Dental Materials Journal 2024; : –

A comparison of the mechanical properties of 3D-printed, milled, and conventional denture base resin materials

Hyeong-Ju YU[1]*, You-Jung KANG[1]*, Yeseul PARK[1], Hoon KIM[2,3] and Jee-Hwan KIM[1]

[1] *Department of Prosthodontics, Oral Science Research Center, Yonsei University College of Dentistry, Seoul, Republic of Korea*
[2] *Research Institute of Agriculture and Life Sciences, College of Agriculture and Life Sciences, Seoul National University, Seoul 08826, Republic of Korea*
[3] *Graphy, Inc. 6th Fl, Ace GasanFORHU, 225 Gasan digital 1-ro, Geumcheon-gu, Seoul 08501, Republic of Korea*
Corresponding author, Jee-Hawn KIM; E-mail: jee917@yuhs.ac

This study investigated the mechanical properties of three denture base resin materials produced by three-dimensional (3D) printing (Group P), computer-aided design/computer-aided manufacturing milling (Group M), and conventional (Group C) methods. Three-point flexural tests were performed before and after thermocycling treatment to evaluate the mechanical properties. Additionally, nanoindentation and dynamic mechanical analysis (DMA) were used to analyze the behavior of the materials. After flexural strength tests, scanning electron microscopy (SEM) was performed to evaluate the fracture cross-section. The results consistently showed that Group P exhibited significantly higher flexural strength and modulus regardless of thermocycling than Groups C and M ($p<0.05$), along with a higher storage modulus in DMA and greater resistance and resilience to nanoindentation deformation. SEM analysis showed that Group C had a relatively smooth cross-section, whereas Groups M and P had torn cross-sections. This study suggests that the 3D-printed material has suitable mechanical properties for hard dental prosthesis applications.

Keywords: 3D printing resin, CAD/CAM, Denture base, Mechanical property, Thermocycling

INTRODUCTION

Dentures are an effective treatment option for edentulous patients. Among the various materials used to fabricate dentures, polymethyl methacrylate (PMMA) has several advantages regarding economic feasibility, physicochemical properties, and aesthetics. Therefore, PMMA has been actively applied in denture manufacturing since the 1930s[1]. However, several characteristics of traditional PMMA dentures fabricated using conventional methods, such as their insufficient resistance to fracture, deformation owing to polymerization shrinkage, and complex processing procedures, require improvement[2,3].

Recent digital dentistry advancements have significantly increased the use of digital technology in denture fabrication[4,5]. This innovative approach is under active implementation and application in clinical settings as it can replace traditional methods, mitigating the drawbacks of polymerization shrinkage, labor, and working time. The utilization of digital dentistry advancements in denture fabrication not only streamlines the process but also enhances processing accuracy through simplified fabrication procedures[6,7].

Computer-aided manufacturing (CAM) involves subtractive machining, in which PMMA blocks are milled. Consequently, wear and tear of the milling machine during the cutting process is unavoidable, and the process is limited to only approximately 90% of the PMMA block[8,9]. In contrast, additive manufacturing methods, such as three-dimensional (3D) printing, enable the fabrication of more complex structures. Additive manufacturing also allows for a reduction in material consumption of nearly 40% and eliminates concerns about milling machine wear and tear[10].

Denture bases are susceptible to fracture owing to prolonged exposure to physical stresses caused by mastication. Therefore, denture bases must possess sufficient strength and resilience to withstand such forces without fracturing[11]. Flexural strength refers to the maximum bending stress at the fracture point, and dentures with higher flexural strength exhibit better resistance to fracture[12]. Sustained flexural stress caused by mastication can lead to creep, resulting in a damping effect and potential deformation. To prevent this, denture bases should exhibit appropriate elasticity and stiffness[13]. Additionally, a humid oral environment can accelerate the moisture absorption of acrylic resin and deteriorate its mechanical properties[14]. Therefore, additional research is required that includes an artificial aging process that reproduces the oral environment.

Recently, clinical practice has considered it necessary to utilize digital materials and methods when manufacturing dentures. However, most research has been on computer-aided design (CAD)/CAM milling and the mechanical properties of existing denture base resin materials, and research on the physical properties of 3D printing materials is limited[15]. A few studies using 3D printing resin have reported that the physical properties of digital materials are significantly lower than those of milling materials and conventional materials, and hence, there is still insufficient evidence for clinical application. To improve mechanical properties, oligomer-based 3D printing resins have been developed.

*These authors contributed equally to this work.
Received Mar 20, 2024: Accepted Jul 31, 2024
doi:10.4012/dmj.2024-080 JOI JST.JSTAGE/dmj/2024-080

Resins containing oligomers can achieve stable and high mechanical properties. The main physical and mechanical properties of the resin are derived from oligomers. Therefore, it is important to determine and synthesize the chemical structure of the resin oligomer. Generally, as the molecular weight of an oligomer increases, physical and mechanical properties increase. However, as the viscosity increases with the molecular weight, monomers are added to control it[16-18]. Previous studies using these oligomer-based resins have reported high physical properties of oligomer-based 3D printing resins; however, these properties were limited to crown materials[19]. Therefore, to fully exploit the advantages of 3D printing technology in the digital age, comparative studies must be conducted on the mechanical properties of 3D-printed dental resin materials for denture bases.

The purpose of this study is to compare the properties of 3D-printed dental resin materials for denture bases with those from milling and conventional manufacturing methods through various methods such as three-point flexural strength testing, dynamic mechanical analysis (DMA), and nanoindentation testing. Additionally, our study aims to observe the changes in the properties of each material under intraoral conditions simulated by thermocycling. The first null hypothesis of this study states that 1) there will be no significant difference in the mechanical properties between denture base resin materials fabricated by 3D printing and denture base resin materials fabricated by conventional methods or by CAD/CAM milling. Moreover, the second null hypothesis states that 2) thermocycling will not significantly affect the mechanical properties of denture base resin materials.

MATERIALS AND METHODS

Figure 1 shows a flowchart describing the experimental procedure.

Specimen preparation

Three groups of acrylic denture base materials were used in this study, including a conventional injection-type resin (Ivocap, Ivoclar Vivadent, Schaan, Liechtenstein), a CAD/CAM milling resin (Vipi Block Gum, VIPI, São Paulo, Brazil), and 3D printing resin (THD, Graphy, Seoul, Korea) (Table 1). Specimens were prepared

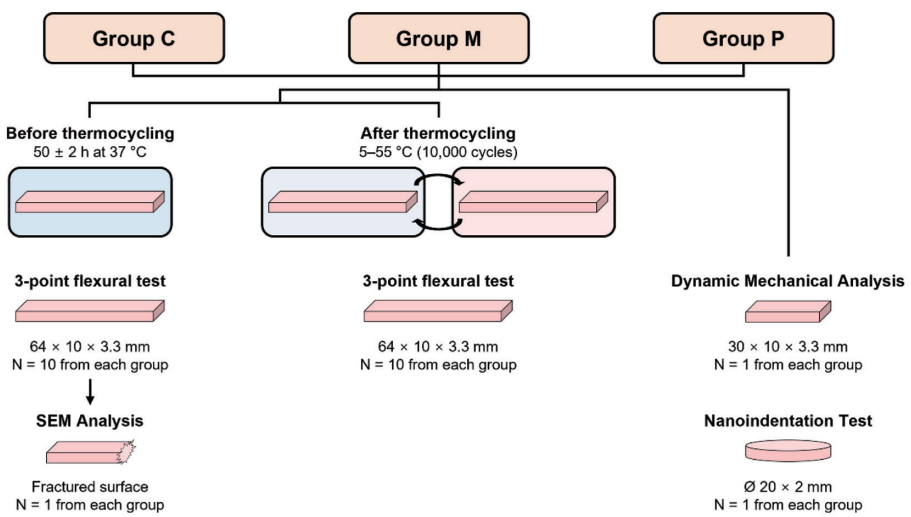

Fig. 1 Flowchart of the experimental procedure.

Table 1 Composition of materials used for the denture base resin and type of fabrication used

Group	Product name	Manufacturer	Composition	Fabrication method
Group C	Ivocap	Ivoclar Vivadent, Schaan, Liechtenstein	Composition powder in %: co-polymer 88.6; polymethyl methacrylate 10.0; benzoyl-peroxide 0.9; pigments <0.5. Composition liquid in %: methyl methacrylate 88.4; dimethacrylate (cross-linking agent) 5.6; co-polymer 6.0.	Conventional
Group M	Vipi Block Gum	VIPI, São Paulo, Brazil	Methyl Polymethacrylate, Biocompatible pigments, EDMA and Fluorescent	Milling
Group P	THD	Graphy, Seoul, Korea	Urethane-dimethacrylate, Urethane acrylate oligomer	3D printing

in various dimensions for each type of experiment. Specifically, for three-point flexural strength tests, specimens were fabricated with dimensions of 64 mm×10 mm(±0.2 mm)×3.3 mm(±0.2 mm) (length×width×height) in accordance with ISO 20795-1:2013[20]. For DMA testing, a single specimen measuring 30×10×3.3 mm (length×width×height) was prepared for each group. Finally, for nanoindentation tests, single disk specimens were fabricated for each group with diameters of 20 mm and thicknesses of 2 mm.

Wax duplicates with the appropriate dimensions for each experiment were first flasked and invested in dental stones to fabricate specimens using conventional injection molding-type resins. Gypsum was poured into a flask, and then wax duplicates were placed inside the flask. After the gypsum was set, the wax was completely removed, and the mold was coated with a separator (Separating Fluid, Ivoclar Vivadent). The pre-measured resin and monomer capsules (SR Ivocap High Impact, Ivoclar Vivadent) were mixed using a commercial mixer (Cap Vibrator, Ivoclar Vivadent) for 5 min before being injected into the flask. The injection process was performed at a pressure of 6 bar according to the injection molding system. The SR Ivocap assembly was polymerized at 100°C for 35 min and then immersed in cold water for 30 min. Finally, the specimens were removed from the flasks and polished according to manufacturer instructions. The CAD/CAM milling specimens were prepared using pre-polymerized denture base-resin blocks. The CAD/CAM milling blocks were cut using an automated cutting machine (Multi Slicer ASM420M, Okamoto, Nagoya, Japan) equipped with a diamond blade (SD400, Noritake, Nagoya, Japan) and polished to obtain rectangular blocks with set dimensions. The 3D-printed samples were designed as solid structures with the appropriate specimen dimensions for each experiment using 3D modeling software (Tinkercad, Autodesk, San Rafael, CA, USA). The design was saved as a standard tessellation language (STL) file, which was used to manufacture specimens using denture-based materials. The specimens were fabricated using a 3D printer (Sprint Ray Pro 95, Sprint Ray, Los Angeles, CA, USA), employing the digital light processing technique according to the manufacturer-recommended parameters. The 3D-printed specimens were cleaned in an ultrasonic bath (UCS-20, Lab Companion, Billerica, MA, USA) containing 90% isopropyl alcohol (Samchun Pure Chemical, Pyeongtaek, Korea). The specimens were then cured for 25 min in a curing machine (Cure-M 102H, Graphy) equipped with light-emitting diodes (LEDs) with a wavelength of 405 nm at a light intensity of 400 mW/cm^2 according to the recommendations of the manufacturer.

Sixty specimens (20 from each group) were prepared for the three-point flexural strength tests. Half of the specimens from each group were stored in distilled water for 50±2 h at 37°C[20]. The other half of the specimens underwent an aging treatment of 10,000 thermal cycles at 5–55°C in distilled water.

Flexural strength and modulus

The flexural strength and modulus were measured in accordance with ISO 20795-1:2013 by conducting three-point flexural strength tests using a jig in a universal testing machine (3366 Series, Instron Engineering, Norwood, MA, USA)[20]. Immediately before testing, the specimen was removed from the water bath, and the wet specimen was placed in the jig. The distance between the specimen supports was 50 mm, and a loading force was applied to the specimens at a crosshead speed of 5 mm/min until fracture. The maximum flexural strength (σ; MPa) was calculated using Equation (1).

$$\sigma=\frac{3Fl}{2wh^2} \quad (1)$$

The flexural modulus (E; MPa) was calculated using Equation (2).

$$E=\frac{Fl^3}{4wh^3d} \quad (2)$$

where F is the fracture load (N), l is the distance between supports (mm), w is the width of the specimen (mm), h is the height of the specimen (mm), and d is the deflection (mm) under load F.

DMA

DMA (Q800, TA Instruments, New Castle, DE, USA) was performed at a mechanical dynamic analysis frequency of 0.01–1,000 Hz to determine the mechanical dynamics and rheological properties of the materials. The strain rate in the single cantilever mode, automatically calculated using the length and width of the specimen and the DMA software, was 0.1%. The chamber was maintained at 37°C and atmospheric pressure during the experiment.

Nanoindentation

Nanoindentation tests were conducted to determine the microscopic surface properties. The nanoindentation tests were performed using a micro-indentation instrument (MCT3 STeP4, Anton Paar, Graz, Austria) equipped with a Vickers diamond indenter. The applied test conditions were a loading time of 10 s, a dwell time of 5 s, and an unloading time of 10 s using indentation loads of 2, 4, 6, 8, and 10 N.

Scanning electron microscopy (SEM)

Fracture surface images of each group were obtained at magnifications of ×40 and ×100 using SEM (S-300N, Hitachi, Tokyo, Japan). One representative specimen was obtained from each group, and all specimens were coated with a 100-nm-thick platinum coating using an ion sputter coater (E-1010, Hitachi) to prepare the samples for SEM analysis.

Statistical analysis

Statistical analyses were performed using SPSS software (SPSS 25.0, SPSS, Chicago, IL, USA). The

normality of all data was assessed using the Shapiro–Wilk test. A one-way analysis of variance was conducted for data following a normal distribution, followed by *post-hoc* tests using Bonferroni correction to analyze the flexural strength and flexural modulus data according to the different fabrication methods. Data that did not follow a normal distribution were analyzed using the Kruskal-Wallis test, with *post-hoc* tests performed using the Mann-Whitney *U* test. Regarding the comparison between before and after the thermocycling process, the data following a normal distribution were analyzed using the independent sample *t*-test, whereas data not following a normal distribution were analyzed using the Mann-Whitney *U* test. All analyses were conducted at a confidence level of 95% (α=0.05).

RESULTS

Flexural strength and modulus

The flexural strength results show that Group P exhibited the highest flexural strength (119.11±9.29 MPa), followed by Group C (95.28±5.59 MPa) and Group M (93.01±6.27 MPa). Regardless of thermocycling, Group P exhibited a statistically significant higher flexural strength (p<0.05) than Groups M and C. After thermocycling, the mean flexural strength of Groups P and M decreased to 117.62±9.81 and 89.62±8.60 MPa, respectively, and that of Group C slightly increased to 96.08±4.13 MPa. Despite these changes, no group exhibited statistically significant differences before and after thermocycling (p>0.05) (Fig. 2 and Table 2).

Group P exhibited the highest flexural modulus (2.38±0.19 GPa), followed by Group M (1.92±0.18 GPa) and Group C (1.42±0.20 GPa). The flexural modulus of Groups P and M were significantly higher than that of Group C (p<0.05). The flexural modulus of all groups increased after thermocycling. Groups P, M, and C exhibited post-thermocycling flexural modulus of 4.06±0.15, 2.18±0.14, and 1.79±0.14 GPa, respectively. Statistically significant differences were observed between the groups (p<0.05) (Fig. 3 and Table 3).

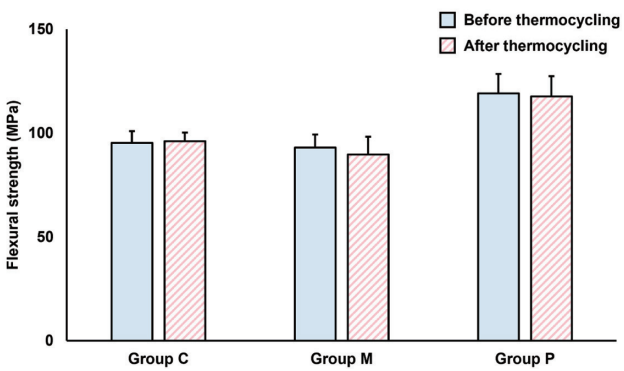

Fig. 2 Comparison of the flexural strengths for the three specimen groups before and after thermocycling. Groups C, M, and P represent the conventional, milling, and 3D printing fabrication methods.

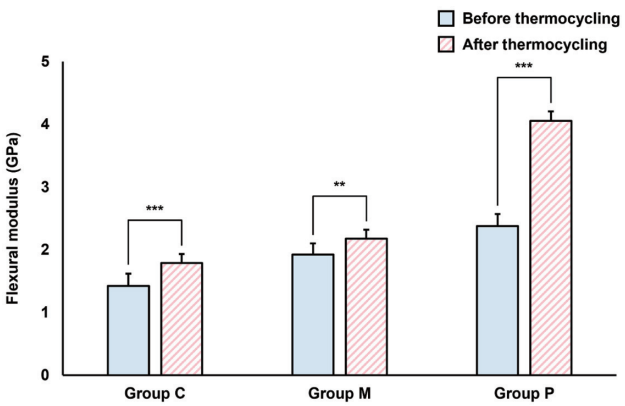

Fig. 3 Comparison of the flexural modulus for the three specimen groups before and after thermocycling. Groups C, M, and P represent the conventional, milling, and 3D printing fabrication methods. Comparison among the groups is expressed as *p<0.05, **p<0.01, and ***p<0.001. Asterisks and horizontal bars indicate statistically significant differences before and after thermocycling, respectively.

Table 2 Mean and standard deviations of the flexural strength among the materials and thermocycling groups (unit: MPa)

	Group C	Group M	Group P	p-value
Before thermocycling	95.28±5.59[b]	93.01±6.27[b]	119.11±9.29[a]	<0.001
After thermocycling	96.08±4.13[b]	89.62±8.60[b]	117.62±9.81[a]	<0.001

Different lowercase letters in each column indicate statistically significant differences (p<0.05).

Table 3 Mean and standard deviations of the flexural modulus among the materials and thermocycling groups (unit: GPa)

	Group C	Group M	Group P	p-value
Before thermocycling	1.42±0.20[b]	1.92±0.18[a]	2.38±0.19[a]	<0.001
After thermocycling	1.79±0.14[c]	2.18±0.14[b]	4.06±0.15[a]	<0.001

Different lowercase letters in each column indicate statistically significant differences (p<0.05).

DMA

DMA was used to evaluate the mechanical properties of the polymer materials under dynamic conditions. The storage and loss modulus of each material were measured at various frequencies (Fig. 4). Groups C, M, and P exhibited storage modulus of 1,814 and 2,522, 3,001.5 and 4,307, and 3,940 and 5,707 MPa at 0.1 and 63 Hz, respectively (Fig. 4a). The storage modulus of all three groups increased with an increasing frequency, indicating a stiffer material response at higher frequencies. Group P exhibited a higher storage modulus at 0.1 Hz than the other materials, which is consistent with the results from the other tests.

Nanoindentation

The applied nanoindentation force was increased from 2 to 10 N in 2 N increments. Group C exhibited indentation depths of 26.7, 38.4, 47.5, 54.2, and 58.3 μm, respectively. After the applied force was removed, the recovered indentation depths were 15.2, 22, 27.5, 31.2, and 32.9 μm, respectively (Fig. 5A). The indentation depths for Group M were 23.8, 33, 40.1, 46.5, and 51.8 μm, respectively, and the recovered indentation depths were 15.8, 20.8, 25, 28.5, and 31.8 μm, respectively (Fig. 5B). The indentation depths for Group P were 22.8, 30.7, 37.5, 42.5, and 48 μm, respectively, and the recovered indentation depths were 14.5, 18.1, 21.7, 24.1, and 27.1 μm, respectively (Fig. 5C). A comparison of the force–depth curves under a 10 N load demonstrated that Group P exhibited the lowest deformation and strongest resilience (Fig. 5D). This indicates that Group P exhibited higher resistance to deformation and a better ability to recover its original shape after the applied force was removed, as compared with the other materials.

SEM

The fractured surfaces of the specimens after the three-point flexural strength test were examined using SEM. Group C exhibited a relatively smooth fracture surface (Figs. 6A, B). Group M exhibited an irregular, grooved, and rough surface with a partially torn cross-section (Figs. 6C, D). Group P resisted the applied force and exhibited a sagging, torn cross-section (Figs. 6E, F).

DISCUSSION

The mechanical properties of dental resins for denture bases fabricated using three different manufacturing methods were evaluated using three-point flexural strength tests before and after thermocycling and DMA and nanoindentation tests. The 3D-printed group exhibited the highest flexural strength and modulus compared to the other groups. Although no significant difference was observed in the flexural strengths before and after thermocycling, there was a significant difference in the flexural modulus.

Flexural strength refers to the maximum stress experienced by a specimen during the flexural strength test immediately before it fractures and indicates the

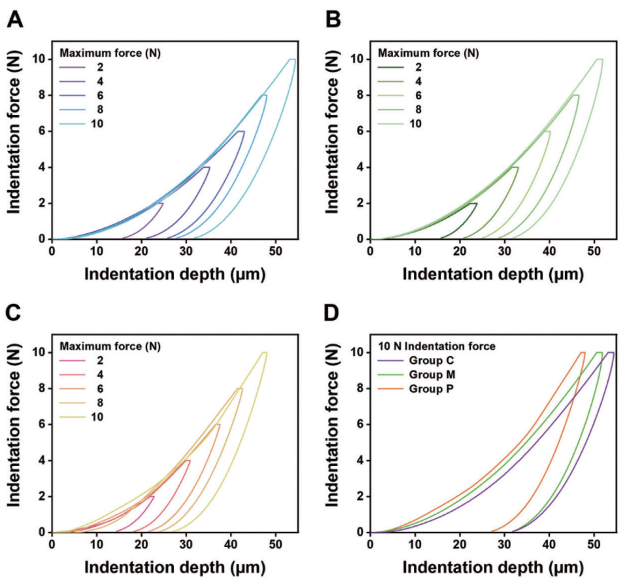

Fig. 5 Pressure-indentation characteristics in the nanoindentation tests.
Indentation deformation behavior of the resins as a function of various indentation loads of 2–10 N for Groups (A) C, (B) M, and (C) P. (D) Comparison of the force–depth curves of Groups C, M, and P at an indentation load of 10 N.

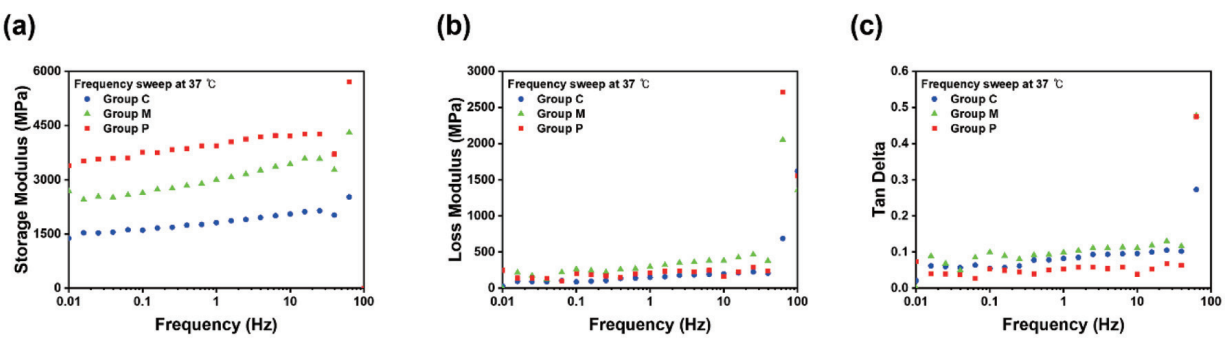

Fig. 4 Dynamic rheological properties as a function of the frequency for Groups C, M, and P at 37°C.
(a) Storage modulus, (b) loss modulus, and (c) loss tangent change graphs.

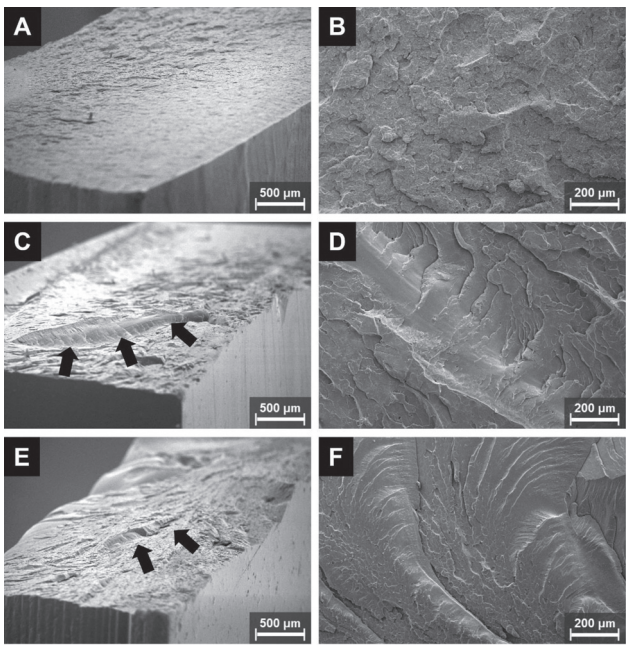

Fig. 6 SEM images (A, C, E: ×40 magnification; B, D, F: ×100 magnification) of the fracture surfaces of each material.
(A, B) Group C, with a smooth and even surface, (C, D) Group M; arrows indicate the jagged, rough appearance and partially torn aspect, and (E, F) Group P; arrows indicate stretching and tearing features.

mechanical strength of the material[21,22]. The flexural modulus is a strength property calculated as the ratio of stress to strain and indicates the resistance of the material to bending[23,24]. These mechanical properties are essential for dental prosthetic materials because they experience various forces in the oral environment[25]. Fiore *et al*. reported that CAD/CAM resin exhibited the highest flexural strength (110.23±4.71 MPa) among the different fabrication methods[26]. The CAD/CAM resin was followed by the 3D-printed (87.34±6.19 MPa) and heat-polymerized resins (80.79±4.37 MPa). Zeidan *et al*. reported that CAD/CAM resin groups exhibited higher flexural strength (120.49 MPa from Avadent and 119.59 MPa from Polident) than other groups using different fabrication methods. The CAD/CAM resin was followed by the heat-polymerized resins (99.00 MPa from vertex) and the 3D-printed resins (61.63 MPa from Nextdent and 28.82 MPa from Harz)[27]. Several experimental studies have shown that CAD/CAM resins exhibit superior flexural strengths and modulus[8,28]. The excellent mechanical properties of the CAD/CAM resin blocks have been attributed to their high polymerization rate and the resulting improvement in their mechanical properties under high-temperature and high-pressure polymerization conditions[3,29]. Prpić *et al*. found that experimental groups of heat-polymerized denture base resins exhibited higher flexural strength than CAD/CAM denture base resins[30]. They also observed that experimental groups of heat-polymerized denture base resins exhibit similar strength levels to CAD/CAM denture base resins. Additionally, Temizci *et al.* also reported that 3D-printed denture base resins have superior flexural strength at 113.53 MPa, followed by CAD/CAM denture base resins at 104.65 MPa and heat-polymerized denture base resins at 95.23 MPa. They also suggested that extending post-curing time prevents insufficient chain cross-linking and enhances the degree of polymerization[31]. Lee *et al.* also observed that experimental groups of 3D-printed and heat-activated denture base resins exhibit higher flexural strengths and flexural modulus than CAD/CAM denture base resins[32]. The differences in the resin polymerization method and the degree of polymerization resulting from the fabrication method are essential factors that can affect the material properties, and relying solely on the fabrication method to determine the strength of the material has limitations. Lee *et al.* argued that factors beyond the fabrication method, such as the polymeric composition of the material and the unique fabrication techniques proposed by manufacturers, also play significant roles in determining the material properties[32]. The 3D-printed denture base resin in this study exhibited excellent flexural strength (119.11±9.29 MPa) compared to the other denture base resins. The CAD/CAM (95.28±5.59 MPa) and heat-cured denture base resins (93.01±6.27 MPa) exhibited similar strengths with slight differences. The 3D-printed denture base resin exhibited the highest flexural modulus (2.38±0.19 GPa), followed by the CAD/CAM (1.92±0.18 GPa) and heat-polymerized resins (1.42±0.20 GPa). The flexural strengths met the ISO standard (65 MPa)[20]. Although the flexural modulus of Groups C and M did not meet the ISO standard (2 GPa), that of Group P met the standard[20]. These results are consistent with those of previous studies[33,34]. The 3D-printed material, reported to show lower strengths in previous studies, exhibited superior physical properties in this study. These experimental results suggest that the 3D-printed material possesses sufficient strength to be used as a hard denture material. These results were attributed to the unique material characteristics and manufacturing methods employed by some manufacturers, as mentioned by Lee *et al.*[32]. Additionally, post-processing techniques designed to increase the polymerization degree of printing resins may be associated with these findings, as mentioned by Temizci *et al.*[31].

Further research is needed to compare different methods for enhancing the properties of printing resin materials and to investigate the effects of the compositional components on the properties of currently available printing resin materials. Polymeric materials used in dentistry exhibit viscoelastic properties. This implies that the materials can undergo deformation over time and possess both elastic and viscous characteristics. These physical characteristics must be considered due to the different viscoelastic properties required for different applications[35]. Dynamic testing uses frequency to measure the storage modulus, loss modulus, and

loss tangent values to evaluate the viscoelasticity of a material. The storage modulus, derived from the deformation and recovery patterns at a specific frequency, represents the elasticity of the material. In contrast, the loss modulus represents the viscosity of a material. The loss tangent is the ratio between the storage and loss modulus and can be used to evaluate the cushioning ability of a material under compressive loads[35]. Soft liner denture materials require relatively low storage modulus and high loss tangent values to effectively absorb external forces[36]. In contrast, hard denture materials require high rigidity, which makes them advantageous due to their high storage modulus values. Hard denture materials should also exhibit minimal deformation under external forces; therefore, high storage modulus and low loss tangent values are preferred[13]. The 3D-printed denture base fabricated in this study exhibited a high storage modulus, demonstrating its ability to rigidly and elastically resist external forces. Although the differences were insignificant, the 3D-printed denture base resin exhibited the lowest loss modulus and tangent values, followed by the CAD/CAM-fabricated and heat-polymerized denture base resins (Figs. 4b, c). The 3D-printed material that exhibited superior strength in the flexural test also demonstrated properties suitable for hard material applications under dynamic testing.

Hardness testing using indentation has been widely adopted to evaluate the properties of dental materials[37]. Hardness is typically analyzed by examining the indentation created using Vickers or Knoop tests[38]. The applied load in these tests typically ranges in 10–500 g. Previous studies have used the Vickers test to compare the surface hardness of the resins from different fabrication types. Zeidan *et al.* reported that CAD/CAM denture base resin exhibit the highest Vickers hardness number at 29.18±3.44, followed by heat-polymerized denture base resin with a Vickers number of 22.44±0.98, and 3D-printed resin with Vickers number of 2.64 ±0.37[39]. Helal *et al.* also observed that CAD/CAM denture base resin exhibited a Vickers number of 26.14±0.39, while the Vickers number for 3D printed denture base resin was 20.06±0.39, showing the superior performance of CAD/CAM denture base resin. These results are similar to previous studies that observed the superior properties of CAD/CAM denture base materials[40]. The magnitude of the applied load can be considered a macro-indentation when compared to the particle size of the resin material to be analyzed[41]. Nanoindentation tests, as employed in this study, allow the load range to be adjusted between 0.0001 and 5 g. Microindentation can be measured by analyzing the indentation at a scale of 1 μm, allowing for the precise characterization of the material properties. In contrast to the conventional method of analyzing hardness using the indentation area, nanoindentation tests allow for continuous observation of the loading and unloading phases using a depth-sensing instrument[42]. The deformation curves and indentation depths were measured in micrometers using nanoindentation under a loading force of 2–10 N. Group P exhibited the smallest indentation depth when the same load was applied. This indicates that Group P exhibited the highest elastic recovery upon load release. Furthermore, Group P exhibited a steeper slope when comparing the indentation curves of the three materials under a 10 N load. Considering the extent of deformation with an increasing load, it can be inferred that Group P exhibited the stiffest properties based on the steepness of the slope. The 3D-printed material exhibited excellent elasticity in terms of deformation recovery and stiffness during the deformation process. Our experimental results are consistent with the flexural strength and dynamic test results. In addition to analyzing the indentation depth, further research is required to compare the hardness and flexural modulus values obtained from nanoindentation experiments with the hardness values obtained from the Vickers experiments and the flexural modulus values.

Thermocycling was used to simulate the conditions of constant exposure to moisture and heat in a clinical environment to mimic practical conditions more realistically[43,44]. Thermocycling at 5–55°C in the presence of water is considered appropriate for aging dental materials. Additionally, Gale and Darvell argued that 10,000 cycles of thermocycling are roughly equivalent to a full year of intraoral cycling in terms of environmental conditions[45]. Accordingly, thermocycling was conducted for 10,000 cycles at 5–55°C in this study to observe the changes in the specimen properties. Ghavami-Lahiji *et al.* determined the flexural strength and hardness as a function of the number of thermocycling cycles[46]. The flexural strength significantly decreased with an increasing number of thermocycling cycles, whereas the hardness exhibited a concurrent trend of decreasing in some experimental groups and increasing in others. Similar to other studies, Ayaz *et al.*[14] and Machado *et al.*[47] also reported a decrease in flexural strength with increasing thermocycling cycles. Although no statistically significant difference was detected in this study, the flexural strengths of Groups M and P decreased after thermocycling. The decrease in flexural strength was attributed to changes in the properties of the polymers. The heat in the thermocycling process increases the water absorption of the resin, which affects the polarity of the polymer and changes the distance between polymer chains. Additionally, the absorbed water acts as a plasticizer, which induces a slip-over phenomenon in the polymer chains and degrades the properties of the polymer[47]. Kawano *et al.* investigated the flexural strength, hardness, and elastic modulus as a function of the number of thermocycling cycles, similar to the abovementioned studies, and reported that the flexural strength of all experimental groups significantly decreased with an increasing number of thermocycling cycles[48]. However, the hardness and elastic modulus exhibited mixed results, with some experimental groups showing an increase and others showing a decrease. In this study, the flexural modulus increased after thermocycling. Chadwick *et al.* reported that the hardness and elastic modulus are influenced by the degree of polymerization of a composite resin matrix and the volume of the filler, which are components of

a polymer[49]. Moreover, Ghavami-Lahiji *et al*. observed an increase in the degree of polymerization with an increasing number of thermocycling cycles[46]. The increase in the degree of polymerization observed during thermocycling can be attributed to the heat-induced polymerization of the residual unpolymerized monomers within the composite resin[50]. Ferracane *et al*. stated that the structural deformation of polymers during thermocycling can induce a relative increase in the filler volume, leading to an increase in the elastic modulus of the material[51]. The causes of the changes in the elastic modulus after thermocycling can be inferred based on these studies. However, if the thermal stress continues to increase and causes significant changes and degradation in the polymer properties, both the hardness and elastic modulus are expected to decrease, similar to the decrease in flexural strength.

Owing to the varied forces experienced in the intraoral environment, various experimental methods should be used, as in this study, to reproduce different situations and evaluate the properties of materials for this application. Because this study principally investigated the mechanical properties of denture base materials, it cannot fully represent actual denture bases and has limitations in suggesting clinical applications for these materials. Therefore, considering clinical practice, additional *in vitro* studies on the manufacturing accuracy and deformation during detachment are necessary, and further clinical studies are required. In addition, this study evaluated only one material for each manufacturing method; thus, multiple materials must be evaluated for each manufacturing method in follow-up research. Furthermore, because the 3D-printed material consistently exhibited stiffness and high strength in all three experiments, in contrast to previously reported results, additional investigations are required to re-evaluate the properties of 3D-printed materials.

CONCLUSIONS

The mechanical properties of 3D-printed, milled, and heat-polymerized denture base resins were compared and analyzed. The following results were obtained under specific experimental conditions:

1. The 3D-printed material exhibited suitable mechanical properties for hard denture applications.
2. Regardless of thermocycling, the 3D-printed denture exhibited a higher flexural strength and modulus than the materials fabricated using the other methods, demonstrating excellent mechanical properties.
3. Although there was no significant change in the flexural strength after thermocycling, the flexural modulus showed an increasing trend after thermocycling.

ACKNOWLEDGMENTS

This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF), funded by the Ministry of Education (2022R1A6A3A01087390).

CONFLICT OF INTEREST

The authors declare no competing interests.

REFERENCES

1) Zarb GA, Hobkirk J, Eckert S, Jacob R. Prosthodontic Treatment for Edentulous Patients: Complete Dentures and Implant-supported Prostheses. 13th ed. Elsevier; 2012.
2) Rodriguez LS, Paleari AG, Giro G, de Oliveira Jr NM, Pero AC, Compagnoni MA. Chemical characterization and flexural strength of a denture base acrylic resin with monomer 2-tert-butylaminoethyl methacrylate. J Prosthodont 2013; 22: 292-297.
3) Infante L, Yilmaz B, McGlumphy E, Finger I. Fabricating complete dentures with CAD/CAM technology. J Prosthet Dent 2014; 111: 351-355.
4) Fang JH, An X, Jeong SM, Choi BH. Digital immediate denture: A clinical report. J Prosthet Dent 2018; 119: 698-701.
5) Bidra AS, Taylor TD, Agar JR. Computer-aided technology for fabricating complete dentures: Systematic review of historical background, current status, and future perspectives. J Prosthet Dent 2013; 109: 361-366.
6) Di Fiore A, Vigolo P, Graiff L, Stellini E. Digital vs conventional workflow for screw-retained single-implant crowns: A comparison of key considerations. Int J Prosthodont 2018; 31: 577-579.
7) Granata S, Giberti L, Vigolo P, Stellini E, Di Fiore A. Incorporating a facial scanner into the digital workflow: A dental technique. J Prosthet Dent 2020; 123: 781-785.
8) de Oliveira Limírio JPJ, Gomes JML, Alves Rezende MCR, Lemos CAA, Rosa CDD, Pellizzer EP. Mechanical properties of polymethyl methacrylate as a denture base: Conventional versus CAD-CAM resin —A systematic review and meta-analysis of in vitro studies. J Prosthet Dent 2022; 128: 1221-1229.
9) Strub JR, Rekow ED, Witkowski S. Computer-aided design and fabrication of dental restorations: Current systems and future possibilities. J Am Dent Assoc 2006; 137: 1289-1296.
10) Alharbi N, Wismeijer D, Osman RB. Additive manufacturing techniques in prosthodontics: Where do we currently stand? A critical review. Int J Prosthodont 2017; 30: 474-484.
11) Kelly E. Fatigue failure in denture base polymers. J Prosthet Dent 1969; 21: 257-266.
12) Zappini G, Kammann A, Wachter W. Comparison of fracture tests of denture base materials. J Prosthet Dent 2003; 90: 578-585.
13) Vaidyanathan J, Vaidyanathan TK. Dynamic mechanical analysis of heat, microwave and visible light cure denture base resins. J Mater Sci Mater Med 1995; 6: 670-674.
14) Ayaz EA, Bağış B, Turgut S. Effects of thermal cycling on surface roughness, hardness and flexural strength of polymethylmethacrylate and polyamide denture base resins. J Appl Biomater Funct Mater 2015; 13: e280-e286.
15) Lourinho C, Salgado H, Correia A, Fonseca P. Mechanical properties of polymethyl methacrylate as denture base material: Heat-polymerized vs. 3D-printed-systematic review and meta-analysis of in vitro studies. Biomedicines 2022; 10: 2565.
16) Li P, Chu Z, Chen Y, Yuan T, Yang Z. One-pot and solvent-free synthesis of castor oil-based polyurethane acrylate oligomers for UV-curable coatings applications. Prog Org Coat 2021; 159: 106398.

17) Kim H, Kim J, Ryu KH, Lee J, Kim HJ, Hyun J, *et al*. Embedded direct ink writing 3D printing of UV curable resin/sepiolite composites with Nano orientation. ACS Omega 2023; 8: 23554-23565.
18) Kim H, Khan T, Ryu KH, Sohn IS, Kim HJ. Nanocomposite materials for 3D printing. In: Maciel MAM, editor. Applications and Industrialisation of Nanotechnology. Manchester, England, One Central Press, 2022. p. 91-113.
19) Kang YJ, Kim H, Lee J, Park Y, Kim JH. Effect of airborne particle abrasion treatment of two types of 3D-printing resin materials for permanent restoration materials on flexural strength. Dent Mater 2023; 39: 648-658.
20) ISO20795-1:2013. Dentistry-Base polymers Part I: Denture base polymers. 2nd ed. Geneva: International Standard Organization; 2013.
21) Steinmassl O, Offermanns V, Stöckl W, Dumfahrt H, Grunert I, Steinmassl PA. In vitro analysis of the fracture resistance of CAD/CAM denture base resins. Materials 2018; 11: 401.
22) Pacquet W, Benoit A, Hatège-Kimana C, Wulfman C. Mechanical properties of CAD/CAM denture base resins. Int J Prosthodont 2019; 32: 104-106.
23) Ayman AD. The residual monomer content and mechanical properties of CAD\CAM resins used in the fabrication of complete dentures as compared to heat cured resins. Electron Phys 2017; 9: 4766-4772.
24) Meng TR Jr, Latta MA. Physical properties of four acrylic denture base resins. J Contemp Dent Pract 2005; 6: 93-100.
25) Gharechahi J, Asadzadeh N, Shahabian F, Gharechahi M. Flexural strength of acrylic resin denture bases processed by two different methods. J Dent Res Dent Clin Dent Prospects 2014; 8: 148-152.
26) Fiore AD, Meneghello R, Brun P, Rosso S, Gattazzo A, Stellini E, *et al*. Comparison of the flexural and surface properties of milled, 3D-printed, and heat polymerized PMMA resins for denture bases: An in vitro study. J Prosthodont Res 2022; 66: 502-508.
27) Zeidan AAE, Sherif AF, Baraka Y, Abualsaud R, Abdelrahim RA, Gad MM, *et al*. Evaluation of the effect of different construction techniques of CAD-CAM milled, 3D-printed, and polyamide denture base resins on flexural strength: An in vitro comparative study. J Prosthodont 2023; 32: 77-82.
28) Al-Dwairi ZN, Tahboub KY, Baba NZ, Goodacre CJ. A comparison of the flexural and impact strengths and flexural modulus of CAD/CAM and conventional heat-cured polymethyl methacrylate (PMMA). J Prosthodont 2020; 29: 341-349.
29) Steinmassl O, Dumfahrt H, Grunert I, Steinmassl PA. Influence of CAD/CAM fabrication on denture surface properties. J Oral Rehabil 2018; 45: 406-413.
30) Prpić V, Schauperl Z, Ćatić A, Dulčić N, Čimić S. Comparison of mechanical properties of 3D-printed, CAD/CAM, and conventional denture base materials. J Prosthodont 2020; 29: 524-528.
31) Temizci T, Bozoğulları HN. Effect of thermal cycling on the flexural strength of 3-D printed, CAD/CAM milled and heat-polymerized denture base materials. BMC Oral Health 2024; 24: 357.
32) Lee DH, Lee JS. Comparison of flexural strength according to thickness between CAD/CAM denture base resins and conventional denture base resins. J Dent Rehabil Appl Sci 2020; 36: 183-195.
33) Lee HH, Lee CJ, Asaoka K. Correlation in the mechanical properties of acrylic denture base resins. Dent Mater J 2012; 31: 157-164.
34) Ucar Y, Akova T, Aysan I. Mechanical properties of polyamide versus different PMMA denture base materials. J Prosthodont 2012; 21: 173-176.
35) Braden M, Stafford GD. Viscoelastic properties of some denture base materials. J Dent Res 1968; 47: 519-523.
36) Murata H, Hamada T, Sadamori S. Relationship between viscoelastic properties of soft denture liners and clinical efficacy. Jpn Dent Sci Rev 2008; 44: 128-132.
37) O'Neill H. Hardness Measurement of Metals and Alloys. 2nd ed. London, Chapman & Hall Ltd, 1967.
38) Ryge G, Foley DE, Fairhurst CW. Micro-indentation hardness. J Dent Res 1961; 40: 1116-1126.
39) Zeidan AAE, Abd Elrahim RA, Abd El Hakim AF, Harby NM, Helal MA. Evaluation of surface properties and elastic modulus of CAD-CAM milled, 3D printed, and compression moulded denture base resins: An in vitro study. J Int Soc Prev Community Dent 2022; 12: 630-637.
40) Helal MA, Fadl-Alah A, Baraka YM, Gad MM, Emam AM. In-vitro comparative evaluation for the surface properties and impact strength of CAD/CAM milled, 3D printed, and polyamide denture base resins. J Int Soc Prev Community Dent 2022; 12: 126-131.
41) Willems G, Lambrechts P, Braem M, Celis JP, Vanherle G. A classification of dental composites according to their morphological and mechanical characteristics. Dent Mater 1992; 8: 310-319.
42) Tabor D. Indentation hardness and its measurement: Some cautionary comments. In: Blau PJ, Lawn BR, editors. Microindentation Techniques in Materials Science and Engineering. ASTM International. 1985. p.0.
43) Rinastiti M, Ozcan M, Siswomihardjo W, Busscher HJ. Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. J Dent 2010; 38: 29-38.
44) de Oliveira JC, Aiello G, Mendes B, Urban VM, Campanha NH, Jorge JH. Effect of storage in water and thermocycling on hardness and roughness of resin materials for temporary restorations. Mater Res 2010; 13: 355-359.
45) Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent 1999; 27: 89-99.
46) Ghavami-Lahiji M, Firouzmanesh M, Bagheri H, Jafarzadeh Kashi TS, Razazpour F, Behroozibakhsh M. The effect of thermocycling on the degree of conversion and mechanical properties of a microhybrid dental resin composite. Restor Dent Endod 2018; 43: e26.
47) Machado AL, Puckett AD, Breeding LC, Wady AF, Vergani CE. Effect of thermocycling on the flexural and impact strength of urethane-based and high-impact denture base resins. Gerodontology 2012; 29: e318-e323.
48) Kawano F, Ohguri T, Ichikawa T, Matsumoto N. Influence of thermal cycles in water on flexural strength of laboratory-processed composite resin. J Oral Rehabil 2001; 28: 703-707.
49) Chadwick RG, McCabe JF, Walls AW, Storer R. The effect of storage media upon the surface microhardness and abrasion resistance of three composites. Dent Mater 1990; 6: 123-128.
50) Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Biebuyck JJ, Mestdagh M, Larbanois P, *et al*. A physico-chemical explanation of the post-polymerization shrinkage in dental resins. Dent Mater 2006; 22: 405-412.
51) Ferracane JL, Berge HX, Condon JR. In vitro aging of dental composites in water--Effect of degree of conversion, filler volume, and filler/matrix coupling. J Biomed Mater Res 1998; 42: 465-472.