

Original Research

# Microhardness and flexural strength of two 3D-printed denture base resins



Cristina Bettencourt Neves<sup>1,\*</sup> , Ana Filipa Chasqueira<sup>1</sup> , Patrícia Rebelo<sup>2</sup> ,  
Mariana Fonseca<sup>2</sup> , Jaime Portugal<sup>1</sup> , Ana Bettencourt<sup>3</sup> 

<sup>1</sup> Universidade de Lisboa, Faculdade de Medicina Dentária, Unidade de Investigação e Ciências Orais e Biomédicas (UICOB), Lisbon, Portugal

<sup>2</sup> Universidade de Lisboa, Faculdade de Medicina Dentária, Lisbon, Portugal

<sup>3</sup> Universidade de Lisboa, Faculty of Pharmacy, Research Institute for Medicines (iMed.Ulisboa), Lisbon, Portugal

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## ABSTRACT

**Objectives:** To evaluate the microhardness and flexural strength of printed and conventionally produced denture base acrylic resins.

**Methods:** A total of 32 parallelepiped specimens (64×10×3.3 mm) were manufactured using two light-cured resins suitable for 3D printing (V-Print Dentbase and Denture 3D+) and two heat-cured resins for conventional production (Probase Hot and Villacryl Rapid) (n=8). After 24-h storage in water, Knoop microhardness (98.12 mN load for 30 s) and three-point flexural strength (1 kN load cell, at a crosshead speed of 5 mm/min and 50 mm between rods) were determined. Microhardness data were analyzed with one-way ANOVA followed by Tukey post-hoc tests. Flexural strength data were submitted to the Kruskal-Wallis test. A significance level of 5% was considered ( $\alpha=0.05$ ).

**Results:** The resins showed statistically significant ( $p<0.001$ ) differences regarding microhardness (V-Print Dentbase < Denture 3D+ < Probase Hot < Villacryl Rapid). No statistically significant ( $p=0.527$ ) differences were found in flexural strength between the four resins.

**Conclusions:** The printed resins had lower microhardness values than conventional resins, but all resins showed similar flexural strength. (Rev Port Estomatol Med Dent Cir Maxilofac. 2022;63(4):198-203)

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\* Corresponding author.

E-mail address: [mneves@edu.ulisboa.pt](mailto:mneves@edu.ulisboa.pt) (Cristina Bettencourt Neves).

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## Microdureza e resistência à flexão de duas resinas de base de prótese impressas em 3D

### R E S U M O

#### Palavras-chave:

Impressão em 3D  
CAD-CAM  
Resinas de base de prótese  
Resistência à flexão  
Dureza

**Objetivos:** Avaliar a microdureza e a resistência à flexão de resinas acrílicas de base de prótese impressas e produzidas pelo método convencional.

**Métodos:** Foi produzido um total de 32 espécimes com forma de paralelepípedo (64×10×3,3 mm) usando duas resinas fotopolimerizáveis para impressão 3D (V-Print Dentbase e Denture 3D+) e duas resinas termopolimerizáveis para fabrico convencional (Probases Hot e Villacryl Rapid) (n=8). Após 24h de armazenamento em água, foi determinada a microdureza Knoop (carga de 98,12 mN durante 30 s) e a resistência à flexão de três pontos (célula de carga de 1 kN a uma velocidade de 5 mm/min e 50 mm entre suportes) em cada espécime. Os resultados de microdureza foram analisados através do teste ANOVA de uma via, seguido de comparações múltiplas de Tukey, e os resultados de resistência à flexão através do teste Kruskal-Wallis. Foi considerado um nível de significância de 5% ( $\alpha=0,05$ ).

**Resultados:** Foram encontradas diferenças estatisticamente significativas ( $p<0,001$ ) na microdureza das resinas (V-Print Dentbase < Denture 3D+ < Probases Hot < Villacryl Rapid). Não se verificaram diferenças estatisticamente significativas ( $p=0,527$ ) entre a resistência à flexão das quatro resinas.

**Conclusões:** As resinas impressas apresentaram menor microdureza do que as resinas de fabrico convencional, mas todas as resinas obtiveram resistência à flexão semelhante. (Rev Port Estomatol Med Dent Cir Maxilofac. 2022;63(4):198-203)

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## Introduction

Dental medicine aims to restore health, function, and aesthetics, regardless of disease or injury to the stomatognathic system.<sup>1</sup> Although total edentulism is decreasing due to improved oral disease control, the increasing average life expectancy and the consequent high number of elderly individuals lead to a higher risk of tooth loss.<sup>2</sup>

Removable dentures are a viable treatment option to replace missing teeth and soft tissue<sup>1-3</sup> since they are easy to fabricate and repair and have good mechanical properties.<sup>4</sup> Several resin polymers with different chemical compositions and types of polymerization reactions are used to produce removable dentures.<sup>5,6</sup> Nevertheless, conventional production of heat-cured polymethylmethacrylate (PMMA) remains the most widely used and is considered the gold-standard fabrication protocol.<sup>3-6</sup> Still, it has some disadvantages, such as the high number of clinical appointments needed,<sup>7</sup> laboratory consuming time,<sup>4</sup> polymerization shrinkage that could lead to maladaptation,<sup>8</sup> allergenic potential due to the presence of residual methyl methacrylate (MMA) monomer,<sup>9-11</sup> susceptibility to bacterial colonization,<sup>12</sup> reduction of mechanical properties over time,<sup>13-15</sup> and difficulty of performing an adequate duplication.<sup>4</sup> Thus, in an attempt to overcome these limitations, new materials and manufacturing techniques have been proposed.<sup>4,16,17</sup>

Digital denture manufacturing techniques reduce the cost and time of clinical and laboratory procedures,<sup>18</sup> besides pro-

viding reproducibility by storing digital information.<sup>7,14</sup> Three-dimensional (3D) printing is a simple and recently introduced additive digital denture manufacturing technique with low material waste.<sup>19,20</sup> Materials developed for this technique are composed of less cytotoxic and more complex dimethacrylate-based monomers than MMA, such as urethane dimethacrylate (UDMA), bisphenol-A ethoxylate dimethacrylate (bis-EMA), triethylene glycol dimethacrylate (TEGDMA), and hydroxyethyl methacrylate (HEMA), and based on light-curing technology.<sup>9,20-24</sup> Also, some authors have shown that light-curing printing techniques provide similar fit accuracy to heat-curing conventional methods.<sup>8,20,21,25</sup> However, the additive technique with incremental layers can affect the polymeric network's cohesive strength, leading to an increased probability of failures or fractures.<sup>21</sup> Therefore, studies evaluating the mechanical properties of 3D-printed resins for dentures are still necessary.

Since dentures must resist masticatory loads and plastic deformation induced by mechanical indentation or abrasion, the study of properties such as microhardness and flexural strength is important for clinical decisions on the materials and techniques to use.<sup>26-29</sup> The aim of this in vitro study was to evaluate the mechanical properties of denture base resins manufactured by 3D printing and conventional techniques, according to the following null hypotheses: 1) there are no differences between the microhardness of the different resins studied; 2) there are no differences between the flexural strength of the different resins.

## Material and methods

A power analysis was performed based on a pilot study to estimate the sample size of both tests ( $n=8$ ) and provide statistical significance ( $\alpha=0.05$ ) at 80% power and an effect size of 0.25.<sup>13,30</sup>

Four denture base resins were selected: V-Print Dentbase (VOCO GmbH, Germany) and Denture 3D+ (NextDent BV, The Netherlands) as light-cured denture base resins suitable for 3D printing and Probase Hot (Ivoclar Vivadent AG, Liechtenstein) and Villacryl Rapid (Zhermack SpA, Italy) as heat-cured denture base resins for conventional production (Table 1). Eight parallelepiped-shaped (64x10x3.3 mm) specimens were prepared from each resin according to the respective manufacturer's instructions and considering specific standards.<sup>31</sup>

Printed specimens were designed with CAD 3D Sprint software (3D Systems, USA), and a standard tessellation language (STL) file was produced. Before printing, the Denture 3D+ liquid formulation was manually shaken for 5 minutes, followed by 1-hour agitation with the LC-3D Mixer machine (NextDent BV, 3D Systems, The Netherlands). The Denture 3D+ specimens were locally printed on a NextDent 5100 3D Printer device (NextDent BV, 3D Systems, The Netherlands), with a layer thickness of 50  $\mu\text{m}$ . After printing, the specimens were cleaned with isopropanol, and a post-processing treatment was executed inside an LC-3DPrint Box (NextDent BV, 3D Systems, The Netherlands) for 30 minutes. V-Print Dentbase specimens were produced by the manufacturer (VOCO GmbH, Germany) from the STL file on a W2P Solflex 650 printing device (W2P Engineering GmbH, Austria), with a layer thickness of 50  $\mu\text{m}$ . Then, specimens were cleaned with isopropanol (2+2 minutes), and the post-processing treatment was executed in the Otofash

G171 device (NK-Optik GmbH, Germany) during two cycles of 2000 flashes.

The heat-cured denture base acrylic resins Probase Hot and Villacryl Rapid were produced by a conventional flasking technique, according to the manufacturer's instructions (Table 1). Probase Hot was produced using a long curing cycle (water until 80°C, further maintenance for 10 hours, and cooling). Villacryl Rapid was produced using a short curing cycle (water until 80°C, increase until 100°C, further maintenance for 20 minutes, and cooling).

After processing, all specimens' edges were grounded with a 600-grit silicon carbide paper (Carbimet Paper Discs, Buehler Ltd., USA) mounted in a rotational grinding and polishing machine (DAP-U, Struers, Denmark). Then, the specimens were soaked in distilled water and manually polished with polishing discs P1200 and P2500 (CarbiMet, Buehler, USA), 2 min each. Finally, each specimen was finished with 6- $\mu\text{m}$ , 3- $\mu\text{m}$ , and 1- $\mu\text{m}$  monocrystalline diamond suspension (MetaDi, Buehler, USA) for 1 minute each. Polished surfaces were cleaned with an ethanol-soaked swab, and specimens were stored in distilled water at 37 $\pm$ 2 °C for 24 h in an oven (Ehret, Germany) before testing.<sup>32</sup>

Knoop microhardness was determined (Duramin, Struers DK 2750, Denmark) with 98.12 mN load for 30 s. The mean of six equidistant measurements made in each specimen was used as their corresponding microhardness value (KHN). Immediately after the microhardness test, flexural strength was evaluated using a 3-point bending device attached to a universal testing machine Instron Model 4502 (Instron, USA), with a 1-kN load cell at 5 mm/min crosshead speed and a span of 50 mm. The width and thickness of each specimen were confirmed at three points with a digital micrometer (Digital Caliper, Mutitoyo, Japan).

**Table 1. Characteristics of the material (composition, ratio, curing method, manufacturer, lot number, and expiration date)**

Material	Composition		Ratio	Curing method	Manufacturer	Lot number (expiration date)	
ProBase Hot	P: PMMA	Li: MMA	P/Li 22,5/10 (g/mL)	Heat-curing: 10 hours in 80°C  Cooling in the same water	Ivoclar Vivadent AG, Liechtenstein	P: VT0090 (12/2023)	Li: L31617 (06/2025)
Villacryl Rapid	P: PMMA	Li: MMA	P/Li 23/10 (g/mL)	Heat-curing: 80-90°C →100°C (10min); 100°C (20 min) Cooling at room temperature	Zhermack SpA, Italy	P: Z091000 (08/2023)	Li: Z091000 (08/2023)
V-Print Dentbase	Li: UDMA Bis-EMA TEGDMA		50-100% 25-50% 5-10%	Light-curing by layer (385nm)	VOCO GmbH, Germany	Li: #2122339 (12/10/2022)	
Denture 3D+ Opaque pink	Li: UDMA Bis-EMA HEMA		75% 10-20% 5-10%	Light-curing by layer (385nm)	NextDent BV, The Netherlands	Li: XG511N02 (20/05/2022)	

P – Powder; Li – Liquid; PMMA – poly(methyl methacrylate); MMA – methyl methacrylate; UDMA – urethane dimethacrylate; bis-EMA – bisphenol-A ethoxyethyl dimethacrylate; TEGMA – triethylene glycol dimethacrylate; HEMA – hydroxyethyl methacrylate

Data were analyzed using IBM SPSS Statistics for Macintosh, version 27 (IBM Corp., USA). After assessing normality and homogeneity of variance (Shapiro-Wilk and Levene tests,  $p > 0.05$ ), microhardness data were analyzed with one-way ANOVA followed by Tukey post-hoc tests. Since normality (Shapiro-Wilk test,  $p = 0.019$ ) and homoscedasticity (Levene test,  $p = 0.013$ ) were not verified for flexural strength, data were analyzed with the non-parametric Kruskal-Wallis test. In all statistical tests, a significance level of 5% ( $\alpha = 0.05$ ) was considered.

**Results**

Microhardness mean values ranged between 11.6 KHN for V-Print Dentbase and 14.9 KHN for Villacryl Rapid (Table 2). ANOVA showed statistically significant ( $p < 0.001$ ) differences in microhardness between the resins tested (Figure 1). Both conventional heat-cured resins showed statistically significant ( $p < 0.001$ ) higher microhardness than the two 3D-printed light-cured resins. Comparing same-type resins, statistically significant higher microhardness values were found for Denture 3D+ ( $p = 0.003$ ) compared to V-Print Dentbase and for Villacryl Rapid ( $p = 0.01$ ) compared to Probase Hot (Figure 1).

Regarding flexural strength, the highest mean value (123.8 MPa) was achieved with V-Print Dentbase, while Villacryl Rapid showed the lowest mean value (101.8 MPa) (Table 2). Despite these results, no statistically significant ( $p = 0.527$ ) differences were found in flexural strength between the four resins studied (Figure 2).

**Discussion**

This study compared the microhardness and flexural strength of denture base resins produced by the digital additive and the conventional techniques. 3D-printed light-cured resins showed lower microhardness and similar flexural strength compared to heat-cured resins produced conventionally.

Higher microhardness in denture base materials is usually related to higher resistance to abrasion and surface wear.<sup>27</sup> In the present study, printed resins based on light-curing dimethacrylate monomers showed lower microhardness than conventional heat-cured PMMA resins. Thus, this study's first null hypothesis is rejected, which agrees with the literature.<sup>28</sup> Different chemical compositions and the higher degree of cure associated

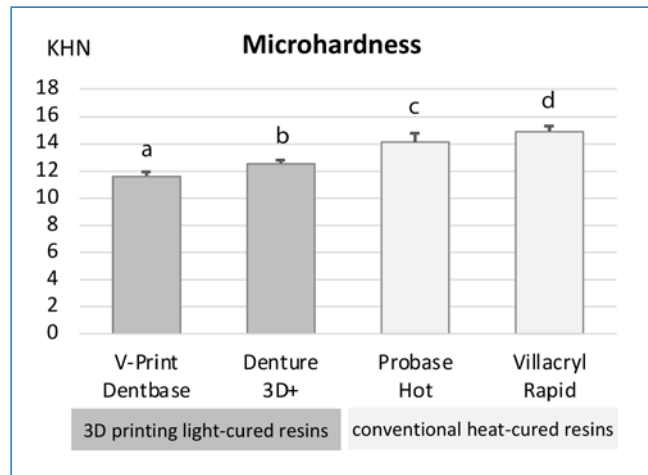


Figure 1. Microhardness (KHN) distribution of denture base acrylic resins' groups (n=8). Bars with different letters were statistically different ( $p < 0.05$ ).

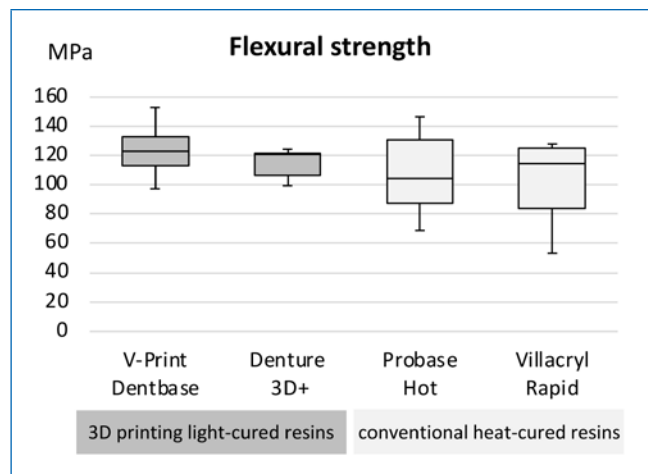


Figure 2. Boxplots of flexural strength (MPa) distribution of denture base acrylic resins' groups (n=8). Differences between resins were not statistically significant ( $p > 0.05$ ).

with heat-cured resins may explain these results. Other studies concluded that 3D-printed acrylics had lower mechanical properties than most other denture base materials.<sup>7,26-28</sup>

**Table 2. Mean, standard deviation, median, interquartile range, and minimum and maximum values of microhardness (KHN) and flexural strength (MPa) per group of resins (n=8)**

Test	Resin	M(SD)	m(IQR)	Min	Max
Knoop Microhardness (KHN)	V-Print Dentbase	11.6(0.34)	11.8(0.53)	11.03	11.98
	Denture 3D+	12.5(0.29)	12.6(0.32)	11.98	13.00
	Probase Hot	14.1(0.69)	13.9(0.80)	13.32	15.53
	Villacryl Rapid	14.9(0.41)	14.79(0.72)	14.33	15.48
Flexural Strength (MPa)	V-Print Dentbase	123.8(18.33)	122.6(31.39)	97.13	152.51
	Denture 3D+	114.5(10.17)	120.5(19.14)	99.58	124.08
	Probase Hot	107.7(27.70)	104.6(48.53)	68.70	146.10
	Villacryl Rapid	101.8(29.08)	114.0(54.36)	53.27	127.85

KHN – Knoop hardness number; MPa – Megapascal; M – mean; SD – standard deviation; m – median; IQR – interquartile range; Min – minimum value; Max – maximum value

Comparing the two 3D-printed resins, V-Print Dentbase showed lower microhardness than Denture 3D+. Despite having the same polymerization type, the different chemical compositions may lead to different mechanical properties.<sup>28</sup> V-Print Dentbase has 20%-50% of bis-EMA in its composition against 10-20% in Denture 3D+ (Table 1), which can explain the reduction in microhardness due to bis-EMA usually increasing the viscosity of resins.<sup>23</sup> Apart from the common monomers, the V-Print Dentbase resin comprises TEGMA, while Denture 3D+ resin contains HEMA. TEGMA monomer is added to reduce the viscosity promoted by bis-EMA but may negatively affect the mechanical properties due to promoting water absorption, increasing polymerization shrinkage, and leading to a greater release of residual monomer.<sup>23,24</sup>

The second null hypothesis was not rejected because the resins did not show significant differences in flexural strength. The light-cured printed materials V-Print Dentbase and Denture 3D+ obtained mean values similar to heat-cured conventional resins Probases Hot and Villacryl Rapid. These results do not agree with the literature, where printed resins have shown lower flexural strength than heat-cured conventional resins.<sup>26,28,33</sup> Previous studies assumed that the bond between the layers of printed resins was weaker than the polymer network within each layer, which could be explained by the shrinkage stress and porosity that occur during light-curing.<sup>21,27,33,34</sup> Also, these polymers were made of different monomers with weaker bonds to the main matrix than MMA, which could result in a plasticizer effect on the core of the resin.<sup>23,24</sup> However, efforts have been made to improve the core properties of printed resins. Factors like the width of each layer, the built orientation settings, and printing with recommended 3D printers can play an important role in optimizing the properties of printed resins to make them comparable to conventional ones.<sup>8,21,25</sup>

Specimens were stored throughout the study in distilled water at 37°C. Like the humid environment of the oral cavity, water immersion exerts a plasticizing effect on resins, capable of interfering with their materials' flexural strength.<sup>13</sup> However, all groups showed flexural strength values above 65 MPa, the minimum value recommended for denture bases according to ISO standards for acrylic resins.<sup>13,31,35</sup>

One of this study's limitations is having been performed in vitro under laboratory-controlled conditions, thus requiring a cautious extrapolation of the results concerning these materials' behaviors in intraoral conditions. In the future, it would be relevant to further consider the biomechanical behavior of these resins submitted to physical, chemical, and mechanical aging to simulate the different biodegradation processes that denture materials are subjected to in the intraoral environment. Another limitation is the variability of the flexural strength results, especially in conventional resins, which may indicate the need for a study with a larger sample.

## Conclusions

The printed resins V-Print Dentbase and Denture 3D+ showed lower microhardness values than the conventional resins Probases Hot and Villacryl Rapid, but no differences were found regarding flexural strength.

## Conflict of interest

The authors have no conflicts of interest to declare.

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## Ethical disclosures

**Protection of human and animal subjects.** The authors declare that no experiments were performed on humans or animals for this study.

**Confidentiality of data.** The authors declare that no patient data appear in this article.

**Right to privacy and informed consent.** The authors declare that no patient data appear in this article.

## CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

**Cristina Bettencourt Neves:** Conceptualization, Methodology, Project administration, Resources, Supervision, Visualization, Writing – review & editing. **Ana Filipa Chasqueira:** Investigation, Methodology, Validation, Visualization, Writing – review & editing. **Patrícia Rebelo:** Data curation, Investigation, Visualization, Writing – original draft. **Mariana Fonseca:** Data curation, Investigation, Visualization, Writing – original draft. **Jaime Portugal:** Conceptualization, Methodology, Formal analysis, Supervision, Visualization, Writing – review & editing. **Ana Bettencourt:** Methodology, Visualization, Writing – review & editing.

## ORCID

Cristina Bettencourt Neves  0000-0003-4327-1372  
 Ana Filipa Chasqueira  0000-0002-4274-7423  
 Patrícia Rebelo  0000-0002-1751-2016  
 Mariana Fonseca  0000-0002-2202-6119  
 Jaime Portugal  0000-0001-5058-6554  
 Ana Bettencourt  0000-0002-8498-5892

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