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Evaluation of Mechanical Properties of Three-dimensional Printed Flexible Denture Resin according to Post-polymerization Conditions: A Pilot Study

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Purpose: The purpose of this study was to evaluate whether three-dimensional (3D)-printed flexible denture resin has suitable mechanical properties for use as a thermoplastic denture base resin material.

Materials and Methods: A total of 96 specimens were prepared using the 3D printed flexible denture resin (Flexible Denture). Specimens were designed in CAD software (Tinkercad) and printed through a digital light-processing 3D printer (Asiga MAX UV). Post-polymerization process was conducted according to air exposure or glycerin immersion at 35°C or 60°C and for 30 or 60 minutes. The maximum flexural strength, elastic modulus, 0.2% offset yield strength, and Vickers hardness of 3D-printed flexible denture resin were assessed.

Result: The maximum flexural strength ranged from 64.46±2.03 to 84.25±4.32 MPa, the 0.2% offset yield strength ranged from 35.28±1.05 to 46.13±2.33 MPa, the elastic modulus ranged from 1,764.70±64.66 to 2,179.16±140.01 MPa, and the Vickers hardness ranged from 7.01±0.40 to 11.45±0.69 kg/mm^2.

Conclusion: Within the limits of the present study, the maximum flexural strength, 0.2% offset yield strength, elastic modulus, and Vickers hardness are sufficient for clinical use under the post-polymerization conditions of 60°C at 60 minutes with or without glycerin precipitation.

Key Words: Mechanical properties; Post-polymerization conditions; 3D printed flexible denture; 3D printed flexible denture resin

Introduction

Removable partial dentures are one of the main treatment options used to replace missing teeth in partially edentulous patients. Non-precious metal alloys used to fabricate metal frameworks in most

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partial dentures have lower densities than gold alloys, making them lighter and cheaper than the ones made of gold alloys, with excellent mechanical properties[1]. In addition, it is known that the corrosion resistance of non-precious metal alloys is as good as that of gold alloys due to the chromium contained in the alloy composition[2]. However, in the case of removable partial dentures including anterior teeth, exposure of the metal clasp may cause a dissatisfying appearance[3]. As an alternative to this, thermoplastic denture base resin can be used as a highly elastic resin material[4,5]. It is lighter than removable partial dentures made of non-precious metal alloys and can reproduce gingival colors, enabling the fabrication of clasps with natural appearance rather than metallic color.

Computer-aided design and manufacturing (CAD/CAM) technology in dentistry is being utilized in various fields. This enables the machining of materials such as zirconia, ceramic, and polymethyl methacrylate (PMMA) as well as customized implant abutments, and shortens the manufacturing time by simplifying the existing complex manufacturing process[6]. However, CAD/CAM systems are limited in terms of reproducibility when processing complex structures, and there is a disadvantage in wasting the material during processing[7]. On the other hand, 3D printers can reproduce complex structures by stacking printing materials and reduce material consumption in making hollow structures[8,9]. Owing to these advantages, the application of 3D printers in the dental field has become popular and various materials for 3D printing that can substitute conventional restorative materials have been released. Among them, the development of a 3D-printing resin material with flexibility suitable for flexible dentures makes it possible to fabricate removable partial dentures using CAD software and 3D printer. This 3D printed flexible denture resin for the fabrication of removable partial dentures is used as a denture base material and has a pink color similar to the gingiva. Due to its flexibility, the denture base itself can be used as a clasp. Therefore, a series of processes required for manufacturing conventional thermoplastic denture base resin such as wax denture molding, flasking, injection-molding, and deflasking are eliminated and significant reduction in working time is expected. However, despite the trials from numerous studies, it has been shown that the additive manufacturing method has low mechanical properties[10]. Therefore, a post-polymerization process is essential to provide sufficient mechanical properties to the final 3D-printed product. Post-polymerization is the process of increasing the mechanical properties and stability of 3D-printed materials using heat and light[11]. Previous studies have reported that the post-polymerization time, temperature, and method of oxygen barrier may affect the mechanical properties of 3D-printing resin[12-14]. To date, many studies on 3D-printing resins have evaluated the properties by comparing and analyzing the properties of existing restorative materials such as glass ceramic or polymer-infiltrated ceramic network to replace these materials. However, to the best of our knowledge, few studies have introduced 3D-printed flexible denture resin of which the mechanical properties need to be evaluated. Therefore, this study aimed to investigate the effects of various post-polymerization methods on the maximum flexural strength, 0.2% offset yield strength, elastic modulus, and surface hardness of 3D-printed flexible denture resin. In addition, we evaluated the mechanical properties of 3D-printed flexible denture resin materials under various post-polymerization conditions. The effects of the presence or absence of precipitation of glycerin, time, and temperature were determined. The null hypothesis was that post-polymerization methods according to glycerin immersion, temperature, and time do not affect the mechanical properties of 3D-printed flexible denture resin.

Materials and Methods

1. Fabrication of Specimens

Six specimens for evaluation of flexural properties were fabricated in bar-shape (64×10×3.3 mm) of according to International Organization for Standardization (ISO) 20795-1[15,16]. Additional six disk-shaped (15×2 mm) specimens were fabricated according to the previous study[17]. Each specimen was designed for each test using CAD software (Tinkercad; Autodesk, San Rafael, CA, USA) (Fig. 1). In particular, the bar-shaped specimen was used in the 3-point bending test according to the ISO 20795-1[15]. The designed file was saved in Standard Triangulated Language (STL) format, and then specimens were sliced in the slicing software (Asiga Composer; Asiga, Sydney, Australia) which was set directly at a 0° angle on the build flatform, a layer thickness of 100 μm, and an exposure time of 6.2 seconds for each layer (Fig. 1).

The 3D printed flexible denture resin (Flexible Denture; Graphy, Seoul, Korea) was processed using a digital light-processing (DLP) 3D printer (Asiga MAX UV; Asiga) with an ultraviolet light emitting-diode light source (385 nm) (Fig. 2). After printing, the residual resin around the specimen was washed with 90% isopropyl alcohol (IPA) solution for 5 minutes, dried, and post-polymerization (405 nm, Form cure; Formlabs, Somerville, MA, USA) was performed. According to the purpose of the study, in the post-polymerization stage, the specimens were divided into subgroups depending on air exposure or glycerin immersion, heating at 35°C or 60°C, and polymerization time of 30 or 60 minutes (n=6 per experimental group).

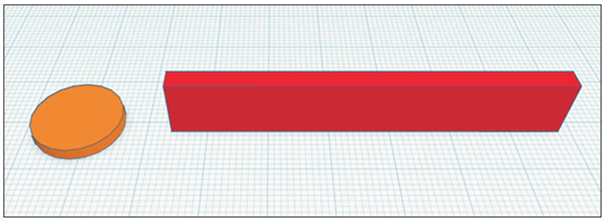

Fig. 1. Specimens designed for each test on the CAD software. CAD: computer-aided design.

2. Three-point Bending

Specimens were stored for 48 hours in a closed container containing distilled water at 37°C before testing and then tested. The span distance of the universal testing machine (EZ-LX; SHIMADZU, Kyoto, Japan) was fixed at 50 mm, each specimen was placed on a support, and a vertical force was applied to the middle of the specimen at a crosshead speed of 5 mm/min to obtain the maximum flexural strength (σ), 0.2% offset yield strength (σ_y) and elastic modulus (E). Flexural properties were calculated using the following formulas:

$$\sigma = \frac{3Fl}{2bh^2}$$

$$E = \frac{F_1 l^3}{4bh^3 d}$$

$$\sigma_y = \frac{3F_2 l}{2bh^2}$$

F is the maximum applied load (N).
l is the distance (mm) between the supports.
b is the width of the test specimen (mm).
h is the height of the specimen (mm).
F_1 is the load (N) at a point in the straight-line por-

Fig. 2. Three-dimensional printed flexible denture resin specimens.

tion of the load/deflection curve, and
d is the deflection (mm) at load F_1
F_2 is the load at 0.2% offset yield point.

3. Vickers Hardness

A Vickers hardness tester (MMT-X; MATSUZAWA, Akita, Japan) was used to measure the hardness of the surfaces. A 136° diamond-shaped indenter was applied to the specimen with a load of 300 g for 25 seconds, and the diagonal length of the indentation was measured. Measurements were repeated three times for each specimen, and the average value was calculated.

An overview of the overall experimental design of this study is presented in Fig. 3.

4. Statistical Analysis

Two-way analysis of variance (ANOVA) was used to confirm the interaction according to temperature and time for the air exposed group and the glycerin group. An independent t-test was used to assess significant differences between and within groups of post-polymerization temperature and time (α=0.05). All statistical analyses were performed using SPSS Statistics 26 software (IBM Co., Armonk, NY, USA).

Result

The average and standard deviation of the maximum flexural strength, 0.2% offset yield strength, elastic modulus, and Vickers hardness values according to the post-polymerization conditions were calculated, as shown in Table 1.

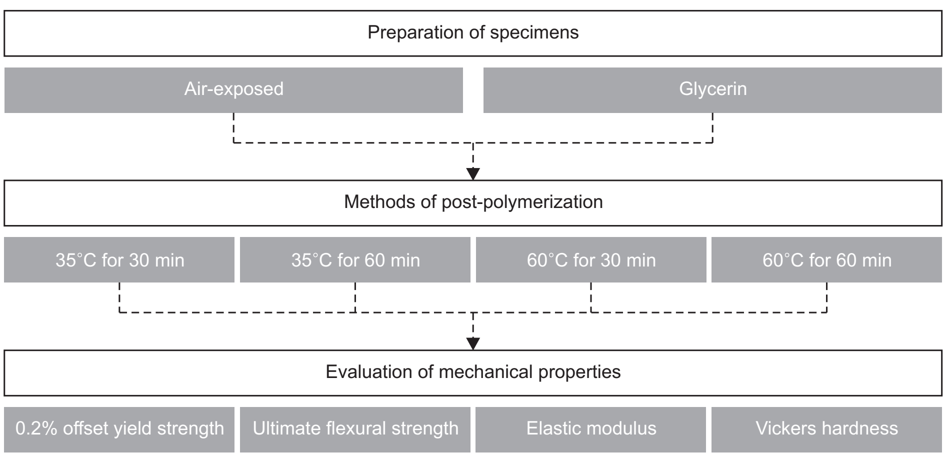

Fig. 3. Experimental schematic.

Table 1. Mean and standard deviation of the mechanical properties of the 3D-printed flexible denture resin according to post-polymerization conditions - either exposure to air or glycerin immersion depending on temperature and time

Mechanical properties	Time (min)	Air exposed 35°C	Air exposed 60°C	Glycerin immersion 35°C	Glycerin immersion 60°C
Maximum flexural strength (MPa)	30	64.46±2.03	81.01±1.56	66.43±2.17	74.65±9.77
	60	73.30±2.36	83.53±0.50	71.16±3.24	84.25±4.32
0.2% offset yield strength (MPa)	30	35.28±1.05	43.83±1.38	36.05±0.65	40.74±4.40
	60	38.85±1.93	44.13±0.91	37.35±1.91	46.13±2.33
Elastic modulus (MPa)	30	1,764.70±64.66	2,147.86±33.39	1,765.09±45.15	1,988.26±231.42
	60	1,935.81±100.89	2,166.08±27.20	1,840.02±87.89	2,179.16±140.01
Vickers hardness (kg/mm^2)	30	7.01±0.40	10.67±1.24	7.15±0.56	10.20±0.44
	60	8.73±1.33	11.45±0.69	9.76±1.06	10.69±0.85

3D: three-dimensional.

1. Three-point Bending

For the maximum flexural strength, two-way ANOVA suggested that temperature, time, and their interaction were significant in the air-exposed group (P<0.001), but not in the glycerin group (P=0.306). The maximum flexural strengths for each time at the same temperature were significantly different at both 35°C (P<0.001) and 60°C (P=0.009) in the air-exposed group. The results for each temperature at the same time were significantly different at both 30 minutes (P<0.001) and 60 minutes (P<0.001) in the air-exposed group (Fig. 4A). There was a significant difference between polymerization times at 35°C (P=0.014) in the glycerin group, but not at 60°C (P=0.052) (Fig. 4B).

Regarding the 0.2% offset yield strength, two-way ANOVA suggested that temperature, time, and their interaction were significant in the air-exposed group (P=0.009), but not in the glycerin group (P=0.077). The 0.2% offset yield strengths for each time at the same temperature were significantly different at 35°C (P=0.003) but not at 60°C (P=0.664) in the air-exposed group, and there were significant differences between each temperature group at both 30 minutes (P<0.001) and 60 minutes (P<0.001) in the air-exposed group (Fig. 5A). For glycerin, there was no significant difference at 35°C (P=0.165) but there was at 60°C (P=0.024). Results for each temperature

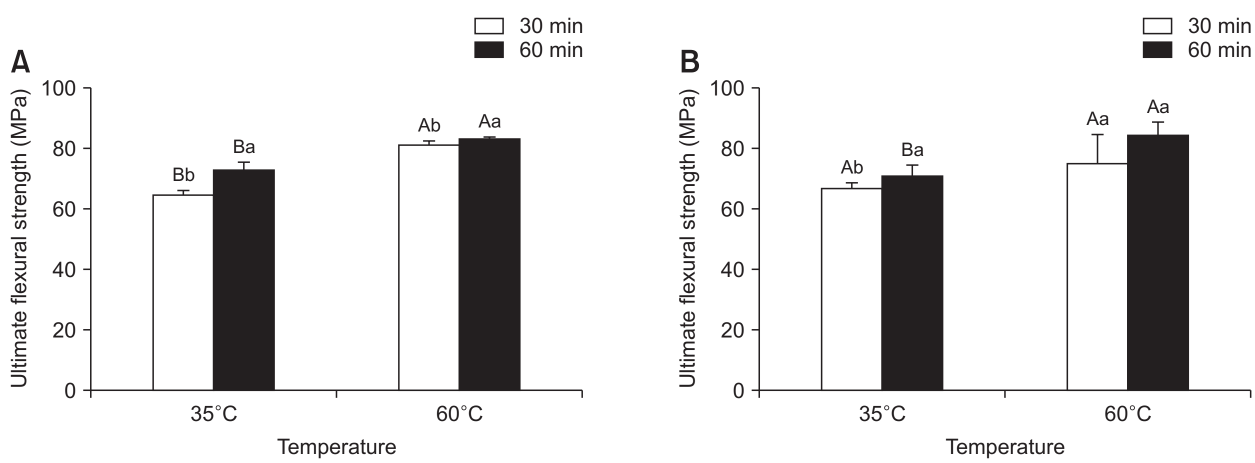

Fig. 4. Maximum flexural strength of 3D-printed flexible denture resin. (A) Air-exposed. (B) Glycerin. Uppercase letters: significant differences within the same time. Lowercase letters: significant differences within the same temperature. 3D: three-dimensional.

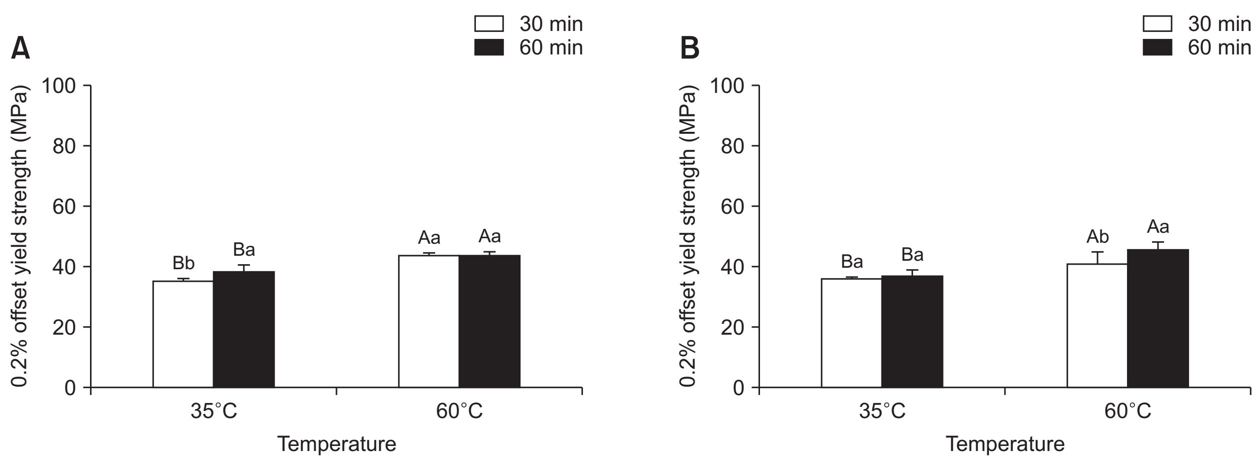

Fig. 5. 0.2% offset yield strength of 3D-printed flexible denture resin. (A) Air-exposed. (B) Glycerin. Uppercase letters: significant differences within the same time. Lowercase letters: significant differences within the same temperature. 3D: three-dimensional.

at the same time showed a significant difference at both 30 minutes (P=0.047) and 60 minutes (P<0.001) in the glycerin group (Fig. 5B).

For the elastic modulus, two-way ANOVA suggested that temperature, time, and their interaction were significant in the air-exposed group (P=0.008) but not in the glycerin group (P=0.336). The elastic modulus at each time at the same temperature were significantly different at 35°C (P=0.006) but not at 60°C (P=0.324) in the air-exposed group. The elastic modulus at each temperature at the same time were significantly different at both 30 minutes (P<0.001) and 60 minutes (P<0.001) in the air-exposed group (Fig. 6A). In the glycerin group, there was no significant difference in the mechanical property according to post-polymerization times at both 35°C (P=0.093) and 60°C (P=0.115). There was no significant difference between each temperature at 30 minutes (P=0.065) in contrast to 60 minutes (P=0.001) in the glycerin group (Fig. 6B).

2. Vickers Hardness

Regarding the Vickers hardness, two-way ANOVA suggested that there were no significant interactions between temperature and time in the air-exposed group (P=0.262), unlike the glycerin group (P<0.003). There was a significant difference in hardness between polymerization times at 35°C (P=0.024) in the

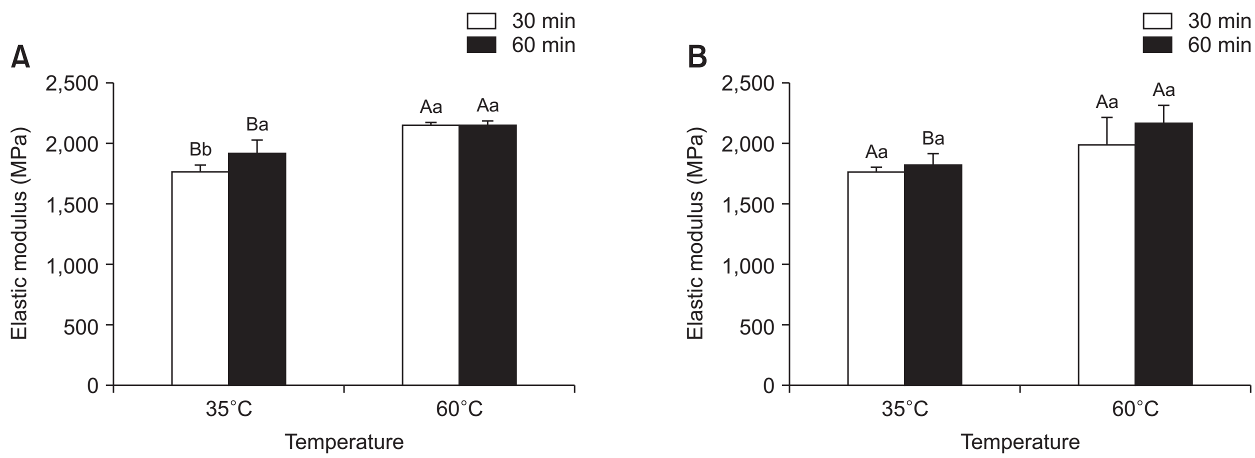

Fig. 6. Elastic modulus of 3D-printed flexible denture resin. (A) Air-exposed. (B) Glycerin. Uppercase letters: significant differences within the same time. Lowercase letters: significant differences within the same temperature. 3D: three-dimensional.

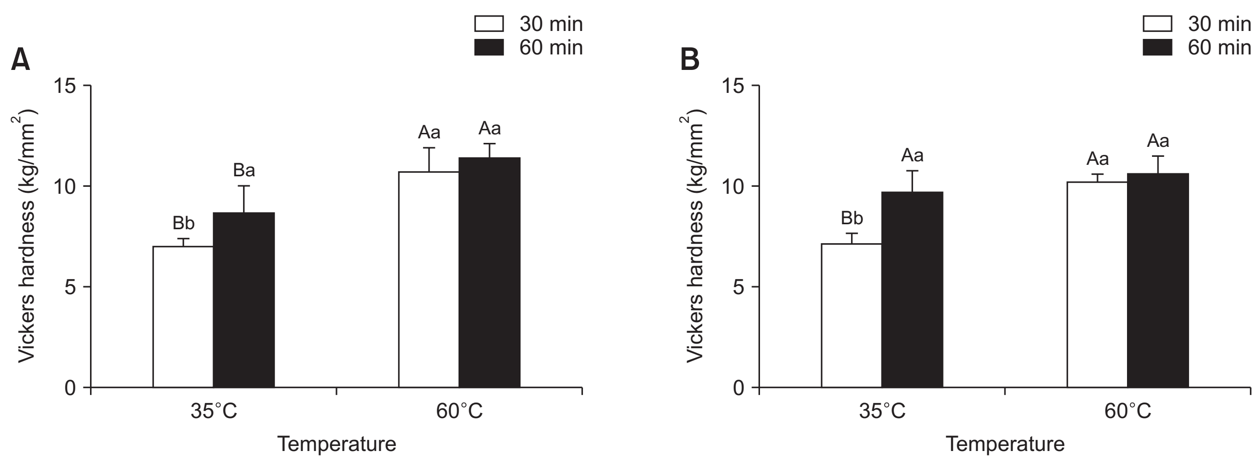

Fig. 7. Vickers hardness of 3D printed flexible denture resin. (A) Air-exposed. (B) Glycerin. Uppercase letters: significant differences within the same time. Lowercase letters: significant differences within the same temperature. 3D: three-dimensional.

air-exposed group but not at 60°C (P=0.209). The hardness at each temperature was significantly different at both 30 minutes (P<0.001) and 60 minutes (P=0.003) in the air-exposed group (Fig. 7A). In the glycerin group, time factor resulted in significant difference in hardness at 35°C (P<0.001) but not at 60°C (P=0.242). Temperature also affected significant difference in hardness at 30 minutes (P<0.001) but not at 60 minutes (P=0.125) (Fig. 7B).

Discussion

Most removable partial dentures are composite structures containing metal framework and resin, where the clasp is made of metal and the artificial teeth and denture base are made of resin. However, appearance of the metallic color of the clasp may dissatisfy the patients. For patients who are reluctant to use a metal clasp, a thermoplastic denture base resin in which the clasp is made of denture base material rather than metal can be used. With the development of 3D printers and materials, 3D-printed flexible denture resins may replace conventional thermoplastic denture base resin. However, the mechanical properties of 3D-printed flexible denture resins according to post-polymerization conditions are not yet known. The purpose of this study was to evaluate the differences in maximum flexural strength, 0.2% offset yield strength, elastic modulus, and surface hardness of 3D-printed flexible denture resin materials according to post-polymerization conditions. In this study, it was found that the mechanical properties changed according to post-polymerization temperature and time. Therefore, the null hypothesis established in this study was rejected.

In this study, specimens were post-polymerized by either air exposure or glycerin immersion as post-polymerization conditions. In general, specimens are cleaned after 3D printing and post-polymerized in a post-polymerization unit exposed to the air whereas some manufacturers recommend post-polymerization processing of the 3D-printed denture base resin by immersing in glycerin. This is based on the results of previous studies demonstrating that the polymerization rate increased when the contact of oxygen with the PMMA surface was blocked during the polymerization reaction. In addition, to improve the properties of a material through post-polymerization, the exposure time and temperature may be important factors. In this study, no significant difference was found in mechanical properties between air exposure and glycerin immersion conditions. However, the mechanical properties increased with increasing post-polymerization time and temperature. Bayarsaikhan et al.[13] evaluated the mechanical properties according to the post-polymerization temperature and time for 3D-printed denture teeth, and they reported a trend of increasing mechanical properties with increasing post-polymerization temperature and time. Bağis and Rueggeberg[18] and Ferracane and Condon[19] reported that the higher the temperature, the stronger the diffusion of free radicals and the faster the polymerization. In addition, the mechanical properties increased as the post-polymerization time increased, but there was no significant difference after 60 minutes[13]. In other studies, the mechanical properties tend to increase as the post-polymerization time increases[20].

The post-polymerization conditions recommended by the manufacturer for the materials used in this study were 60 minutes at 5°C~35°C without glycerin immersion. However, within the conditions of this study, the best mechanical properties were observed at 60°C at 60 minutes post-polymerization, regardless of the presence of glycerin. According to the ISO 20795-1 regulation for a denture base polymer, the maximum flexural strength of either a heat-polymerized or thermoplastic polymer must be 65 MPa or more, and the elastic modulus must be 2,000 MPa or more. For self-curing polymers, maximum flexural strength must be 60 MPa or more and an elastic modulus, 1,500 MPa or more. Although the

3D-printed flexible denture resin tested in this study is not a thermoplastic polymer, it is commonly used as a flexible denture base material. Therefore, according to a clinically preferred maximum flexural strength of 65 MPa and an elastic modulus of 2,000 MPa, the preferred maximum flexural strength was satisfactory in all conditions except for the air-exposed/35°C/30 minutes post-polymerization condition. The preferred elastic modulus was acceptable under air-exposed/60°C/30 minutes, air-exposed/60°C/60 minutes, and glycerin/60°C/60 minutes post-polymerization conditions. The post-polymerization conditions for acceptable maximum flexural strength and elastic modulus were air exposure under 60°C and 30 minutes, air exposure under 60°C, and 60 minutes, and glycerin immersion under 60°C and 60 minutes, respectively.

Comparing the mechanical properties of the thermoplastic denture base resin with a previous study and post-polymerization at 60°C and 60 minutes with and without glycerin, Hamanaka et al.[21] measured the maximum flexural strength, flexural strength at the proportional limit, and elastic modulus of six types of clinically used thermoplastic denture base resins stored for 30 days in distilled water at 37°C according to ISO 20795-1. The results are as follows. The maximum flexural strength ranged from 38.17±1.64 MPa to 93.04±3.60 MPa, the flexural strength in the proportional limit ranged from 11.69±3.85 MPa to 37.97±7.98 MPa, and the elastic modulus ranged from 880±0.04 MPa to 2,510±0.15 MPa[21]. Ayaz et al.[22] measured the Vickers hardness of two types of polyamide-based thermoplastic denture base resin; when 100 g of force was applied to each specimen for 30 seconds, hardness values of 8.8±1.22 kg/mm^2 and 8.0±1.45 kg/mm^2 were reported, respectively. Ali et al.[23] measured Vickers hardness for heat-polymerized resins and self- polymerized resins. When a force of 300 g was applied to each specimen for 15 seconds, hardness values of 17.0±0.4 kg/mm^2 and 16.0±0.4 kg/mm^2 were reported, respectively. Alhareb et al.[24] measured the Vickers hardness of heat-polymerized resins. A force of 300 g applied for 10 seconds lead to the hardness of 17.60±0.79 kg/mm^2. Fueki et al.[25] conducted a scratch test between thermoplastic denture base resin and PMMA, and reported that thermoplastic resin was more easily damaged on the surface compared to PMMA. Hamanaka et al.[26] evaluated abrasion resistance between thermoplastic denture base resin and heat-polymerized PMMA using nanoindentation, and reported that the abrasion resistance of thermoplastic denture base resin was lower than that of heat-polymerized PMMA.

When comparing the mechanical properties of 3D-printed flexible denture resin with previous studies of thermoplastic denture base resin, it has been claimed that the evaluated 3D-printed flexible denture resin has sufficient mechanical properties for use as a flexible denture base material under specific post-polymerization conditions. A limitation of this preliminary study is that this 3D-printed flexible denture resin was not directly tested with a thermoplastic denture base resin under the same environment, which should be assessed in future studies. Future research directions include fatigue, impact strength, absorption and solubility, color stability, denture base-fitting accuracy, and cytotoxicity tests. In addition, the bonding strength between 3D-printed flexible denture resin and 3D-printed denture teeth and evaluation of bonding strength between 3D-printed flexible denture resin and denture relining material is required.

Conclusion

1. The 3D-printed flexible denture resin showed the highest mechanical properties at post-polymerization conditions of 60°C and 60 minutes, regardless of the presence of glycerin, and acceptable maximum flexural strength and elastic modulus of 65 MPa and 2,000 MPa or more, respectively.

2. Further studies have yet to follow to determine whether this resin is suitable for clinical use, including comparative studies with thermoplastic denture base resin.

Conflict of Interest

No potential conflict of interest relevant to this article was reported.

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