Flexural strength and Vickers hardness of milled and 3D-printed resins for provisional dental restorations

Ana Luiza Caetano Souza1*, Jorge Luiz de Oliveira Cruvinel Filho1, Sicknan Soares da Rocha1

Aim: Various forms of temporary resins are offered on the market; however, the properties of temporary resins obtained by milling and 3D printing have not been fully examined. This study aimed to compare the flexural strength and Vickers hardness of milled and 3D-printed resins. Methods: Three resins were tested: Evolux PMMA (milled resin), Cosmos Temp (3D-printed resin), and Structur 2 SC (bis-acrylic resin, group control). Specimens were prepared with rectangular shapes (n = 12) for flexural strength measurements and disc shapes (n = 9) for Vickers hardness tests. Flexural strength tests were performed at a crosshead speed of 0.75 mm/min, and the Vickers hardness was measured under a load of 20 N for 10 s. The obtained data were subjected to the Kruskal–Wallis test. Results: A significant difference (p < 0.05) in flexural strength was observed among the three sample groups: Evolux PMMA (111.76 MPa), Structur 2 SC (87.34 MPa), and Cosmos Temp (56.83 MPa). No significant difference (p < 0.05) was observed between the Vickers hardness values of Structur 2 SC (33.37 VHN) and Evolux PMMA (29.11 VHN); however, both materials were statistically superior to Cosmos Temp (10.90 VHN). Conclusion: While the mechanical properties of the milled resin were superior or similar to those of the bis-acrylic resin, the 3D-printed resin was statistically inferior to both the milled and bis-acrylic resins.

Keywords: Dental restoration, temporary. Flexural strength. Hardness tests. Computer-aided design.
Introduction

In recent years, bis-acrylic resins have attracted significant research interest among dental professionals. They comprise urethane dimethacrylate (UDMA), organic matrix components of composite resins, and microparticle fillers. These materials have important advantages for clinical applications include the ease of manipulation, high mechanical resistance, low polymerization shrinkage, less exothermic reaction, and low level of unpleasant odor\(^1\). Owing to these important characteristics, bis-acrylic resins are rapidly replacing polymethylmethacrylate (PMMA)-based resins in transitory restorations used by conventional techniques\(^4\).

More recently, with the new advances in computer-aided design/computer-aided manufacturing (CAD/CAM) technology, other forms of temporary resins have become available. The CAD/CAM system has been widely utilized in the field of dentistry owing to its ability to manufacture high-quality prosthetic restorations\(^5,6\). Using this technique, durable and esthetically pleasing parts are produced with high speed and efficiency while ensuring proper quality control\(^7\). The obtained CAD/CAM interim resins are highly resistant to hot, cold, and moist environments\(^8\).

The CAD/CAM fabrication process of interim restorations can be implemented using subtractive manufacturing (SM) or additive manufacturing (AM) technology. The SM technique is based on removing material from a larger block to attain the shape of a virtually designed object\(^9\). AM technology, also known as 3D printing technology, is defined as the process of joining materials in a layer-by-layer manner to fabricate objects based on their 3D models\(^10\). Note that AM technology considerably differs from SM technology because it minimizes the material waste\(^11-13\).

The literature on the physical and mechanical properties of PMMA-based temporary resins obtained by milling (SM) and 3D printing (AM) is very scarce. In this context, flexural strength and Vickers hardness tests represent important methods for evaluating the behavior of these materials and determining their limitations.

This study aimed to evaluate the influence of the manufacturing method, such as SM and AM technologies, on the flexural strength and Vickers hardness of resins for transitory restorations using a bis-acrylic resin as a control, and two null hypotheses were formulated prior to the study: (H\(_0\)\(^1\)) stating that there is no significant difference in flexural strength among the three resins and (H\(_0\)\(^2\)) stating that there is no significant difference in Vickers hardness among the three resins.

Materials and methods

Flexural strength and Vickers hardness values were assessed for three different materials used in provisional restorations: bis-acrylic resin (BR), milled resin (MR), and 3D-printed resin (PR) (Table 1).
<table>
<thead>
<tr>
<th>Type</th>
<th>Code</th>
<th>Resin</th>
<th>Manufacturer</th>
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<tbody>
<tr>
<td>Bis-acrylic</td>
<td>BR</td>
<td>Structur 2 SC</td>
<td>Voco, Porto Alegre, Brasil</td>
</tr>
<tr>
<td>Milled</td>
<td>MR</td>
<td>Evolux PMMA</td>
<td>Blue Dent, Pirassununga, Brasil</td>
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<tr>
<td>3D-printed</td>
<td>PR</td>
<td>Cosmos Temp</td>
<td>Yller, Pelotas, Brasil</td>
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For flexural strength measurements, specimen standards (25 × 2 × 2 mm, n = 12) were fabricated according to ADA–ANSI specification #27\textsuperscript{14}. BR was mixed according to manufacturer’s instructions using a self-mixing gun and then injected into custom-fabricated silicone rubber molds with dimensions of 25 × 2 × 2 mm (Zetalabor, Zhermack, BadiaPolesine, Italy). After 5 min, the blocks were retrieved from the molds.

MR specimens with dimensions of 25 × 2 × 2 mm were virtually designed using the CAD software Ceramill Mind (Amann Girrbach, Koblach, Austria) and milled from an Evolux PMMA block (101 × 101 × 20 mm) using a milling machine Ceramill Motion 2 (Amann Girrbach, Koblach, Austria).

PR specimens were also virtually designed using the Ceramill Mind software and then printed on a stereolithography (SLA) printer (D30, Rapid Shape, Heimsheim, Germany). After printing, the specimens were cleaned with 90% isopropyl alcohol for 5 min according to manufacturer’s specifications and post-polymerized with 3000 flashes of ultraviolet light (385 nm) in a UV-A type 3 post-polymerization light-box (Flashlight plus, Shera Material Technology, Lemforde, Germany). The sizes of the three resin specimens were measured with a digital caliper (Fowler/Sylvac UltraCal Mark IV Electronic Caliper, Crissier, Switzerland) for the standard quality assessment. Subsequently, the specimens were polished by grinding on wet silicon carbide papers (200, 400, and 800 grit).

For the Vickers hardness test, specimens (n = 9) with disk shapes (diameter: 8 mm, thickness: 2 mm) were fabricated according to ADA–ANSI specification #27\textsuperscript{11} following the manufacturing procedure established for flexural strength test samples. All specimens were stored in a water bath at 37 ± 1 °C for 24 h.

A three-point bending test was performed using an Instron 4411 universal testing machine (Instron Testing Instruments, Canton, MA, USA) with a crosshead speed of 0.75 mm/min (n = 12). The Vickers hardness test was conducted using a micro-Vickers hardness tester (HMV-G20, Shimadzu, Tokyo, Japan) with a 20-N load applied for 10 s. Five readings were obtained for the top and bottom parts of the test specimens, and their values were averaged for each sample.

The obtained data were checked for normal distribution using the Shapiro–Wilk test. As the data were not normally distributed, statistical significance among the different specimen group was tested with the non-parametric Kruskal–Wallis test, using the SPSS software (Version 25.0, Chicago, IL, USA). The level of significance was set to 0.05.
**Results**

The obtained flexural strength and Vickers hardness values are listed in Table 2.

<table>
<thead>
<tr>
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<th>Structur 2 SC (BR)</th>
<th>Evolux PMMA (MR)</th>
<th>Cosmos Temp (PR)</th>
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<tr>
<td>Flexural Strength (MPa)</td>
<td>87.34</td>
<td>111.76</td>
<td>56.83</td>
</tr>
<tr>
<td>Vickers Hardness (VHN)</td>
<td>33.37</td>
<td>29.11</td>
<td>10.90</td>
</tr>
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</table>

The results of the Kruskal–Wallis tests revealed the existence of a significant difference among the flexural strengths of the BR, MR, and PR groups ($p < 0.05$). The MR group exhibited the highest flexural strength (111.76 MPa) followed by the BR (87.34 MPa) and PR (56.83 MPa) groups. Meanwhile, no significant difference in Vickers hardness was observed between the BR (33.17 VHN) and MR (29.11 VHN) groups ($p > 0.05$). In contrast, the PR group (10.90 VHN) was statistically inferior to the other two groups ($p < 0.05$) (Table 3).

**Discussion**

Considering the significance level of 5% ($\alpha = 0.05$), the null hypothesis ($H_01$) stating that there is no significant difference in flexural strength among the three resins was rejected, opting for the alternative hypothesis ($H_1$). For the Vickers hardness tests, the null hypothesis ($H_02$) was rejected, despite no statistical difference between the bis-acrylic and milled resins was observed.

Bis-acrylic resins based on multifunctional methacrylic acid esters have emerged as the materials of choice for provisional restorations because of their easy intra-oral manipulation and mechanical properties comparable to those of conventional materials available in the powder/liquid form. However, mixing and filling the over-impression may lead to the incorporation of voids, compromising the mechanical strength of these materials, which significantly limited their application in multiple extensive prostheses.
The restorations fabricated using the CAD/CAM system are more accurate and easier to manipulate than bis-acrylic resins. In addition, they are predicted to possess good mechanical properties and thus represent a viable solution for long-term/long-span interim restorations, which require high strength and color stability.

The results obtained for the Evolux PMMA milled resin group were in agreement with the findings of previous studies. Dureja et al. reported the highest flexural strengths for CAD/CAM resin blocks. The same results were obtained in a study that evaluated the flexural strength and microhardness of three transitory resins fabricated by printing, milling, and conventional methods. The highest and lowest flexural strength means were obtained for the milled and printed resins, respectively. In an evaluation of various resins for occlusal devices, 3D-printed resins also demonstrated lower wear and fracture resistances than those of the milled and conventionally fabricated resins.

After examining the flexural strengths and marginal accuracies of conventional and CAD/CAM transitory resins under the influence of thermal cycling, Yao et al. found that the CAD/CAM resins exhibited the best behavior even after 5000 thermal cycles. This improved stability resulted from the high polymerization efficiency during the fabrication of resin blocks.

Hardness can be used as an indicator of density, and it was hypothesized that a denser material would be more resistant to wear and surface deterioration. However, surface hardness alone is not an indicator of the overall rigidity and strength and cannot be used to predict the clinical behavior of long-span prostheses. This limitation shows the need for conducting other mechanical tests such as measurements of flexural strength, which is generally considered the main indicator of the mechanical response of a restorative material.

With the results similar to those obtained in the present study, Perea-Lowery et al. demonstrated that during the evaluation of mechanical properties, including flexural strength, Martens hardness, Vickers hardness, and elastic modulus, the 3D-printed resin materials were not as resistant to stress and aging as the pressed or milled resin materials. All tests revealed that the pressed and milled resins exhibited higher mean values than those of the 3D-printed resins.

This inferior behavior of 3D-printed resins is a result of the high residual monomer content and greater porosity, which is minimized through the industrial manufacturing of PMMA blocks for milled resins. This process allows the production of polymers with high density, which increases the strength of restorations.

Conventional resins based on PMMA are mono-functional resins with linear molecules, low molecular weight, and low strength. In addition to the intrinsic characteristics of these resins, they cannot be polymerized under pressure, and the high content of residual monomers contributes to their inferior mechanical behavior. Additionally, the blocks for the milling process prevent heating and polymerization shrinkage. These properties of PMMA resins are preserved by CAD/CAM technology and have enabled their application in transitory dental restorations.
In addition to the superior mechanical properties of the produced resins, the CAD/CAM process based on the milling method is more accurate in terms of anatomical shape, marginal adaptation, and occlusal/interproximal contacts. The milling process can be performed using a simplified procedure with a significantly reduced operating time\(^{18}\). However, despite the increasing availability and ease of operating the software and milling equipment (subtractive process), they require considerable investments for their acquisition.

Concomitantly, the increasing application of the 3D printing process has promoted a substantial expansion of digital flow in dentistry\(^{10}\), allowing the production of objects with different degrees of complexity and limited material waste\(^{11,13}\). 3D printing requires less expensive equipment as compared with milling machines in addition to the low fabrication time. Within the timeframe of approximately 20 min, which is required for milling a single block, it is possible to simultaneously print several objects positioned on the same tray\(^{21}\). Therefore, 3D printing is becoming increasingly popular in dentistry. 3D-printed resins are widely used in the production of cast models, surgical templates for guiding implant surgeries, maxillofacial prostheses, and orthodontic appliances and have been recently applied for provisional restorations\(^{21}\).

The inferior mechanical behavior of 3D-printed resins is related to several factors, including printing technology, light intensity and wavelength, CAD design, printing orientation, layer thickness, post-processing procedures, and material characteristics\(^{20,21,25-28}\). Previous studies have shown that the orientation angle during printing (0º, 45º, or 90º) strongly influences the printing accuracy and mechanical properties (such as flexural strength) of 3D-printed resins\(^{20,21,29,30}\). This phenomenon is likely correlated with the thicknesses and union of various resin layers; however, no consensus has been reached regarding this issue in the literature. Furthermore, various resin properties (such as viscosity, critical energy for polymerization, and photon penetration depth) and printing hardware capabilities (such as photon length and power density) may also influence the manufacturing outcome\(^{30}\).

The mechanical properties evaluated in this study (flexural strength and Vickers hardness) are directly related to the degree of material polymerization, which depends on several factors affecting the printing process. Thus, better process control and improvements of the processing and post-processing phases are required for 3D printing. In this context, manufacturers of 3D printers should provide detailed guidelines for printing protocols and polymerization methods as these factors potentially influence the clinical performance of printed materials\(^{29,30}\).

Owing to the numerous advantages of the 3D printing process, including the elimination of material waste, lower fabrication time, and reduced cost, additional studies must be performed in the future to better utilize this important technique for dental applications.

In the present study, an SLA printer was used, and samples with an orientation angle of 0º were prepared for flexural strength and Vickers hardness tests. A probable limitation of this study was that other orientation angles were not considered, suggesting that additional tests are needed in the future.
Conclusion
From the results of this study, the following conclusions have been drawn.
1. The highest mean flexural strength was obtained for the Evolux PMMA milled resin followed by the Structur 2 SC bis-acrylic and 3D-printed Cosmos Temp resins with a significant difference among the three groups of samples.
2. The Vickers hardness of the 3D-printed resin was statistically lower than those of the bis-acrylic and milled resins, which did not exhibit any significant differences.

Author contributions
Ana Luiza Caetano Souza: Contributed to conception, design, data acquisition, drafted the manuscript
Jorge Luiz de Oliveira Cruvinel Filho: Contributed to conception, design, data interpretation and critically revised the manuscript
Sicknan Soares da Rocha: Contributed to conception, design, data interpretation, performed all statistical analyses, drafted and critically revised the manuscript and critically revised the manuscript
All authors actively participated in the discussion of the manuscript’s findings, and have revised and approved the final version of the manuscript.

Conflicts of interest
None.

Data availability
Datasets related to this article will be available upon request to the corresponding author.

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orientation, positioning, and curing influence dimensional accuracy and flexural