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RESEARCH AND EDUCATION

Influence of postpolymerization methods and artificial aging procedures on the fracture resistance and flexural strength of a vat-polymerized interim dental material

Michael D. Scherer, DMD, MS,[a] Basir A. Barmak, MD, MSc, EdD,[b] Mutlu Özcan, DDS, DMD, PhD,[c] and Marta Revilla-León, DDS, MSD[d]

ABSTRACT

Statement of problem. The influence of postpolymerization methods and artificial aging procedures on the fracture resistance and flexural strength of additively manufactured interim polymers remains unclear.

Purpose. The purpose of this in vitro study was to evaluate the effect of the conditions (dry and water- and glycerin-submerged) and time (25, 30, 35, 40, and 45 minutes) of postpolymerization methods with and without artificial aging procedures on the fracture resistance and flexural strength of an additively manufactured interim material.

Material and methods. Bar specimens (25×2×2 mm) were manufactured from an interim resin (NexDent C&B MFH N1) with a 3-dimensional printer (NexDent 5100) as per the manufacturer's recommendations. Three groups were created based on the postpolymerization condition: dry (D group) and submerged in a container with water (W group) or glycerin (G group) inside the ultraviolet polymerization machine (LC-3DPrint Box). Each group was divided into 5 subgroups (D1 to D5, W1 to W5, and G1 to G5) depending on the polymerizing time (25, 30, 35, 40, and 45 minutes) (n=20). Each subgroup was divided into nonaged and aged subgroups. The aged groups were treated in a mastication simulator. Fracture strength was measured on a universal testing machine. The flexural strength was calculated as per International Organization for Standardization (ISO) 10477-2018. The Kolmogorov-Smirnov test demonstrated that data were normally distributed. The 3-way ANOVA test was used to analyze the data (α=.05).

Results. A significant main effect was found on the fracture strength analysis for each of the 3 factors: postpolymerization condition ($F[2, 449]=81.00$, $P<.001$), treatment duration ($F[4, 449]=2.84$, $P=.024$), and aging procedure ($F[1, 449]=7.62$, $P=.006$). The only significant 2-way interaction was between postpolymerization condition and treatment duration ($F[8, 449]=3.12$, $P=.002$). Furthermore, a significant main effect was found on the flexural strength for each of the 3 factors including postpolymerization condition ($F[2, 449]=82.55$, $P<.001$), treatment duration ($F[4, 449]=2.85$, $P=.024$), and artificial aging procedure ($F[1, 449]=6.72$, $P=.010$). The only significant 2-way interaction was between postpolymerization condition and treatment duration ($F[8, 449]=3.33$, $P=.001$). Dry postconditions at 25 minutes and nonaged procedures obtained the significantly highest fracture resistance and flexural strength values.

Conclusions. Postpolymerization conditions and duration time affected the fracture resistance and flexural strength of the additively manufactured interim material assessed. Artificial aging procedures significantly decreased the fracture resistance and flexural strength of the additively manufactured interim dental material. (J Prosthet Dent 2021;■:■-■)

Vat-polymerization additive manufacturing (AM) technologies provide a relatively new fabricating method for interim restorations[1,2]; however, the dental literature that evaluated the biocompatibility,[3] mechanical properties,[4-14] and clinical behavior of AM interim restorations is scarce.[15]

The vat-polymerization additive manufacturing workflow comprises 3 important steps to generate a physical object directly from a virtual design.[1,16,17] Data processing, the first step, includes the design and specifications required to manufacture the virtual design of a restoration. Typical data processing steps include the printing and support parameters and the slicing of the standard tessellation language (STL) file, which is performed by using a computer-aided manufacturing (CAM) software program.[18] Manufacturing, the second step, is defined as the additively manufacturing of the interim

[a]Assistant Clinical Professor, Department of Restorative Dentistry, School of Dentistry, Loma Linda University, Loma Linda, Calif; and Private practice, Sonora, Calif.
[b]Assistant Professor, Department of Clinical Research and Biostatistics, Eastman Institute of Oral Health, University of Rochester Medical Center, Rochester, NY.
[c]Professor and Head, Division of Dental Biomaterials, Clinic of Reconstructive Dentistry, Center of Dental Medicine, University of Zürich, Zürich, Switzerland.
[d]Assistant Professor and Assistant Program Director AEGD Residency, Comprehensive Dentistry Department, College of Dentistry, Texas A&M University, Dallas, Texas; and Affiliate Faculty Graduate Prosthodontics, Restorative Dentistry Department, School of Dentistry, University of Washington, Seattle, Wash; and Researcher at Revilla Research Center, Madrid, Spain.

Clinical Implications

Twenty-five minutes of dry polymerization after processing is recommended to maximize the fracture resistance and flexural strength of the interim dental polymer tested.

restoration with a 3-dimensional (3D) printer.[1,2,17] Finally, postprocessing procedures involve the processes that need to be performed on the AM object, such as removing the object from the build platform, cleaning any unpolymerized liquid resin remaining on the object, implementing appropriate polymerizing methods, and removing the support structures from the object.[1,2,17]

Dental studies have reported the influence of the manufacturing methods on the mechanical properties of AM interim dental restorations by varying the manufacturing printing and supportive parameters such as the layer thickness,[11] print orientation,[5-8,12] build platform position,[6,7,10] and postprocessing polymerization protocols.[7,11,12] Previous studies have described a negative effect of artificial aging procedures on the color stability,[13] marginal and internal discrepancies,[15] fracture resistance, and flexural strength of AM interim restorations.[12,13]

Vat-polymerization technologies use photosensitive polymers that are polymerized layer-by-layer using different light sources.[1,2,18] The polymerization reaction is inhibited by oxygen diffusing from the ambient atmosphere into resin during the light polymerization procedure.[19-23] Water- or glycerin-submerged polymerization conditions provide reduced oxygen conditions compared with air.[21,23] However, the influence of varying postpolymerizing processing techniques in AM on the fracture resistance and flexural strength, with and without artificial aging procedures, of the AM interim restorations remains uncertain.

The purpose of this in vitro investigation was to evaluate the effect of the postpolymerization conditions (dry, water-submerged, and glycerin-submerged) and postpolymerization times (25, 30, 35, 40, and 45 minutes) on the fracture resistance and flexural strength, with and without artificial aging procedures, of a vat-polymerized AM interim dental material. The null hypothesis was that no significant difference would be found on the fracture resistance and flexural strength across the different polymerization conditions and times tested, with and without artificial aging procedures.

MATERIAL AND METHODS

A bar design (25×2×2 mm) was generated by using a computer-aided design (CAD) software program (Blender, version 2.77a; The Blender Foundation). The bar dimensions followed the International Organization for Standardization (ISO) 10477-2018.[24] The STL file was exported and used to manufacture all the specimens with an interim dental material (NextDent C&B MFH N1; 3D systems) and a 3D printer (Nextdent 5100; 3D Systems) at a constant room temperature of 23 °C and as per the resin mixing and printing parameters recommended by the manufacturer. The 3D printer was calibrated by following the manufacturer's recommendations. The manufacturer of the printer reports a resolution of 1920×1080 pixels.

Three groups were created based on the conditions of the postprocessing procedures performed, namely polymerization procedures in dry conditions (D group) or submerged in a container with room-temperature (23 °C) distilled water (W group) or glycerin (G group) inside the chamber of the ultraviolet (UV)-polymerization machine (LC-3DPrint Box; 3D Systems). Each group was further divided into 5 subgroups (D1 to D5, W1 to W5, and G1 to G5) depending on the postpolymerizing time used (Table 1). To standardize the manufacturing procedures, all the specimens of the same subgroup were manufactured from the same bottle of resin at the same time and in the same position on the build platform of the printer. The bar was oriented so that the layer was perpendicular to the load to be applied in the fracture resistance test (Fig. 1). All the postprocessing procedures were performed by a prosthodontist (M.S.) wearing nitrile gloves who had more than 10 years of experience in handling 3D polymer printers.

To fabricate the D1 subgroup specimens, the manufacturing workflow of the manufacturer was

Table 1. Additively manufactured interim dental material grouped as per different postpolymerization conditions and protocols

Group	Material Selected	Rinsing Procedures	UV-Polymerization Procedures (LC-3D Print Box; 3D Systems)
D1	AM interim dental material (NexDent C&B MFH N1; 3D Systems)	Ultrasonic bath of 91% isopropyl alcohol (IPA) (Isopropyl alcohol 91%; Cumberland Swan) for 3 min followed by a second clean 91% IPA bath of 2 min	25 min in dry conditions
D2 (control)			30 min in dry conditions
D3			35 min in dry conditions
D4			40 min in dry conditions
D5			45 min in dry conditions
W1			25 min water-submerged
W2			30 min water-submerged
W3			35 min water-submerged
W4			40 min water-submerged
W5			45 min water-submerged
G1			25 min glycerin-submerged
G2			30 min glycerin-submerged
G3			35 min glycerin-submerged
G4			40 min glycerin-submerged
G5			45 min glycerin-submerged

AM, additively manufactured; D, dry postpolymerization condition; G, glycerin-submerged condition; W, water-submerged condition.

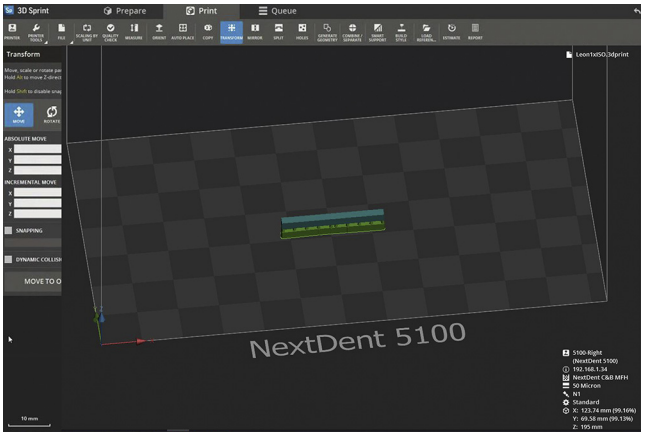

Figure 1. Print orientation of bar specimens.

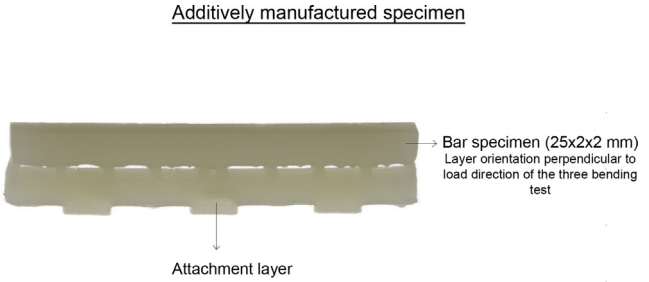

Figure 2. Additively manufactured specimen fabricated with layer orientation perpendicular to load direction of 3-point bend test.

followed. After printing, the specimens were removed from the build platform with a removal tool provided by the manufacturer. Afterward, specimens were fully submerged in an ultrasonic bath (TriClean Ultrasonic Cleaner U-10LHREC; BrandMax) with 91% isopropyl alcohol (Isopropyl alcohol 91%; Cumberland Swan) for 3 minutes and subsequently in a second ultrasonic bath with clean 91% isopropyl alcohol for 2 minutes. Specimens were placed on a paper towel and dried in ambient air. Specimens were then placed in the UV-polymerization machine (LC-3DPrint Box; 3D Systems) with full spectrum (300-550 nm) UV-light exposure for 30 minutes as per the recommendations provided by the manufacturer.

In the D2, D3, D4, and D5 subgroups, the same manufacturing procedures as for the D1 subgroup were followed, except for polymerization time, which varied among the groups. The polymerization time was 25 minutes for the D2 group, 35 minutes for the D3 subgroup, 40 minutes for the D4 subgroup, and 45 minutes for the D5 subgroup. In the W1, W2, W3, W4, and W5 groups, the same manufacturing procedures as for the D1, D2, D3, D4, and D5 groups, respectively, were followed except for the postpolymerization conditions in which the specimens were submerged in a glass container with room-temperature distilled water (23 °C) and placed inside the chamber of the same UV-polymerization unit. In the G1, G2, G3, G4, and G5 groups, the same manufacturing procedures as for the D1, D2, D3, D4, and D5 groups, respectively, were followed except for the postpolymerization conditions in which the specimens were submerged in a glass container with room-temperature (23 °C) glycerin (Glycerin 10311601, USP; Humco) and placed inside the chamber of the same UV-polymerization unit.

Twenty specimens per group were manufactured and stored in a black container until measurements were completed. Supports were removed from all specimens (Fig. 2). Scanning electron microscope (SEM) images of the superior aspect of the bar were obtained at 10kV, ×1000 magnification (Zeiss Supra V50; Carl Zeiss) from the D, G, and W subgroup specimens.

Each subgroup was randomly divided by using a shuffled deck of cards into 2 groups as per the accelerating artificial aging procedures (mastication simulation): nonaged and aged subgroups. Nonaged specimens were stored at room temperature (23 °C). For 1 of the hydrolytically aged (×8000 cycles between 5 °C and 55 °C) subgroups, 1 million cycles (distilled water, Ø6-mm stainless steel indenter) were applied in a mastication simulator (CS-4.8 Chewing Simulator; Mechatronik) with a loading force of 80 N on the occlusal plane for 12×10^6 cycles at 1.7 Hz, with a lateral movement of 1 mm, a vertical movement of 0.5 mm, and a downward and lateral speed of 60 mm/s. Four specimens were cycled at the same time, 1 in each individual chamber. At the end of the artificial aging procedures, the specimens were evaluated at a time interval no longer than 48 hours.

Three-point fracture strengths of the specimens were measured as per ISO 10477-2018[24] on a universal testing machine (Instron Model 8501; Instron Corp). Loading was applied at a crosshead speed of 1 mm/min until the specimen fractured. Load at failure was recorded, and flexural strength was calculated as per the specimen's dimensions. The flexural strength (σ in MPa) for a bar specimen under a load in a 3-point bend test was calculated by using the following formula: $\sigma = \frac{3\times Fmax\times L}{2\times b\times d^2}$, where Fmax is the fracture strength, L is the length, b is the with, and d is the thickness of the specimens.[24]

The Kolmogorov-Smirnov test demonstrated that the data were normally distributed. Therefore, 3-way ANOVA was conducted on a sample of 450 interim dental materials to examine the main effects and interaction effects of the postpolymerization condition, treatment duration, and artificial aging procedure on the fracture resistance and flexural strength of vat-polymerized interim dental material (α=.05). All statistical testing was performed by using a statistical software program (IBM SPSS Statistics, v26.0; IBM Corp).

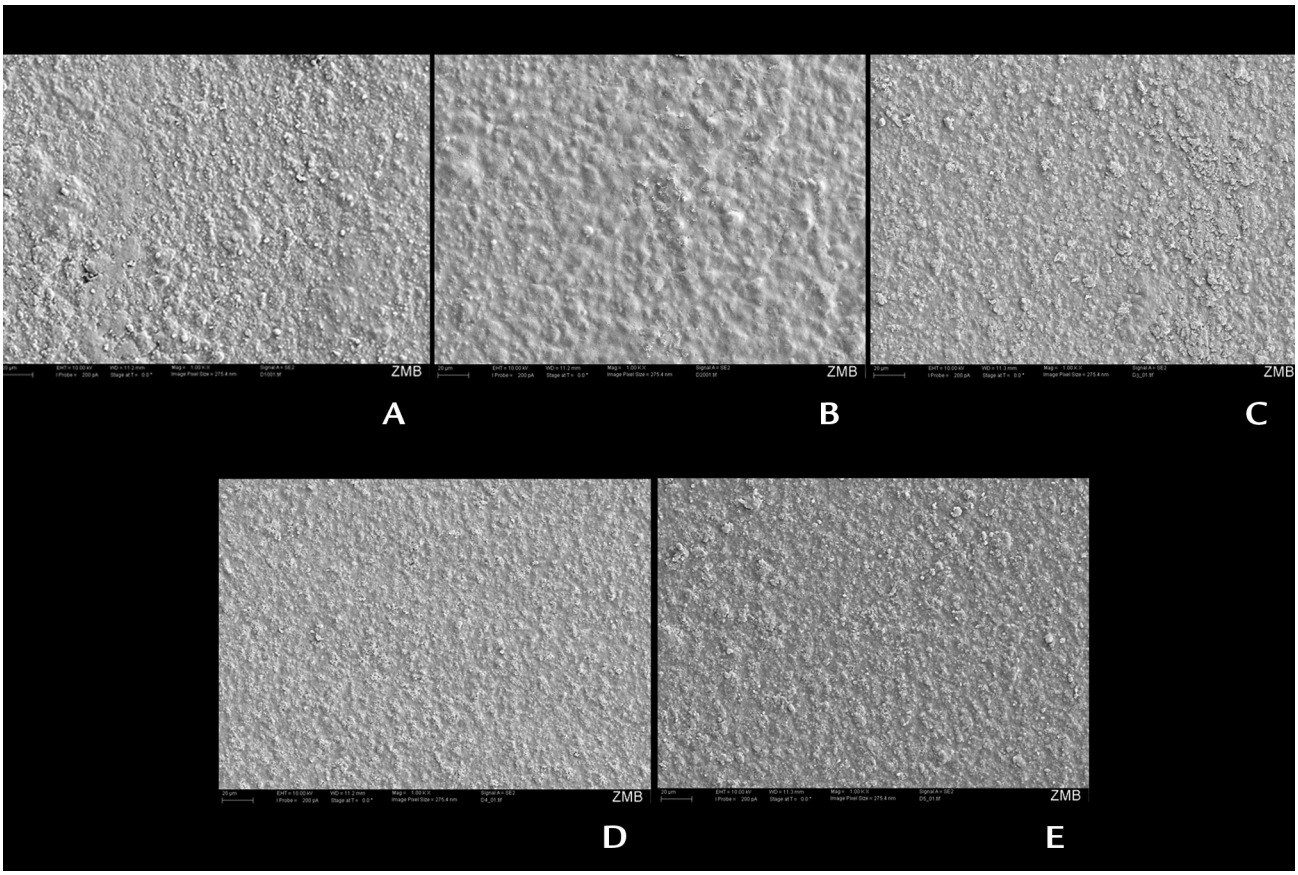

Figure 3. Scanning electron microscope images obtained at ×1000 magnification. A, D1 subgroup (25-minute polymerization time). B, D2 subgroup (30-minute polymerization time). C, D3 subgroup (35-minute polymerization time). D, D4 subgroup (40-minute polymerization time). E, D5 subgroup (45-minute polymerization time). D, Dry postpolymerization condition.

RESULTS

Except for the G5 subgroup, SEM images revealed similar surface characteristics among the different groups tested, namely the D (Fig. 3), W (Fig. 4), and G subgroups (Fig. 5). The surface irregularity might be a consequence of the additively manufacturing process. The G5 subgroup, however, presented different surface characteristics, and disorganized tangled fibers can be observed. The diameter of the fibers was less than 1 μm (Fig. 6).

Regarding fracture resistance (Table 2), a significant main effect was found for each of the 3 factors: postpolymerization condition ($F[2, 449]=81.00$, $P<.001$), treatment duration ($F[4, 449]=2.84$, $P=.024$), and artificial aging procedure ($F[1, 449]=7.62$, $P=.006$). No significant 3-way interaction was detected. The only significant 2-way interaction was shown between the postpolymerization condition and treatment duration ($F[8, 449]=3.12$, $P=.002$). More specifically, in comparing the 3 postpolymerization conditions with the Tukey post hoc test, a significant difference in fracture resistance was detected between dry and water polymerizations in favor of the dry condition (mean difference=12.85, SE=1.08, and $P<.001$); between dry and glycerin polymerizations in favor of the dry condition (mean difference=10.46, SE=1.08, and $P<.001$); but not between water and glycerin polymerizations ($P=.071$). In addition, in comparing the 5 treatment durations with the Tukey post hoc test, a significant difference in fracture resistance was detected between the 25- and 30-minute treatment durations in favor of the 25-minute treatment duration (mean difference=4.5, SE=1.39, and $P<.012$). None of the other pairwise comparisons detected a significant difference. Furthermore, in comparing the aged and nonaged conditions, the significant difference in fracture resistance was in favor of the nonaged group (mean difference=2.41, SE=0.881, and $P<.001$) (Fig. 7A).

Regarding flexural strength (Table 2), a significant main effect was found for each of the 3 factors: the postpolymerization condition ($F[2, 449]=82.55$, $P<.001$), treatment duration ($F[4, 449]=2.85$, $P=.024$), and artificial aging procedure ($F[1, 449]=6.72$, $P=.010$). No significant 3-way interaction was detected. The only significant 2-way interaction was between the postpolymerization condition and treatment duration ($F[8, 449]=3.33$, $P=.001$). More specifically, in comparing the 3 postpolymerization conditions with the Tukey post hoc test, a

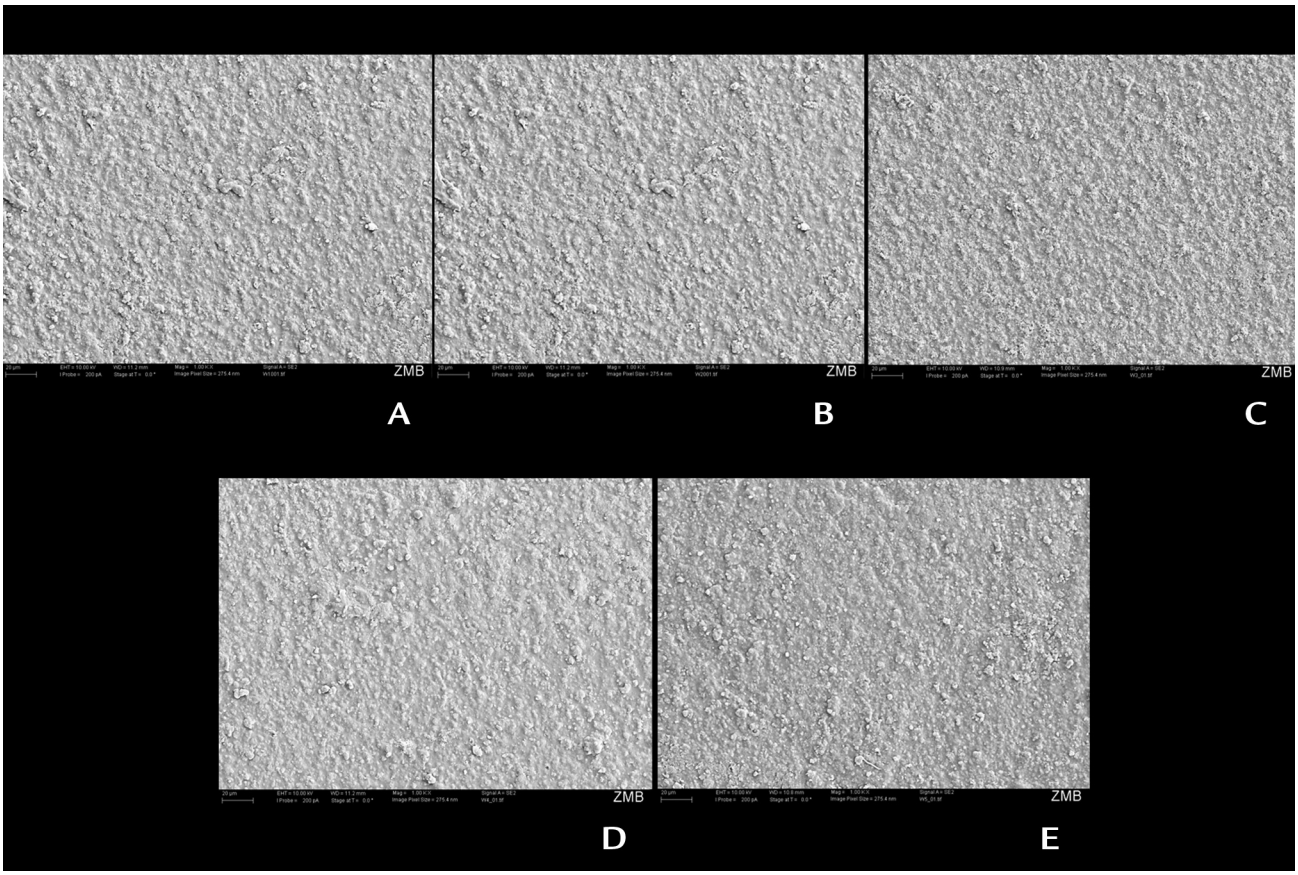

Figure 4. Scanning electron microscope images obtained at ×1000 magnification. A, W1 subgroup (25-minute polymerization time). B, W2 subgroup (30-minute polymerization time). C, W3 subgroup (35-minute polymerization time). D, W4 subgroup (40-minute polymerization time). E, W5 subgroup (45-minute polymerization time). W, Water-submerged postpolymerization condition.

significant difference in flexural strength was detected between the dry and water polymerizations in favor of the dry condition (mean difference=59.4, SE=4.98, and $P<.001$); between the dry and glycerin polymerizations in favor of the dry preparation (mean difference=48.6, SE=4.98, and $P<.001$); but not between the water and glycerin polymerizations ($P=.079$). In addition, in comparing the 5 treatment durations with the Tukey post hoc test, a significant difference in flexural strength was detected between the 25- and 30-minute treatment durations in favor of the 25-minute treatment duration (mean difference=20.88, SE=6.43, and $P=.011$). None of the other pairwise comparisons detected a significant difference. Furthermore, in comparing the aged and nonaged procedure, the significant difference in fracture resistance was in favor of the nonaged group (mean difference=10.4, SE=4.06, and $P=.011$) (Fig. 7B).

DISCUSSION

The postpolymerization conditions, time duration, and artificial aging procedures had a significant effect on the fracture resistance and flexural strength of the AM interim material evaluated. The dry postpolymerization condition for 25 minutes without aging procedures obtained the highest fracture resistance and flexural strength values. Therefore, the null hypothesis was rejected.

To standardize the manufacturing of the specimens and minimize the variables that could have influenced the results obtained, the printing and supportive parameters were maintained constant among the groups, and all specimens were manufactured from the same bottle of resin. Each subgroup was manufactured at a time with the same printing parameters, build platform position, and print orientation.[7,11,12] Furthermore, the bar specimen used follows the proposed International Organization for Standardization standard dimension,[24] and the layer orientation of the bar specimens was positioned perpendicular to the load direction of the 3-bend test.[5] Finally, the printer and UV-polymerization machine used were those recommended by the manufacturer of the interim material tested; also, the D2 subgroup represented the protocol recommended by the manufacturer.

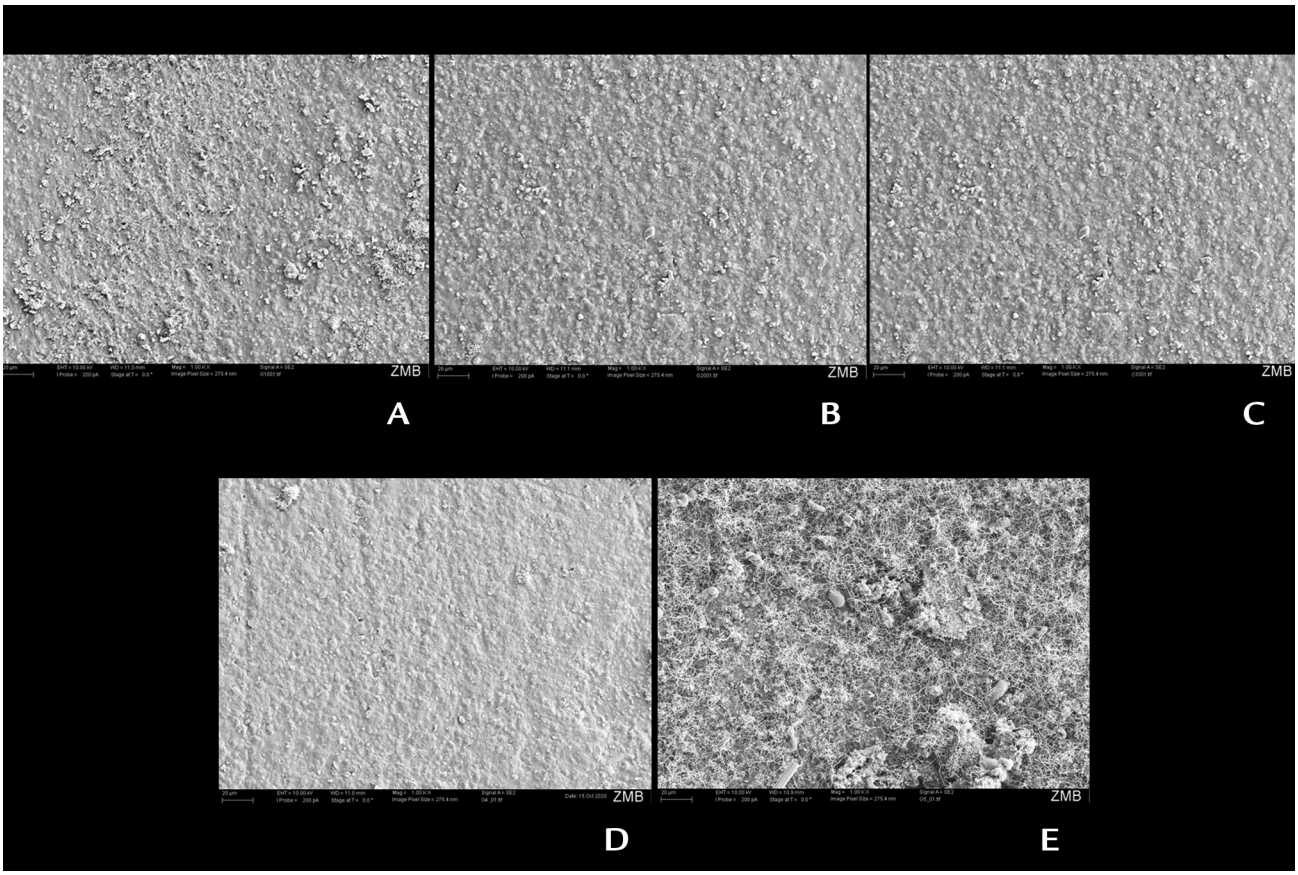

Figure 5. Scanning electron microscope images obtained at ×1000 magnification. A, G1 subgroup (25-minute polymerization time). B, G2 subgroup (30-minute polymerization time). C, G3 subgroup (35-minute polymerization time). D, G4 subgroup (40-minute polymerization time). E, G5 subgroup (45-minute polymerization time). G, Glycerin-submerged postpolymerization condition.

The results of the present study indicated that the dry postpolymerization condition obtained the highest fracture resistance and flexural strength mean values compared with the water- and glycerin-submerged postpolymerization conditions. Specimens treated with water-submerged postpolymerization condition obtained significantly higher fracture resistance and flexural strength mean values compared with the glycerin-submerged postpolymerization conditions. The interim dental material might absorb water if submerged in water or glycerin, which may explain the lower mechanical property measurements obtained in the W and G groups. Although the dry condition obtained the highest mean values for the mechanical properties tested, further studies are needed to assess other properties such as the degree of polymerization conversion, water absorption, color stability, or manufacturing accuracy.

In the present investigation, the postpolymerization times did demonstrate significant effects on the mechanical properties tested. The manufacturer of the interim material evaluated recommends dry postpolymerization conditions for 30 minutes (D2 subgroup); however, the same polymerization condition with a reduced polymerization time of 25 minutes obtained significantly higher fracture resistance and flexural strength mean values. Therefore, decreasing the postpolymerization time beyond the manufacturer-

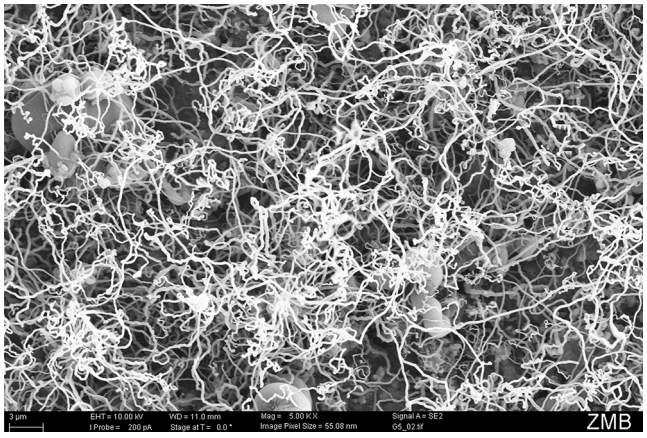

Figure 6. Scanning electron microscope images obtained at ×500 magnification of G5 subgroup (45-minute polymerization time) specimen. G, Glycerin-submerged postpolymerization condition.

Table 2. Fracture resistance and flexural strength values computed among different subgroups tested

Postpolymerization Condition	Treatment Duration (min)	Artificial Aging	Fracture Resistance (N) (Mean ±Standard Deviation)	Flexural Strength (MPa) (Mean ±Standard Deviation)
Dry	25	No	61.20 ±12.14	286.86 ±56.93
		Yes	59.97 ±9.79	281.10 ±45.89
	30	No	58.63 ±3.34	274.85 ±15.64
		Yes	57.14 ±7.33	267.84 ±34.34
	35	No	56.36 ±8.73	264.18 ±40.92
		Yes	55.73 ±10.22	261.22 ±47.92
	40	No	61.63 ±12.76	288.91 ±59.80
		Yes	60.19 ±11.33	282.14 ±53.12
	45	No	64.81 ±8.01	303.81 ±37.54
		Yes	61.26 ±10.08	287.18 ±47.23
Glycerin	25	No	54.51 ±14.84	255.52 ±69.54
		Yes	50.48 ±7.64	238.31 ±35.78
	30	No	47.34 ±10.81	221.89 ±50.68
		Yes	49.25 ±7.00	232.00 ±32.60
	35	No	49.87 ±6.33	233.78 ±29.67
		Yes	49.01 ±7.06	229.94 ±33.29
	40	No	52.57 ±7.54	246.41 ±35.34
		Yes	47.86 ±8.22	226.38 ±40.40
	45	No	48.39 ±7.34	226.82 ±34.41
		Yes	43.02 ±6.48	201.03 ±29.63
Water	25	No	49.04 ±8.45	229.90 ±39.62
		Yes	47.81 ±6.97	227.15 ±37.57
	30	No	43.97 ±9.46	206.09 ±44.34
		Yes	39.71 ±9.78	190.86 ±35.34
	35	No	52.29 ±13.86	245.12 ±64.97
		Yes	48.18 ±9.10	226.78 ±28.71
	40	No	45.50 ±10.30	213.27 ±48.27
		Yes	43.95 ±6.52	202.93 ±29.02
	45	No	50.78 ±11.97	238.04 ±56.11
		Yes	47.22 ±6.82	224.25 ±26.94

recommended protocol resulted in better mechanical properties compared with those of the established recommended protocols.

The SEM images revealed changes in surface morphology for the glycerin-submerged polymerization conditions, notably the G5 subgroup, which exhibited the most distinct surface changes. Specimens in the G5 group exhibited disorganized tangled fiber surface characteristics, possibly from the plasticized surface or disrupted chain configuration of the polymer; however, studies are needed to better understand this finding.

Artificial aging procedures significantly impacted the mechanical properties tested in all groups, a finding consistent with those of previous studies.[12] In the present study, the artificial aging produced a mean reduction in the fracture resistance mean values of 2.66% in the D group, of 6.58% in the W group, and of 8.58% in the G group. Similarly, the artificial aging procedures produced a mean reduction in the flexural strength mean values of 2.66% in the D group, of 7.00% in the W group, and of 5.28% in the G group. The clinical impact of this effect should be further analyzed.

Previous studies have analyzed the effect of the polymerizing postprocessing procedures and artificial aging methods on the mechanical properties of AM interim dental materials.[7,12,13] However, none have evaluated the effect of different postpolymerization conditions and times on the fracture resistance and flexural strength of AM interim dental materials with and without artificial aging procedures. Therefore, direct comparisons with previous studies are not possible.

Unkovskiy et al[7] evaluated the flexural strength of dental resin for processing surgical guides polymerized with 3 different UV-polymerization machines. Although the authors reported no significant difference among the flexural strength mean values, the results were not directly comparable as the material used was not marketed for interim dental restorations and a different vat-polymerization technology and printer were used.

Reymus et al[12] compared the fracture resistance between conventional, milled, and AM interim 3-unit fixed dental prostheses. The authors analyzed the impact of postpolymerization procedures by using 3 UV-polymerization machines and artificial aging methods on the AM specimens. Fracture resistance values varied significantly among the specimens postprocessed in different UV-polymerization machines. Furthermore, most of the groups obtained significantly lower fracture resistance mean values after artificial aging procedures. Scotti et al[13] evaluated the flexural strength of conventional and AM interim dental materials, reporting that the AM interim material tested obtained flexural strength similar to that of a bis-acryl interim dental material. However, comparisons with the results of the present study are difficult because of the diverse specimen dimensions and the manufacturing technology and printer selected.

Limitations of the present study include the limited interim dental materials tested, the single 3D printer and UV-polymerization machine tested, the limited postprocessing procedures evaluated, and the restricted mechanical properties analyzed. Further studies are needed to further analyze the mechanical properties, chemical composition, degree of conversion, biocompatibility, and clinical behavior of the AM interim dental materials before their routine clinical use can be recommended.

CONCLUSIONS

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

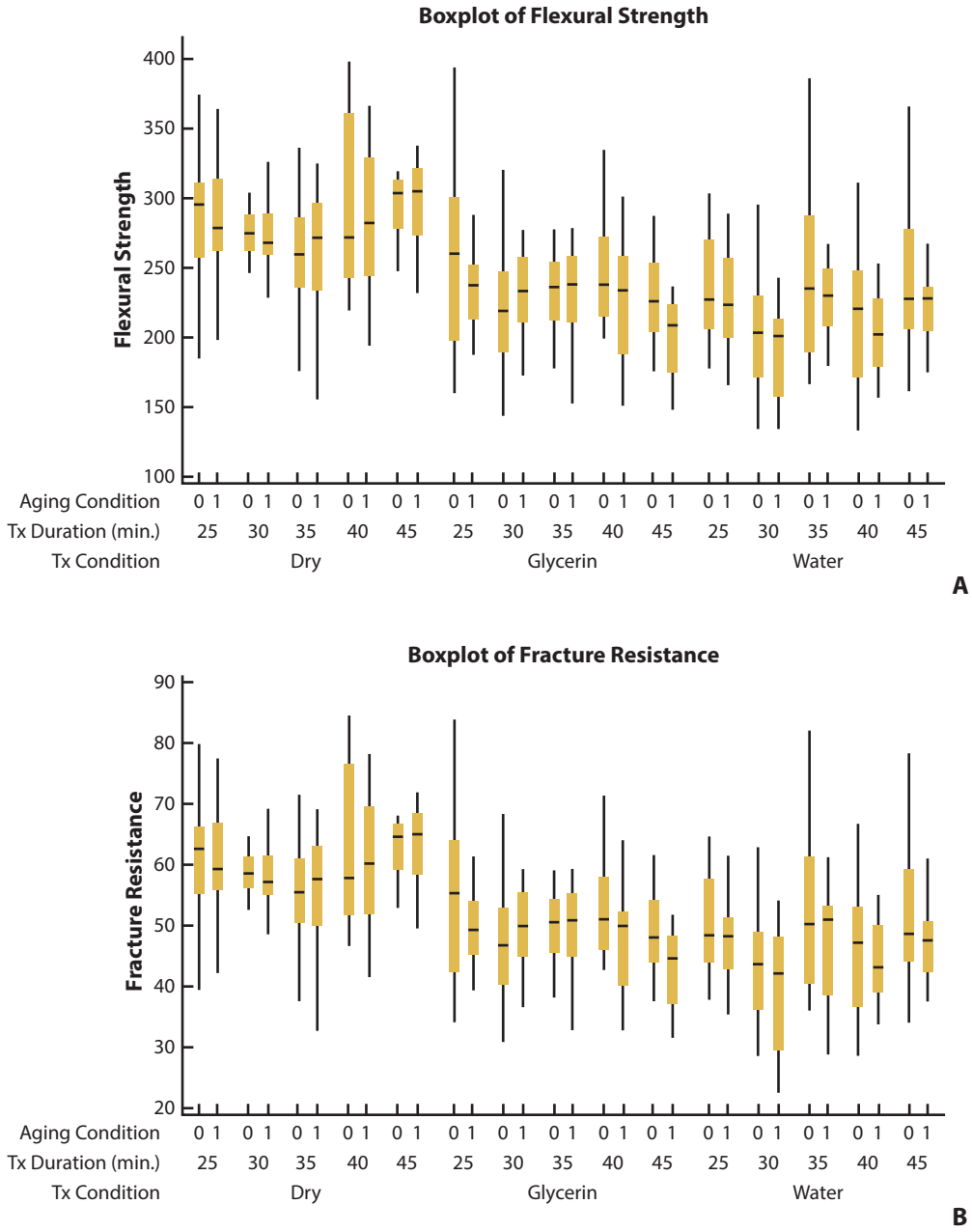

Figure 7. Box plots of different postpolymerization conditions (dry and submerged in container with water or glycerin) with and without artificial aging procedures. A, Fracture resistance values. B, Flexural strength values.

1. The postpolymerization conditions tested significantly affected the fracture resistance and flexural strength of the AM interim dental material evaluated. Dry postpolymerization conditions obtained the highest fracture resistance and flexural strength mean values compared with the water- and glycerin-submerged conditions.
2. The postpolymerization time had a significant effect on the fracture resistance and flexural strength of the AM interim dental polymer tested. The 25-minute postpolymerization time obtained the highest fracture resistance and mean flexural strength values.
3. Artificial aging procedures significantly decreased the fracture resistance and flexural strength of the AM interim dental polymer tested in all groups.

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Corresponding author:
Dr Marta Revilla-León
3302 Gaston Ave, Room 713
Dallas, TX 75246
Email: revillaleon@tamu.edu

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