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RESEARCH AND EDUCATION

Influence of polymerization protocols on the physical and mechanical properties of a 3D printed resin

Fabio A.P. Rizzante, DDS, MSc, PhD, MBA,[a] Michael Azzer, DMD,[b] Nima G. Moghaddam, DMD,[c] Thomas Watson, DMD,[d] Guilherme F. Moura, DDS, MSc, PhD,[e] and Adilson Y. Furuse, DDS, MSc, PhD[f]

ABSTRACT

Statement of problem. Fast prototyped, or 3-dimensionally (3D) printed, materials enhance clinical efficiency when compared with other manufacturing methods. Nevertheless, standardization and information regarding the influence of different postprocessing protocols on the final physical and mechanical properties of 3D printed parts is lacking.

Purpose. The purpose of this in vitro study was to evaluate the effect of different polymerization methods and times on the flexural strength, microhardness, and color stability of a 3D printed resin (OnX; SprintRay).

Material and methods. A total of 40 disks (Ø10×2 mm) and 40 bars (10×2×2 mm) were 3D printed, washed, and subdivided into 4 groups (n=10) according to the polymerization protocol: VALO Grand light polymerization unit for 40 and 120 seconds (VG40s andVG120s) and ProCure 2 polymerization chamber for 1 and 2 cycles (PC×1 and PC×2). The bars were stored in distilled water at 37 °C for 24 hours, and a 3-point bend test was performed with a universal testing machine with an 8-mm span and a downward movement at a rate of 0.5 mm/minute until fracture. The disks were polished with abrasive disks. Color stability was assessed after polymerization (baseline), after 1 and 7 days in dark, dry storage at 37 °C, and after 3 days of artificial aging in deionized water at 60 °C. Values of b* were used to calculate yellow shift/Δb* values after 3 days of artificial aging. Microhardness after 7 days in dark, dry storage was assessed with a Knoop indenter. The data were assessed for homogeneity using the Levene test and for normality using the Shapiro-Wilk test. Two-way ANOVA (flexural strength, microhardness, and Δb* tests) and 3-way repeated-measures ANOVA (color stability test) were followed by the Tukey HSD post hoc test (α=.05 for all tests).

Results. For microhardness, the polymerization unit (P<.001), polymerization cycles (P=.003), and interaction between both factors (P=.005) were significantly different, with VG40s=VG120s>PC×1>PC×2. For flexural strength, the polymerization unit (P<.001), polymerization cycles (P<.001), and interaction between both factors (P<.001) were significantly different, with VG120s=PC×1=PC×2>VG40s. For color stability, the polymerization unit (P=.009), time (P<.001), and interaction between time and polymerization unit (P<.001) and time, polymerization unit, and cycle (P=.01) were significantly different. After 3 days of artificial aging, PC×1=PC×2>VG40s=VG120s. Significantly different Δb* was found for polymerization unit (P<.001) and polymerization cycles (P=.002), with VG120s<VG40s=PC×2≤PC×1.

Conclusions. Resins polymerized using VG120s produced similar or better microhardness, flexural strength, and color stability results than PC while significantly decreasing the postpolymerization time. Specimens polymerized with PC×2 showed the lowest microhardness. Excessively increased polymerization time may jeopardize the properties of 3D printed parts. (J Prosthet Dent 2025;133:1091.e1-e6)

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[a]Associate Professor and Assistant Dean for Innovation, James B. Edwards College of Dental Medicine, Medical University of South Carolina (MUSC), Charleston, SC.
[b]Graduate student, James B. Edwards College of Dental Medicine, Medical University of South Carolina (MUSC), Charleston, SC.
[c]Graduate student, James B. Edwards College of Dental Medicine, Medical University of South Carolina (MUSC), Charleston, SC.
[d]Graduate student, James B. Edwards College of Dental Medicine, Medical University of South Carolina (MUSC), Charleston, SC.
[e]Assistant Professor, Department of Reconstructive and Rehabilitation Sciences, James B. Edwards College of Dental Medicine, Medical University of South Carolina (MUSC), Charleston, SC.
[f]Associate Professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo (USP), Bauru, SP.

Clinical Implications

This study provided insights into more efficient postprocessing methods and into the influence of different postpolymerization methods on the final properties of 3D printed materials. The potential negative effects of additional polymerization cycles should be considered.

The manufacturing of dental restorations, whether interim or definitive, has seen a significant shift towards the integration of computer-aided design and computer-aided manufacturing (CAD-CAM) technologies. This digital workflow relies on a combination of optical scanners, CAD software programs, and manufacturing units.[1,2]

Additive manufacturing, or 3-dimensional (3D) printing, has gained popularity because of its enhanced efficiency compared with milling methods. It delivers comparable accuracy at a reduced cost and minimizes waste.[1,2] Three-dimensional printing in dentistry typically uses versatile stereolithography (SLA) or digital light-processing (DLP) technologies for surgical guides, study casts, and interim and definitive restorations.[1–3]

Three-dimensionally printed interim restorations have been reported to have similar or better properties compared with traditional chairside interim materials such as acrylic and bisacrylic resins while being fully integrated with the digital workflow.[2–4] Nevertheless, concerns arise from the lack of standardization and information regarding the influence of different postprocessing protocols on the final properties of 3D printed parts. Different materials may benefit from different polymerization protocols, including polymerization time, energy, and temperature, which may not be achieved by some polymerization chambers.[1,5–10] This concern is especially relevant for hobbyist and open-platform printers, with no validated protocol for different 3D printed resins.

Despite the marketing of polymerization chambers capable of reducing polymerization times from 30 to 60 minutes to approximately 15 minutes,[4] few validated devices are available. Therefore, even with a fully validated workflow, the time required for postprocessing could hinder its chairside application.[1] Additionally, broad-spectrum light polymerization units have been proposed as a means of increasing the efficiency of the postpolymerization process.[1] However, the authors are unaware of this polymerization method being tested with resins marketed for definitive restoration.

Because of the wide variety of material composition and the lack of standardization of postpolymerization protocols, a better understanding of these processes is needed. Defining the balance between polymerization time and the parameters for different polymerization chambers ensures optimal material properties and avoids undesirable consequences such as photodegradation or insufficient polymerization that could increase the toxicity of the 3D printed device.[11–15]

This study evaluated the effect of different polymerization methods and polymerization times on the microhardness, flexural strength, and color stability of a 3D printed resin (OnX; SprintRay) recommended for interim implant-supported prostheses and definitive denture teeth (OnX; SprintRay). The evaluation was conducted using a validated workflow, including a 3D printer (Pro95; SprintRay) and polymerization chamber (ProCure 2; SprintRay), as well as an alternative polymerization method (VALO Grand; Ultradent Products, Inc) with different polymerization times. The null hypotheses were that no difference in microhardness, flexural strength, or color stability would be found across different polymerization methods and that no difference in these properties would be found considering different polymerization times.

MATERIAL AND METHODS

This study followed a 2×2 factorial design having as study factors polymerization units at 2 levels (PC-ProCure 2; SprintRay and VG-VALO Grand light polymerization unit; Ultradent Products, Inc) and polymerization time at 2 levels (PC for the predetermined OnX resin profile cycle time/PC×1 and double the recommended time/PC×2 and VG for 40 seconds/VG40s and 120 seconds/VG120s). The response variables were the flexural strength, microhardness, and color stability of a 3D printed resin (OnX; SprintRay).

For the flexural strength test, 8 rectangular bars (10×2×2 mm) were used per group, while color stability and microhardness tests used 10 disk-shaped specimens (Ø10×2 mm) per group. The resin specimens were designed by using an open-source software program (Meshmixer; Autodesk Inc) exported in standard tessellation language (STL) format and subsequently imported into a nesting software program (Dashboard; SprintRay). All specimens were printed using the automated resin profiles and a 3D printer (Pro95 DLP; SprintRay) at a 90-degree angle, with a layer thickness of 100 μm,[1,3] and medium supports automatically generated by the software program. The supports were placed at the sides of the specimens, avoiding critical test areas.[1]

After being printed, the specimens were sprayed with 99.5% isopropyl alcohol (IPA) followed by an air stream to eliminate nonpolymerized resin according to the manufacturer's instructions. The specimens were subdivided based on polymerization units and times (Table 1).

Table 1. List of resin, polymerization units, and respective polymerization times

Polymerization unit	*Manufacturer*	*Polymerization times/cycles*	*Characteristics*
ProCure 2	SprintRay	1 and 2 cycles	Wavelength: 385 42 individual LEDs Max temperature: 80 °C
VALO grand	Ultradent Products, Inc	40 S and 2 min	Wavelength: 385 to 515 nm Standard mode-1000 mW/cm^2
Material	*Manufacturer*	*Characteristics*	
OnX Bleach resin	SprintRay	Methacrylate oligomers/monomers and acrylic monomers with nanoceramic fillers	

All disk specimens had 1 surface polished using a polishing machine (Ecomet 3000; Buehler Ltd.) and a sequence of 600-, 1200-, and 2000-grit abrasive disks under water cooling. All microhardness and color measurements were performed on the polished surface.[2,16]

The Knoop microhardness test was performed after 7 days of dark, dry storage at 37 ºC. Microhardness was measured with a 0.5-N load applied for 15 seconds and a 0.5-mm/minute crosshead speed using a microhardness tester (Micromet 5114 tester; Buehler Ltd). Three readings were made, with a 500-µm separation between each indentation. The indentations were assessed using the built-in stereomicroscope (×10), and the largest diagonal was used to calculate the Knoop hardness,[2,16] with the mean value considered the final result.

Bar-shaped specimens for flexural strength were stored in distilled water at 37 °C for 24 hours before testing. Subsequently, a 3-point bend test was performed using a universal testing machine (OM150; Odeme Dental Research) with an 8-mm span and a downward movement of 0.5 mm/minute until specimen fracture.[1,2] The force applied (N) was recorded and then converted to MPa using the formula: $FS=3Fl/2bd^2$, where FS is the flexural strength in MPa, F is the loading force at the fracture point in N, l is the length of the support span (8 mm), and b and d are the width and thickness of the specimen cross-section as measured with digital calipers (Digimatic Caliper 500-196-30; Mitutoyo America Corp).

The color stability test was performed on the same specimens used for the Knoop microhardness test with a spectrophotometer (Vita Easyshade V; Vita Zahnfabrik) at different timepoints: immediately after polymerization (baseline), after 1 and 7 days in dark, dry storage at 37 °C, and after 3 days of artificial aging in deionized water at 60 °C.[1,2,16] For each timepoint, the spectrophotometer was calibrated using the built-in calibration tool. The specimens were positioned over a white background, dried with absorbent paper when needed, and evaluated with the device positioned perpendicular to the specimen surface. Two measurements per specimen were made until both results aligned.[1] The degree of color change was calculated using the formula: $\Delta E00=[(\Delta_L/K_L\times S_L)+(\Delta_C/K_C\times S_C)^2+(\Delta_H/K_H\times S_H)^2+R_T\times(\Delta_C/K_C\times S_C)\times(\Delta_H/K_H\times S_H)]^{0.5}$, where ΔL′, ΔC′, and ΔH′ represent the differences in the readings of luminosity, chroma, and hue respectively, between the baseline and the different time points. RT refers to the interaction between chroma and hue in the blue region, SL, SC, and SH are weighting functions to adjust total color difference in L*, a*, and b* coordinates, and KL, KC, and KH are correction terms for experimental conditions. In addition, the results of b were used to calculate the yellow shift (Δb*) between baseline and 3 days after artificial aging.

The results were tested for homogeneity with the Levene test and for normality with the Shapiro-Wilk test. A 2-way analysis of variance (ANOVA) was used for microhardness, flexural strength, and Δb* tests (polymerization method and time). A 3-way repeated-measures ANOVA was used for the color stability test (time, polymerization method, and polymerization time). ANOVA tests were followed by the Tukey HSD test (α=.05).

RESULTS

Results for microhardness and flexural strength are shown in Table 2. For microhardness, the polymerization method (P<.001), polymerization cycles (P=.003), and interaction between both factors (P=.005) were significantly different. Overall results showed VG>PC and 1 cycle/40 seconds>2 cycles/120 seconds. When assessing all groups, VG40s=VG120s>PC×1>PC×2.

For flexural strength, the polymerization method (P<.001), polymerization cycles (P<.001), and interaction between both factors (P<.001) were significant. Overall results for flexural strength showed PC>VG and 2 cycles/120 seconds>1 cycle/40 seconds. When assessing all groups, VG120s=PC×1=PC×2>VG40s.

For color stability, the polymerization unit (P=.009), time (P<.001), and interaction between time and polymerization unit (P<.001), and time, polymerization unit,

Table 2. Results of Knoop microhardness and flexural strength

	Microhardness (KHN)	**Flexural strength (MPa)**
VG40s	30.59 (1.16)A	101.22 (7.46)B
VG120s	30.4 (3.94)A	120.76 (5.36)A
PC×1	26.11 (3.25)B	122.29 (3.62)A
PC×2	20.85 (1.26)C	119.91 (5.57)A

Different letters indicate statistically significant differences between polymerization protocols (P<.05)

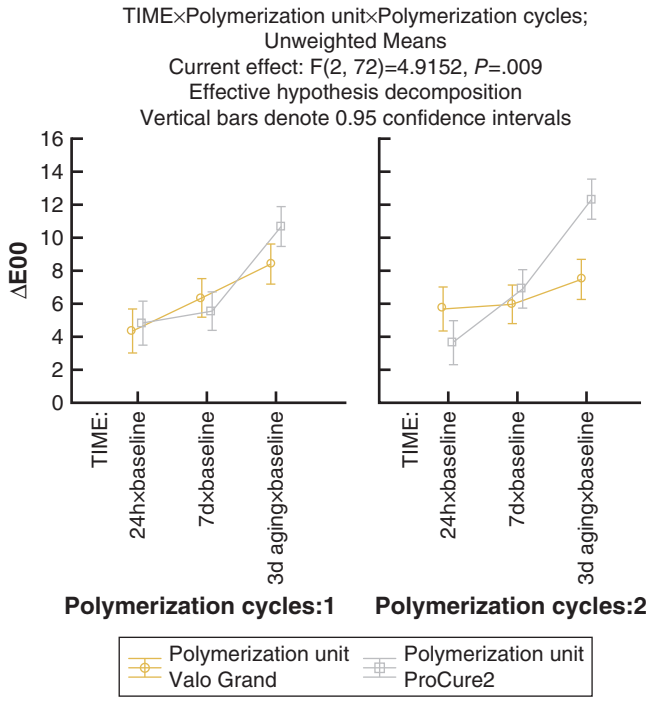

Figure 1. Degree of color alteration (ΔE_{00}) for different groups and timepoints.

and polymerization cycle (P=.01) were significantly different. Overall, groups polymerized with PC exhibited a higher degree of color alteration compared with specimens with VG. Additionally, specimens assessed after 3 days of aging showed a higher degree of color alteration than those evaluated after 24 hours and 7 days, except for VG120s. While all groups showed a similar degree of color stability after 24 hours and 7 days, after artificial aging, the groups polymerized with PC showed a higher degree of color alteration than groups polymerized with VG (Fig. 1, Table 3).

Results for Δb after 3 days artificial aging showed a similar tendency. Polymerization unit (P<.001) and polymerization cycles (P=.002) were significantly different. All groups showed negative b values compared with baseline. Groups polymerized with PC showed higher alteration compared with groups polymerized with VG. VG120s showed the lowest (closest to zero) Δb value, while PC×1 showed the highest Δb values (Table 3).

DISCUSSION

The null hypotheses that no difference in microhardness, flexural strength, or color stability would be found across different polymerization methods and that no difference in these properties would be found considering different polymerization times were rejected as there were observed differences in properties considering the different polymerization protocols and polymerization times.

This study used OnX resin (SprintRay), which has a fully validated workflow for use in a "closed ecosystem" comprised of a 3D printer and polymerization chamber, with recommended settings automatically provided by the manufacturer. Despite the manufacturers recommendation, increasing the polymerization time and temperature during postpolymerization might result in better properties as shown by previous studies with composite resins.[12,13] In addition, another study reported the potential for enhanced physical and mechanical properties of 3D printed resins polymerized using the VALO Grand light polymerization unit (Ultradent Products, Inc), which could decrease the polymerization time and be a more efficient option for the chairside use of 3D printed parts.[1] Nevertheless, the authors are unaware of a previous study that assessed the use of a light-polymerization device on a 3D printed resin for interim implant-supported prostheses and definitive denture teeth.

Considering the results for microhardness, both groups using the VG yielded the highest results, regardless of the polymerization time (40 and 120 seconds), followed by PC with 1 cycle. The microhardness values observed in this study for VG and PC 1 cycle were consistent with those reported previously (approximately 30 KHN).[6]

These results can be explained by the higher energy density of VG combined with its close proximity to the specimen surface during polymerization when compared with PC, resulting in sufficient energy to polymerize the surface effectively despite the reduced polymerization time.[1,17] Additionally, the extended wavelength of VG may be more suitable for activating the photo initiators in the resin.

Interestingly, polymerization using PC for 2 cycles resulted in the lowest surface microhardness. This could

Table 3. Degree of color alteration (ΔE_{00}) for different groups and timepoints and yellow shift (Δb) after 3 days of artificial aging

	24 hours-initial	7 days-initial	3 days aging-initial	Δb*
VG40s	4.35 (1.5)Aa	6.35 (1.52)Aab	8.39 (1.37)ABb	−5.38 (1.49)B
VG120s	5.69 (2.41)Aa	5.95 (1.77)Aa	7.47 (1.85)Aa	−3.12 (2.7)A
PC×1	4.82 (2.85)Aa	5.55 (2.23)Aa	10.65 (2.39)BCb	−8.09 (0.81)C
PC×2	3.64 (0.98)Aa	6.9 (1.64)Ab	12.31 (1.8)Cc	−6.86 (0.83)BC

*Separate statistical analysis (independent from ΔE_{00} results)

Uppercase letters indicate significant differences between rows in same column (different groups considering same timepoint).

Lowercase letters indicate significant differences between columns in same row (same group at different timepoints).

be explained by the prolonged exposure to heat (60 °C according to SprintRay) and light, which can lead to the degradation of the material surface through photodegradation.[12,14]

A decrease in the values of flexural strength was found when groups were polymerized at 80 °C, but no difference for surface microhardness has been reported previously.[12] In this study, only the results for microhardness were influenced by the extra polymerization cycle of PC, which may be a result of different resin composition and higher inorganic filler content for OnX when compared with the resin used in the previous study.[12]

The microhardness values were higher than in a previous study evaluating an unfilled 3D printed resin[16] but lower than composite resin and CAD-CAM blocks.[15] A material with higher surface microhardness should be more resistant to physical and chemical factors in the mouth, which could prevent wear and potential long-term cytotoxic effects on 3D printed restorations.[15]

Similar to other commercially available resins, information about the composition of OnX is limited. According to a recent study,[6] OnX seems to have bisphenol A-glycidyl methacrylate (bis-GMA) or bisphenol A-ethyl methacrylate (bis-EMA), triethylene glycol dimethacrylate (TEGDMA), and possibly some newly developed monomers as part of its organic content, associated with approximately 37% inorganic content comprised of 1- to 5-μm irregular particles of SiO_2 and Yb_2O_3 and 0.2- to 0.5-μm spherical particles of SiO_2.

Results for flexural strength were similar for all groups except VG40s, which may indicate that, despite adequate results for surface microhardness, the lower exposure time is not capable of promoting adequate polymerization depth. Moreover, because of the similar results of PC×1 and PC×2, no influence of the prolonged polymerization cycle on the surface was reflected in the overall mechanical properties of the material. Nevertheless, clinicians should be careful with repeated polymerization cycles to reproduce a glazed surface, as it is unclear whether the material properties would be jeopardized by such practice.

A recent systematic review reported that 3D printed parts have an average of 140 MPa mean flexural strength.[7] In addition, a recent study reported similar results for OnX flexural strength when printed and postpolymerized using the SprintRay ecosystem (similar to PC×1 group in this study).[6] Both recent[6] and present studies have shown slightly lower flexural resistance when compared with the results reported by the manufacturer (131.0 ±11.6 and 122.29 ±3.62 MPa compared with 147 MPa). These results can be attributed to differences in methods, since the manufacturer does not describe their protocol.

Considering color stability results, all groups showed a high degree of color alteration (ΔE>3.3 threshold).[1,8,16] Nevertheless, after artificial aging in water at 60 °C, specimens polymerized with VG showed lower color change than specimens polymerized with PC, which indicates a more stable polymer.[1,8,16]

No differences were found in color stability considering the polymerization times. Nevertheless, as the time storage increased, a trend for PC with higher color alteration was found when polymerized for 2 cycles compared with 1 cycle; this may indicate some degree of surface alteration when longer polymerization cycles are used, probably because of the prolonged increase in temperature.[14]

Although the results cannot be directly extrapolated because the reported tested energies may not accurately represent the energy reaching the tested specimens, the authors of another study[8] demonstrated that increasing light intensity led to improved color stability. However, higher light intensity associated with longer exposure times also demonstrated a tendency towards greater color alteration, similar to the present results.[8]

Despite their many advantages, 3D printed resins show poor color stability. The present results were consistent with those of other recent studies showing discoloration above clinical thresholds in less than 7 days of storage in different media.[1,2,9,10,16] Such behavior has been attributed to water absorption and hydrolysis because of the association of methacrylate matrix and low filler content.[18,19]

This study included the assessment of Δb, which represents a shift to either the yellow or blue wavelength spectrum after artificial aging. All groups showed negative Δb, indicating no increase in the b* color coordinate. Although the composition of 3D printed resins is not well understood, these results suggest there are only photoinitiators of type I (without amine as coinitiators) instead of traditional type 2 photoinitiators (camphorquinone).[20] This suggestion is also supported by the different wavelengths of 3D printer and postpolymerization units.

Although the use of VG resulted in similar or better results for the properties tested in the present study, with the advantage of not requiring additional equipment and reducing time (2 minutes compared with 7 minutes from the PC×1), caution is required when assessing these results: not all polymerization units provide enough energy density, beam distribution, or polymerization spectrum. Therefore, the results observed for VG may not be observed for other light polymerization units. Also, only 1 resin was tested in this study, and results may change due to differences in formulation.

It is also noteworthy that the washing solution and postpolymerization parameters are dependent on each

resin, as the composition of 3D printed resins are not standardized. These findings emphasize the impact of solvent selection and polymerization time on the characteristics of the 3D printed restorative polymers, suggesting potential optimization strategies for improved surface quality and mechanical behavior.[5]

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Resins for 3D printing postpolymerized using VALO Grand for 120 seconds produced similar or better microhardness, flexural strength, and color stability results compared with the Procure 2 polymerization chamber while significantly decreasing the postpolymerization time.
2. Specimens polymerized with 2 cycles on ProCure 2 showed the lowest microhardness values. The excessive increase in polymerization time (or polymerization cycles) may jeopardize the properties of 3D printed parts.

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Corresponding author:
Dr Fabio A.P. Rizzante
Medical University of South Carolina (MUSC)
James B. Edwards College of Dental Medicine
173 Ashley Avenue, BSB 335C
Charleston, SC 29425
Email: rizzante@musc.edu

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