quadrant.

Table 5. Mean (SD) flexural strength (FS) and flexural modulus (FM) of the evaluated resins according to post-curing time.

Material	FS (MPa)					FM (GPa)				
	0 min	5 min	10 min	15 min	20 min	0 min	5 min	10 min	15 min	20 min
COS	19.5±2.7 Bd	84.9±5.3 Bc	107.8±9.2 Ab	118.0±6.3 Aab	122.9±4.7 Aa	0.3±0.1 Bd	2.2±0.2 Bc	2.9±0.3 Ab	3.1±0.1 Aab	3.3±0.2 Aa
SMA	21.9±2.1 Bb	89.9±6.3 ABa	99.6±7.0 Aa	97.6±5.7 Ba	97.3±14.1 Ba	0.4±0.1 Bb	2.5±0.1 Aa	2.7±0.2 ABa	2.5±0.1 Ba	2.7±0.1 Ba
RES	34.2±3.7 Ac	82.5±5.7 Bb	97.1±5.7 Aa	98.5±4.8 Ba	96.7±4.5 Ba	0.8±0.1 Ab	2.4±0.1 Ba	2.6±0.2 Ba	2.6±0.1 Ba	2.5±0.2 Ba
PRI	33.7±4.3 Ab	96.3±4.3 Aa	99.0±3.9 Aa	97.4±6.4 Ba	100.8±4.2 Ba	0.7±0.1 Ab	2.7±0.2 Aa	2.7±0.2 ABa	2.6±0.3 Ba	2.8±0.1 Ba

Within a demarcated quadrant: Similar letters indicate no significant difference ($p < 0.05$). Upper-case letters compare different materials within the same post-curing time (vertical). Small-case letters compare different post-curing times for the same material (horizontal).

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Table 6. Microhardness of the tested resins, according to the post curing time and measurement location.

Material	Depth (mm)	Post-curing time (minutes)				
		0	5	10	15	20
Cosmos Temp 3D	0	3.01 ± 1.28 Bd	13.24 ± 2.44 Ac	17.65 ± 1.65 ABb	19.88 ± 1.40 Aab	21.47 ± 1.51 Aa
	1	4.30 ± 0.52 ABb	11.16 ± 1.37 ABCa	11.02 ± 0.69 Ca	11.05 ± 1.51 Ba	11.93 ± 1.63 Ba
	2	4.51 ± 0.51 ABb	10.55 ± 1.68 BCa	10.67 ± 0.64 Ca	9.98 ± 0.54 Ba	10.77 ± 0.70 Ba
	3	4.58 ± 0.59 ABb	9.38 ± 1.81 Ca	9.18 ± 1.41 Ca	9.57 ± 1.00 Ba	10.33 ± 1.10 Ba
	4	5.26 ± 0.91 Ab	9.75 ± 1.40 Ca	10.22 ± 1.32 Ca	10.87 ± 0.77 Ba	11.74 ± 0.84 Ba
	5	3.96 ± 0.91 ABd	12.30 ± 1.81 ABc	16.89 ± 0.82 Bb	19.11 ± 0.80 Aab	20.64 ± 0.86 Aa
SmartPrint Bio Temp	0	7.93 ± 2.84 Ac	21.12 ± 0.77 Ab	21.38 ± 0.73 Aab	21.41 ± 0.80 Aab	22.85 ± 0.60 Aa
	1	9.18 ± 1.94 Ac	21.07 ± 0.64 Ab	20.92 ± 0.97 Ab	21.30 ± 1.31 Aab	22.60 ± 0.63 Aa
	2	8.96 ± 1.05 Ac	18.74 ± 0.90 Bb	19.36 ± 0.53 Bb	21.11 ± 0.88 Aa	22.52 ± 0.70 Aa
	3	8.37 ± 0.93 Ac	19.71 ± 0.82 ABb	20.61 ± 1.71 Ab	21.11 ± 0.84 Aab	22.20 ± 0.89 ABa
	4	7.99 ± 1.22 Ac	19.66 ± 1.01 ABb	20.72 ± 1.04 Aab	20.64 ± 0.54 Ab	21.92 ± 0.87 ABa
	5	7.39 ± 2.25 Ac	20.44 ± 0.74 ABab	20.91 ± 1.03 Aa	19.49 ± 0.97 Ab	20.89 ± 0.71 Bab
Resilab 3D Temp	0	3.83 ± 0.59 Bd	11.68 ± 0.68 Ac	16.93 ± 0.61 Ab	17.93 ± 0.99 Ab	19.80 ± 1.10 Aa
	1	7.16 ± 0.85 Ad	11.25 ± 0.53 Ac	16.40 ± 0.61 Ab	17.30 ± 1.45 Ab	18.60 ± 1.53 Aa
	2	7.16 ± 0.61 Ae	9.29 ± 0.64 Bd	15.86 ± 0.59 Ac	17.10 ± 0.70 Ab	18.58 ± 1.53 Aa
	3	7.46 ± 0.60 Ad	9.72 ± 0.85 Bc	15.68 ± 0.89 Ab	17.51 ± 1.25 Aa	18.70 ± 1.68 Aa
	4	6.84 ± 0.69 Ad	11.57 ± 0.62 Ac	15.59 ± 1.11 Ab	17.54 ± 1.20 Aab	19.03 ± 1.28 Aa
	5	4.12 ± 0.67 Bd	11.80 ± 0.49 Ac	16.45 ± 1.00 Ab	17.82 ± 0.91 Aab	19.70 ± 1.13 Aa
Prizma 3D Bio Prov	0	9.54 ± 0.93 Ad	16.97 ± 1.01 Ac	18.13 ± 1.37 Abc	19.70 ± 1.13 Ab	21.77 ± 0.93 Aa
	1	10.11 ± 0.79 Ac	15.47 ± 1.06 ABb	16.41 ± 1.30 ABb	18.55 ± 0.70 ABa	19.21 ± 0.74 BCa
	2	9.93 ± 0.63 Ac	14.41 ± 0.39 Bb	16.19 ± 1.56 Bab	17.80 ± 1.65 Ba	17.56 ± 0.67Ca
	3	10.22 ± 0.68 Ac	14.28 ± 1.68 Bb	15.57 ± 1.46 Bb	18.19 ± 1.20 ABa	18.075 ± 0.58Ca
	4	10.26 ± 0.86 Ac	14.52 ± 1.19 Bb	16.02 ± 1.50 Bb	18.21 ± 1.04 ABa	18.54 ± 0.76 BCa
	5	9.49 ± 0.95 Ac	16.479 ± 0.81 Ab	17.01 ± 1.44 ABb	18.99 ± 0.90 ABa	20.04 ± 0.75 ABa

*Similar letters indicate no significant differences. Upper-case letters compare depths, within the same post-curing time (Vertical). Lower-case letters compare different post-curing times for the same depth (Horizontal) ($p<0.05$)

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2.2 Artigo 2: Microtensile bond strength of resin cements to 3-D printed and milled resins for provisional fixed restorations

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Abstract

This study evaluated the microtensile bond strength (μTBS) of two resin cements to three 3-D printed resins and one PMMA-based CAD/CAM material, which are indicated for provisional resin-bonded fixed partial dentures. Blocks (5 x 5 x 5 mm) of the evaluated 3-D-printed resins (Cosmos3DTemp / Yller; Resilab 3D Temp / Wilcos and SmartPrint BioTemp, / MMTech) were printed (Photon, Anycubic Technology Co.). The Control was a PMMA-based milled material (VitaCAD-Temp, VITA). Half the specimens were sandblasted with aluminum oxide abrasive particles and the other half was left untreated. Two resin cements were tested: PanaviaV5 (Kuraray Noritake) and RelyX Ultimate (3M Oral Care). The treated surfaces of two resin blocks were bonded with the corresponding resin cement and a 5 N load was applied for 5 minutes before light-activation. After 24 hours, the bonded blocks were sectioned into 1 x 1 mm side sticks. Half of beams were tested for μTBS in a universal testing machine (EzTest, Shimadzu; 1 mm/min load) until failure. The other half was thermocycled (5000 cycles, 30s dwell-time, 5s transfer time) before tensile testing. Data was analyzed by four-way Generalized Linear Model (material*sandblasting*cement*aging). VITA exhibited the lowest μTBS, regardless of the resin cement type, sandblasting and thermocycling. The μTBS of resin cements to 3D printed resins was above 20 MPa for all the evaluated resin cement, surface treatment and measuring time conditions. Sandblasting significantly improved the μTBS of VIT to both resin cements, especially after thermal aging, but did not improve the μTBS of the 3D printed resins. Differences in the composition and manufacturing method of indirect resin materials can affect their bond strength. Sandblasting seemed not beneficial for 3D printed, although is a crucial step for adhesive cementation of milled temporary resins.

Keywords: Computer-aided design; Computer-aided manufacturing; 3D printing; Provisional restoration; micro-tensile, bond strength.

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Introduction

Temporary restoration is a crucial step for fixed, dental and implant supported prosthodontics, because it will represent an intermediate step between the diagnosis and subsequent treatment,[1,2] help model the soft tissues after bone or tissue grafts,[3] and allow the patient to evaluate the shade, shape, size, position and overall comfort of the proposed rehabilitation in function before cementation of definitive restorations.[4–6] Recently, the development of CAD/CAM technologies for applications in Dentistry, introduced software design of temporary and definitive fixed restorations.[7–9] The first approaches to digital design and manufacturing consisted mainly of subtractive milling of the planned restoration from a pre-polymerized or pre-sintered block of the restorative material.[10–13] Despite the importance of the development, the main disadvantage associated with subtractive CAD/CAM methods is that much of the material is wasted as a result of the milling process.[13–15]

Recent developments on CAD/CAM technologies introduced a new type of additive manufacturing, also known as 3D-printing, where the restorations are fabricated in incremental layers restricted to the contour of the desired shape, thus, the amount of discarded material is reduced.[14,15] Among resin 3D-printing techniques, digital light processing offers advantages for chairside use, because is a relative fast processing, low cost and offers a high resolution.[16,17] Thus, technique consist of a DLP projector, that is in intimate contact with a resin-filled container, and emits light through an intermediary screen. The material composition includes photoinitiators that are activated by the light emitted by the DLP printer,[18] and polymerize the overlying resin in the desired shape and depth to form a layer.[15] The process is subsequently repeated until all the layers are printed, and the restoration is complete. Despite the convenience of this technological advance, and because the clinical application of new techniques advances at a faster rate than the research that validates them,[19] there are still concerns related to the design and manufacturing processes such layer thickness,[20] printing angulation,[21,22] and the post-curing protocols.[23] Other concerns, however, are related to the chemical and mechanical compatibility of 3D-printed resins with other restorative materials.[24–26]

Mechanical performance of 3D-printed resins has been the subject of recent studies[27–29] that have focused mainly on the tensile,[16] flexural[17,30–32] and compressive strength of these materials[33] and other properties such as microhardness,[34–36] degree of conversion,[30,37,38] color stability[30,39] and accuracy of the restorative resins.[21,40–42]

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However, there is a lack of information related to the possibility to bond to 3D-printed resin indirect restorations.[43,44] Adhesive cementation of 3D-printed resins would largely increase the clinical applications of these materials and make for an attractive treatment option in cases where long-term provisional restorations are required.[3,45] Moreover, there are no clear guidelines on the best approach to prepare the surface of 3D-printed resins for bonding, either by chemical or mechanical treatments, although previous studies [46,47] have proposed the application of airborne particle abrasion (APA) [43,44,46–48] and chemical primers[49,50] as a mean to increase the bond strength between other resin-based materials.

Thus, considering the importance of adequately retained provisional fixed restorations, and the insufficient information about clinical protocols for adhesive cementation of 3D printed resins, the purpose of this study was to evaluate the effect of surface treatments on the microtensile bond strength (µTBS) of two resin cements to four digitally manufactured restorative resins, after 24 hours of water storage or thermocycling. The following null hypotheses were tested: (1) regardless of the type of cement or APA approach, there would not be differences on the µTBS of the evaluated 3D printed and milled resins; (2) regardless of the type of cement, different APA protocols would not produce changes on the µTBS of the different resin; (3) regardless of the material and APA treatment, there would not be differences between the evaluated cements; and (4) there would be differences on the µTBS before and after thermal cycling.

Materials and Methods

Analysis of the emission spectrum of the 3D-printer

Qualitative emission spectrum from the 3D-printer (Photon, Anycubic Technology Co., Shenzen, China) was obtained using a calibrated spectrophotometer (Flame S-VIS-NIR, Ocean Insight, Orlando, FL, USA) associated to a 600 µm fiber optic cable with a cosine corrector (CC-3-UV-S, Ocean Insight, Orlando, FL, USA) with 6.35 mm diameter. The spectrophotometer was connected to a software (OceanView version 2.0.7, Ocean Insight, Orlando, FL) and the emission data was exported to a spreadsheet software (Excel 2016; Microsoft, Redmond, WA, USA). The cosine corrector was fixed with the active area perpendicular to the printer screen, and the printer was set to run a

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screen test. The emitted wavelength range was recorded 3 times and the mean value for each wavelength was calculated.

Tested Materials and experimental design.

Four different resins, indicated for fabrication of temporary fixed restorations using CAD/CAM technology were selected for this study. Three are designed for additive manufacturing in digital light processing (DLP) 3D-printers: Cosmos Temp 3D (COS, Yller Biomateriais S.A., Pelotas, RS, Brazil); Smart Print Bio Temp (SMA, MM Tech Projetos Tecnológicos Ltda, São Carlos, SP, Brazil) and Resilab 3D Temp (RLB, Wilcos do Brasil Ltda, Petrópolis, RJ, Brazil). The fourth material is designed for subtractive manufacturing by milling processing (CAD/CAM): VitaCAD Temp (VIT, Vita Zahnfabrik, Bad Säckingen, Germany). Specifications about the composition, lot number, and shade of the tested products are presented in Table 1.

The 3D printed samples were designed using a 3D-figure processing software (MatterControl v.2.20.1.10422, MatterHackers, CA, USA) and exported to a printer slicer software (Chitubox 64, Chitu Systems, GD, China) using the manufacturer indicated parameters for exposure and off time. Layer height was set to 50 µm at 0° angulation for all the materials and experiments.[33] Specimens for the 3D-printed materials were manufactured using the same root STL files to ensure equal specimen characteristics and printed. Then, specimens were washed with isopropyl alcohol under agitation for 10 minutes and post cured with violet light (Wash and Cure 2.0, Anycubic Technology Co., Shenzen, China). For the milled resin, the samples were obtained from a CAD/CAM block (CTM-40) using a low-speed diamond- wafering blade (Isomet 1000 Precision Saw; Buehler Co., Lake Bluff, IL, USA) at 200 rpm with 150 g load.

Morphology of the surface of the resin

For the 3D-printed materials, three plates (5 mm length x 8 mm width x 1 mm height) were printed for each resin. For the milled resin, plates of the same dimensions were separated from a resin block. After all plates were prepared, one half of the plate was covered with isolating tape (Temflex 1700, 3M Electrical Markets Division, Austin, TX, USA) and the other half was treated by airborne particle abrasion (APA) using a dental air abrasion unit (Microetcher II, Danville Engineering, San Ramon, CA, USA) with alumina particles (50 µm, Bio-Art, São Carlos, SP, Brazil) perpendicular to the surface of the resin block, at 10 mm distance for 10 s at 0.2 MPa.[47,51] The samples were

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then ultrasonicated for 3 min in distilled water. The other half of the plate was left untreated (No air abrasion, NAA). Then, the resin plates were stored in a desiccator with silica gel for 24 hours before sputter coating with gold (Desk ll, Denton Vacuum Inc., NJ, EUA) and examined using an SEM (JSM IT 300; Jeol, Tokyo, Japan) at 400X magnification.

Microtensile bond strength (µTBS)

For each evaluated resin, 64 cubic-shaped samples (5x5x5 mm) were fabricated using the previously described equipment and procedures (Figure 1A). For each material, the obtained blocks were randomly divided into four groups (16 cubes per group, 1 pair of blocks per bonded specimen) according to the surface treatments, APA or NAA and two resin cements (Panavia V5, PAN, Kuraray, and Rely X Ultimate, REX, 3M Oral Care). For the 3D-printed blocks, the face of the cubes where the fabrication supports were attached was painted using a water-resistant varnish (Colorama, CEIL Ind. Ltda., São Paulo, SP), Brazil, and for the milled resin, one of the faces was randomly selected for painting. The side of the block opposing the painted face was treated with APA or not (NAA) (Figure 1B). Regardless the surface treatments (APA or NAA), the adhesive (for REX) or the primer (for PAN) were applied to provisional resins, followed by their respective resin cement.[52]

A pair of blocks that received the same surface treatment were used for bonding. To ensure adequate alignment of the resin blocks during cementation, one block was inserted into a heavy-body silicone matrix with drainage holes on each side of the silicone matrix to allow the exit of any excess cement. The matrix fitted the block snuggly, while leaving the bonding surface exposed. The resin cements were mixed, and a thin layer of cement was applied on the previously treated surfaces of the blocks (Figure 1C). A second block was placed into the silicone matrix, with the bonding faces of each block facing each other. After seating the block in position, a 5 N load was applied for 5 minutes before removing any excess cement.[46] Then, the cemented specimens were removed from the silicone matrix and complementary 20 s light curing cycles (Valo, Ultradent Products Inc., South Jordan, UT, 1060 mW/cm2 emittance) were applied on each side of the blocks. Excess of resin cement was removed from the cemented blocks, and they were stored in distilled water for 24 h at 37 ◦C (Figure 1D) and sectioned into approximately 1 × 1 mm specimens or sticks (cross sectional area of

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1 mm^2) using a low-speed diamond-wafering blade (Isomet 1000 Precision Saw; Buehler Co., Lake Bluff, IL, USA) at 200 rpm with 150 g load (Figure 1D).

For each block, approximately 16 stick were obtained and divided in two groups, one group was tested immediately, and the other half was stored in water for 7 days before the application of thermal cycling (OMC 300 TSX, Odeme Dental Research, Luzema, SC, Brazil) for 5,000 cycles (5 °C to 55°C, 30 s dwell time, 5 seconds transfer time) before µTBS testing (Figure 1E).[53,54] For the µTBS test, each stick was fixed to a custom microtensile testing device using cyanoacrylate glue (Super Bonder Power Flex, Loctite, São Paulo, SP Brazil) with an accelerator (Zap Zip Kicker, Pacer Technology, Ontario, CA, USA) (Figure 1E).[24,55] The device was placed in a Universal testing machine (EZ-test-500N, Shimadzu Co., Kyoto, Japan), and a tensile load (1 mm/min) was applied until failure (Figure 1G).[54] The sides of the sticks were measured with a digital caliper (Mitutoyo Corp., Kawasaki, Japan) to calculate the bonded area and for posterior calculation of the µTBS strength (MPa) from the load (N) at failure. The mean µTBS value of all the sticks obtained from the same cemented block was considered as the µTBS of the specimen. All measurements were performed by a trained operator, blinded to the group being tested.

Failure pattern analysis

For the failure pattern analysis, the fractured specimens were dried, sputter-coated with gold (Desk ll, Denton Vacuum Inc., NJ, EUA) and examined using SEM at 250X magnification (JSM IT 300; Jeol, Tokyo, Japan). The failure patterns were classified as: (1) Cohesive fracture within the resin cement, (2) Adhesive failure between the cement and the provisional restorative resin, (3) Mixed failure, and (4) Cohesive fracture within the provisional resin.[24]

Statistical analysis

For the µTBS analysis, pre-test failures were treated as left-censored data, and a value corresponding to the mean between 0 and the lowest measured value in the group was assigned to the stick.[54] The mean value of all the evaluated sticks from each block was considered as the µTBS of the specimen and used for statistical analysis. Data for µTBS was analyzed by Generalized linear model (between-subject factors: “Provisional Resin”*”Air-abrasion” *”Resin Cement”; between-subject factor: “Time”), and the Bonferroni method was used to correct for multiple testing ($p < 0.05$). For the failure

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pattern analysis, the incidence rate of each fracture type was calculated as a percentage for each group. The statistical analysis was performed using SAS 9.3 (SAS Institute, Cary, NC, USA).

Results

Analysis of the emission spectrum of the 3D-printer

Information about the emission spectrum the used DLP 3D-printer is presented in Figure 2. The emission of the printer ranges from 395 to 425 nm with a maximal peak of 408 nm. Hence, the emission spectrum corresponds mostly to violet light.

Surface Morphology

Representative images of the APA and NAA surface for each resin are presented in Figure 2. In general, the microphotograph images show evident differences between the NAA and APA regions for all resins. The APA areas of all materials present a rough, irregular morphology, characterized by the presence of groves and edges, although for VIT, the created defects appear shallower compared to the 3D printed resins, although there is a perceptible roughening of the surface. On the other hand, the NAA surfaces appear smooth and undamaged, both for the 3D-printed resins and the milled acrylic resin.

Microtensile Bond Strength

Mean μTBS values are reported in Table 2. The GLM analysis indicated that the quadruple interaction between factors “Provisional resin”*”Resin cement”*”Air abrasion”*”Time” was significant ($p < 0.001$). In general, 3D printed resins exhibited significantly higher bond strength than the milled resin VIT, regardless of the type of resin cement, the air abrasion treatment or not, and the evaluation time (24 h or thermocycled). For the 3D-printed resins, differences among resin cements were mostly material dependent. At the 24-hour evaluation REX produced a higher μTBS for COS without APA, while PAN was significantly higher for SMA combined with APA. After thermocycling, REX had significantly higher μTBS values for COS combined with APA, and for RSL without AA. For the other group comparisons there were no significant differences. The application of APA did not result in a clear trend of higher μTBS for the evaluated 3D-printed resins.

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Regarding VIT, the µTBS values were significantly higher at the 24-hour evaluation when REX was used for the APA treated group, and after thermocycling regardless of the APA treatment. It must be considered that VIT presented the highest amount of pre-test failures for all the evaluated materials, making impossible the measurement of the µTBS on the NAA group after thermal cycling when PAN was used. As for the effect of APA, the application of APA produced significantly higher µTBS values for VIT after 24 hours when REX was used, and for both resin cements after thermal cycling.

Failure pattern

The rate of incidence of each failure pattern, according to the material, resin cement, air abrasion and evaluation time are presented in Figure 3. Also, representative SEM images of each failure type are presented in Figure 4. Regardless of the resin cement and APA treatment, a higher rate of Type 2 failures (between resin and cement) was observed at both evaluation times. Regarding the APA groups, for PAN at the 24-hours evaluation, the rate of Type 4 failures (cohesive within provisional material) was higher than on the NAA groups. For VIT on the other hand, the most frequent type of failure where Type 2, and there were no Type 4 failures on any evaluated condition. Also, for all groups, the rate of Type 2 failures increased after thermal cycling.

Discussion

The results of this study showed that 3D printed resins are a clinically adequate material for long-term, temporary fixed restorations on esthetic regions, where adhesive cementation is required to obtain adequate retention and adaptation. Based on the findings of this study, the first null hypothesis was rejected because there were significant differences on the µTBS of the evaluated resins. In general, the 3D printed resins obtained a higher µTBS than the milled resin VIT, regardless of the APA procedure, resin cement used, and evaluation time. Also, despite the a few statistically significant differences between the 3D-printed resins, those differences are unlikely to be clinically relevant, because the evaluated materials exhibited a µTBS of approximately 20 MPa or higher regardless, of the type of resin cement, surface treatment and evaluation time, comparable to the values reported previously for indirect resin composites.[48] In this study, differences on the polymerization of the provisional

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restorative material are unlikely to affect the evaluated resins, because despite the limited penetrability of violet light,[18] each layer had a controlled thickness of only 50 µm, which was keep identical for all the materials. Also, the manufacturer of COS reports that it contains the type I photoinitiator known as Lucirin-TPO, which has an adequate absorption for light in the wavelength range of the selected printer.[18] It would be expected that the other 3D-printer resins present similar photoinitiators optimized for the emission spectrum of 3D-printers.[18]

On the other hand, comparison of the 3D-printed resins with the pre-polymerized milled material, showed that all the 3D-printed resins had a higher µTBS. This result could be explained by a joint copolymerization of the unreacted monomers on the 3D-printed resins with the monomers on the resin cements,[43] that does not occur on VIT. The evaluated 3D-printed resins are methacrylate-based materials and therefore, a high affinity between the unreacted monomers on the 3D-printed resins and those on the resin cement could be expected. On the other hand, VIT is a pre-polymerized block of high molecular weight, densely crosslinked acrylate polymers,[12] manufactured under high temperature and pressure conditions.[9] The block of VIT presents a very high degree of conversion and absence of photoinitiators, thus exhibiting little reactivity of residual monomers to copolymerize with the resin cement.[7] This could also explain why a previous study reported that debonding is a weak point of temporary restorations made from VIT.[10] This result is in line with previous studies that demonstrated bonding of indirect resin restorations strongly depends on the micro-retentions created by the APA treatment.[10,46,50]

The second null hypothesis was accepted because for all the evaluated material, the application of APA influenced the µTBS under some of the evaluated conditions. Traditional surface treatment of indirect resin restorations indicates using APA with alumina abrasive particles to create micro-mechanical retentions.[46] Hence, this study intended to determine if this principle also applies to 3D-printed restorations, or if the use of resin cements combined with primers containing functional monomers could result in adequate bond strength between the restorative resin and the resin cement. For the 3D-printed resins, when PAN was used, APA only produced a significant increase on the µTBS of COS at the 24-hour evaluation and for RSL after thermal-cycling. For the other 3D-printed resins, there were no differences regardless of the evaluation time.

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On the other hand, when REX was used, SMA presented a higher µTBS on the NAA groups at both evaluation times.

For the other 3D-printed resins, there were no significant differences between the APA and the NAA protocols. These results corroborated with a previous study, where the application of mechanical treatment on the surface of 3D-printed resins did not increase the shear bond strength of acrylic and bis-acrylic resins.[43] For VIT, the application of APA significantly improved the µTBS to both resin cements, especially after thermal aging. As mentioned before, bonding to pre-polymerized, milled resins heavily depends on the creation of intricate mechanical interlocking between the restorative material and the cementing agent.[50] However, the findings of this study do not support the application of APA as a standard surface conditioning of 3D-printed resins for adhesive cementation. Although the analysis of the SEM micrographs demonstrated notorious differences between the APA and NAA surfaces of all the evaluated resins, the roughening of the surface produced by APA did not translate into a remarkably higher µTBS on the 3D-printed resins. Also, it is important to highlight that for the 3D-printed resins, there was a higher rate of type 4 fractures (cohesive fracture within the provisional resin) on the APA treated samples, compared to the NAA groups. As observed in the SEM images, these failures were characterized by the separation of the printed layers within the sample, thus suggesting that the tensile strength of the material was surpassed.

For 3D-printed resins, several factors such as the layer thickness,[15,31] specimen angulation,[22,27,29,33] as well as the washing[28,38] and post curing,[23,27,30,37] protocols may weaken the cohesivity of the printed specimen and affect the truthfulness of the µTBS evaluation. Because the samples in this study were printed at 0° angulation, the layers on the blocks were perpendicular to the applied load.[33] This could have favored the incidence of type 4 failures, because of the delamination between the printed layers, that at 0° angulation have the smallest possible contact area. Hence, printing at a different angulation might be recommendable for restorations that will be subjected to tensile load because the applied forces will be directed in a more favorable direction[16,32] and there will be a greater contact area between the printed layers.[32] Further research addressing the influence of the build angle on the ultimate tensile strength of 3D-printed resins is required to confirm this supposition. Nonetheless, the obtained results confirm that there is not a clear benefit on the application of APA for the adhesive cementation of

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temporary 3D-printed restorations, because it could produce superficial damage to the 3D-printed restoration, without significantly improving its bond strength.

The third research hypothesis was rejected because significant differences were identified between the resin cements. The observed differences; however, are inconclusive. On the NAA groups, REX presented a higher μTBS than PAN for COS at the 24-hours evaluation. For the APA treated groups, REX produced a higher μTBS for RSL and VIT after thermal cycling. The more stable union between VIT and REX compared to PAN, may be produced by the chemical compatibility between the silane contained on the universal bonding agent and the silicon particles contained on the resin block.[49] Also, for the 3D-printed resins, co-polymerization with the adhesive and the resin cement may explain the maintained μTBS values. On the other hand, the absence of dental tissues led to a modification on the application mode for PAN, by applying Tooth Primer on the surface of one of the resin blocks. Although the Tooth Primer is intended to be placed on dental tissues, this primer also contains an accelerator for the self-curing reaction of PAN, and for that reason it was applied to ensure that the resin cement would be evaluated under the most adequate polymerization conditions.[52] However, the acidic pH of the primer is not neutralized on the absence of ions from the tooth and may affect the long term performance of the resin cement, by inhibiting the catalytic components in charge of the post-cure reaction on the resin cement.[24–26]

The fourth and final hypothesis was upheld for the 3D-printed resins, and rejected for the milled material, because thermal cycling produced differences on the μTBS of VIT. The results showed that for the 3D-printed resins, thermal cycling did not result in significant differences on the μTBS when REX was used. For VIT, the μTBS decreased on the NAA groups with both evaluated resin cements. It has been proposed that thermal cycling is a useful tool to predict the mode of failure of a material. On that regard, the findings of this study showed that thermal cycling produced an increase on the rate of Type 2 failures, on all the evaluated materials, which was confirmed by the increased number of pre-test failures. Also, despite the application of a statistical compensation, the higher number of pre-test failures may have influenced the μTBS results after thermal cycling, because a reduced number of sticks was evaluated compared to the 24-hour evaluation. Considering that the specimens with the weaker μTBS are more prone to failure, those exhibiting a higher μTBS may have survived the thermal cycling process

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and artificially overestimated the bond strength of the resin cements to the provisional restorative material.

It must be considered that even though a long-term evaluation of the μTBS of provisional restorative materials may not seem as relevant as for definitive restorations, the obtained results can be used to estimate the predictability of long term temporary fixed restorations. Although clinically it would be uncommon to bond temporary restorations, there are clinical scenarios that require long-term, fixed temporization, such as changes on the vertical dimension,[10] unclear prognosis for teeth before a definitive complex rehabilitation,[10] and bone and tissue regeneration before implant placement.[3] Also, the evaluation of the μTBS of resin cements to a recently developed kind of 3D printed, temporary restorative materials is important to provide validated information and avoid unnecessary steps on the cementation procedure.

Conclusions

Based on the findings of this study, there were no significant differences on the TBS of the 3D printed resins to the evaluated resin cements, while the milled acrylic resins presented lower bond strength values.

Also, specific surface treatment procedures are required for 3D-printed and milled resins, because of differences in the material manufacturing process. Hence, a combination of APA and adhesive/primer is advised to obtain a durable bonding of temporary, milled restorations. On the other hand, airborne particle abrasion does not result in a significant benefit for the cementation of 3D-printed, fixed provisional restorations.

Temporary 3D printed resins showed similar TBS to the evaluated cements and were resistant to thermal aging. Conversely, the milled 3D resin showed a significant decrease on the TBS after thermal aging.

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References

1. Gratton DG, Aquilino SA. Interim restorations. Dent Clin North Am. 2004;48(2):487–97.

2. Miura S, Fujisawa M, Komine F, Maseki T, Ogawa T, Takebe J, et al. Importance of interim restorations in the molar region. J Oral Sci. 2019;61(2):195–9.

3. Tarnow D, Chu S, Salama M, Stappert C, Salama H, Garber D, et al. Flapless Postextraction Socket Implant Placement in the Esthetic Zone: Part 1. The Effect of Bone Grafting and/or Provisional Restoration on Facial-Palatal Ridge Dimensional Change—A Retrospective Cohort Study. Int J Periodontics Restorative Dent. 2014;34(3):323–31.

4. Winter A, Erdelt K, Giannakopoulos N, Schmitter M, Edelhoff D, Liebermann A. Impact of Different Types of Dental Prostheses on Oral Health–Related Quality of Life: A Prospective Bicenter Study of Definitive and Interim Restorations. Int J Prosthodont. 2021;34(4):441–7.

5. Hahnel S, Scherl C, Rosentritt M. Interim rehabilitation of occlusal vertical dimension using a double-crown-retained removable dental prosthesis with polyetheretherketone framework. J Prosthet Dent . 2018;119(3):315–8. https://doi.org/10.1016/j.prosdent.2017.02.017

6. Peng M, Li C, Huang C, Liang S. Digital technologies to facilitate minimally invasive rehabilitation of a severely worn dentition: A dental technique. J Prosthet Dent. 2021;126(2):167–72. https://doi.org/10.1016/j.prosdent.2020.05.012

7. Skorulska A, Piszko P, Rybak Z, Szymonowicz M, Dobrzinski M. Review on Polymer, Ceramic and Composite Materials for CAD/CAM Indirect Restorations in Dentistry—Application, Mechanical Characteristics and Comparison. Materials

.

(Basel). 2021;14(1592).

8. Ruse ND, Sadoun MJ. Resin-composite blocks for dental CAD/CAM applications. J Dent Res. 2014;93(12):1232–4.

9. Mainjot AK, Dupont NM, Oudkerk JC, Dewael TY, Sadoun MJ. From Artisanal to CAD-CAM Blocks: State of the Art of Indirect Composites. J Dent Res. 2016;95(5):487–95.

10. Huettig F, Prutscher A, Goldammer C, Kreutzer CA, Weber H. First clinical experiences with CAD/CAM-fabricated PMMA-based fixed dental prostheses as long-term temporaries. Clin Oral Investig. 2016;20(1):161–8.

11. Çagri Ural, Burgaz Y, Saraç D. In vitro evaluation of marginal adaptation in five. Quintessence Int (Berl). 2010;41(7):585–90.

12. Başaran EG, Ayna E, Vallittu PK, Lassila LVJ. Load bearing capacity of fiber-reinforced and unreinforced composite resin CAD/CAM-fabricated fixed dental prostheses. J Prosthet Dent. 2013;109(2):88–94.

13. da Silva LH, de Lima E, Miranda RB de P, Favero SS, Lohbauer U, Cesar PF, et al. Dental ceramics: a review of new materials and processing methods. Braz Oral Res. 2017;31(Suppl e58):133–46. https://doi.org/10.1590/1807-3107BOR-2017.vol31.0058

14. Della Bona A, Cantelli V, Britto VT, Collares KF, Stansbury JW. 3D printing restorative materials using a stereolithographic technique: a systematic review. Dent Mater. 2021;37(2):336–50. https://doi.org/10.1016/j.dental.2020.11.030

15. Kessler A, Hickel R, Reymus M. 3D printing in dentistry-state of the art. Oper Dent. 2020;45(1):30–40.

.

16. Alsandi Q, Ikeda M, Arisaka Y, Nikaido T, Tsuchida Y, Sadr A, et al. Evaluation of mechanical and physical properties of light and heat polymerized udma for dlp 3d printer. Sensors. 2021;21(10):1–10.

17. Lin CH, Lin YM, Lai YL, Lee SY. Mechanical properties, accuracy, and cytotoxicity of UV-polymerized 3D printing resins composed of Bis-EMA, UDMA, and TEGDMA. J Prosthet Dent. 2020;123(2):349–54. https://doi.org/10.1016/j.prosdent.2019.05.002

18. Palin WM, Leprince JG, Hadis MA. Shining a light on high volume photocurable materials. Dent Mater. 2018;34(5):695–710. https://doi.org/10.1016/j.dental.2018.02.009

19. Wilson NHF, Lynch CD. The teaching of posterior resin composites: Planning for the future based on 25 years of research. J Dent. 2014;42(5):503–16. http://dx.doi.org/10.1016/j.jdent.2014.02.014

20. Tahayeri A, Morgan MC, Fugolin AP, Bompolaki D, Athirasala A, Pfeifer CS, et al. 3D printed versus conventionally cured provisional crown and bridge dental materials. Dent Mater. 2018;34(2):192–200. http://dx.doi.org/10.1016/j.dental.2017.10.003

21. Osman R, Alharbi N, Wismeijer D. Build Angle: Does It Influence the Accuracy of 3D-Printed Dental Restorations Using Digital Light-Processing Technology? Int J Prosthodont. 2017;30(2):182–8.

22. Revilla-León M, Jordan D, Methani MM, Piedra-Cascón W, Özcan M, Zandinejad A. Influence of printing angulation on the surface roughness of additive manufactured clear silicone indices: An in vitro study. J Prosthet Dent. 2020;1–7. https://doi.org/10.1016/j.prosdent.2020.02.008

.

23. Reymus M, Stawarczyk B. In vitro study on the influence of postpolymerization and aging on the Martens parameters of 3D-printed occlusal devices. J Prosthet Dent. 2020;125(5):817–23. https://doi.org/10.1016/j.prosdent.2019.12.026

24. Soto-Montero J, Nima G, Dias CTDS, Price RBT, Giannini M. Influence of beam homogenization on bond strength of adhesives to dentin. Dent Mater. 2021;37(2):e47–58. https://doi.org/10.1016/j.dental.2020.10.003

25. Bedran-Russo A, Leme-Kraus AA, Vidal CMP, Teixeira EC. An Overview of Dental Adhesive Systems and the Dynamic Tooth–Adhesive Interface. Vol. 61, Dental Clinics of North America. 2017. p. 713–31.

26. Sanares AME, Itthagarun A, King NM, Tay FR, Pashley DH. Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites. Dent Mater. 2001;17(6):542–56.

27. Reymus M, Fabritius R, Keßler A, Hickel R, Edelhoff D, Stawarczyk B. Fracture load of 3D-printed fixed dental prostheses compared with milled and conventionally fabricated ones: the impact of resin material, build direction, post-curing, and artificial aging—an in vitro study. Clin Oral Investig. 2020;24(2):701–10.

28. Mayer J, Stawarczyk B, Vogt K, Hickel R, Edelhoff D, Reymus M. Influence of cleaning methods after 3D printing on two-body wear and fracture load of resin-based temporary crown and bridge material. Clin Oral Investig. 2021;

29. Revilla-León M, Meyers MJ, Zandinejad A, Özcan M. A review on chemical composition, mechanical properties, and manufacturing work flow of additively manufactured current polymers for interim dental restorations. J Esthet Restor Dent. 2019;31(1):51–7.

.

30. Kim D, Shim JSJS, Lee D, Shin SH, Nam NE, Park KH, et al. Effects of post-curing time on the mechanical and color properties of three-dimensional printed crown and bridge materials. Polymers (Basel). 2020;12(11):1–20.

31. Park SM, Park JM, Kim SK, Heo SJ, Koak JY. Flexural strength of 3D-printing resin materials for provisional fixed dental prostheses. Materials (Basel). 2020;13(18):1–14.

32. Keßler A, Hickel R, Ilie N. In vitro investigation of the influence of printing direction on the flexural strength, flexural modulus and fractographic analysis of 3D-printed temporary materials. Dent Mater J. 2021;

33. Alharbi N, Osman R, Wismeijer D. Effects of build direction on the mechanical properties of 3D-printed complete coverage interim dental restorations. J Prosthet Dent. 2016;115(6):760–7. http://dx.doi.org/10.1016/j.prosdent.2015.12.002

34. Revilla-león M, Morillo JA, Att W, Özcan M. Chemical Composition, Knoop Hardness, Surface Roughness, and Adhesion Aspects of Additively Manufactured Dental Interim Materials. J Prosthodont. 2020;0:1–8.

35. Grzebieluch W, Kowalewski P, Grygier D, Rutkowska-Gorczyca M, Kozakiewicz M, Jurczyszyn K. Printable and machinable dental restorative composites for CAD/CAM application—comparison of mechanical properties, fractographic, texture and fractal dimension analysis. Materials (Basel). 2021;14(17).

36. Simoneti DM, Pereira-Cenci T, dos Santos MBF. Comparison of material properties and biofilm formation in interim single crowns obtained by 3D printing and conventional methods. J Prosthet Dent. 2020;1–5. https://doi.org/10.1016/j.prosdent.2020.06.026

37. Perea-Lowery L, Gibreel M, Vallittu PK, Lassila L. Evaluation of the mechanical

.

properties and degree of conversion of 3D printed splint material. J Mech Behav Biomed Mater. 2021;115(November 2020):104254. https://doi.org/10.1016/j.jmbbm.2020.104254

38. Mayer J, Reymus M, Wiedenmann F, Edelhoff D, Hickel R, Stawarczyk B. Temporary 3D printed fixed dental prosthesis materials: Impact of post printing cleaning methods on degree of conversion as well as surface and mechanical properties. Int J Prosthodont. 2021;1–29.

39. Revilla-León M, Umorin M, Özcan M, Piedra-Cascón W. Color dimensions of additive manufactured interim restorative dental material. J Prosthet Dent. 2020;123(5):754–60. https://doi.org/10.1016/j.prosdent.2019.06.001

40. Choi JW, Ahn JJ, Son K, Huh JB. Three-dimensional evaluation on accuracy of conventional and milled gypsum models and 3D printed photopolymer models. Materials (Basel). 2019;12(21):1–10.

41. Nestler N, Wesemann C, Spies BC, Beuer F, Bumann A. Dimensional accuracy of extrusion- and photopolymerization-based 3D printers: In vitro study comparing printed casts. J Prosthet Dent. 2021;125(1):103–10. https://doi.org/10.1016/j.prosdent.2019.11.011

42. Kim J, Lee DH. Influence of the Postcuring Process on Dimensional Accuracy and Seating of 3D-Printed Polymeric Fixed Prostheses. Biomed Res Int. 2020;2020.

43. Lim N-K, Shin S-Y. Bonding of conventional provisional resin to 3D printed resin: the role of surface treatments and type of repair resins. J Adv Prosthodont. 2020;12(5):322.

44. Jeong KW, Kim SH. Influence of surface treatments and repair materials on the shear bond strength of CAD/CAM provisional restorations. J Adv Prosthodont.

.

2019;11(2):95–104.

45. Zalkind M, Hochman N. Laminate veneer provisional restorations: A clinical report. J Prosthet Dent. 1997;77(2):109–10.

46. Soares CJ, Giannini M, de Oliveira MT, Paulillo LAMS, Martins LRM. Effects of surface treatments of laboratory-fabricated composites on the microtensile bond strength to a luting resin cement. J Appl Oral Sci. 2004;12(1):45–50.

47. Spitznagel FA, Horvath SD, Guess PC, Blatz MB. Resin bond to indirect composite and new ceramic/polymer materials: A review of the literature. J Esthet Restor Dent. 2014;26(6):382–93.

48. Makishi P, André CB, Silva JPLE, Bacelar-Sá R, Correr-Sobrinho L, Giannini M. Effect of storage time on bond strength performance of multimode adhesives to indirect resin composite and lithium disilicate glass ceramic. Oper Dent. 2016;41(5):541–51.

49. Tokunaga E, Nagaoka N, Maruo Y, Yoshihara K, Nishigawa G, Minagi S. Phosphate group adsorption capacity of inorganic elements affects bond strength between cad/cam composite block and luting agent. Dent Mater J. 2021;40(2):288–96.

50. Nagasawa Y, Eda Y, Shigeta H, Ferrari M, Nakajima H, Hibino Y. Effect of sandblasting and/or priming treatment on the shear bond strength of self-adhesive resin cement to CAD/CAM blocks. Odontology. 2021;(0123456789):14–7. https://doi.org/10.1007/s10266-021-00635-y

51. D'Arcangelo C, Vanini L. Effect of three surface treatments on the adhesive properties of indirect composite restorations. J Adhes Dent.2007;9(3):319–26. http://www.ncbi.nlm.nih.gov/pubmed/17655072

.

52. de Araújo Neto VG, Soto-Montero J, de Castro EF, Feitosa VP, Rueggeberg FA, Giannini M. Effects of shades of a multilayered zirconia on light transmission, monomer conversion, and bond strength of resin cement. J Esthet Restor Dent. 2021;(March):1–11.

53. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent. 1999;27(2):89–99.

54. Armstrong S, Breschi L, Ozcan M, Pfefferkorn F, Ferrari M, Van Meerbeek B, et al. Academy of Dental Materials guidance on in vitro testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (TBS) approach. Dent Mater. 2017;33(2):133–43. http://dx.doi.org/10.1016/j.dental.2016.11.015

55. Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, et al. Influence of three specimen fixation modes on the micro-tensile bond strength of adhesives to dentin. Dent Mater J. 2007;26(5):694–9.

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Figures

Figure 1. Schematic representation of the sample preparation for the µTBS test. A- 5 x 5 x 5 mm side, resin blocks where either 3D-printed or cut from a pre-polymerized block; B- The obtained cubes were divided into groups and the bonding surface was treated by airborne particle abrasion or left untreated according to the corresponding treatment; C- The corresponding primer and resin cement were applied on the bonding surface, and a second block was placed over the resin cement layer; D- The obtained cemented blocks were stored in water for 24 hours prior to µTBS stick preparation; E- 1 x 1 mm side, µTBS stick specimens were obtained from the cemented resin blocks and divided in two different groups according to the time of evaluation (24 hours or thermocycling); and F- The specimens were fixed in a testing jig using cyanoacrylate glue and tested under tensile load.

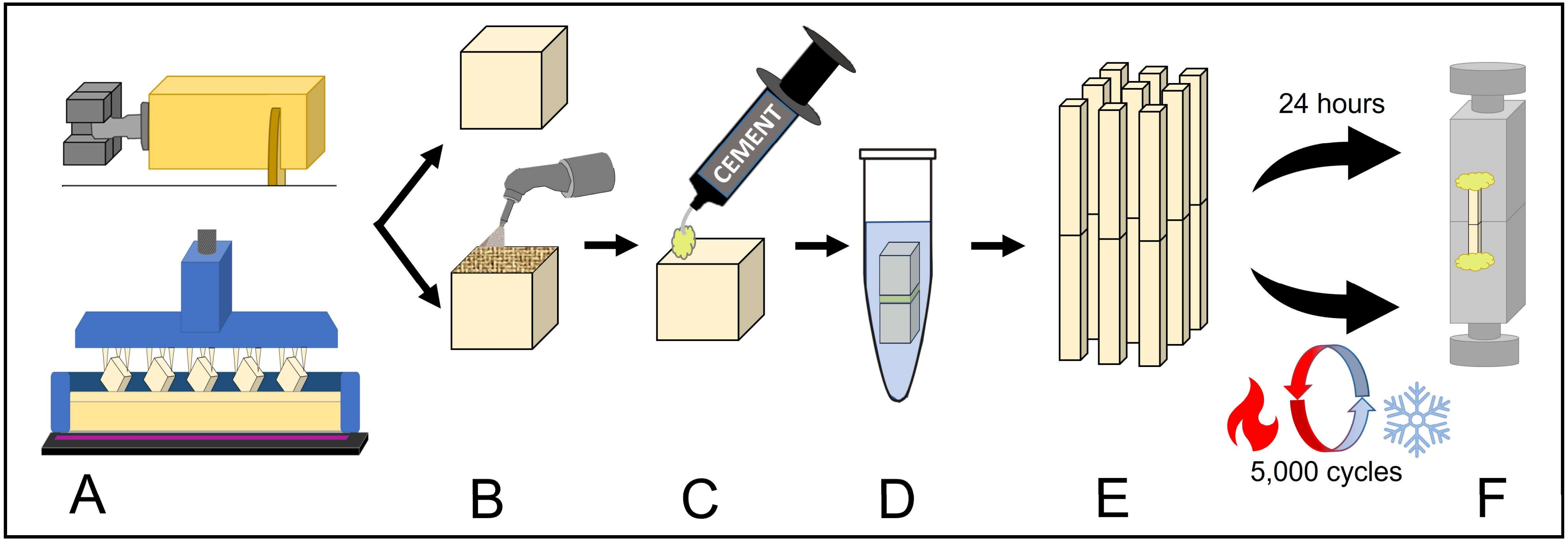

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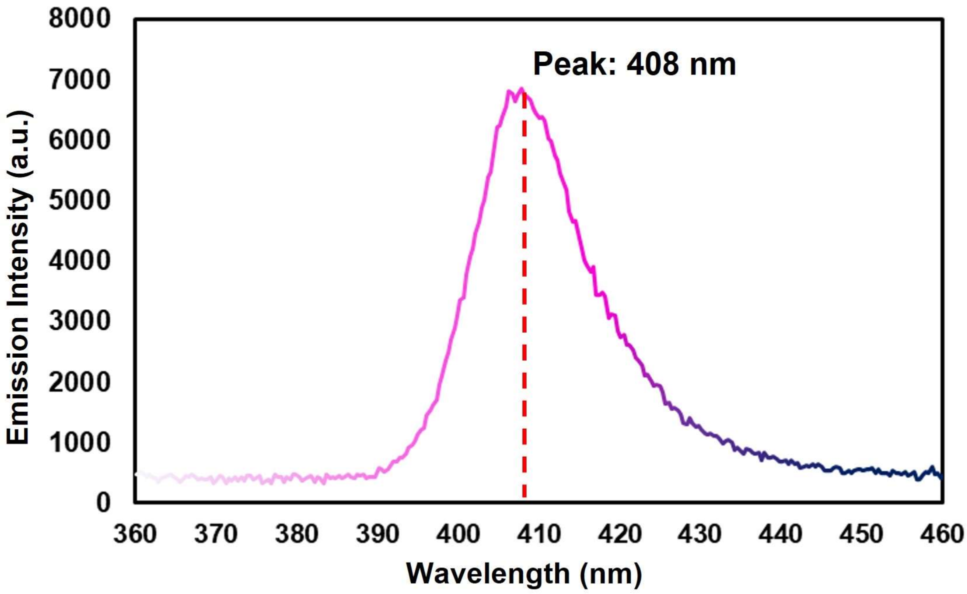
Figure 2. Spectral emission and maximal emission peak of the used 3D-printer.



Abbreviation: a.u.: arbitrary units.

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Figure 3. Representative SEM images (x250 magnification) of the restorative resins reveal different textures between the air abraded and the non-air abraded surfaces. The air abraded resin surfaces present an irregular morphology, characterized by the presence of groves and edges, while the non-air abraded surfaces exhibit a smooth surface.

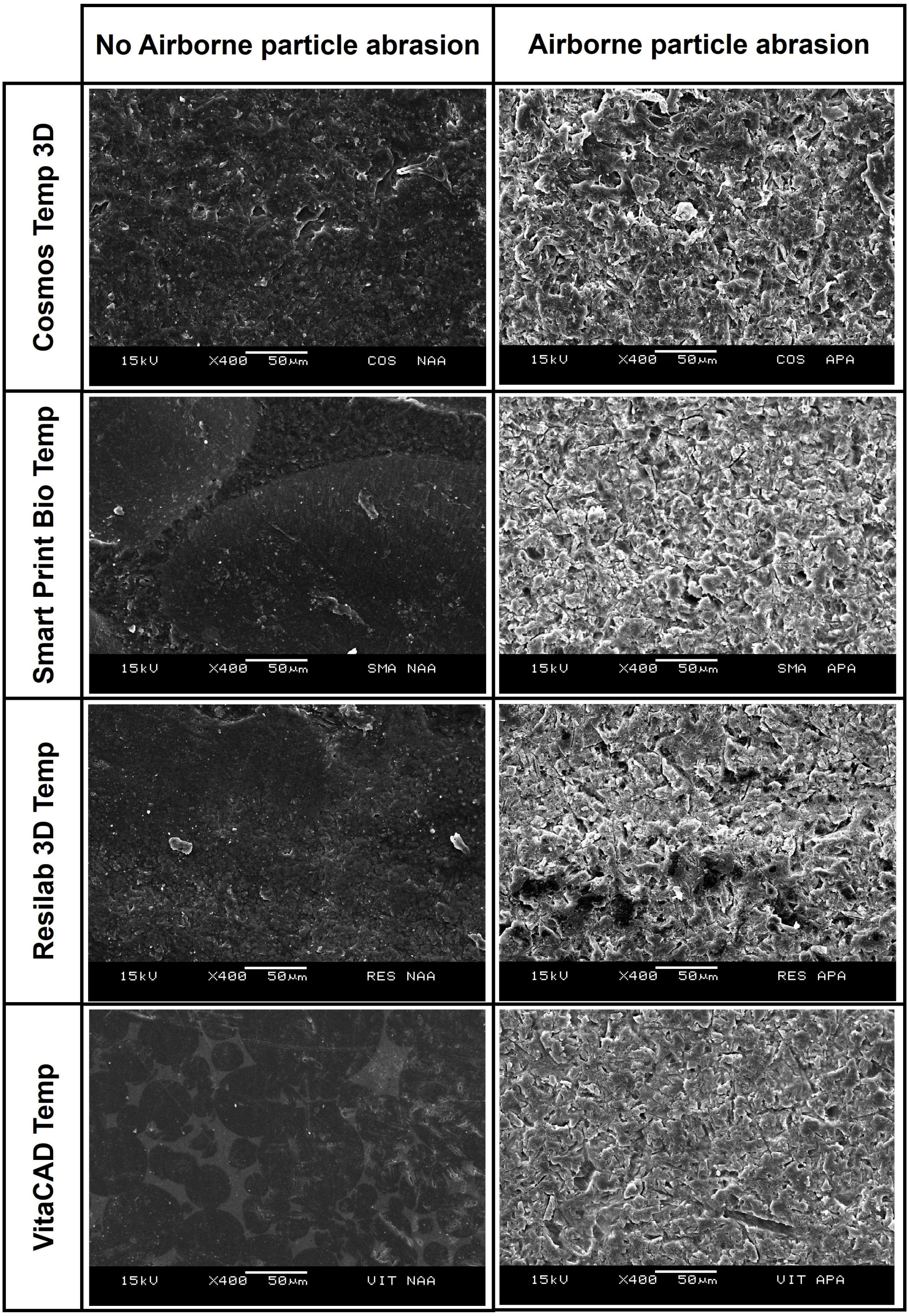

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Figure 4. Distribution of failure modes after 24-h and 5,000 cycles of thermal aging, according to airborne particle abrasion treatment A- for Panavia V5, and B- for RelyX Ultimate.

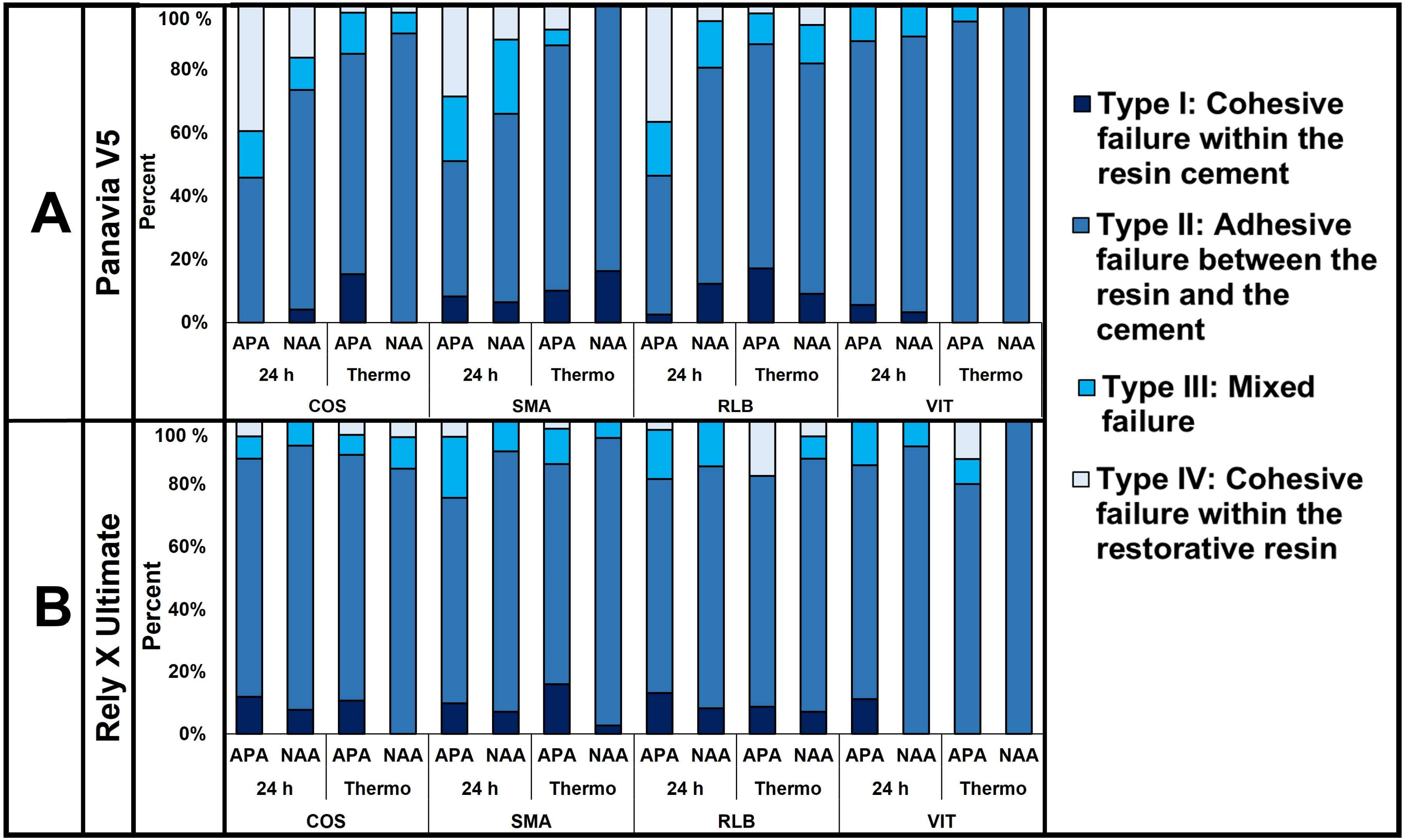

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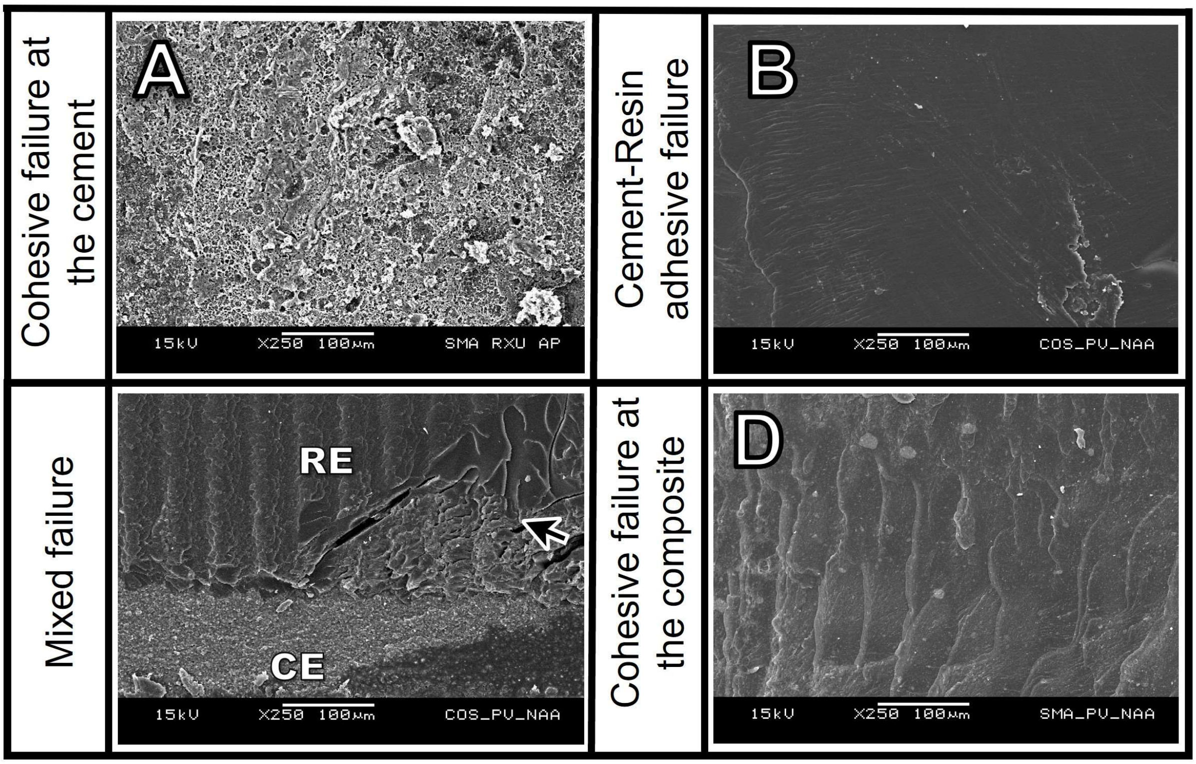
Figure 5. Representative SEM images of each failure type. A- Type 1: Cohesive failure at the resin cement. The arrow indicates areas where the filler particles can be observed, and the surface of the restorative resin is completely covered by remaining resin cement (Air abraded, Smart Print Bio Temp and Rely X Ultimate, after 24 hours); B- Type 2: Adhesive failure between the resin cement and the temporary resin. The image shows the smooth surface of the temporary resin without any reminiscent resin cement over the surface (Non-abraded, Cosmos 3D Temp and Panavia V5 after thermal cycling) C- Type 3: Mixed failure. The image shows the fractured resin cement layer, and the exposed surface of the temporary resin. Also, the pointer indicates the presence of areas where separation of the layers of the temporary resin occurred. (Non-abraded, Cosmos 3D Temp and Panavia V5 after 24 hours); D- Type 4: Cohesive failure within restorative resin. The pointer indicates the presence of fracture lines within the layers of the temporary resin, where delamination is visible (Non-abraded, Smart Print Bio Temp and Panavia V5 after 24 h).



Abbreviations: CE: Resin Cement; RE: Restorative resin

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Table 1. Classification, brand names, lot number, compositions, and shade of the evaluated materials.

Classification	Brand name (Abbreviations) and Lot number	Composition	Shade
3D Printed resin	Cosmos Temp 3D (COS) Lot 00008288	Methacrylate oligomers, diphenyl-2,4,6-trimethylbenzoyl phosphine oxide (TPO), titanium dioxide, carbon black.	A1
	Smart Print Bio Temp (SMA) Lot PTPB1010/20	Methacrylic ester monomers, stabilizer, fillers, pigments, photoinitiators, accelerators.	B1
	Resilab 3D Temp (RES) Lot 1417	Information not avaliable	A1
Milled resin	VitaCAD Temp (VIT) Lot 48000	Poly(methyl methacrylate), silicon dioxide, pigments	1 M2T
Resin Cement	Panavia V5 (PAN) Lot 450053	Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass, Silanated fluoroalminosilicate glass Paste B: Colloidal silica, Bis-GMA, Hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass, Silanated aluminum oxide, accelerators, camphorquinone, pigments	A1
	RelyX Ultimate (REX) Lot 7405361	Base Paste: Methacrylate monomers, radiopaque silanated fillers, initiator components, stabilizers, rheological additives Catalyst Paste: Methacrylate monomers, Radiopaque alkaline fillers, initiator components, stabilizers, pigments, rheological additives, fluorescence dye, Dual cure activator for Universal Adhesive.	A1

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Table 2 Mean (95 % C.I.) microtensile bond strength of evaluated resins, according to evaluation time, resin cement (Panavia V5 or RelyX Ultimate), and airborne particle abrasion treatment, in MPa.

Time of measurement	Restorative Resin	Resin cement			
		Panavia V5		RelyX Universal	
		APA	NAA	APA	NAA
24 hours of water storage	COS	27.5 (24.4 – 30.6) aA [0]	21.0 (18.5 – 23.6) bB [1]	27.3 (24.2 – 30.4) aA [0]	29.5 (26.2 – 32.8) aA [1]
	SMA	28.2 (25.0 – 31.4) aA [0]	27.1 (24.0 – 30.1) aA [1]	20.0 (17.6 – 22.5) bB [0]	29.1 (25.8 – 32.3) aA [2]
	RES	25.4 (22.5 – 28.3) aA [0]	23.9 (21.1 – 26.7) abA [0]	27.3 (24.0 – 31.1) aA [0]	27.2 (24.1 – 30.3) aA [0]
	VIT	8.6 (7.3 – 9.9) bB [1]	8.5 (7.2 – 9.8) cA [2]	14.6 (12.6 – 16.5) cA [1]	3.8 (3.1 – 4.5) bB [3]
5,000 Thermal cycles	COS	21.4 (18.8 – 24.0) bB* [1]	22.4 (19.7 – 25.0) aA [1]	31.0 (26.7 – 33.3) aA [1]	25.9 (22.9 – 28.9) aA [0]
	SMA	26.9 (23.8 – 29.9) aA [0]	25.2 (22.3 – 28.1) aA [0]	22.5 (19.8 – 25.1) bA [1]	26.9 (23.8 – 29.9) aA [0]
	RES	30.9 (27.5 – 34.3) aA* [1]	22.2 (19.6 – 24.9) aB [0]	31.2 (27.8 – 34.7) aA [1]	27.4 (24.3 – 30.5) aA [0]
	VIT	7.1 (6.0 – 8.2) cB [3]	0.0 (0.0 – 0.0) bB* [8]	15.6 (13.5 – 17.6) cA [2]	1.8 (1.4 – 2.2) bA* [5]

Lower case letters compare restorative resins within the same treatment, resin cement and time. Upper case letters compare different resin cement within the same restorative resin, treatment, and time. Connective bars indicate significant different between treatments within the same resin composite, resin cement and time. (*) Differ from 24h within the same resin composite, resin cement and treatment. Values between [ ] indicate the number of pre-test failures for the group.

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3. DISCUSSÃO

Os resultados deste estudo demonstram que o tempo de pós-cura pode afetar a cor e as propriedades mecânicas das resinas para restaurações provisórias manufaturadas em impressoras 3D. Portanto, é necessário ajustar detalhadamente o tempo de exposição à luz de pós-cura, para obter um equilíbrio adequado entre estética e resistência mecânica em restaurações impressas em 3D. Um dos mais relevantes achados, está em que tempos de pós-cura razoáveis para fabricação de provisórios no consultório, conseguem produzir propriedades mecânicas adequadas, em contraste com outros estudos em que foram aplicados tempos de até 2 horas de pós cura.(D. Kim et al., 2020) No relativo à alteração da cor, apesar de ter sido encontrados alguns valores estatisticamente significantes de ΔE_{00}, a alteração da cor com até 20 minutos de pós-cura foi aceitável para algumas das resinas avaliadas, segundo o parâmetro relatado para resinas temporárias impressas. (Revilla-León, Umorin, et al., 2020)

No entanto, foi comprovado que a alteração da cor é material dependente e existe uma severa falta de padronização nos parâmetros de perceptibilidade e aceitabilidade para a alteração de cor em resinas provisórias, por isso é recomendável fazer estudos com metodologias adequadas e que incluam condições clínicas. Existem vários limites de aceitabilidade propostos para alteração de cor em materiais dentários; no entanto, a maioria deles foi obtida usando cerâmicas (Paravina et al., 2015) ou resinas compostas (Rocha et al., 2019; Salas et al., 2018) as quais apresentam propriedades ópticas e de superfície diferentes das resinas impressas em 3D para restaurações provisórias. Também, outro estudo que avaliou as alterções de cor em resinas para impressão 3D não explica como foram estabelecidos os critérios de avaliação e aceitabilidade. (D. Kim et al., 2020) Portanto, apesar das limitações metodológicas, os parâmetros de aceitabilidade estabelecidos em um estudo anterior usando materiais semelhantes, condições de avaliação e ferramenta de medição foram selecionados para manter a comparabilidade entre os resultados. (Revilla-León, Umorin, et al., 2020)

O efeito da polimerização ns propriedades mecânicas como KHN e FS de materiais resinosos já foi amplamente estudado. (Bouschlicher et al., 2004; B. M. Fronza et al., 2017; Bruna Marin Fronza et al., 2015; Mendonça, Soto-Montero, de Castro, et al., 2021; Rueggeberg et al., 2009) É sabido que a polimerização é reduzida nas regiões mais internas do material, e isso afeta a rigidez, força e resistência às forças mastigatórias de restaurações provisórias.(Reymus et al., 2020; Reymus & Stawarczyk, 2020b) Este estudo avaliou o efeito da profundidade na microdureza das resinas impressas, confirmando que o centro do corpo de prova apresenta dureza menor que as camadas externas. No entanto, não foi possível avaliar a resistencia e módulo flexurais em profundidade devido a limitações metodológicas, como o tamanho e formato das amostras para o teste de flexão em 3 pontos.(Mendonça, Soto-Montero, Castro, et al., 2021) Embora foi observado que aumentar o tempo de exposição à luz de pós-cura pode compensar uma baixa profundidade de cura, fatores como a alteração da cor dos materiais podem limitar a possibilidade de pós-cura prolongada.

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Além disso, é recomendável tomar precauções para evitar ou reduzir os ajustes oclusais em restaurações impressas, porque as camadas externas mais duras podem ser removidas, expondo a resina interna, que apressentam menor resistência às forças oclusais. Finalmente, apesar das restaurações indiretas como coroas dificilmente excederem 2 mm de espessura em situações clinicas, o padrão de microdureza encontrado pode ser importante para outro tipo de restaurações mais espessas e volumosas, como onlays ou próteses adesivas, pois o núcleo da restauração apresentaria propriedades mecânicas inferiores às das áreas diretamente expostas à luz. Contudo, foi encontrado que do ponto de vista de retenção, a cimentação adesiva deste tipo de restaurações, fabricadas com resina em impressora 3D aparece como uma opção viável, porque apressentaram uma resistência de união por microtração maior do que a de uma resina para fabricação por fresagem, em todas as condições avaliadas.

Apesar desse resultado promissor, não foi possível encontrar estudos clínicos que avaliassem a performance clínica de restaurações fixas fabricadas com impressoras 3D de tecnologia SLA ou DLP e a evidência disponível consiste unicamente de escassos e isolados relatos de casos clínicos.(Katreva et al., 2018; Nour et al., 2021) Torna-se evidente então, a necessidade de efetuar estudos clínicos que permitam validar o uso destas tecnologias na fabricação de restaurações provisórias fixas de longa duração, principalmente em condições que dependem da adesão do cimento ao substrato para dar retenção à restauração, como em próteses adesivas, facetas ou onlays. A importância de validar o uso das resinas impressas aumenta especialmente quando é considerado que o material pré-polimerizado para fresagem apressentou uma resistência de união significativamente mais baixa à das resinas impressas. Acredita-se, que as resinas impressas são favorecidas pela copolimerização dos monômeros não reagidos na restauração com os monômeros do cimento resinoso.(Lim & Shin, 2020) Destaca-se também que nas resinas para impressão 3D houve poucas diferenças significativas entre os grupos tratados com jateamento com alumina e sem jateamento. Estudos prévios já relataram que o tratamento mecânico com jateamento de partículas na superfície de resinas impressas não aumentou a resistência de união a outros materiais resinosos.(Albahri et al., 2021; Lim & Shin, 2020) Os resultados obtidos confirmaram que não há um benefício evidente da aplicação de jateamento com partículas abrasivas de óxido de alumínio prévio à cimentação adesiva na resistência de união de restaurações provisórias impressas, e que pelo contrário, este procedimento pode danificar a superfície do material.

Como o material pré-polimerizado apresenta uma rede polímeros de acrilato de alto peso molecular, densamente reagidos,(Başaran et al., 2013) e fabricados em condições de alta pressão e temperatura, é observada uma baixa reatividade da superfície da restauração para limita a interação com o cimento resinoso, pois o material tem alto grau de conversão e ausência de fotoiniciadores.(Skorulska et al., 2021) A baixa reatividade do material pré-polimerizado pode explicar também, os resultados obtidos por estudos prévios que reportaram a retenção como o ponto fraco das restaurações fresadas de resina e indicaram que a união deste tipo de restaurações depende significativamente das retenções micromecânicas criadas por procedimentos de modificação da

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rugosidade da superfície, como o jateamento com partículas de óxido de alumínio.(Huettig et al., 2016; Nagasawa et al., 2021; Soares et al., 2004)

Nas resinas impressas foi observada maior incidência de fraturas coesivas na resina da restauração, principalmente nos espécimes jateados. Vários estudos avaliaram a influência de fatores como a espessura da camada, (A. Kessler et al., 2020; Park et al., 2020) a angulação da amostra, (Nawal Alharbi et al., 2016; Revilla-León et al., 2019; Revilla-León, Jordan, et al., 2020; Reymus et al., 2020), e os processos de lavagem (Mayer, Reymus, et al., 2021; Mayer, Stawarczyk, et al., 2021) e pós-cura, (D. Kim et al., 2020; Perea-Lowery et al., 2021; Reymus et al., 2020; Reymus & Stawarczyk, 2020a) que afetam as propriedades das resinas para impressão 3D. As configurações e procedimentos aplicados nestas etapas, podem enfraquecer a resistência coesiva da amostra e afetar a veracidade da avaliação da resistência de união por microtração. Neste estudo, os espécimes foram impressos em angulação de 0°, por esse motivo, as camadas dos blocos estavam em posição perpendicular à carga aplicada. (Nawal Alharbi et al., 2016) Em vista desse resultado, imprimir os espécimes em uma angulação diferente pode ser recomendável para restaurações que serão submetidas às cargas de tensão e tração, porque as forças serão aplicadas em um vetor de movimento mais favorável (Alsandi et al., 2021; Keßler et al., 2021) e com área de contato maior entre as camadas impressas, (Keßler et al., 2021) reduzindo a delaminação coesiva do material. Além disso, é importante considerar que as diferenças encontradas na microdureza dos espécimes avaliados em profundidade podem sugerir uma polimerização desigual das resinas impressas, o que também pode influenciar na incidência de falhas coesivas.(Mendonça, Soto-Montero, de Castro, et al., 2021)

Contudo, resultou evidente a necessidade de efetuar pesquisas adicionais abordando a influência do ângulo de construção na resistência de união das resinas para impressora 3D, usando metodologias como a microtração e não unicamente avaliação em cisalhamento.(Albahri et al., 2021; Jeong & Kim, 2019; Lim & Shin, 2020; Revilla-león et al., 2020) Finalmente, embora a avaliação da resistência de união ao longo prazo de materiais restauradores provisórios pode ter pouca relevância clínica, os resultados obtidos podem ser usados para estimar a previsibilidade de restaurações fixas provisórias. O uso de protocolos adequados para cimentação adesiva de restaurações provisórias de longo prazo é de alta importância no sucesso clínico de alguns tratamentos complexos, como os que requerem alterações na dimensão vertical, (Huettig et al., 2016) regeneração óssea e tecidual antes da colocação do implante, (Tarnow et al., 2014) ou tratamentos expectantes em dentes com prognóstico pouco claro antes de uma reabilitação definitiva. (Huettig et al., 2016)

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4. CONCLUSÃO

O processo de pós-cura deve ser monitorado e ajustado cuidadosamente para cada material, para conseguir as melhores propriedades mecânicas e minimizar a alteração de cor das restaurações impressas, principalmente porque o processo de pós-cura produz alterações nas resinas para impressora 3D. No geral, maiores tempos de exposição, produziram maiores alterações de cor.

Foi comprovado que o processo de pós-cura produz um aumento significativo nas propriedades mecânicas de resistência flexural e microdureza das resinas para impressão 3D avaliadas, e que tempos de 5 a 10 minutos de pós-cura são suficientes para conseguir propriedades mecânicas comparáveis às de outros materiais para restaurações provisórias, com alterações de cor aceitáveis. Contudo, nas resinas avaliadas existe uma tendência de apresentar menores valores de microdureza na parte central interna das amostras.

Foi comprovado que as resinas para impressão 3D podem ser consideradas uma opção adequada para fabricação de restaurações provisórias de longo prazo, pois demonstraram uma boa resistência de união aos cimentos resinosos e são resistentes ao envelhecimento por termociclagem. Além disso, elas apresentam uma melhor resistência de união do que a encontrada nos materiais pré-polimerizados para manufatura subtrativa, sem necessidade de tratamentos mecânicos adicionais como jateamento com partículas de óxido de alumínio, que pode danificar a superfície das restaurações.

Em condições de desenho, manufatura e pós-processamento controladas, as resinas para restauração provisória fixa processadas por impressão 3D, podem oferecer uma performance estética e mecânica semelhante ou superior à obtida dos materiais tradicionais ou pré-polimerizados para fresagem, sendo também uma opção clínica adequada para fabricação de restaurações estéticas provisórias.

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REFERENCIAS[1]

Aati, S., Akram, Z., Shrestha, B., Patel, J., Shih, B., Shearston, K., Ngo, H., & Fawzy, A. (2021). Effect of post-curing light exposure time on the physico–mechanical properties and cytotoxicity of 3D-printed denture base material. *Dental Materials*, 1–11. https://doi.org/10.1016/j.dental.2021.10.011

Albahri, R., Yoon, H. I., Lee, J. D., Yoon, S., & Lee, S. J. (2021). Shear bond strength of provisional repair materials bonded to 3D printed resin. *Journal of Dental Sciences*, *16*(1), 261–267. https://doi.org/10.1016/j.jds.2020.05.003

Alharbi, N, Alharbi, A., & Osman, R. (2021). Mode of bond failure between 3D-printed denture teeth and printed resin base material; effect of fabrication technique and dynamic loading. An in-vitro study. *The International Journal of Prosthodontics*. https://doi.org/10.11607/ijp.6992

Alharbi, Nawal, Osman, R., & Wismeijer, D. (2016). Effects of build direction on the mechanical properties of 3D-printed complete coverage interim dental restorations. *Journal of Prosthetic Dentistry*, *115*(6), 760–767. https://doi.org/10.1016/j.prosdent.2015.12.002

Almarza, R., Ghassemieh, E., Shahrbaf, S., & Martin, N. (2020). The effect of crown fabrication process on the fatigue life of the tooth-crown structure. *Materials Science and Engineering C*, *109*(April 2018), 110272. https://doi.org/10.1016/j.msec.2019.110272

Alsandi, Q., Ikeda, M., Arisaka, Y., Nikaido, T., Tsuchida, Y., Sadr, A., Yui, N., & Tagami, J. (2021). Evaluation of mechanical and physical properties of light and heat polymerized udma for dlp 3d printer. *Sensors*, *21*(10), 1–10. https://doi.org/10.3390/s21103331

Alsandi, Q., Ikeda, M., Nikaido, T., Tsuchida, Y., Sadr, A., Yui, N., Suzuki, T., & Tagami, J. (2019). Evaluation of mechanical properties of new elastomer material applicable for dental 3D printer. *Journal of the Mechanical Behavior of Biomedical Materials*, *100*(July), 103390. https://doi.org/10.1016/j.jmbbm.2019.103390

[1] De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors - Vancouver Group. Abreviatura dos periódicos em conformidade com

Bae, S., Hong, M. H., Lee, H., Lee, C. H., Hong, M., Lee, J., & Lee, D. H. (2020). Reliability of metal 3d printing with respect to the marginal fit of fixed dental prostheses: A systematic review and meta-analysis. *Materials*, *13*(21), 1–14. https://doi.org/10.3390/ma13214781

Başaran, E. G., Ayna, E., Vallittu, P. K., & Lassila, L. V. J. (2013). Load bearing capacity of fiber-reinforced and unreinforced composite resin CAD/CAM-fabricated fixed dental prostheses. *Journal of Prosthetic Dentistry*, *109*(2), 88–94. https://doi.org/10.1016/S0022-3913(13)60022-0

Bouschlicher, M. R., Rueggeberg, F., & Wilson, B. M. (2004). Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. *Operative Dentistry*, *29*(6), 698–704. http://www.ncbi.nlm.nih.gov/pubmed/15646227

Çagri Ural, Burgaz, Y., & Saraç, D. (2010). In vitro evaluation of marginal adaptation in five. *Quintessence International*, *41*(7), 585–590.

Choi, J. W., Ahn, J. J., Son, K., & Huh, J. B. (2019). Three-dimensional evaluation on accuracy of conventional and milled gypsum models and 3D printed photopolymer models. *Materials*, *12*(21), 1–10. https://doi.org/10.3390/ma12213499

da Silva, L. H., de Lima, E., Miranda, R. B. de P., Favero, S. S., Lohbauer, U., Cesar, P. F., Lima, E. de, Miranda, R. B. de P., Favero, S. S., Lohbauer, U., & Cesar, P. F. (2017). Dental ceramics: a review of new materials and processing methods. *Brazilian Oral Research*, *31*(Suppl e58), 133–146. https://doi.org/10.1590/1807-3107BOR-2017.vol31.0058

Della Bona, A., Cantelli, V., Britto, V. T., Collares, K. F., & Stansbury, J. W. (2021). 3D printing restorative materials using a stereolithographic technique: a systematic review. *Dental Materials*, *37*(2), 336–350. https://doi.org/10.1016/j.dental.2020.11.030

Fronza, B. M., Ayres, A. P. A., Pacheco, R. R., Rueggeberg, F. A., Dias, C. T. S., & Giannini, M. (2017). Characterization of inorganic filler content, mechanical properties, and light transmission of bulk-fill resin composites. *Operative Dentistry*, *42*(4), 445–455. https://doi.org/10.2341/16-024-L

Fronza, Bruna Marin, Rueggeberg, F. A., Braga, R. R., Mogilevych, B., Soares, L. E. S., Martin, A. A. A., Ambrosano, G. G. G., & Giannini, M. (2015). Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites. *Dental Materials*, *31*(12), 1542–1551. https://doi.org/10.1016/j.dental.2015.10.001

Fuh, J. Y. H., Lu, L., Tan, C. C., Shen, Z. X., & Chew, S. (1999). Curing characteristics of acrylic photopolymer used in stereolithography process. *Rapid Prototyping Journal*, *5*(1), 27–34. https://doi.org/10.1108/13552549910251855

Grzebieluch, W., Kowalewski, P., Grygier, D., Rutkowska-Gorczyca, M., Kozakiewicz, M., & Jurczyszyn, K. (2021). Printable and machinable dental restorative composites for CAD/CAM application—comparison of mechanical properties, fractographic, texture and fractal dimension analysis. *Materials*, *14*(17). https://doi.org/10.3390/ma14174919

Huettig, F., Prutscher, A., Goldammer, C., Kreutzer, C. A., & Weber, H. (2016). First clinical experiences with CAD/CAM-fabricated PMMA-based fixed dental prostheses as long-term temporaries. *Clinical Oral Investigations*, *20*(1), 161–168. https://doi.org/10.1007/s00784-015

Jeong, K. W., & Kim, S. H. (2019). Influence of surface treatments and repair materials on the shear bond strength of CAD/CAM provisional restorations. *Journal of Advanced Prosthodontics*, *11*(2), 95–104. https://doi.org/10.4047/jap.2019.11.2.95

Jockusch, J., & Özcan, M. (2020). Additive manufacturing of dental polymers: An overview on processes, materials and applications. *Dental Materials Journal*, *39*(3), 345–354. https://doi.org/10.4012/dmj.2019-123

Katreva, I., Dikova, T., & Tonchev, T. (2018). 3D Printing – an Alternative of Conventional Crown Fabrication: a Case Report. *Journal of IMAB - Annual Proceeding (Scientific Papers)*, *24*(2), 2048–2054. https://doi.org/10.5272/jimab.2018242.2048

Keßler, A., Hickel, R., & Ilie, N. (2021). In vitro investigation of the influence of printing direction on the flexural strength, flexural modulus and fractographic analysis of 3D-printed temporary materials. *Dental Materials Journal*. https://doi.org/10.4012/dmj.2020-147

Kessler, A., Hickel, R., & Reymus, M. (2020). 3D printing in dentistry-state of the art. *Operative Dentistry*, *45*(1), 30–40. https://doi.org/10.2341/18-229-L

Kessler, Andreas, Reichl, F. X., Folwaczny, M., & Högg, C. (2020). Monomer release from surgical guide resins manufactured with different 3D printing devices. *Dental Materials*, *36*(11), 1486–1492. https://doi.org/10.1016/j.dental.2020.09.002

Kim, D., Shim, J. S. J. S., Lee, D., Shin, S. H., Nam, N. E., Park, K. H., Shim, J. S. J. S., & Kim, J. E. (2020). Effects of post-curing time on the mechanical and color properties of three-dimensional printed crown and bridge materials. *Polymers*, *12*(11), 1–20. https://doi.org/10.3390/polym12112762

Kim, J., & Lee, D. H. (2020). Influence of the Postcuring Process on Dimensional Accuracy and Seating of 3D-Printed Polymeric Fixed Prostheses. *BioMed Research International*, *2020*. https://doi.org/10.1155/2020/2150182

Lim, N.-K., & Shin, S.-Y. (2020). Bonding of conventional provisional resin to 3D printed resin: the role of surface treatments and type of repair resins. *The Journal of Advanced Prosthodontics*, *12*(5), 322. https://doi.org/10.4047/jap.2020.12.5.322

Lin, C. H., Lin, Y. M., Lai, Y. L., & Lee, S. Y. (2020). Mechanical properties, accuracy, and cytotoxicity of UV-polymerized 3D printing resins composed of Bis-EMA, UDMA, and TEGDMA. *Journal of Prosthetic Dentistry*, *123*(2), 349–354. https://doi.org/10.1016/j.prosdent.2019.05.002

Mainjot, A. K., Dupont, N. M., Oudkerk, J. C., Dewael, T. Y., & Sadoun, M. J. (2016). From Artisanal to CAD-CAM Blocks: State of the Art of Indirect Composites. *Journal of Dental Research*, *95*(5), 487–495. https://doi.org/10.1177/0022034516634286

Mayer, J., Reymus, M., Wiedenmann, F., Edelhoff, D., Hickel, R., & Stawarczyk, B. (2021). Temporary 3D printed fixed dental prosthesis materials: Impact of post printing cleaning methods on degree of conversion as well as surface and mechanical properties. *The International Journal of Prosthodontics*, 1–29. https://doi.org/10.11607/ijp.7048

Mayer, J., Stawarczyk, B., Vogt, K., Hickel, R., Edelhoff, D., & Reymus, M. (2021). Influence of cleaning methods after 3D printing on two-body wear and fracture load of resin-based temporary crown and bridge material. *Clinical Oral Investigations*. https://doi.org/10.1007/s00784-021-

03905-9

McCarty, M. C., Chen, S. J., English, J. D., & Kasper, F. (2020). Effect of print orientation and duration of ultraviolet curing on the dimensional accuracy of a 3-dimensionally printed orthodontic clear aligner design. *American Journal of Orthodontics and Dentofacial Orthopedics*, *158*(6), 889–897. https://doi.org/10.1016/j.ajodo.2020.03.023

Mendonça, B. C. de, Soto-Montero, J., Castro, E. F. De, Pecorari, V. G. A., Rueggeberg, F. A., & Giannini, M. (2021). Flexural strength and microhardness of bulk-fill restorative materials. *Journal of Esthetic and Restorative Dentistry*, *33*(4), 628–635. https://doi.org/10.1111/jerd.12727

Mendonça, B. C. de, Soto-Montero, J. R., de Castro, E. F., Kury, M., Cavalli, V., Rueggeberg, F. A., & Giannini, M. (2021). Effect of extended light activation and increment thickness on physical properties of conventional and bulk - filled resin - based composites. *Clinical Oral Investigations*, *0123456789*. https://doi.org/10.1007/s00784-021-04296-7

Methani, M. M., Cesar, P. F., de Paula Miranda, R. B., Morimoto, S., Özcan, M., & Revilla-León, M. (2020). Additive Manufacturing in Dentistry: Current Technologies, Clinical Applications, and Limitations. *Current Oral Health Reports*, *7*(4), 327–334. https://doi.org/10.1007/s40496-020-00288-w

Nagasawa, Y., Eda, Y., Shigeta, H., Ferrari, M., Nakajima, H., & Hibino, Y. (2021). Effect of sandblasting and/or priming treatment on the shear bond strength of self-adhesive resin cement to CAD/CAM blocks. *Odontology*, *0123456789*, 14–17. https://doi.org/10.1007/s10266-021-00635-y

Nestler, N., Wesemann, C., Spies, B. C., Beuer, F., & Bumann, A. (2021). Dimensional accuracy of extrusion- and photopolymerization-based 3D printers: In vitro study comparing printed casts. *Journal of Prosthetic Dentistry*, *125*(1), 103–110. https://doi.org/10.1016/j.prosdent.2019.11.011

Nour, M., Bshara, A. N., Abou, J., John, N., & Rizk, C. K. (2021). Clinical Performance of Two Types of Primary Molar Indirect Crowns Fabricated by 3D Printer and CAD / CAM for Rehabilitation of Large Carious Primary Molars. *European Journal of Dentistry*, *January*.

https://doi.org/10.1055/s-0040-1721905

Osman, R., Alharbi, N., & Wismeijer, D. (2017). Build Angle: Does It Influence the Accuracy of 3D-Printed Dental Restorations Using Digital Light-Processing Technology? *The International Journal of Prosthodontics*, *30*(2), 182–188. https://doi.org/10.11607/ijp.5117

Paravina, R. D., Ghinea, R., Herrera, L. J., Bona, A. D., Igiel, C., Linninger, M., Sakai, M., Takahashi, H., Tashkandi, E., & Del Mar Perez, M. (2015). Color difference thresholds in dentistry. *Journal of Esthetic and Restorative Dentistry*, *27*(S1), S1–S9. https://doi.org/10.1111/jerd.12149

Park, S. M., Park, J. M., Kim, S. K., Heo, S. J., & Koak, J. Y. (2020). Flexural strength of 3D-printing resin materials for provisional fixed dental prostheses. *Materials*, *13*(18), 1–14. https://doi.org/10.3390/ma13183970

Perea-Lowery, L., Gibreel, M., Vallittu, P. K., & Lassila, L. (2021). Evaluation of the mechanical properties and degree of conversion of 3D printed splint material. *Journal of the Mechanical Behavior of Biomedical Materials*, *115*(November 2020), 104254. https://doi.org/10.1016/j.jmbbm.2020.104254

Prpić, V., Schauperl, Z., Ćatić, A., Dulčić, N., & Čimić, S. (2020). Comparison of Mechanical Properties of 3D-Printed, CAD/CAM, and Conventional Denture Base Materials. *Journal of Prosthodontics*, *29*(6), 524–528. https://doi.org/10.1111/jopr.13175

Revilla-León, M., Jordan, D., Methani, M. M., Piedra-Cascón, W., Özcan, M., & Zandinejad, A. (2020). Influence of printing angulation on the surface roughness of additive manufactured clear silicone indices: An in vitro study. *Journal of Prosthetic Dentistry*, 1–7. https://doi.org/10.1016/j.prosdent.2020.02.008

Revilla-León, M., Meyers, M. J., Zandinejad, A., & Özcan, M. (2019). A review on chemical composition, mechanical properties, and manufacturing work flow of additively manufactured current polymers for interim dental restorations. *Journal of Esthetic and Restorative Dentistry*, *31*(1), 51–57. https://doi.org/10.1111/jerd.12438

Revilla-león, M., Morillo, J. A., Att, W., & Özcan, M. (2020). Chemical Composition, Knoop Hardness, Surface Roughness, and Adhesion Aspects of Additively Manufactured Dental Interim Materials. *Journal of Prosthodontics*, *0*, 1–8. https://doi.org/10.1111/jopr.13302

Revilla-León, M., Umorin, M., Özcan, M., & Piedra-Cascón, W. (2020). Color dimensions of additive manufactured interim restorative dental material. *Journal of Prosthetic Dentistry*, *123*(5), 754–760. https://doi.org/10.1016/j.prosdent.2019.06.001

Reymus, M., Fabritius, R., Keßler, A., Hickel, R., Edelhoff, D., & Stawarczyk, B. (2020). Fracture load of 3D-printed fixed dental prostheses compared with milled and conventionally fabricated ones: the impact of resin material, build direction, post-curing, and artificial aging—an in vitro study. *Clinical Oral Investigations*, *24*(2), 701–710. https://doi.org/10.1007/s00784-019-02952-7

Reymus, M., & Stawarczyk, B. (2020a). In vitro study on the influence of postpolymerization and aging on the Martens parameters of 3D-printed occlusal devices. *Journal of Prosthetic Dentistry*, *125*(5), 817–823. https://doi.org/10.1016/j.prosdent.2019.12.026

Reymus, M., & Stawarczyk, B. (2020b). Influence of Different Postpolymerization Strategies and Artificial Aging on Hardness of 3D-Printed Resin Materials: An In Vitro Study. *The International Journal of Prosthodontics*, *33*(6), 634–640. https://doi.org/10.11607/ijp.6634

Rocha, R., Fagundes, T., Caneppele, T., & Bresciani, E. (2019). Perceptibility and Acceptability of Surface Gloss Variations in Dentistry. *Operative Dentistry*. https://doi.org/10.2341/18-184-c

Rueggeberg, F. A., Cole, M. A., Looney, S. W., Vickers, A., & Swift, E. J. (2009). Comparison of manufacturer-recommended exposure durations with those determined using biaxial flexure strength and scraped composite thickness among a variety of light-curing units: Masters of esthetic dentistry. *Journal of Esthetic and Restorative Dentistry*, *21*(1), 43–61. https://doi.org/10.1111/j.1708-8240.2008.00231.x

Salas, M., Lucena, C., Herrera, L. J., Yebra, A., Della Bona, A., & Pérez, M. M. (2018). Translucency thresholds for dental materials. *Dental Materials*, *34*(8), 1168–1174. https://doi.org/10.1016/j.dental.2018.05.001

Simoneti, D. M., Pereira-Cenci, T., & dos Santos, M. B. F. (2020). Comparison of material properties and biofilm formation in interim single crowns obtained by 3D printing and conventional methods. *Journal of Prosthetic Dentistry*, 1–5. https://doi.org/10.1016/j.prosdent.2020.06.026

Skorulska, A., Piszko, P., Rybak, Z., Szymonowicz, M., & Dobrzinski, M. (2021). Review on Polymer, Ceramic and Composite Materials for CAD/CAM Indirect Restorations in Dentistry - Application, Mechanical Characteristics and Comparison. *Materials*, *14*(1592).

Soares, C. J., Giannini, M., de Oliveira, M. T., Paulillo, L. A. M. S., & Martins, L. R. M. (2004). Effects of surface treatments of laboratory-fabricated composites on the microtensile bond strength to a luting resin cement. *Journal of Applied Oral Science*, *12*(1), 45–50.

Söderberg, J. (2013). Automating amateurs in the 3D printing community: Connecting the dots between deskiling and user-friendliness. *Work Organization, Labour & Globalization*, *7*(1), 124–140.

Sulaiman, T. A. (2020). Materials in digital dentistry—A review. *Journal of Esthetic and Restorative Dentistry*, *32*(2), 171–181. https://doi.org/10.1111/jerd.12566

Tahayeri, A., Morgan, M. C., Fugolin, A. P., Bompolaki, D., Athirasala, A., Pfeifer, C. S., Ferracane, J. L., & Bertassoni, L. E. (2018). 3D printed versus conventionally cured provisional crown and bridge dental materials. *Dental Materials*, *34*(2), 192–200. https://doi.org/10.1016/j.dental.2017.10.003

Tarnow, D., Chu, S., Salama, M., Stappert, C., Salama, H., Garber, D., Sarnachiaro, G., Sarnachiaro, E., Gotta, S., & Saito, H. (2014). Flapless Postextraction Socket Implant Placement in the Esthetic Zone: Part 1. The Effect of Bone Grafting and/or Provisional Restoration on Facial-Palatal Ridge Dimensional Change—A Retrospective Cohort Study. *International Journal of Periodontics & Restorative Dentistry*, *34*(3), 323–331. https://doi.org/10.11607/prd.1821

Uzun, G. (2008). An overview of dental CAD/CAM systems. *Biotechnology and Biotechnological Equipment*, *22*(1), 530–535. https://doi.org/10.1080/13102818.2008.10817506

Zhang, Z. chen, Li, P. lun, Chu, F. ting, & Shen, G. (2019). Influence of the three-dimensional printing technique and printing layer thickness on model accuracy. *Journal of Orofacial Orthopedics*, *80*(4), 194–204. https://doi.org/10.1007/s00056-019-00180-y

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