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Original Article

Effects of postpolymerization time and temperature on the flexural properties and hardness profile of three-dimensional printed provisional resin

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Abstract *Background/purpose:* Three-dimensional (3D) printing technique was widely used for provisional restorations in clinical use. However, the effects of post-polymerization temperature and time on the flexural properties and hardness profile were not fully elucidated yet. The purpose of this study is to investigate the effects of post-polymerization temperature and time on the flexural properties and hardness profile of the provisional restoration.

Materials and methods: 3D-printing provisional resin was printed and post-polymerized at various temperatures (room temperature, 40 °C, 60 °C and 80 °C) and periods (0, 15, 30, 60, 90 and 120 min of photopolymerization). Afterwards, the flexural strength, flexural modulus, surface hardness, and internal hardness at different depth were evaluated.

Results: The group post-polymerized without concurrent heating had significantly shallow depth of cure comparing to the heating counterparts. The surface hardness of the groups post-polymerized at different temperatures did not show any difference. All groups with post-polymerization temperature at 40 °C, 60 °C and 80 °C and post-polymerization time ranged between 15 and 90 min, had curing depth between 3 and 4 mm. Group post-polymerized without concurrent heating has significantly shallow depth of cure comparing to the heating counterparts.

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Conclusion: Post-polymerization at an elevated temperature, preferably 60 °C, is suggested. The wall thickness of the 3D-printing provisional prosthesis thinner than 3–4 mm is recommended.

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Introduction

Provisional restorations play an important role in fixed dentistry. Nowadays, three-dimensional (3D) printing provisional restorations used in dentistry were fabricated mainly from photopolymerization-based 3D printers. Photopolymerization-based 3D printing resins were composed of monomers, oligomers, diluent and photoinitiator.¹ Photoinitiator is the key component to initiate photopolymerization reaction. It can be activated by specific wavelength of light and generate highly active free radicals. These radicals will open the double bonds of the acrylates or methacrylates and initiate a series of propagation reactions of the resin. The exposure time of each layer was normally set to be the exposure period after which the current layer of polymerizing resin is strong enough to withstand the peeling force between the solidified resin and the bottom of the resin tray. After the exposure of each layer, the 3D printer proceeds the peeling action and ready to print the next layer. At this time point, the polymerizing resin in the current layer is still propagating and have not entered the termination stage yet.

All 3D-printed models before post-polymerization are in green state, which means that they were only partially polymerized and post-polymerization process can maximize the degree of polymerization.² During post-polymerization, continuous exposure to ultraviolet light (UV) or near-ultraviolet light can complete the propagating polymer chains, and bring the photopolymerization reaction into termination stage.^{3,4}

Increasing temperature moderately is able to improve energy and fluidity and provide higher degree of cross-linking reaction. Bagis and Rueggeberg founded that thermal energy can enhance the degree of conversion of resins after post-polymerizations.⁵ Many material properties, such as surface hardness, flexural strength, glass transition temperature (T_g) and color stability, were also improved.⁵

So far, the studies about 3D printing provisional restorations were still limited. Most literatures focused on the parameter setting for 3D printing process.^{6,7} Green state needs to go through the post-polymerization process to reach the designed mechanical properties and biocompatibility.^{8,9} Therefore, the purpose of this study was to investigate effects of post-polymerization on the flexural properties and hardness profile of 3D-printed provisional resins.

Materials and methods

The 3D printing resin for provisional restorations used in this study was AA temp (Enlighten Materials Co., Taipei,

Taiwan), which, at the time of the experiment, was the only resin with FDA approval in Taiwan. It was certified as a Class II medical device by both the U.S. FDA and Taiwan FDA. The initiation wavelength of AA Temp was 365~405 nm.

All 3D-printed specimens were designed using either Autodesk Meshmixer (Autodesk Inc., San Rafael, CA, USA) or Exocad Dental CAD software (Exocad GmbH, Darmstadt, Germany) prior to printing. The samples of 3D model were exported and saved as STL files, exported into MiiCraft Ultra 125 DLP 3D printer (Young Optics Inc., Hsinchu City, Taiwan) and printed with a layer thickness of 50 μ m. After printing, all printed specimens were cleaned with 95% alcohol for 15 min. The specimens were then dried and received post-polymerization treatment using a 405 nm FormCure (Formlabs Inc., Somerville, MA, USA) post-polymerization unit at room temperature, 40, 60 and 80 °C for 0, 15, 30, 60, 90 and 120 min. The specimens in the green state (GS) (specimens without post-polymerization) were used as a control.

According to the standard ISO 10477:2018, six 3D-printed 2×2×25 mm bars per group were prepared randomly from build platform of the 3D printer.¹⁰ The flexural strength and flexural modulus were measured using the 3-point bending method, with QC-513B a universal test machine (Cometech Testing Machines Co., Taichung City, Taiwan) at a cross-head speed of 1 mm per minute.

The surface hardness of the 10×10×10 mm specimens ($n = 18$) was measured using the HMV-2 micro Vickers hardness tester (Shimadzu Corporation, Kyoto, Japan) with applying fixed force of 100 g (0.98 N) for 15 s.

The micro Vickers hardness tester was also indirectly used to evaluate the extent of polymerization throughout the cross-section of the 3D printed specimens. To understand the depth of cure of the 3D printing model after post processing treatments, 10×10×10 mm specimens were cut in half by using an IsoMet®1000 precision sectioning saw (Buehler Ltd., Lake Bluff, IL, USA). The internal Vickers hardness (VHN) was measured as a function of depth of material at 1 mm intervals. All specimens were also examined using the HMV-2 micro Vickers hardness tester with applying fixed load of 100 g (0.98 N) for 15 s. The average value was calculated from six different locations on each interval of the specimen ($n = 6$).

The flexural strength, flexural modulus, and surface Vickers hardness data were analyzed using a two-way analysis of variance (ANOVA) followed by the Tukey post hoc test, utilizing the statistical software program SPSS® version 24.0 (IBM SPSS Inc., Chicago, IL, USA) with a significance level (α) of 0.05. The analysis of the internal hardness results was conducted using a one-way ANOVA, followed by Dunnett's test for comparing all depths with 80% of the surface hardness.

Results

Flexural strength and flexural modulus of AA Temp post-polymerized with different temperature/time combinations were shown in Figs. 1 and 2, respectively. It can be clearly seen that all groups with post-polymerization have significantly higher flexural strength and modulus than those with no post-polymerization. 15 min of post-polymerization at any ambient temperature can already elevate the flexural strength above 50 MPa, which is the minimal requirement for provisional restoration, as stated in ISO 10477:2018.¹⁰ When post-polymerization time increased up to 90 min, an increasing trend of flexural strength and modulus can be observed at all ambient temperatures. While there was no significant difference between 90-min and 120-min post-polymerization times, except for the modulus at 40 °C ($P < 0.001$), extending the post-polymerization time to 120 min resulted in a decrease in the average values of flexural strength and modulus. At an identical post-polymerization time, a heating temperature of 60 °C resulted in the highest flexural strength and modulus. However, there was no significant difference in the flexural strength among different heating temperatures when the post-polymerization time was set at 0 min ($P > 0.05$). Furthermore, with increasing temperature, both the flexural strength and modulus exhibited an upward trend under the same post-polymerization time. Nevertheless, when the temperature was raised to 80 °C, there was a significant decrease in both flexural strength and modulus ($P < 0.05$). The highest value in flexural strength and modulus was shown in 60 °C/90 min group.

The surface hardness analysis of AA Temp post-polymerized for different temperature/time combinations was showed in Fig. 3. It can be seen that surface hardness of groups with post-polymerization were significantly higher than the groups without post-polymerization ($P < 0.001$). With increasing post-polymerization time, the surface hardness at all post-polymerization temperature were getting slightly higher. However, there was no significant difference for all groups ($P > 0.05$). It was

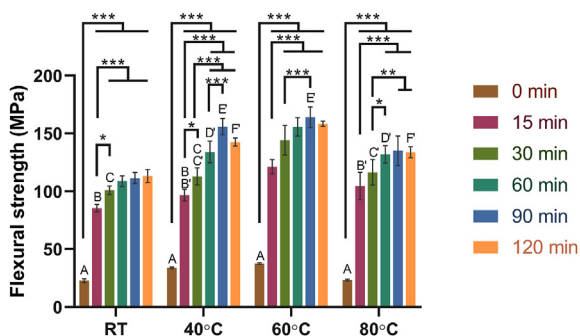


Figure 1 Flexural strength of specimens after post-polymerization for different time/temperature combinations. Asterisks indicate statistically significant differences between the different times at the same temperature ($*P < 0.05$, $**P < 0.01$, $***P < 0.001$). Different letters stand for statistically significant differences ($P < 0.05$) between different temperatures within the same time. Abbreviation: RT (Room temperature), min (minutes).

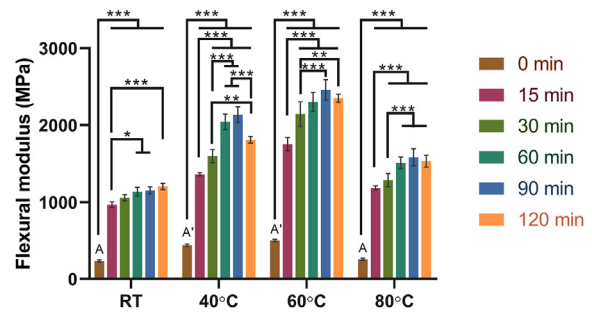


Figure 2 Flexural modulus of specimens after post-polymerization for different time/temperature combinations. Asterisks indicate statistically significant differences between the different times at the same temperature ($*P < 0.05$, $**P < 0.01$, $***P < 0.001$). Different letters stand for statistically significant differences ($P < 0.05$) between different temperatures within the same time. Abbreviation: RT (Room temperature), min (minutes).

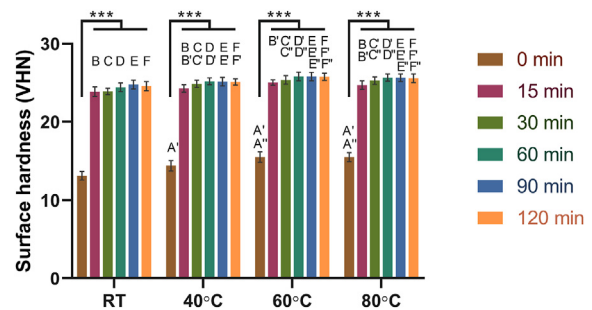


Figure 3 The surface Vickers hardness of specimens after post-polymerization for different time/temperature combinations. Asterisks indicate statistically significant differences between the different times at the same temperature ($*P < 0.05$, $**P < 0.01$, $***P < 0.001$). Different letters stand for statistically significant differences ($P < 0.05$) between different temperatures within the same time. Abbreviation: VHN (Vickers hardness), RT (Room temperature), min (minutes).

observed that a heating temperature of 60 °C led to significantly higher surface hardness compared to the room temperature group for post-polymerization times of 0, 15, 30, and 60 min ($P < 0.05$). Furthermore, a heating temperature of 80 °C also resulted in significantly higher surface hardness for post-polymerization times of 0, 30, and 60 min ($P < 0.05$).

The internal hardness of AA Temp post-polymerized for different temperature/time combinations was showed in Table 1. The assessment of internal hardness served as a means to determine the resin's depth of cure.^{10–12} The internal hardness that was larger than 80% of the surface hardness was defined as cured resin, a concept elucidated by Bouschlicher et al. and Aravamudhan et al.^{11,12} It can be observed from Table 1 that, although the hardness values at all the depths were significantly higher than 80% of the surface hardness, without post-polymerization, the internal hardness at all depths had comparable values with the surface hardness, regardless of the ambient temperature. When the post-polymerization time increased to

Table 1 The internal Vickers hardness (VHN) of the 3D printing provisional resin after post-polymerization with different temperature/time combinations. The first column stands for the surface hardness, and the 2nd column to 6th column are measurement points located 1 mm–5 mm deep from the surface of the cube. Groups with internal Vickers hardness less than 80% of the surface hardness were in bold. Asterisks indicate statistically significant differences compared to 80% of the surface hardness (* $P < 0.05$, ** $P < 0.01$, *** $P < 0.001$).

Curing time (minutes)	Curing temperature (°C)	80% of surface hardness	Internal hardness (Mean \pm SD)					
			0 [Surface]	1 mm	2 mm	3 mm	4 mm	5 mm
0	RT	10.64 \pm 0.25	13.3 \pm 0.31***	13.33 \pm 0.31***	13.19 \pm 0.36***	13.33 \pm 0.40***	13.31 \pm 0.37***	13.17 \pm 0.29***
	40	11.65 \pm 0.21	14.57 \pm 0.27***	14.54 \pm 0.29***	14.51 \pm 0.35***	14.49 \pm 0.32***	14.6 \pm 0.2***	13.92 \pm 0.25***
	60	12.57 \pm 0.2	15.72 \pm 0.25***	15.57 \pm 0.3***	15.58 \pm 0.24***	15.59 \pm 0.25***	15.6 \pm 0.22***	14.38 \pm 0.30***
	80	12.53 \pm 0.17	15.67 \pm 0.22***	15.53 \pm 0.25***	15.53 \pm 0.23***	15.51 \pm 0.20***	15.45 \pm 0.24***	14.42 \pm 0.19***
15	RT	19.21 \pm 0.24	24.02 \pm 0.31***	18.73 \pm 0.21***	18.43 \pm 0.26***	17.17 \pm 0.17***	14.18 \pm 0.15***	13.45 \pm 0.29***
	40	19.56 \pm 0.17	24.45 \pm 0.21***	19.86 \pm 0.21**	19.74 \pm 0.19	19.59 \pm 0.14	14.88 \pm 0.13***	14.08 \pm 0.15***
	60	20.16 \pm 0.15	25.2 \pm 0.19***	20.93 \pm 0.19***	20.46 \pm 0.18**	20.17 \pm 0.16	16.63 \pm 0.15***	15.43 \pm 0.20***
	80	19.91 \pm 0.19	24.88 \pm 0.23***	20.83 \pm 0.21***	20.41 \pm 0.18***	20.11 \pm 0.23	16.55 \pm 0.21***	15.2 \pm 0.18***
30	RT	19.25 \pm 0.19	24.07 \pm 0.24***	19.53 \pm 0.2*	18.82 \pm 0.17***	17.84 \pm 0.23***	14.82 \pm 0.23***	13.83 \pm 0.18***
	40	20.03 \pm 0.22	25.03 \pm 0.27***	20.94 \pm 0.12***	20.23 \pm 0.14	20.12 \pm 0.13	15.17 \pm 0.16***	14.87 \pm 0.23***
	60	20.43 \pm 0.17	25.53 \pm 0.22***	21.31 \pm 0.24***	20.85 \pm 0.25**	20.44 \pm 0.24	16.82 \pm 0.25***	15.92 \pm 0.26***
	80	20.36 \pm 0.15	25.45 \pm 0.19***	21.2 \pm 0.21***	20.83 \pm 0.24***	20.41 \pm 0.2	16.78 \pm 0.21***	15.52 \pm 0.25***
60	RT	19.68 \pm 0.18	24.6 \pm 0.23***	19.93 \pm 0.19	19.83 \pm 0.19	18.95 \pm 0.19***	15.18 \pm 0.19***	14.97 \pm 0.26***
	40	20.29 \pm 0.17	25.37 \pm 0.21***	21.97 \pm 0.16***	21.31 \pm 0.18***	20.85 \pm 0.14***	16.48 \pm 0.22***	15.62 \pm 0.13***
	60	20.79 \pm 0.14	25.98 \pm 0.17***	22.47 \pm 0.22***	21.64 \pm 0.15***	20.93 \pm 0.15	17.52 \pm 0.21***	16.53 \pm 0.14***
	80	20.67 \pm 0.15	25.83 \pm 0.19***	22.36 \pm 0.23***	21.52 \pm 0.22***	20.79 \pm 0.18	17.41 \pm 0.26***	16.47 \pm 0.19***
90	RT	19.96 \pm 0.17	24.95 \pm 0.22***	20.28 \pm 0.13**	20.03 \pm 0.19	19.98 \pm 0.19	15.51 \pm 0.16***	15.17 \pm 0.28***
	40	20.25 \pm 0.17	25.32 \pm 0.21***	22.35 \pm 0.18***	21.7 \pm 0.13***	21.07 \pm 0.18***	16.97 \pm 0.16***	15.93 \pm 0.19***
	60	20.77 \pm 0.17	25.97 \pm 0.22***	22.78 \pm 0.14***	21.9 \pm 0.13***	21.2 \pm 0.13***	17.73 \pm 0.19***	16.55 \pm 0.08***
	80	20.65 \pm 0.14	25.82 \pm 0.17***	22.22 \pm 0.22***	21.47 \pm 0.21***	20.78 \pm 0.21	17.6 \pm 0.2***	16.52 \pm 0.21***
120	RT	19.8 \pm 0.17	24.75 \pm 0.21***	20.94 \pm 0.17***	20.23 \pm 0.16***	20.03 \pm 0.16*	15.98 \pm 0.15***	15.45 \pm 0.27***
	40	20.23 \pm 0.16	25.28 \pm 0.2***	22.6 \pm 0.15***	21.83 \pm 0.18***	21.63 \pm 0.21***	17.43 \pm 0.14***	16.5 \pm 0.20***
	60	20.75 \pm 0.15	25.93 \pm 0.19***	23.07 \pm 0.19***	22.42 \pm 0.15***	21.98 \pm 0.19***	18.13 \pm 0.15***	16.97 \pm 0.18***
	80	20.6 \pm 0.15	25.75 \pm 0.19***	22.13 \pm 0.2***	21.41 \pm 0.22***	20.61 \pm 0.21	17.63 \pm 0.29***	17.77 \pm 0.18***

Abbreviation: RT (Room temperature), SD (Standard deviation).

The internal hardness represented in bold are significantly lower than 80% of the surface hardness.

15 min, the hardness values at 1, 2, and 3 mm depths for conditions 40 °C/15 min, 60 °C/15 min, and 80 °C/15 min exceeded 80% of the surface hardness. This indicates that the depth of cure for 40 °C/15 min, 60 °C/15 min, and 80 °C/15 min extended to 3 mm, while for RT/15 min, the curing depth was less than 1 mm. When the post-polymerization time duration was extended to 30 min, the depth of cure for 40 °C/30 min, 60 °C/30 min and 80 °C/30 min still reached to 3 mm. However, for RT/30 min, the curing depth increased to less than 2 mm. The increasing depth of cure over time can only be observed in the groups without heating treatment during post-polymerization. After heating treatment, the hardness values at depths of 4 and 5 mm within each group were significantly lower than 80% of the surface hardness ($P < 0.001$). This suggests that the groups subjected to heating treatment during different post-polymerization times had curing depths of less than 4 mm.

Discussion

Regardless of the ambient temperature, the flexural strength of the groups with post-polymerization were statistically higher than the groups without post-polymerization ($P < 0.001$). According to ISO 10477:2018, which is the most commonly used standard for provisional prosthesis, the recommended minimum flexural strength value is 50 MPa.¹⁰ The groups without post-polymerization were obviously not satisfied with the fundamental strength. On the contrary, the groups with post-polymerization met the standard. The group with post-polymerization at 60 °C had highest flexural strength among all groups. The value was between 85.61 ± 3.22 and 164.05 ± 8.96 MPa. Haselton et al. compared total of 13 commercial methacrylate-based or bis-acryl resins for conventional provisional restorations, and the flexural strength ranged between 56.2 ± 4.1 and 123.6 ± 13.6 MPa.¹³ Digholkar et al. evaluated the provisional prosthesis fabricated using poly (methyl methacrylate) (PMMA) computer-aided design/computer-aided manufacturing (CAD/CAM) blanks, yielding a value of 104.2 ± 12.8 MPa.¹⁴ Compared to the aforementioned studies, it was founded that the flexural strength of AA Temp 3D printing provision resin under appropriate post-polymerization treatment could be comparable to conventional or CAD/CAM blanks provisional resins.

Flexural modulus is an indicator of stiffness. When the ambient temperature heating up to 60 °C, the flexural modulus of AA Temp could be enhanced with post-polymerization. Regardless of ambient temperature and post-polymerization time duration, the groups with post-polymerization were significantly higher than the groups without post-polymerization ($P < 0.001$). The values of post-polymerization groups were between 968.9 ± 37.4 and 2460.1 ± 133.8 MPa. The groups that underwent post-polymerization at 60 °C for over 30 min or the groups post-polymerized at 40 °C for 60 and 90 min exhibited a flexural modulus exceeding 2000 MPa. This value is comparable to that of conventional methacrylate-based provisional resins (2000 ± 100 MPa).¹⁵

Digholkar et al. compared the surface hardness of provisional resins using 3D printing, CAD/CAM and conventional fabrication.¹⁴ The results indicated that the group fabricated using 3D printing exhibited the highest surface hardness.¹⁴ According to ISO 4049 standard, the suggested curing depth for provisional restorations is 1.5 mm.¹⁶ Moreover, DeWald et al. and Aravamudhan et al., suggested that the curing depth of the provisional restoration can be defined as the depth where the internal hardness is higher than of 80% of the surface hardness.^{12,17} In Table 1, the depth where its internal hardness lower than 80% of the surface hardness was highlight in bold. It is interesting to see that groups with post-polymerization temperature at 40 °C, 60 °C and 80 °C and post-polymerization time ranged between 15 and 120 min, had curing depth between 3 and 4 mm. It suggested that energy introduced during post-polymerization with elevated temperature can effectively work on provisional crown with wall thickness less 4 mm. However, for large restorations such as a pontic, the internal structure may not be polymerized well during post-polymerization with elevated temperature.

Munoz et al. suggested that heating treatment up to 60 °C could shorten the curing time of resin-based materials without affecting the polymerization.¹⁸ Our results were shown a good accordance. It was found in our data that groups with post-polymerization at 60 °C showed higher flexural strength, flexural modulus, surface hardness and internal hardness than the counterparts at room temperature, 40 °C, and 80 °C. The behavior of flexural strength and flexural modulus in response to varying temperature and post-polymerization time reveals important insights about the 3D printing material's properties. The trend of flexural strength and flexural modulus increasing is observed as the temperature rises for the same post-polymerization time duration, continuing until the temperature surpasses 60 °C. However, a decline in values is noted at 80 °C. Similarly, at a constant heating temperature, as the post-polymerization time is extended, both flexural strength and flexural modulus increase. However, the values of the groups with heating treatment decreased when the post-polymerization time was extended to 120 min. This phenomenon is likely indicative of photodegradation, a process where prolonged exposure to light can lead to a decline in material properties.¹⁹ In our previous study, we assessed the cytotoxicity of two provisional 3D printing resins (AA TEMP; Enlighten Materials, and C&B; NextDent). The findings revealed that excessive post-polymerization exposure also has the potential to induce cytotoxicity in 3D printing provisional resins.²⁰ Consequently, these findings underscore the importance of achieving a balance between temperature and post-polymerization time to prevent overexposure and photodegradation, highlighting the significance of optimal conditions for maximizing material performance.

In conclusion, it can be inferred that heating at the appropriate temperature during the proper post-polymerization time is crucial for achieving maximal flexural strength, modulus, and hardness in 3D-printed provisional prostheses. This enables 3D-printed provisional resins to attain comparability with conventional provisional resins. Manufacturers should specify the curing machine and post-

polymerization temperature in the instruction for use to ensure better clinical outcomes.

Declaration of competing interest

The authors have no conflicts of interest relevant to this article.

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