

Effect of Aging on the Mechanical Properties of CAD/CAM–Milled and 3D-Printed Acrylic Resins for Denture Bases

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Purpose: The purpose of this study was to investigate the mechanical properties of acrylic resins at different aging times for denture bases manufactured using the conventional method, microwave processing, milling, and 3D printing. **Materials and Methods:** A total of 160 rectangular samples ($64 \times 10 \times 3.3 \pm 0.03$ mm) were prepared, divided among the four main resin groups, and subdivided into four analysis times (T0, T1, T2, and T3), resulting in 10 samples per subgroup. The samples were stored in distilled water at $37^\circ \pm 2^\circ\text{C}$ for 24 hours (T0), then subjected to thermocycling at temperatures of $5^\circ \pm 1^\circ\text{C}$ and $55^\circ \pm 1^\circ\text{C}$ in different numbers of cycles: 5,000 (T1); 10,000 (T2); and 20,000 (T3). The mechanical properties evaluated were surface microhardness, flexural strength, and modulus of elasticity. Statistical differences between resin groups and aging time were evaluated using two-way analysis of variance ($P < .05$). **Results:** The 3D-printed resin showed the significantly lowest values of microhardness, flexural strength, and modulus of elasticity compared to other resins ($P < .001$). **Conclusions:** The CAD/CAM–milled denture resin showed mechanical properties similar to those of traditional resins (conventional and microwave-processed). The 3D-printing resin did not show adequate mechanical properties for long-term clinical use. Despite this, new studies are developing better properties of this resin for long-term use. *Int J Prosthodont* 2024;37(suppl):s5–s11. doi: 10.11607/ijp.8376

Polymethyl methacrylate (PMMA) is the acrylic polymer most used to manufacture denture bases due to its acceptable esthetics, easy manipulation, biocompatibility, and low cost.¹ The method traditionally used for the manufacture of complete dentures is heat curing via conventional (bain-marie) or microwave processing.^{1,2} However, with the introduction of CAD/CAM technology in dentistry, the manufacture of dentures by milling and 3D printing has become possible.³

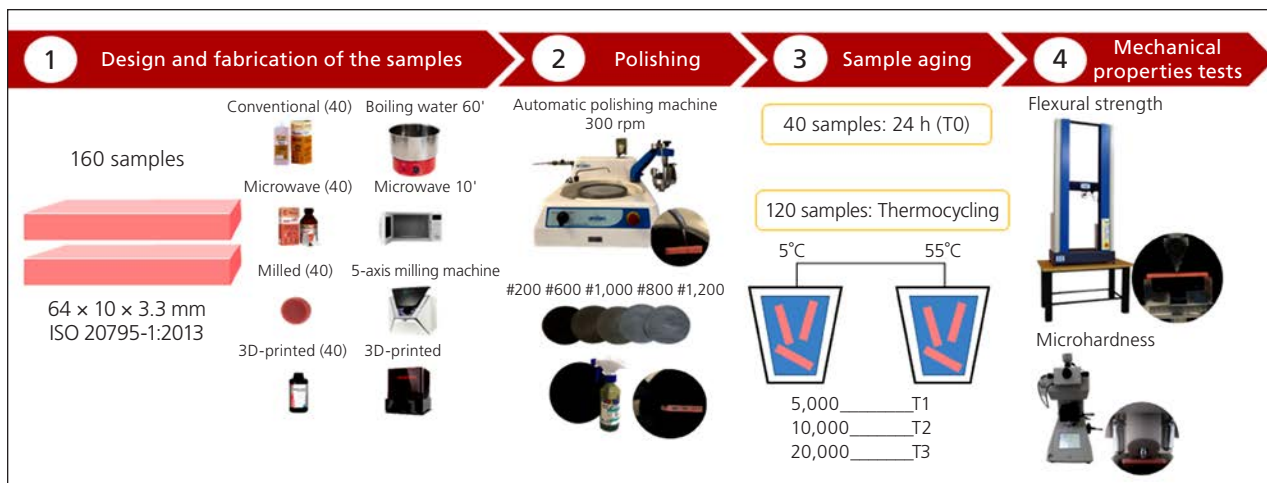
Dentures manufactured using the CAD/CAM method were introduced to reduce clinical time, facilitate the duplication of prostheses, optimize dimensional accuracy, and improve their mechanical properties,^{3,4} given that traditional dentures have a porous surface and low resistance.¹ The milling method uses acrylic resin blocks

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Table 1 Acrylic Resins Used for Denture Base Manufacturing

Material	Brand	Proportion	Manufacture method
Conventional	Onda Cryl, Clásico	14 g powder to 6.5 mL liquid	60 min bain-marie
Microwave	Onda Cryl, Clásico	14 g powder to 6.5 mL liquid	10 min microwave
Milled	Blue Dent, Articon	Blocks condensed	Milling
3D-printed	Liquid resin, Smart Dent	Liquid resin	3D printer

**Fig 1** Visual representation of the study design.

condensed under high pressure and heat, where the polymerization process occurs under standardized conditions, aiming to reduce porosity.⁵ Meanwhile, 3D printing uses a liquid resin, in which the prosthesis is manufactured by deposition of resin layers that are simultaneously light-cured by ultraviolet light, which present less material waste.^{3,6}

The mechanical properties of denture base resins are regularly tested for surface microhardness, flexural strength, and modulus of elasticity.⁷ The hardness of the material determines its resistance to wear, and dentures made with a low-hardness material can be damaged by mechanical brushing, causing pigmentation and retention of bacterial plaque, thus reducing the useful life of the dentures.^{8,9} Dentures must have high flexural strength and a high modulus of elasticity, as they are subjected to repeated flexural forces during mastication that induce internal tensions in the acrylic resin, which cause fatigue failure over time.^{10,11} Having a high strength will reduce the chances of cracks and fissures propagating, which prevents fractures, while the high modulus of elasticity decreases the chances of plastic deformation.¹⁰ Dentures capable of sustaining greater flexure in combination with high cyclic loading may be less subject to clinical failure.¹¹

Therefore, the purpose of this study was to investigate the mechanical properties of acrylic resins at different aging times for denture bases manufactured using the conventional method, microwave processing, milling, and 3D printing. The two hypotheses evaluated in this study were: (1) that significant differences would be found between heat-cured resins and those manufactured by the CAD/CAM system; and (2) that no significant differences would be found over aging times.

MATERIALS AND METHODS

Sample Design and Fabrication

Four acrylic resins for denture bases were selected for this study based on the manufacturing method: conventional (bain-marie), microwave processing, milling, and 3D printing (Table 1). A total of 160 rectangular samples (64 × 10 × 3.3 ± 0.03 mm) were prepared according to the ISO 20795-1:2013 International Standard,¹² evenly divided among the four main resin groups, and further evenly subdivided into four analysis times (T0, T1, T2, and T3), resulting in 10 samples per subgroup (Fig 1).

For the preparation of conventional and microwave-processed samples, molds were made in the sample dimensions, with laboratory silicone (Zetalabor, Zhermack)



Fig 2 All materials used for the confection of conventional and microwave-processed samples, including the mold and muffle used.

and light-addition silicone (Elite HD+, Zhermack) included in plastic muffles (Mufla VIPI-STG, Vipi Odonto Products) with a special type IV plaster (Durone, Dentsply Sirona) (Fig 2). The same resin (medium pink color, Onda Cryl, Clássico) was used for the conventional and microwave processing methods, handled according to the manufacturer's instructions (Table 1). The resin was inserted into the molds, maintained under a load of 14.71 kN for 2 minutes in a hydraulic press (Maxx 1, Essence Dental), and kept on the bench for 30 minutes. The conventional method samples were polymerized in a water bath for 60 minutes in boiling water (100°C), while microwave-processed samples were polymerized for 3 minutes with a power of 30%, followed by 4 minutes with a power of 0%, and 3 minutes with a power of 60% in a microwave (Brastemp). After polymerization, edge irregularities and excess resin were removed using a maxicut (Vicking).^{11,13}

The samples made using the milled and 3D-printing methods were first designed using CAD software (Exocad) according to the sample dimensions. The CAD standard mosaic language files were sent to the CAM software of the milling machine and the 3D printer. PMMA blocks (medium pink color, BlueDent, Articon) were milled in a 5-axis milling machine (SilaMill R5, Siladent) to obtain the milled samples, while a liquid resin (medium pink color, SmartDent) was used in a stereolithographic printer with

digital light-processing technology (MoonRay Model S, VertySystem) to obtain 3D-printed samples.⁸

All samples were subjected to standardized finishing and polishing using sandpaper discs in the following granulation sequence: #200, #600, #1,000 (Carbi-Met, Buehler), and #800, #1,200 (MicroCut, Buehler) coupled to an automatic polishing machine (Auto-Met 250, Buehler) under constant water irrigation at 300 rpm for 30 seconds on each face. After finishing with sandpaper, the samples were polished with a polycrystalline diamond solution (MetaDi Supreme, Buehler) passed on all flat faces, and applied for 5 seconds on a felt disc attached to an automatic polishing machine at 300 rpm. The specimens were then cleaned using ultrasound (UltraSonic Clean, UNIQUE) for 5 minutes to remove the residue. The 64 × 10 × 3.3 mm measurements were confirmed with a digital caliper with a resolution of 0.01 mm (Digimatic, Mitutoyo) at 5 points for ± 0.03 mm (Fig 3).^{11,13–15}

Sample Aging

Before mechanical testing, all samples from the four resin manufacture groups were randomized according to aging time. The samples were stored in distilled water in an incubator (Incubadora BOD, Cienlab) at 37° ± 2°C for 24 hours before the first mechanical tests (T₀).¹⁶ After the initial analysis, all samples were subjected



Fig 3 Samples were made of each resin manufacturing type according to the established measurements: (a) conventional, (b) microwave-processed, (c) milled, and (d) 3D-printed.

to thermocycling (Model MSCT-3, Convel) in distilled water with alternating 30-second baths at a temperature of $5^{\circ} \pm 1^{\circ}\text{C}$ and $55^{\circ} \pm 1^{\circ}\text{C}$ (70 seconds per cycle; 30-second residence time; 5-second transfer time) in different numbers of cycles: 5,000 (T1), 10,000 (T2), and 20,000 (T3) (Fig 1).^{16,17} Under the conditions presented, thermocycling represents a 6-month clinical aging of the acrylic resin for every 5,000 cycles.^{18,19}

Mechanical Properties Tests

Surface microhardness was evaluated with a microhardness tester (HMV-2T, Shimadzu) equipped with a Knoop diamond according to ASTM E384-11 guidelines.²⁰ Three markings were made on each sample at 500- μm distances with a static vertical load of 0.24 N for 10 seconds. A single operator (V.A.A.B.) measured the longest diagonal of each marking, and the mean of the three measurements was defined as the microhardness value (kgf/mm^2) of the sample.²¹

The flexural strength and modulus of elasticity were tested using a three-point bending test on a universal testing machine (EMIC) according to the ISO

20795-1:2013 guidelines¹² for denture base polymers. The samples were positioned on circular support beams with a span of 50 mm each. A load cell of 100 kg/F was used to apply a constant load to the center of the sample at a crosshead speed of 5 mm/minute until fracture. The moment of fracture was designated as the moment when the applied load decreased to 0. Data were recorded using a software program (Tesc, Intermetric). The flexural strength and modulus of elasticity were calculated using the following equations:

- Flexural strength (MPa) = $3Fl/2bh^2$
- Modulus of elasticity (MPa) = $Fl^3/4bh^3d$

In these equations, F is the maximum load, l is the distance between the supports, b is the width, h is the height, and d is the deflection.^{15,22}

Statistical Analysis

Data sets were analyzed using statistical software (SigmaPlot version 14.5, Systat Software). Continuous measures with means and standard deviations were

**Table 2** Knoop Microhardness Test Results According to Group and Aging Time

Group	T0	T1	T2	T3
Conventional	21.64 ± 0.85 ^{Aa}	21.04 ± 1.17 ^{Aa}	21.04 ± 0.53 ^{Aa}	19.93 ± 0.45 ^{Ab}
Microwave	20.95 ± 0.47 ^{Ba}	20.70 ± 0.71 ^{Aa}	20.50 ± 0.27 ^{Aa}	20.44 ± 0.63 ^{Aa}
Milled	21.35 ± 0.41 ^{ABa}	20.91 ± 0.41 ^{Aa}	21.18 ± 0.32 ^{Aa}	20.02 ± 0.19 ^{Ab}
3D-printed	17.45 ± 1.10 ^{Ca}	14.50 ± 1.27 ^{Bb}	14.52 ± 0.79 ^{Bb}	11.72 ± 0.69 ^{Bc}

T0 = 24 hours; T1 = 6 months; T2 = 12 months; T3 = 24 months.

Different capital letters in columns and different lowercase letters in rows show a significant difference ($P < .05$).

Data are presented in kgf/mm² as mean ± SD values.

Table 3 Flexural Strength Test Results According to Group and Aging Time

Group	T0	T1	T2	T3
Conventional	73.12 ± 4.57 ^{Aa}	73.08 ± 4.88 ^{Aa}	72.95 ± 4.84 ^{Aa}	72.29 ± 4.63 ^{Aa}
Microwave	76.41 ± 6.01 ^{Aa}	76.34 ± 4.47 ^{Aa}	76.27 ± 5.97 ^{Aa}	76.15 ± 4.42 ^{Aa}
Milled	78.38 ± 3.99 ^{Aa}	78.37 ± 4.95 ^{Aa}	77.02 ± 3.71 ^{Aa}	70.66 ± 3.96 ^{Ab}
3D-printed	87.70 ± 3.61 ^{Ba}	65.21 ± 2.19 ^{Bb}	58.31 ± 1.96 ^{Bc}	51.68 ± 9.40 ^{Bd}

T0 = 24 hours; T1 = 6 months; T2 = 12 months; T3 = 24 months.

Different capital letters in columns and different lowercase letters in rows show a significant difference ($P < .05$).

Data are presented in MPa as mean ± SD values.

Table 4 Modulus of Elasticity Test Results According to Group and Aging Time

Group	T0	T1	T2	T3
Conventional	2,025.03 ± 111.76 ^{Aa}	2,017.31 ± 52.57 ^{Aa}	2,010.55 ± 90.73 ^{Aa}	1,944.47 ± 65.98 ^{Aa}
Microwave	2,041.98 ± 84.96 ^{Aa}	2,017.23 ± 65.10 ^{Aa}	1,995.17 ± 40.30 ^{Aa}	1,979.64 ± 82.51 ^{Aa}
Milled	2,189.64 ± 421.79 ^{Aa}	2,038.53 ± 40.03 ^{Aab}	2,013.20 ± 27.86 ^{Aab}	1,985.53 ± 25.01 ^{Ab}
3D-printed	1,284.75 ± 400.09 ^{Ba}	788.06 ± 55.32 ^{Bb}	728.36 ± 119.41 ^{Bb}	710.83 ± 110.70 ^{Bb}

T0 = 24 hours; T1 = 6 months; T2 = 12 months; T3 = 24 months.

Different capital letters in columns and different lowercase letters in rows show a significant difference ($P < .05$).

Data are presented in MPa as mean ± SD values.

calculated for all groups and tests. Statistical differences between resin groups and aging time were evaluated using two-way analysis of variance (ANOVA). All tests were performed at a significance level of $P < .05$.

RESULTS

Surface Microhardness

The 3D-printed resin had the lowest microhardness value, regardless of the aging time ($P < .001$). The microwave-processed resin was the only resin that did not show a significant decrease in microhardness during aging ($P > .05$). Conventional and milled resins showed significant decreases in microhardness at 24 months of aging ($P < .05$), while for 3D-printed resin showed a nonsignificant decrease between 6 months and 12 months of aging ($P = 1.00$) (Table 2).

Flexural Strength

The 3D-printed resin showed the significantly highest flexural strength value at 24 hours compared to the

other resins ($P < .001$); however, the flexural strength decreased significantly at 6, 12, and 24 months ($P < .05$), presenting the significantly lowest flexural strength values at these times ($P < .001$). Conventional and microwave-processed resins did not show a significant decrease in flexural strength during aging ($P > .05$), while milled resins showed a significant decrease in flexural strength only after 24 months of aging ($P < .05$) (Table 3).

Modulus of Elasticity

The 3D-printed resin had a significantly lower modulus of elasticity than the other resins, regardless of the aging time ($P < .001$). Conventional and microwave-processed resins did not show a significant decrease in elastic modulus during aging ($P > .05$). The milled resin showed a significant decrease in the modulus of elasticity only with 24 hours of aging compared to other resins at that time ($P = .037$), while the 3D-printed resin at 6, 12, and 24 months of aging did not show significant differences between them ($P > .05$) but showed a significant decrease in relation to 24 hours of aging ($P < .001$) (Table 4).

DISCUSSION

The two-way ANOVA results rejected the two hypotheses evaluated in this study. The first hypothesis was rejected because although significant differences were found between 3D printed resin and traditional resins, milled resins did not show significant differences from traditional resins. The second hypothesis was also rejected because significant differences were found in the aging times, except for the microwave-processed resin.

The surface microhardness provides information about the density of the material and its resistance to wear.^{8,9} According to the specifications of ANSI/ADA specification 12-2002,²³ the Knoop hardness of denture base resins must be $> 15 \text{ kgf/mm}^2$ to avoid excessive wear of the material. In the present study, resins manufactured by conventional methods, microwave processing, and milling had values $> 15 \text{ kgf/mm}^2$ at all aging times, while the 3D-printed resin only met this goal at 24 hours of aging ($17.45 \pm 1.10 \text{ kgf/mm}^2$), which is a significantly low value ($P < .001$). The 3D-printed resin had the lowest microhardness values, reaching a value of $11.72 \pm 0.69 \text{ kgf/mm}^2$ at 24 months of aging, which makes it the most favorable resin for pigmentation and retention of bacterial biofilms.²¹ This low value for the 3D-printed resin may be associated with its polymerization process that is not carried out under heat.^{3,6} Farina et al²⁴ reported that heating PMMA increases the degree of monomer conversion, reducing the presence of residual monomers and the effect plasticizer, resulting in greater hardness.

The denture base subjected to flexural testing at different aging times simulates the ability to succeed intraorally under high functional loads during mastication and parafunction over time,⁵ with flexural strength being one of the main determinants of the mechanical properties of acrylic resin, in which its high strength is directly linked to a high degree of monomer conversion.^{15,25} According to the ISO 20795-1:2013 standard,¹² acrylic resins must not reach values $< 65 \text{ MPa}$. The conventional, microwave-processed, and milled resins exhibited values $> 65 \text{ MPa}$ at all aging times, even with a significant decrease in milling after 24 months of aging ($P < .05$). The 3D-printed resin had values $> 65 \text{ MPa}$ only at 24 hours and 6 months of aging, reaching a resistance of $51.68 \pm 9.40 \text{ MPa}$. These results corroborate the study by Prcić et al,⁸ who showed that printed resins have lower flexural strength than conventional and 3D-printing resins. The flexural strength of the 3D-printed resin demonstrates a limitation in the useful time of a temporary prosthesis, which may present a great chance of fracture during this period.^{5,8,15}

The modulus of elasticity evaluates the deformation capacity before fracture, which, according to the

ISO 20795-1:201312 standard, should not present values $< 2,000 \text{ MPa}$. The conventional resin showed values $< 2,000 \text{ MPa}$ at 24 months of aging, while the microwave-processed resin showed values $< 2,000 \text{ MPa}$ at 12 and 24 months; however, neither resin showed a significant decrease in relation to the 24-hour aging time ($P = .716$ and $.846$, respectively). The milled resin also presented values $< 2,000 \text{ MPa}$ in the 24-months aging samples; however, it was the highest value among the resins at this time ($1,985.53 \pm 25.01 \text{ MPa}$), while the 3D-printed resin had values $< 2,000 \text{ MPa}$ in 24-hour aging samples and decreased over time, reaching $710.83 \pm 110.70 \text{ MPa}$ after 24 months of aging. Therefore, the 3D-printed resin presents a greater chance of permanent plastic deformation compared to other resins, which can consequently cause clinical changes in dimensions, leading to occlusal changes and retention loss.^{15,21}

According to the results of the present study, the milled resin presented the best mechanical properties but was not significantly different from traditional resins, while the 3D-printing resin presented the lowest mechanical property values and was negatively affected by aging, limiting its use to provisional complete dentures.⁸ Recent studies^{26,27} have begun to introduce nanoparticles into 3D printing resins to improve their mechanical and biologic properties, which is an alternative that appears to increase the resin's resistance and prolong its use time.²⁶ These studies are introducing new materials in 3D-printed prostheses to be more durable and achieve more long-term use, showing satisfactory results with zirconia and TiO_2 nanocomposites. Additionally, the industry has been making changes to the composition of 3D-printed resins for greater strength and long-term durability.²⁸

Although aging simulates oral conditions, this study is limited by its in vitro design, which limits the exact replication of clinical situations. Future studies are warranted for further investigation of the degree of conversion and presence of residual monomers, dimensional stability, impact resistance, and cytotoxicity of resins manufactured by the CAD/CAM method, in addition to investigating the mechanical properties of 3D-printing resin incorporated with nanoparticles.

CONCLUSIONS

Based on the results of this in vitro study, the following conclusions were drawn: (1) The CAD/CAM milled resin showed mechanical properties similar to those of traditional resins (conventional and microwave-processed); and (2) the 3D-printing resin did not show adequate mechanical properties for long-term clinical use. Despite this, new studies are developing better properties of this resin for long-term use.

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The authors declare no conflicts of interest.

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