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ORIGINAL ARTICLE

Flexural Properties and Hardness of CAD-CAM Denture Base Materials

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Abstract

Purpose: To compare flexural strength, elastic modulus, and surface hardness of computer aided design and computer aided manufacturing CAD-CAM milled, 3D-printed, and heat-polymerized denture base resins.

Materials and methods: A total of 120 specimens were fabricated from heat-polymerized acrylic resin (HP), milled resin (Avadent and IvoCad), and 3D-printed resin (ASIGA, FormLabs, and NextDent). The specimens were divided into 6 groups according to the type of denture base material (n = 20/material) (10/flexural properties and 10/hardness). Flexural strength and elastic modulus of the specimens were evaluated by 3-point bending test and surface hardness by Vickers hardness test. To test flexural properties, the specimens were fabricated according to ISO 20795-1:2013 standards ($64 \times 10 \times 3.3 \pm 0.2$ mm). The dimensions for hardness test were $15 \times 10 \times 2.5 \pm 0.2$ mm. Scanning electron microscope was used to evaluate the surface morphology of the fractured specimens. The means and standard deviations were calculated, followed by one-way ANOVA and Tukey post-hoc test ($\alpha = 0.05$).

Results: Milled resins showed significantly higher values for flexural strength, elastic modulus, and surface hardness, followed by HP and then 3D-printed resins ($p < 0.001$). Within milled groups, flexural strength of AvaDent was significantly higher than IvoCad ($p < 0.001$), while elastic modulus and hardness didn't show significant difference. Within 3D-printed resins, ASIGA showed the highest flexural strength and elastic modulus, insignificantly with FormLabs ($p = 0.595$) and significantly with NextDent ($p = 0.008$). ASIGA also showed significantly the highest hardness among the 3D-printed groups. No significant difference was found between FormLabs and NextDent in flexural strength ($p = 0.357$), elastic modulus ($p = 1.00$), or surface hardness ($p = 0.987$).

Conclusion: CAD-CAM milled resins had greater flexural properties and hardness compared to heat-polymerized acrylic resin and 3D-printed resins. Although 3D-printed samples showed the lowest values of tested properties, the flexural strength and modulus were above clinically acceptable values.

KEYWORDS
3D-printing, CAD-CAM, denture base resin, flexural properties, milled, surface hardness

Heat-polymerized acrylic resin (HP) has been widely used in the fabrication of dental prostheses, however it still has some deficiencies. Conventionally used HP shows inferior mechanical properties and a high degree of polymerization shrinkage (linear and volumetric) which results in dimensional inaccuracies of the end product. To overcome this, resin composition modifications, different polymerization techniques, resin reinforcements with fillers and nano-fillers, as well as the implementation of digital technologies for removable prostheses fabrication, have been suggested.[1] The fabrication of a dental prosthesis from a pre-polymerized acrylic puck has eliminated the issue of polymerization shrinkage, since the prosthesis is milled out of an already polymerized and shrunken block of material to the final dimensions, thus producing better adaptation fit and high mechanical performance.[2,3]

Digital technology is increasingly used in the dental field, particularly in computer-aided design and computer-aided manufacturing (CAD-CAM) of dental restoration.[4] CAD-CAM technology allows the fabrication of dental prostheses with high accuracy, in less time and with less discomfort to the patient compared to traditional methods; in addition, it offers direct duplication of an existing denture.[5,6] Two methods are available for CAD-CAM fabrication of dental restorations: 3D-printing (additive) and milling methods (subtractive).[5]

Fabrication of dentures is more common using the milling technique.[5] The accuracy of milled dentures is related to the material used and the milling tools (number and size of milling burs).[7,8] High strength and hardness of the milled material are due to decreased structural defects within the final material and presence of inorganic fillers.[7,9] Additionally, a number of studies reported that milled CAD-CAM fabricated specimens present superior color stability compared to heat-, self-, and light-cured acrylics[10] or 3D-printed acrylic resin.[11] However, subtractive production of dentures produces large amounts of waste and requires a vast amount of raw material.[12] On the other hand, 3D-printing of a dental prosthesis is accomplished by layering of the material. Therefore, it is considered less expensive than milling due to reduced material waste and absence of wear of milling tools.[4] In addition, 3D-printing allows the production of several objects at the same time and printing of large complex designs.[13,14] 3D-printing offers a more sustainable production source with minimal waste but with lower accuracy, poorer retention, and low printing resolution.[11]

Flexural properties are major properties that need to be considered in the fabrication of dentures since they are subjected to flexural stress during mastication that could lead to deformation or fracture in the long term.[15] Surface hardness is another important property of denture base material to resist surface abrasion that would consecutively reduce microbial colonization and provide color stability of denture bases.[16] Therefore, denture base materials are requested to have acceptable strength and hardness and comply with values recommended by ISO 20795-1:2013 for flexural strength (65 MPa) and modulus (2 GPa).[17]

Previous studies investigated the properties of HP and CAD-CAM denture base materials. However, studies comparing HP, milled, and 3D-printed resins remain limited. Meanwhile contradicting results have been reported regarding their mechanical and physical properties. Some studies reported superior strength of HP over milled specimens,[18,19] while other studies reported opposite results.[20,21] Similarly, higher hardness of milled resin than HP was reported,[9,18,21] as well as insignificant difference between both materials.[19,22] Therefore, this study evaluated different properties of multiple brands of denture base materials including two milled resins and three 3D-printed resins, that to the authors knowledge, were not compared in a previous study. The aim of this study was to compare the flexural properties and hardness of CAD-CAM milled and 3D-printed denture base materials to heat-polymerized acrylic resin. The study hypothesis is that HP and CAD-CAM fabricated denture base materials will have similar flexural strength, elastic modulus, and surface hardness.

MATERIALS AND METHODS

Based on previous studies,[9,23] a power analysis technique was used to calculate the sample size with the power of 80% and level of significance 0.05. Hence, the sample size calculation confirmed that 120 specimens were required ($n = 10$). One type of HP (Major.Base.20, Major Prodotti Dentari, Moncalieri, Italy), two types of milled acrylic discs, AvaDent (AvaDent, Global Dental Science Europe, Tilburg, The Netherlands) and IvoCad (IvoBase CAD, Ivoclar Vivadent, Schaan, Liechtenstein), and three types of 3D-printed resins, ASIGA (ASIGA DentaBase, Asiga pty Ltd, Alexandria, Australia), FormLabs (FormLabs Denture Base LP, MA), and Denture 3D+ (NextDent B.V., Soesterberg, The Netherlands) were investigated in this study. Acrylic specimens were fabricated with required dimensions per test according to ISO 20795-1:2013 standards; $64 \times 10 \times 3.3 \pm 0.2$ mm for flexural properties (flexural strength and elastic modulus). The dimensions of specimens for hardness test were$15 \times 10 \times 2.5 \pm 0.2$ mm. Specimens (120, 60/flexural properties and 60/hardness) were divided into 6 groups ($n = 10$/test) according to the material type: HP, Avadent, IvoCad, ASIGA, FormLabs, and NextDent.

HP specimens were processed and polymerized using a conventional water bath technique at 70°C for 90 minutes followed by 100°C for 30 minutes, as detailed in a previous study.[23] After complete polymerization, specimens were finished where by any resin flashes were removed using tungsten carbide bur.

For milled specimens (AvaDent and IvoCad), prepolymerized polymethylmethacrylate (PMMA) blocks were fixed on a sectioning machine (IsoMet 5000 Linear Precision Saw, Buehler, IL) and sectioned to the desired dimensions using a diamond saw in wet conditions.[9,24]

The 3D-printed specimens were designed using open software (123D design, Autodesk, version 2.2.14, CA) and

TABLE 1 Materials and equipment used in the present study

Group	Material	Manufacturing technique	Resin mixing unit	Processing unit	Thickness of layer	Orientation	Post-processing steps and unit	Finishing and polishing
Heat-polymerized acrylic resin	Major.Base.20, Major Prodotti Dentari, Moncalieri, Italy	Conventional water bath processed	Hand mixed	KaVo Elektrotechnisches Werk GmbH, Leutkirch, Germany	Full thickness of specimen	NA	Deflasking	Tungsten carbide bur to remove excess acrylic followed by 1200 grit sand paper (MicroCut PSA; Buehler, IL, USA) using Metaserv 250 grinder-polisher; Buehler GmbH)
AvaDent	AvaDent denture base puck (AvaDent, Global Dental Science Europe, Tilburg, The Netherlands)	Cut	Pre-polymerized	Cut into desired shape using precision saw (IsoMet 5000 Linear Precision Saw, Buehler, USA)	Full thickness of specimen	Horizontal within the disc	None	
IvoCad	IvoBase CAD (Ivoclar Vivadent, Schaan, Liechtenstein)							
ASIGA	ASIGA DentaBase, (Asiga pty Ltd, Alexandria, Australia)	3D-Printing	(LC-3D Mixer, NextDent, 3D systems, Vertex Dental B.V., Soesterberg, Netherland)	ASIGA Max Printer (DLP)	50 μm/layer	90°	Washing with Isopropyl Alcohol	
FormLabs	FormLabs Denture Base LP (FormLabs, Somerville, MA, USA)			Form 2 Printer (SLA)				
NextDent	Denture 3D+ (NextDent B.V., Soesterberg, The Netherlands)			NextDent 5100 3D Printer (SLA)				

then converted to a standard tessellation language (STL) file. The printing order was sent to three printers (ASIGA; ASIGA, Form 2; FormLabs, and NextDent 5100 3D system, NextDent). The parameters of each printer were adjusted according to manufacturer's recommendations (Table 1). After all specimens were prepared and excess acrylic and supports were trimmed, polishing was performed with 1200-grit sandpaper (MicroCut PSA; Buehler) using a polishing machine (Metaserv 250 grinder-polisher; Buehler GmbH) in wet conditions. One investigator polished all specimens and then reevaluated the specimens' dimensions to 0.01 μm accuracy using a digital caliber. Specimens with proper dimensions were stored in distilled water at 37°C for 48 hours before testing.

Evaluation of flexural strength, elastic modulus, and surface hardness were performed as explained in a previous study.[23] The specimens were tested immediately after retrieval from water and without drying. Flexural strength and elastic modulus were measured by three-point bending test using a universal testing machine (Instron Model 8871; Instron Corp., Canton, MA). Surface hardness was calculated by using a hardness tester (Wilson Hardness; ITW Test & Measurement, GmbH, Shanghai, China) with a Vickers diamond indenter (25-gf load for 30 seconds).

Scanning electron microscopy tool (SEM, TESCAN VEGA3 LM model, Czech Republic at 20 kV) was applied to evaluate the surface morphology of the fractured specimens of all groups (HP, AvaDent, IvoCad, ASIGA, FormLabs, and

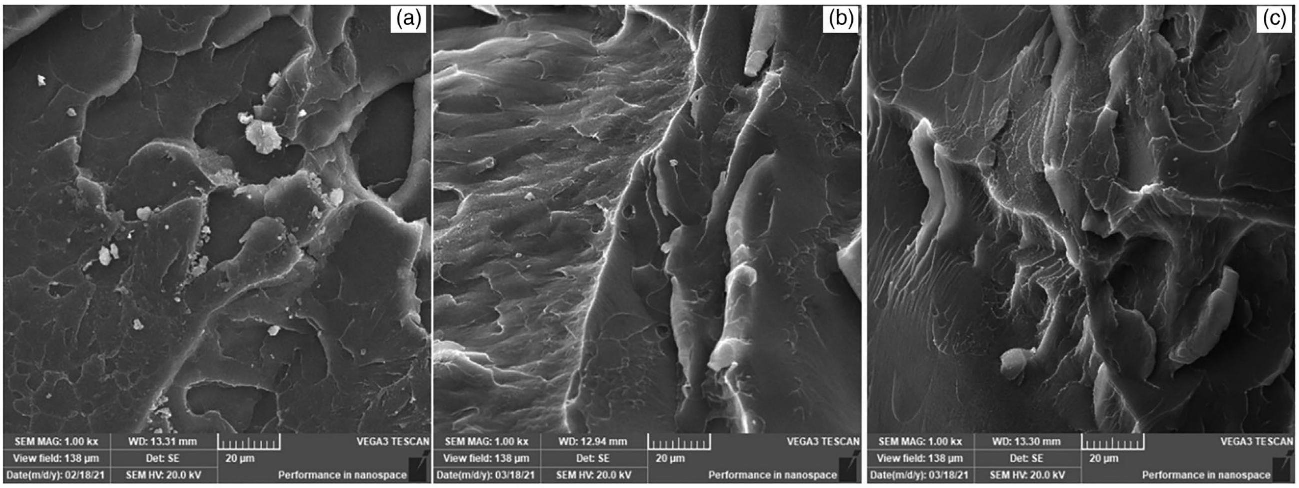

FIGURE 1 Representative SEM images for fractured specimen: a, HP; b, AvaDent; and c, IvoCad

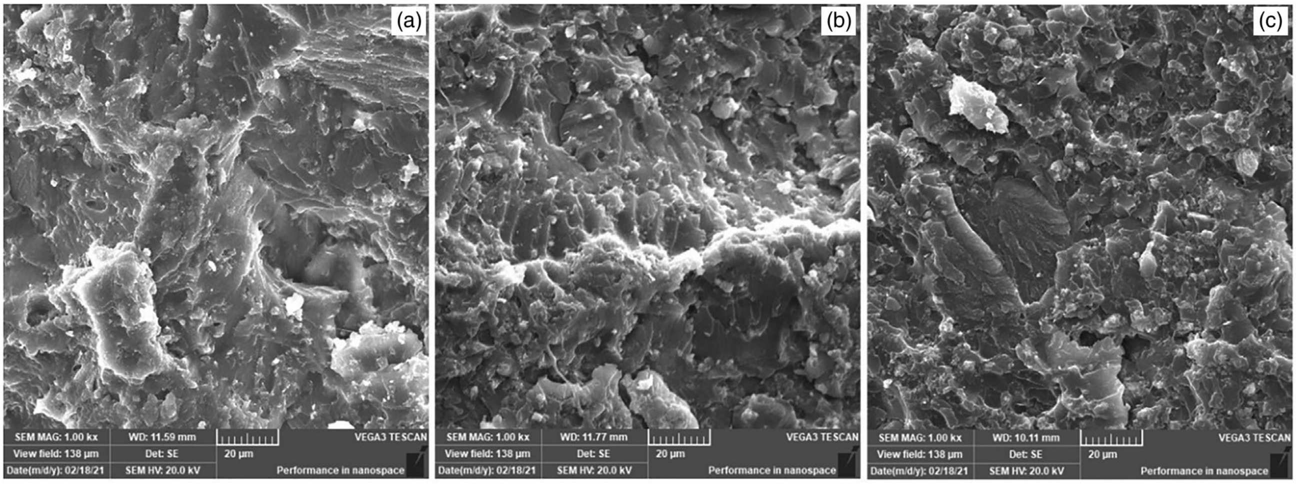

FIGURE 2 Representative SEM images for fractured specimen: a, ASIGA; b, FormLabs; and c, NextDent

NextDent). SEM micrographs were displayed at representative magnification of ×1000 for all the specimens to highlight their important surface features (Figs 1 and 2).

Normality of the data was tested first using Shapiro-Wilk which yielded insignificant results confirming normal distribution of data. Hence, parametric tests were used for analysis. However, means and standard deviations were calculated first for descriptive presentation of the data followed by one-way ANOVA and Tukey post-hoc test for inferential statistical analysis. All p-values less than 0.05 were considered statistically significant.

RESULTS

The mean values, standard deviations, ANOVA, and post hoc results are presented in Table 2. There were significant differences between tested materials for all properties based on one-way ANOVA results ($p < 0.001$).

With regards to flexural strength, AvaDent (94.5 ± 2.2 MPa) and IvoCad (87.6 ± 1.1 MPa) showed the highest flexural strengths followed by HP (82.7 ± 1.6 MPa), then 3D-printed resins: ASIGA (70.1 ± 0.8 MPa), FormLabs (69.1 ± 1.5 MPa), and NextDent (67.7 ± 1.2 MPa). AvaDent and IvoCad showed significantly higher flexural strength when compared with other groups ($p < 0.001$). Also, AvaDent was significantly higher than IvoCad ($p < 0.001$). Within 3D-printed resins, ASIGA showed the highest flexural strength insignificantly with FormLabs ($p = 0.595$) and significantly with NextDent ($p = 0.008$). No significant difference was found between FormLabs and NextDent ($p = 0.357$).

The highest elastic modulus values were recorded with IvoCad (9507.4 ± 479.1 MPa) and AvaDent (8547.5 ± 397.5 MPa) that were significantly different from all other tested groups ($p < 0.001$). The elastic moduli of 3D-printed resins were significantly lower in comparison with HP ($p < 0.001$). Comparing 3D-printed resins, ASIGA (5258.9 ± 325.9 MPa) showed the highest elastic modulus

TABLE 2 Mean and standard deviations (SD) and significance between materials for all tested properties

	Properties		
	Flexural strength (MPa)	Elastic modulus (MPa)	Hardness (VHN)
Materials	Mean (SD)	Mean (SD)	Mean (SD)
HP	82.7 (1.6)	6458.8 (296.6)	39.6 (9.9)
AvaDent	94.5 (2.2)	8547.5 (397.5)[a]	46.3 (2.9)[a]
IvoCad	87.6 (1.1)	9507.4 (479.1)[a]	46.9 (5.9)[a]
ASIGA	70.1 (0.8)[a]	5258.9 (325.9)[b]	31.1 (7.5)
FormLabs	69.1 (1.5)[a,b]	4792.1 (421.6)[b,c]	17.5 (2.8)[b]
NextDent	67.7 (1.2)[b]	4750.3 (288.2)[c]	15.2 (0.15)[b]
One-way ANOVA	F = 587.278	F = 223.089	F = 39.571
	$p < 0.001$*	$p < 0.001$*	$p < 0.001$*

Same superscript letters per column denotes statistically insignificant difference ($p > 0.05$).

insignificantly with FormLabs ($p = 0.075$), and significantly with NextDent ($p = 0.041$). No significant difference was found between FormLabs and NextDent ($p = 1.00$).

The highest mean of surface hardness was found with IvoCad (46.9 ± 5.9) followed by AvaDent (46.3 ± 2.9) and HP (39.6 ± 9.9), respectively, while the lowest values were recorded with 3D-printed resins, ASIGA (31.1 ± 7.5), FormLabs (17.5 ± 2.8), and NextDent (15.2 ± 0.15). Tukey pairwise comparisons revealed that there were no statistically significant differences between IvoCad vs AvaDent ($p = 1.00$), and FormLabs vs NextDent ($p = 0.987$), while ASIGA showed significant differences with FormLabs and NextDent ($p < 0.001$).

Representative SEM images of the fractured surfaces of all groups of specimens are shown in Figures 1 and 2. In HP specimens (Fig 1A), lamellae with minor blunt steps and some areas with smooth background represented brittle to intermediate mode of fracture. For milled specimens (AvaDent, and IvoCad), compact surfaces with uniform sharp lamellae were found with AvaDent (Fig 1B), while random, slightly sharp lamellae with compact resin and features of ductile fracture mode were observed for IvoCad surface (Fig 1C). On the other hand, the surface topography of the 3D-printed fractured specimens displayed irregular surfaces with absence of normal lamellae. However, faint small lamellae with minor cracks were found, as well as some small voids (Fig 2). Moreover, a number of particles were also observed as they appeared in a bright contrast highlighting the layered structure of these specimens.

DISCUSSION

This study was conducted to evaluate the flexural properties and surface hardness of denture base resins fabricated through different manufacturing techniques (HP, CAD-CAM milled, and CAD-CAM 3D-printed). The results of this study revealed the statistical variation between the different classifications of resins with regards to all tested properties resulting in rejection of the study hypothesis.

Flexural strength is the stress absorbed by the material just before its failure, while the modulus of elasticity represents the rigidity/flexibility of the material.[9,20] The higher the value, the more rigid the material.[20] The denture base must have certain properties to withstand occlusal forces. It has to show some degree of flexibility to reduce the possibility of fracture while maintaining its shape to distribute the load equally to underlying structures.[21] The use of 3-point bending test has been recommended by ISO 20795-1:2013 [17] to measure the flexural properties of polymers. On the other hand, hardness is a measure of the resistance to localized plastic deformation induced by either mechanical indentation or abrasion.[9,16]

According to the results of this study, milled CAD-CAM denture base resins showed the highest flexural properties and surface hardness compared to HP and 3D-printed resins. Similar findings were reported by previous studies for flexural properties (flexural strength and modulus)[20,21,25–28] and surface hardness.[9,18,21,29]

The higher values of flexural properties obtained for milled specimens might be related to the unique manufacturing process of the pre-polymerized pucks used for denture milling.[30] These CAD-CAM PMMA pucks are manufactured under strict conditions of high pressure and temperature.[31] During this manufacturing process, the PMMA chains are elongated by near optimum polymerization resulting in longer chains of PMMA, denser structure with minimal intermolecular distance, fewer unreacted monomer, and lower porosity.[6,20,32] Additionally, since these specimens are prepared of pre-polymerized pucks, there is no risk of further shrinkage after specimen production which improves fit and eliminates the possible stress incorporation as seen in heat-polymerized resin.[2,5,20] The higher hardness of milled CAD-CAM resin could also be related to its processing under high temperature and pressure, resulting in less residual monomer and minimal plasticizing effect.[33]

Contrary to the results of this study, are the findings of Ayman,[18] and Pacquet et al[19] who reported superior flexural strength of HP over milled resin. The contradicting results might be related to discrepancy of specimen size between the two studies [this one (64 × 10 × 3.3 mm) and Pacquet et al[19] (65 × 10 × 2.5 mm), or the difference in chemical composition of the tested materials between this study (Major.Base.20, AvaDent, IvoCAD, Asiga, NextDent, and FormLabs) and Paquet et al[19] and Ayman[18] (HP- Probase Hot, IvoCAP, and IvoCAD vs. Vertex and CAD/CAM Polident d.o.o.). In disagreement with our findings, some studies reported insignificant differences between the hardness of milled CAD-CAM and HP.[19,22] The reason for variation of results with this study might result from the different materials tested in these studies.

The literature is scarce in investigational studies evaluating the properties of 3D-printed resins. Currently, few studies[9,23] reported similar results to those seen in this study. 3D-printed resin showed significantly lower flexural strength and surface hardness values compared to milled and HP. The chemical composition of the photopolymerized resin (used for 3D-printing) and structure of final product may be one of the sources of deficit in flexural strength results.[9] The reduced flexural strength and surface hardness of 3D-printed resin compared to milled and HP could result from its fabrication in layers, post-curing factors, and the presence of residual monomer.[23] The printing orientation in a perpendicular direction to the surface being tested for hardness may have resulted in more separation between the layers during load application, resulting in lower hardness values. This has been suggested by Kebler et al[34] who reported low interlayer bonds that are easily separated with shear forces.

The 3D-printed specimens were additively created through the gradual curing of thin layers of the resin material until the final shape was achieved. The thickness and orientation of the layers could play a major role in surface and mechanical properties of the final product.[34,35] In this study, the layers were printed in 50 μm thickness in order to improve the curing of the resin by decreasing the layer thickness and minimizing the polymerization shrinkage between the layers.[34] Standardizing the layer thickness between different printers and materials eliminated the possible effect of thickness discrepancy on the tested properties. However, this production process (3D-printing) of the specimens is prone to void inclusion within or between consecutive layers resulting in weaker structure. Additionally, specimens were printed at 90° orientation to the build platform resulting in the layers being parallel to the applied load once the specimens were horizontally supported on the jig for flexural strength testing. Based on this, one would assume that this printing orientation might produce the lowest flexural strength and that bonding between consecutive layers would be weaker than molecular bonding within each layer. However, the reverse was confirmed in a previous study.[35] Because all 3D-printed specimens in this study were manufactured at the same orientation, this comparison between the different build orientations is not valid. Additionally, comparison between similar print orientation in this study and Unkovskiy et al[35] was not possible owing to the differences in specimens' dimensions. Due to the processing technique of 3D-printed resin, a lower conversion rate is anticipated compared to other forms of PMMA used in this study, resulting in lower mechanical properties.[36]

Based on the SEM images, HP specimens showed smooth background with few faint lamellae indicative of brittle type of fracture. On the other hand, milled specimens showed compact structure and sharp projections suggestive of ductile fracture. On the opposite was the topography of 3D-printed specimens where great irregularity and absence of organized projections/lamella, in addition to void inclusion and layering of structure, were detected. These features were linked to intermediate to brittle type of fractures and weaker build structure.

Nevertheless, and regardless of the type of resin, all the groups in this study demonstrated flexural strength and moduli above the minimum required value by the ISO 20795-1:2013[17] set at 65 MPa and 2 GPa, respectively, and in contrast to Ucar et al[37] who reported acceptable flexural strength for different HP, but not modulus.

The results of this study revealed that milled resin showed the highest flexural strength, elastic modulus, and surface hardness compared to HP and 3D-printed resins which had the lowest values for all tested properties. 3D-printed resins require further improvement in the chemical composition or the manufacturing process to improve their mechanical properties for clinical applicability.

This study investigated different types of denture base materials fabricated by different technologies and including different brands of milled and 3D-printed resins. However, the tests were not performed in conditions simulating those in the oral cavity, i.e., the specimens were not subjected to thermal or pH changes. Future studies should investigate the properties of CAD-CAM denture base materials after aging to determine the durability of these materials in clinical use. Also, the factors that may influence the mechanical properties of 3D-printed resins need further investigations such as the post curing time, thickness and orientation of printed layers, and reinforcement with fillers.

CONCLUSIONS

Material types and fabrication techniques affected the flexural properties and hardness of denture base resins. Milled denture base resins had the highest flexural strength, modulus, and hardness among the tested groups, followed by HP and the 3D-printed resin groups, respectively. Regardless of the order of tested groups, the lowest reported flexural strength and elastic modulus values conformed to the ISO recommended values and were above the clinically accepted values, suggesting the suitability of these materials (3D-printed resins) for clinical use.

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