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RESEARCH AND EDUCATION

Influence of postprocessing rinsing solutions and duration on flexural strength of aged and nonaged additively manufactured interim dental material

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ABSTRACT

Statement of problem. Additive manufacturing procedures for fabricating interim restorations include rinsing postprocessing procedures. However, the impact of different rinsing solutions and times on flexural strength is unknown.

Purpose. The purpose of this in vitro study was to assess the influence of the rinsing solutions and duration, as well as accelerated aging (thermocycling) procedures, on the flexural strength and Weibull characteristics of an additively manufactured interim dental material.

Material and methods. A bar design (25×2×2 mm) file was used to fabricate all the specimens with 3D printing and an interim material (Nextdent C&B MFH). Five groups were created based on the rinsing solution used during the postprocessing procedures: 91% isopropyl alcohol (IPA) (control or IPA-91), 99% IPA (IPA-99 group), bio-ethyl alcohol 100% (BE group), tripropylene glycol monomethyl ether (TPM) 100% (TPM group), and water miscible formula (Resinaway) (RA group). Each group was divided into 4 subgroups depending on the total rinsing time: 5, 6, 7, and 8 minutes (5, 6, 7, and 8 subgroups). Additionally, each subgroup was distributed between nonaged and aged thermocycling procedures (n=10). Flexural strength measurements were made by using a universal testing machine. Two-parameter Weibull distribution values, including the Weibull modulus, scale (m), and shape (0), were calculated. Three-way ANOVA and pairwise multiple comparison Tukey tests were used to analyze the data (α=.05).

Results. Three-way ANOVA showed that the rinsing solution (P<.001), rinsing time (P=.004), and thermocycling procedures (P<.001) were significant predictors of the flexural strength values obtained. The IPA-91 and IPA-99 groups obtained the highest flexural strength, while the RA, TPM, and BE groups obtained the lowest flexural strength. The 7- and 8-minute subgroups obtained the highest flexural strength, while the 5-minute subgroup obtained the lowest flexural strength. The nonaged specimens obtained significantly higher mean flexural strength values than the aged specimens.

Conclusions. The vat-polymerized additively manufactured interim dental material tested with differing rinsing solutions and times demonstrated significant differences in the flexural strength values measured. Accelerated artificial aging procedures significantly decreased the flexural strength of the vat-polymerized interim dental material tested. (J Prosthet Dent 2022;■:■-■)

Vat-polymerization additive manufacturing (AM) technologies, including digital light processing (DLP) methods, can be selected to fabricate interim restorations.[1,2] The mechanical properties of AM interim materials have been analyzed[3-17] in terms of manufacturing accuracy,[9,14] chemical composition,[7]

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Clinical Implications

When using the material and printer tested, the 91% and 99% IPA postprocessing rinsing solutions with a total rinsing time of 7 or 8 minutes are recommended to maximize the flexural strength properties.

color,[3,10] surface roughness,[7,9,12] marginal and internal discrepancies,[11,14,17] mechanical properties,[3,4,6,8,9,13,16] microbial adhesion,[9] wear,[5] and influence of accelerated aging procedures.[3,4,8,17]

Based on the established data, a correlation exists between the manufacturing methods (printing and postprocessing parameters) and the properties of the AM interim dental restoration.[4,6-9,11,13,14,16,18] An important relationship exists among the technology, printer, and material selected to manufacture the AM interim dental restoration.[18] However, the optimal printing and postprocessing parameters regarding rinsing solution and duration and their effect on the mechanical properties of AM interim materials remain uncertain.[4,6-9,11,13,14,16,18]

Previous studies have reported a significant decrease in the flexural strength,[3,4,8] color stability,[3] and marginal and internal discrepancies[17] of AM interim dental restorations after artificial aging procedures. However, the flexural strength values and Weibull characteristics between aged and nonaged AM interim dental materials when varying postprocessing rinsing protocols remain unclear.

The purpose of the present study was to assess the influence of the rinsing solutions (IPA 91%, IPA 99%, bio-ethyl alcohol 100%, TPM 100%, and water miscible formula), total rinsing times (5, 6, 7, and 8 minutes), and accelerated aging (thermocycling) procedures on the flexural strength and Weibull characteristics of vat-polymerized AM interim material (Nextdent C&B MFH, N1; 3D Systems). The null hypotheses were that no significant difference would be found in the flexural strength and Weibull characteristics of specimens fabricated by using different rinsing solutions and times and that no significant difference would be found in the flexural strength and Weibull characteristics of aged and nonaged specimens.

MATERIAL AND METHODS

A bar (25×2×2 mm) designed according to the International Organization for Standardization (ISO) 10477-2018[19] was obtained by using a computer-aided design (CAD) software program (Blender, v2.77a; The Blender Foundation). The virtual design was exported in a standard tessellation language (STL) file and used to manufacture all the specimens.

Three groups were created based on the rinsing solution used to postprocess the specimens: 91% IPA (control or IPA-91), 99% IPA (IPA-99 group), bio-ethyl alcohol 100% (BE group), tripropylene glycol methyl ether (TPM) 100% (TPM group), and water miscible formula (RA group) (Table 1). The IPA-91 group was considered the control group, as this is the rinsing solution recommended by the manufacturer.

All the specimens were manufactured with a DLP printer (Nextdent 5100; 3D Systems) and an interim resin (Nextdent C&B MFH Shade N1; 3D Systems) at a constant room temperature of 23 °C. A new bottle of interim resin was used. The printer was calibrated according to the manufacturer's recommendations, and the specimens of each group were manufactured at the same time. Also, the position of the specimen on the build platform, the layer thickness (50 μm), the printing orientation, and the supportive material were identical in all the groups. All specimens were oriented so that the layer was perpendicular to the load to be applied in the fracture resistance test.

For the IPA-91-5 subgroup, specimens were fully submerged in an ultrasonic bath (TriClean Ultrasonic Cleaner U-10LHREC; BrandMax) with 91% IPA (isopropyl alcohol 91%; Cumberland Swan) for 3 minutes and subsequently in a second ultrasonic bath with a clean 91% IPA alcohol for 2 minutes. For the IPA-91-6, the same protocol as for the IPA-91-5 subgroup was followed, except for the rinsing time. Both the first and second rinsing baths were extended to 3 minutes. For the IPA-91-7 subgroup, the same protocol as for the IPA-91-5 subgroup was followed, except for the rinsing time. The time in the first rinsing bath was extended to 4 minutes, and the second to 3 minutes. For the IPA-91-8 subgroup, the same protocol as for the IPA-91-5 subgroup was followed, except for the rinsing time. Both the first and second rinsing baths lasted for 4 minutes. For the IPA-99, BE, TPM, and RA groups, the protocol was similar to that for the IPA-91 group with the same rinsing solvent and time (Table 1).

All the specimens were polymerized in the UV-polymerization machine (LC-3DPrint Box; 3D Systems) with full-spectrum (300 to 550 nm) UV-light exposure for 30 minutes according to the manufacturer's recommendations. The support material was removed from all specimens with a tool provided by the manufacturer, and the surface from which supports had been removed was marked with a black pen. The specimens were then stored in a black container until the measurements were made.

A total of 20 specimens per subgroup were randomly divided into 2 more subgroups, nonaged and aged (n=10), by using a shuffled deck of cards. In the aged

Table 1. Manufacturing procedure details of groups tested

Group	Rinsing Solution	Rinsing Time
IPA-91-5 (control)	Isopropyl alcohol 91%	3+2 min
IPA-91-6		3+3 min
IPA-91-7		4+3 min
IPA-91-8		4+4 min
IPA-99-5	Isopropyl alcohol 99%	3+2 min
IPA-99-6		3+3 min
IPA-99-7		4+3 min
IPA-99-8		4+4 min
BE-5	Bio-ethyl alcohol 100%	3+2 min
BE-6		3+3 min
BE-7		4+3 min
BE-8		4+4 min
TPM-5	TPM 100%	3+2 min
TPM-6		3+3 min
TPM-7		4+3 min
TPM-8		4+4 min
RA-5	Water miscible formula (Resinaway; Monocure 3D)	3+2 min
RA-6		3+3 min
RA-7		4+3 min
RA-8		4+4 min

BE, bio-ethyl alcohol; IPA, isopropyl alcohol; RA, Resinaway; TPM, tripropylene glycol methyl ether.

group, the specimens were subjected to thermocycling procedures, which involved 6000 cycles of 3 consecutive rounds each: 20 seconds (dwell time) at 5 °C, 5 seconds (transfer time) at ambient air temperature (23 °C), and 20 seconds (dwell time) at 55 °C.

Flexural strength measurements (MPa) were made for all the specimens according to ISO 10477:2018.[19] A universal testing machine (Universal Testing Machine; ZwickRoell) was used at a crosshead speed of 1 mm/min on a 10-mm span. The specimens were loaded to failure, and fracture load (N) data were recorded. The flexural strength (σ) was computed by using the following formula: $\sigma = \frac{3 \times Fmax \times L}{2 \times b \times d^2}$, where Fmax is the failure load (force) at the fracture point (N), L is the length of the support span (10 mm), b is the width of the specimen, and d is the thickness of the specimen.[19]

The Weibull distribution maximum likelihood estimation without a correction factor, including the Weibull modulus, scale (m), and shape (0) to interpret the predictability and reliability of the flexural strength tests, was performed with a statistical software program (Minitab Software V.16; Minitab).[20] Additionally, specimens of all groups were observed under a scanning electron microscope (Zeiss Supra V50; Carl Zeiss NTS GmbH). These images were obtained at 5 kV and ×1000 and ×250 magnifications.

The flexural strength (MPa) data were used for the statistical analysis. The Shapiro-Wilk and Kolmogorov-Smirnov tests revealed that the data were normally distributed ($P>.05$). Three-way ANOVA followed by post hoc multiple comparison Tukey tests were used. A statistical software program (IBM SPSS Statistics for Windows, v26; IBM Corp) was used to perform the statistical analysis ($\alpha=.05$).

Table 2. Flexural strength values (MPa) of groups tested

Group	Thermocycling	Flexural Strength, Mean ±Standard Deviation
IPA-91-5 (control)	Nonaged	271.3 ±16.82
	Aged	197.8 ±43.39
IPA-91-6	Nonaged	261.9 ±25.11
	Aged	216.5 ±16.14
IPA-91-7	Nonaged	280.4 ±18.85
	Aged	227.6 ±33.76
IPA-91-8	Nonaged	280.5 ±16.67
	Aged	236.2 ±35.35
IPA-99-5	Nonaged	279.3 ±23.3
	Aged	199.4 ±33.57
IPA-99-6	Nonaged	270.7 ±18.42
	Aged	217.6 ±24.96
IPA-99-7	Nonaged	273.1 ±39.08
	Aged	218.8 ±24.29
IPA-99-8	Nonaged	276.0 ±15.31
	Aged	228.2 ±26.94
BE-5	Nonaged	239.8 ±30.46
	Aged	188.6 ±18.98
BE-6	Nonaged	238.6 ±24.72
	Aged	201.0 ±18.31
BE-7	Non-aged	254.9 ±24.89
	Aged	206.0 ±24.45
BE-8	Nonaged	232.4 ±16.16
	Aged	200.2 ±4.87
TPM-5	Nonaged	244.2 ±7.57
	Aged	190.8 ±12.31
TPM-6	Nonaged	230.7 ±9.65
	Aged	215.2 ±21.6
TPM-7	Nonaged	241.1 ±19.11
	Aged	207.3 ±19.71
TPM-8	Nonaged	239.7 ±22.33
	Aged	221.5 ±21.19
RA-5	Nonaged	244.0 ±35.77
	Aged	202.1 ±24.59
RA-6	Nonaged	252.7 ±14.01
	Aged	197.9 ±25.42
RA-7	Nonaged	256.6 ±12.58
	Aged	203.4 ±39.63
RA-8	Nonaged	257.4 ±21.72
	Aged	192.5 ±20.85

BE, bio-ethyl alcohol; IPA, isopropyl alcohol; RA, Resinaway; SD, standard deviation; TPM, tripropylene glycol methyl ether.

RESULTS

Three-way analysis of variance (ANOVA) showed that only the rinsing solution (df=4, MS=12 882, F=20.93, $P<.001$), rinsing time (df=3, MS=2736, F=4.45, $P=.004$), and thermocycling procedures (df=1, MS=230 338, F=374.28, $P<.001$) were significant predictors of the flexural strength mean values obtained (Table 2, Fig. 1A).

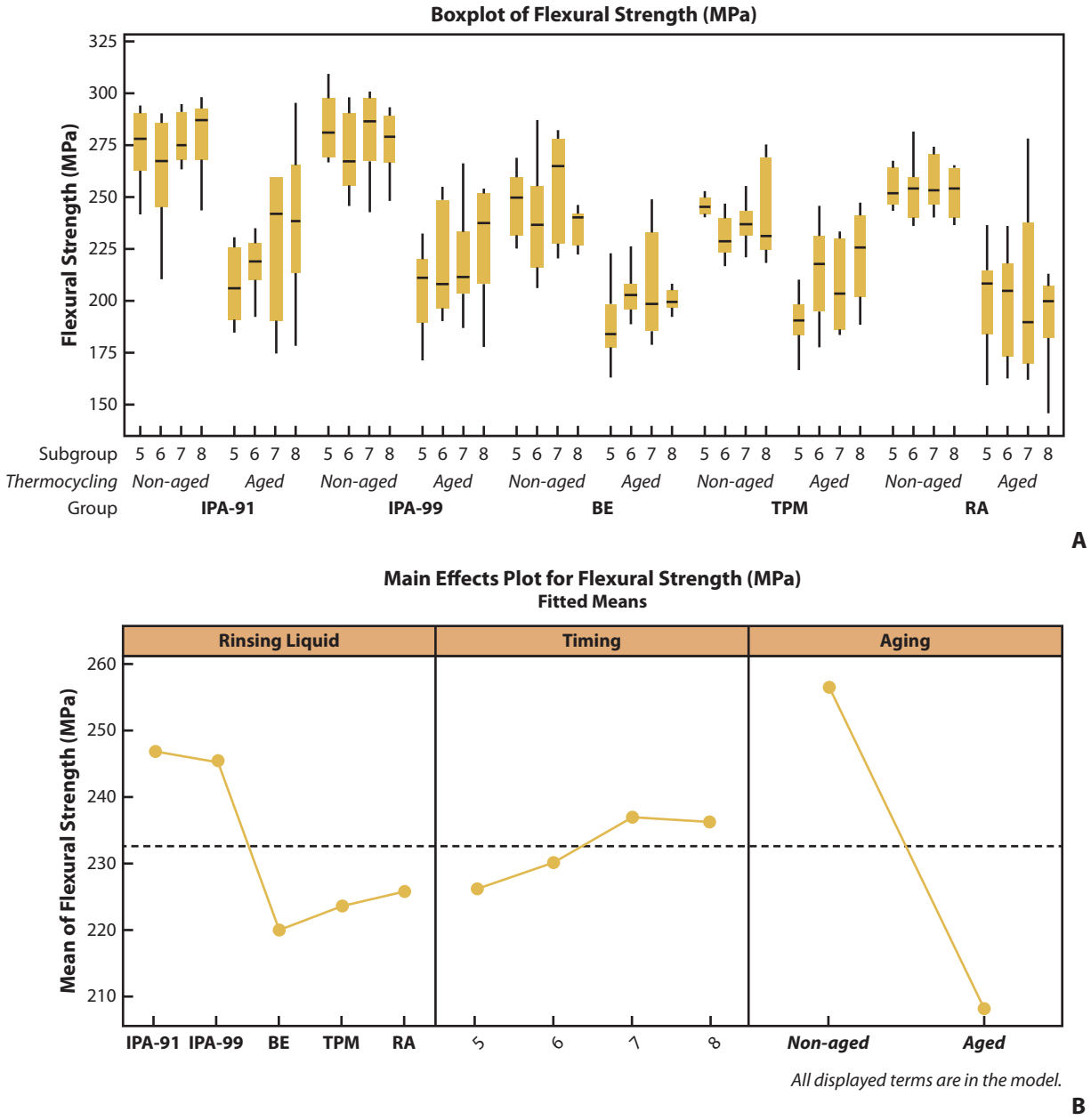
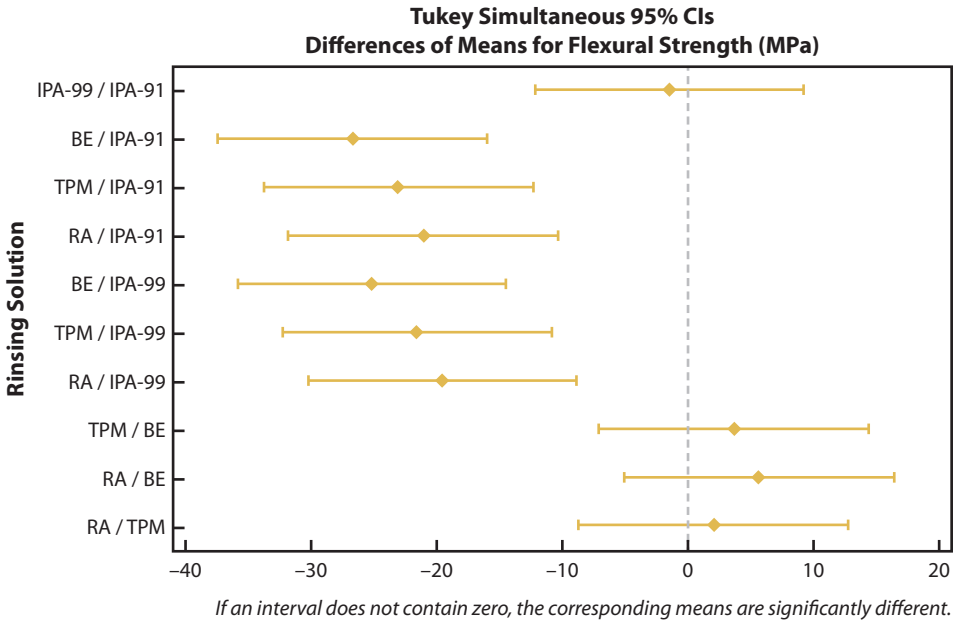


Figure 1. A, Boxplot of flexural strength values obtained. B, Main effects plot for flexural strength obtained.

Artificial aging methods explained the 43.4% of variation in the flexural strength values computed, while the rinsing solution tested explained the 9.7% of variation found in the flexural strength values.

The Tukey pairwise comparison showed significant mean value differences in flexural strength among the different rinsing solutions tested (Fig. 1B). The IPA-91 (246.88 MPa) and IPA-99 groups (245.37 MPa) obtained the highest mean flexural strength values, while the RA (225.83 MPa), TPM (223.81 MPa), and BE (220.19 MPa) groups obtained the lowest mean flexural strength values. The Tukey pairwise comparison showed significant mean value differences in flexural strength among the rinsing times (Fig. 1C). The 7-minute (236.92 MPa) and 8-minute (236.45 MPa) subgroups obtained the highest mean values for flexural strength, while the 5-minute (226.02 MPa) subgroup obtained the lowest mean values for flexural strength.

The Tukey pairwise comparison showed significant mean value differences in flexural strength between the aged and nonaged specimens (Fig. 1D). The nonaged specimens (256.41 MPa) obtained significantly higher



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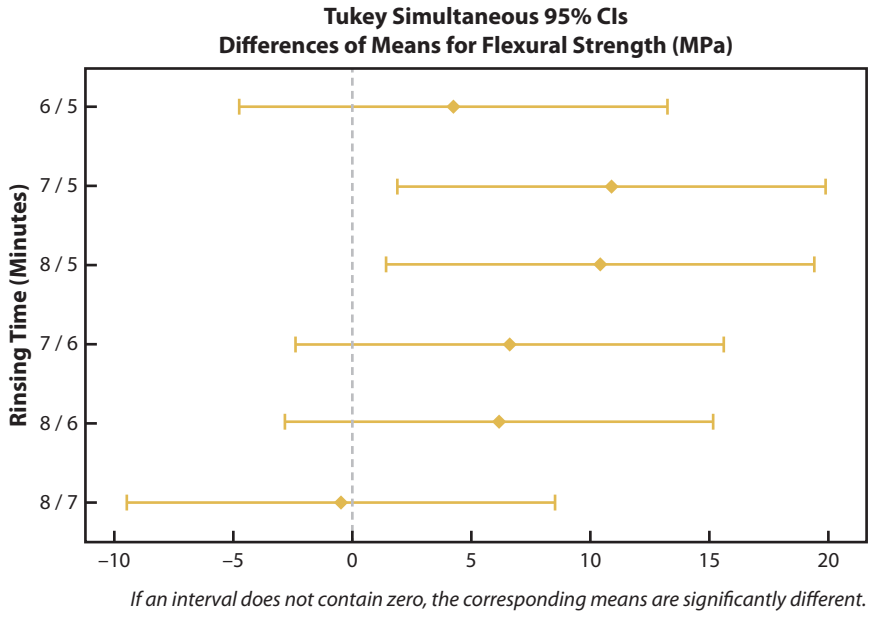


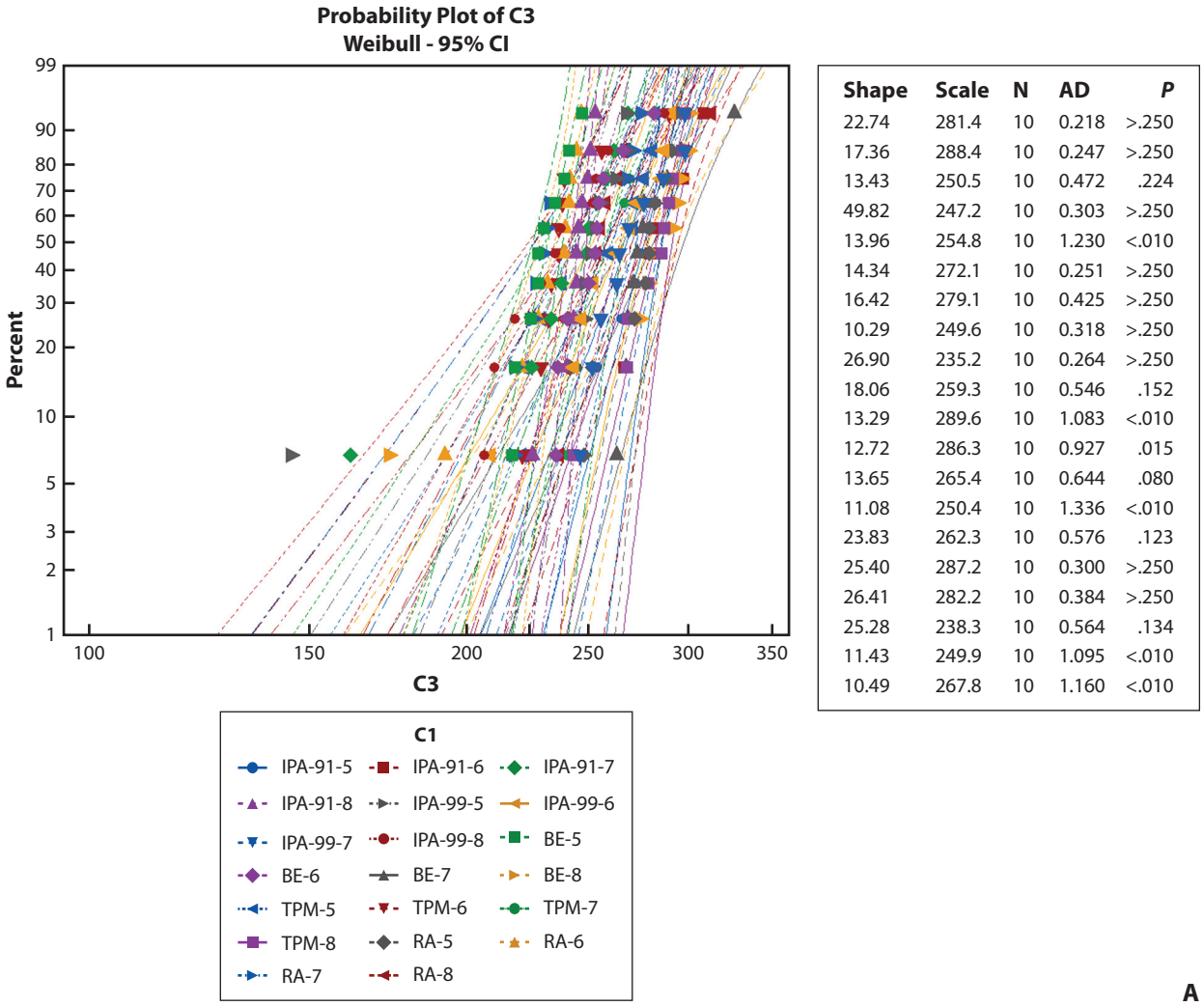
D

Figure 1. (*continued*). C, Tukey simultaneous 95% CIs flexural strength values for different rinsing solutions tested. D, Tukey simultaneous 95% CIs flexural strength values for different rinsing times tested. BE, bio-ethyl alcohol; IPA, isopropyl alcohol; RA, Resinaway; TPM, tripropylene glycol methyl ether.

mean values for flexural strength than the aged specimens (208.42 MPa).

The Weibull distribution presented the highest shape for the nonaged IPA-91-8 subgroup (49.82) compared with other nonaged groups (10.49 to 26.41), while the Weibull distribution presented the highest shape for the aged RA-5 subgroup (19.81) compared with other aged groups (7.55 to 14.22) (Fig. 2).

The SEM images of the specimens showed a relatively smooth surface with no visible pits or traces of different layers. All the groups presented a smooth depression between strands, with an approximate distance of 50 μm between the horizontal strands. No differences were observed among the IPA-91, IPA-99, BE, and TPM groups; additionally, no differences were observed between nonaged and aged specimens in each group and



Shape	Scale	N	AD	*P*
22.74	281.4	10	0.218	>.250
17.36	288.4	10	0.247	>.250
13.43	250.5	10	0.472	.224
49.82	247.2	10	0.303	>.250
13.96	254.8	10	1.230	<.010
14.34	272.1	10	0.251	>.250
16.42	279.1	10	0.425	>.250
10.29	249.6	10	0.318	>.250
26.90	235.2	10	0.264	>.250
18.06	259.3	10	0.546	.152
13.29	289.6	10	1.083	<.010
12.72	286.3	10	0.927	.015
13.65	265.4	10	0.644	.080
11.08	250.4	10	1.336	<.010
23.83	262.3	10	0.576	.123
25.40	287.2	10	0.300	>.250
26.41	282.2	10	0.384	>.250
25.28	238.3	10	0.564	.134
11.43	249.9	10	1.095	<.010
10.49	267.8	10	1.160	<.010

A

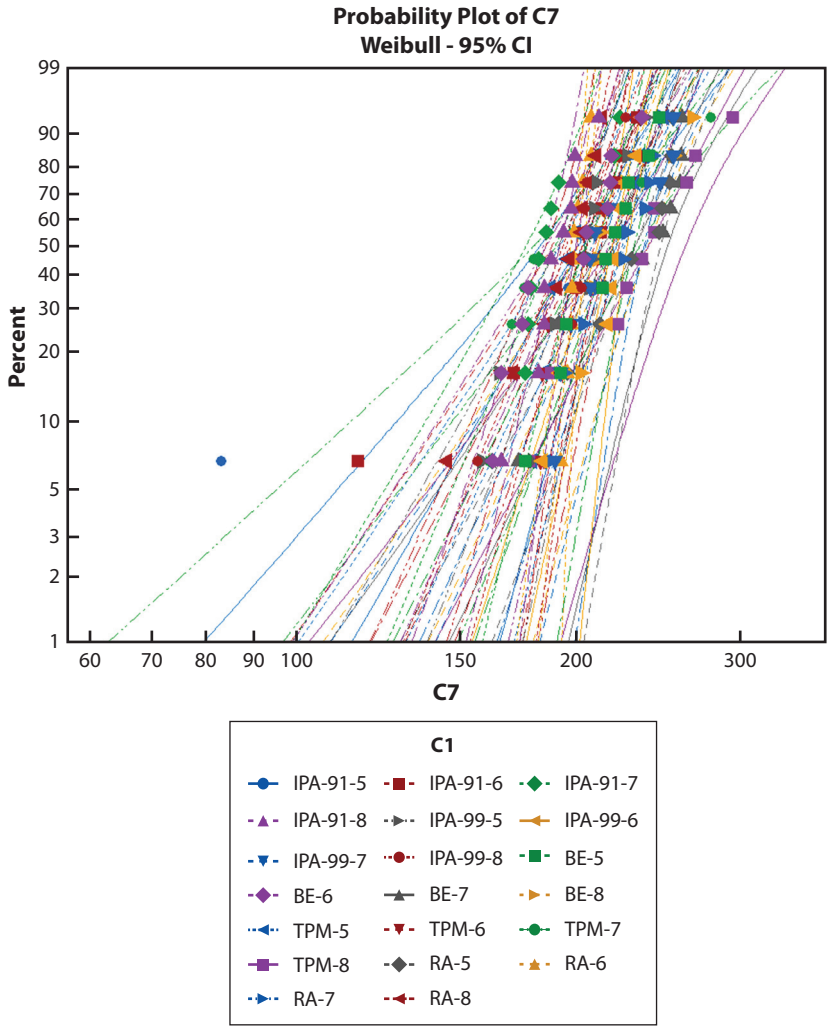
Figure 2. Weibull modulus for groups tested. A, Before thermocycling.

among the IPA-91, IPA-99, BE, and TPM groups. In the nonaged RA group, the same findings were found as in the other groups. However, the specimens of the aged RA group presented a different SEM image. After thermocycling, superficial cracks were observed in all specimens (Figs. 3, 4). No differences were observed among the RA subgroups.

DISCUSSION

The results of the present study demonstrated that the interim specimens fabricated with different rinsing solutions and total rinsing times by using a DLP printer led to significant differences in the flexural strength values obtained. Furthermore, accelerated artificial aging methods revealed a significant effect, with decreased flexural strength values. Therefore, the null hypotheses were rejected.

Based on the results obtained in the present study, the IPA rinsing solution at 91% or 99% concentration demonstrated higher mean values for flexural strength than when using BE, TPM, or RA rinsing solutions. The manufacturer of the printer and material evaluated recommends the 91% IPA rinsing solution; however, clinicians and dental laboratory technicians may have used alternative rinse solutions with little understanding of the impact. Reasons for using alternatives include availability and access to IPA, regulatory concerns about using IPA internationally, flammability risk of IPA compared with alternative solutions, perception that the alternative rinse solutions were milder, cost concerns, and volatile chemical smell of alcohols compared with alternatives. The selection of BE, TMP, or RA solvent decreased the mean flexural strength value by 21%, 10%, and 9%, respectively, compared with the 91% IPA solvent. However, superficial crack images were observed on



Shape	Scale	N	AD	P
7.550	211.4	10	0.879	.020
9.657	211.0	10	0.588	.111
10.16	197.2	10	0.867	.021
18.22	196.1	10	0.299	>.250
10.76	212.1	10	0.460	.237
19.81	222.9	10	0.469	.227
9.554	228.6	10	0.775	.036
15.22	208.1	10	0.601	.099
12.59	224.3	10	0.177	>.250
9.664	208.6	10	0.436	>.250
9.200	241.2	10	0.734	.045
9.124	229.8	10	0.800	.031
9.188	216.8	10	0.554	.144
12.22	216.0	10	0.494	.203
5.625	219.5	10	0.485	.212
8.004	250.6	10	0.205	>.250
11.90	239.1	10	0.579	.120
44.20	202.5	10	0.532	.166
13.63	230.5	10	0.312	>.250
14.32	200.4	10	0.361	>.250

B

Figure 2. (*continued*). B, After thermocycling. BE, bio-ethyl alcohol; IPA, isopropyl alcohol; RA, Resinaway; TPM, tripropylene glycol methyl ether.

thermocycled specimens of the RA group, which suggests its use is contraindicated.

The total rinsing time tested in the present study varied from 5 to 8 minutes. However, variations in the mean flexural strength effect were found among the rinsing solutions tested. In the 91-IPA group, the IPA-91-8 and IPA-91-7 subgroups obtained the highest mean values for flexural strength. While the manufacturer of the printer and material tested recommends 5 minutes of total rinsing time with 91% IPA solvent, the mean flexural strength increased when the total rinsing time increased. Further studies are needed to assess different rinsing solvents and total rinsing time, with parameters such as manufacturing accuracy, surface roughness, or microstructure of the interim restoration.

To limit the number of variables that can influence the outcome of AM specimens,[4] all the specimens were manufactured by using the same resin bottle, printing parameters, position on the building platform, and polymerization postprocessing procedures. Additionally, the ISO-recommended dimensions for the bar-shaped specimens were followed,[19] and the layer orientation of the specimens was perpendicular to the load direction of the 3-bend test to maximize the mechanical properties.[15]

Dental literature assessing the flexural strength of AM interim dentals is scarce.[3,4] Scotti et al[3] evaluated the flexural strength of AM interim specimens (C&B MHF; Nextdent), reporting a mean flexural strength of 105.10 ±9.80 MPa; however, details concerning the technology, printer, printing parameters, or postprocessing methods used were not reported. Therefore, comparisons with the results of the present study are challenging. Similarly, Scherer et al[4] assessed the flexural strength of aged and nonaged interim resin specimens (C&B MHF; Nextdent) with different postpolymerization times and conditions.[4] For the dry conditions and 30-minute rinse time, a mean ±standard deviation flexural strength value of 274.85 ±5.64 MPa for nonaged specimens and of 267.84 ±34.34

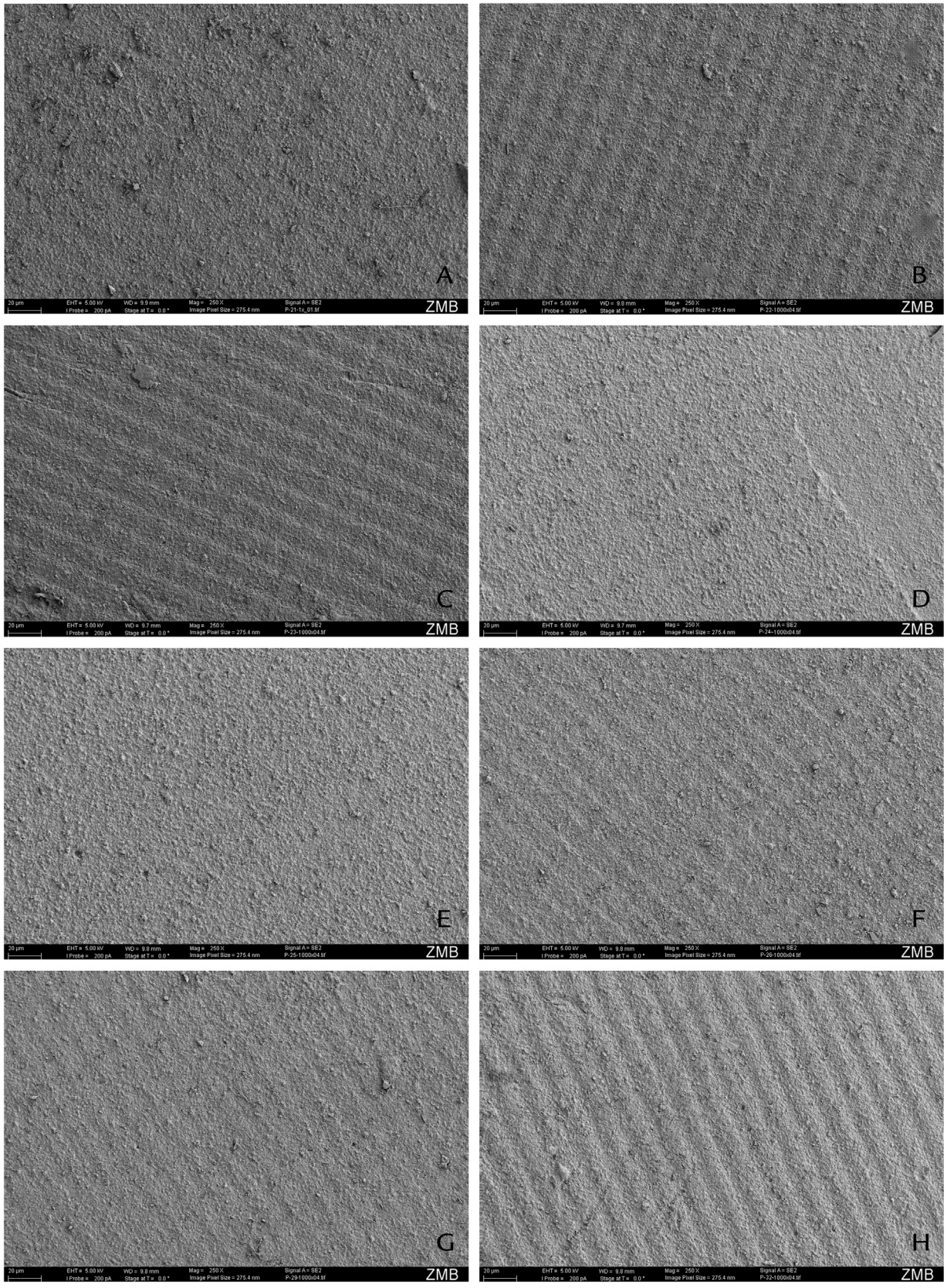


Figure 3. Representative scanning electron microscope images. Original magnification ×250 A, Nonaged IPA-91 group. B, Aged IPA-91 group. C, Nonaged IPA-99 group. D, Aged IPA-99 group. E, Nonaged BE group. F, Aged BE group. G, Nonaged TPM group. H, Aged TPM group. BE, bio-ethyl alcohol; IPA, isopropyl alcohol; TPM, tripropylene glycol methyl ether.

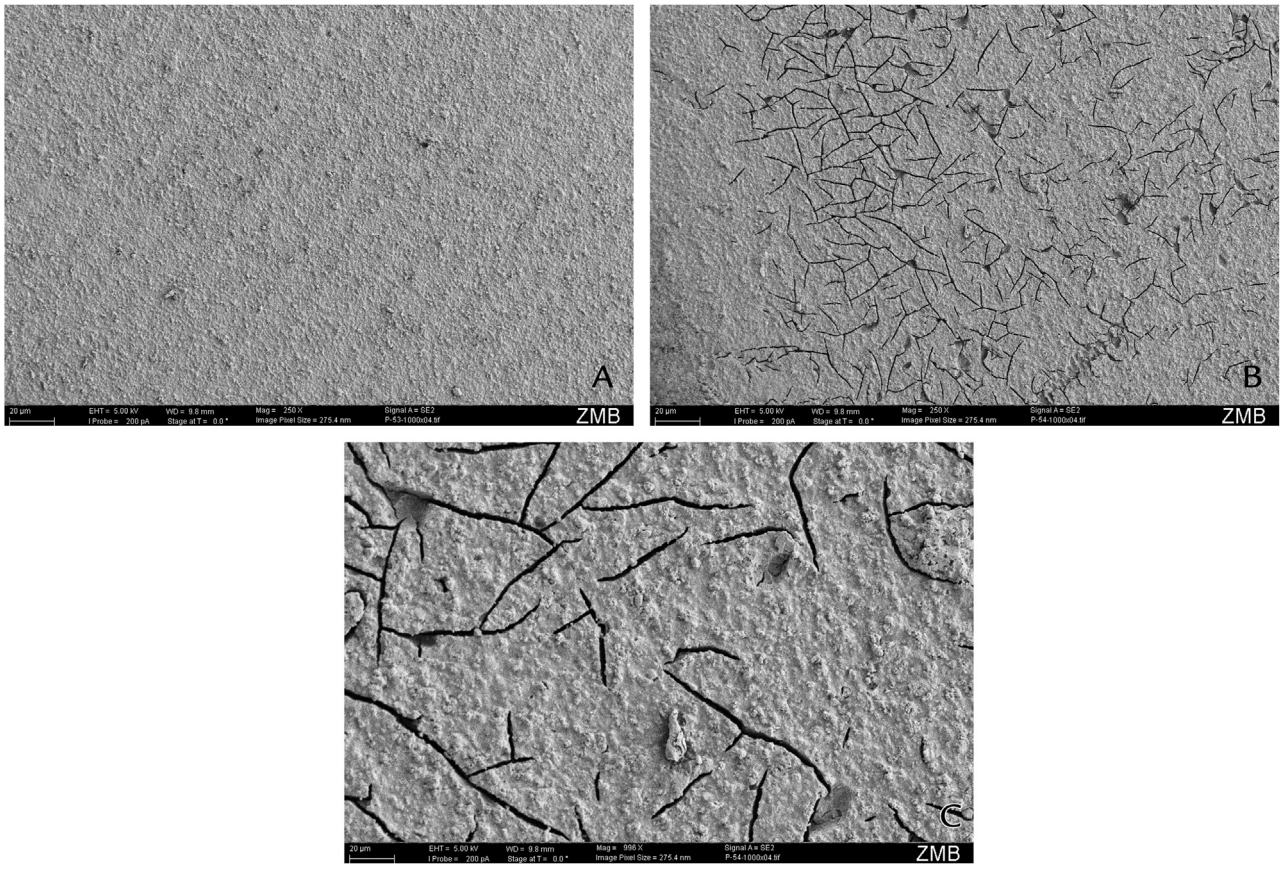


Figure 4. Representative scanning electron microscope images for RA-group. A, Nonaged RA group. B, Aged RA group. C, Aged RA group. Original magnification: A, B, ×250 magnification; C, ×996 magnification. RA, Resinaway.

MPa for aged specimens were reported, consistent with the results in the present study.

Additional studies have assessed the mechanical properties of AM interim dental restorations;[6,8,11,13,16,21] however, inhomogeneous research methodologies including crown-shape specimens, manufacturing procedures, and measurement techniques make a comparison with the results of the present study difficult.

The results obtained in the present study should not be applied to different manufacturing workflows with a different printer or interim material. Furthermore, even with the same printer and material, variations in the printing parameters and postpolymerization procedures would lead to different outcomes.[18] While the aforementioned factors related to the choice of rinse solution may influence rinse protocols from a user perception, the results of this study may justify the routine use of the 91% or 99% IPA solvent for postprocessing procedures of AM interim restorations.

Thermocycling procedures aim to simulate the deterioration of the material in the oral environment[22] and have been reported to affect the flexural strength of AM interim materials.[3,4,8] In the present study, a significant difference was obtained between nonaged and aged specimens, with the aged RA group demonstrating superficial cracks and a general appearance of degradation of the surface of the restoration, resulting in a restoration that may fail during clinical use. While the effect is multifactorial, matrix degradation and water absorption of the interim material are the likely cause of this degradation.[23] According to ISO 4049 and the American National Standards Institute (ANSI)/American Dental Association (ADA) specifications,[24] an interim fixed prosthesis material must have a minimum strength of 50 MPa under 3-point bend testing. Therefore, all the specimens obtained a clinically acceptable flexural strength.

Limitations of the present study included the limited vat-polymerization technologies, printers, and materials tested. Additional studies are recommended to further evaluate the effect of different rinsing solutions and rinsing times on other mechanical properties and the manufacturing accuracy of AM interim dental restorations.

CONCLUSIONS

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

1. The vat-polymerized AM interim dental material tested was manufactured by using differing rinsing

solutions and times and demonstrated significant differences in the flexural strength values measured.
2. The 91% and 99% IPA solutions for 7-minute and 8-minute rinsing obtained the highest mean values for flexural strength compared with the other rinsing solutions and times evaluated.
3. Accelerated artificial aging procedures significantly decreased the flexural strength of the vat-polymerized interim dental material tested.
4. The Weibull distribution of flexural strength values on the aged specimens measured was lower than that on the nonaged specimens.
5. In the RA group, SEM analysis demonstrated superficial cracks in the aged specimens, which might contraindicate its clinical use.

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