Title: Influence of the layer thickness on the flexural strength of aged and non-aged additively manufactured interim dental material

Running title: INFLUENCE OF LAYER THICKNESS ON FLEXURAL STRENGTH

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This article has been accepted for publication and undergone full peer review but has not been through the copyediting, typesetting, pagination and proofreading process, which may lead to differences between this version and the Version of Record. Please cite this article as doi: 10.1111/jopr.13582.

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Conflict of Interest

The authors did not have any conflict of interest, financial or personal, in any of the materials described in this study.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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Received February 23, 2022

Accepted July 25, 2022

ABSTRACT

Purpose. To measure the flexural strength and Weibull characteristics of aged and non-aged printed interim dental material fabricated with different layer thickness.

Material and methods. Bars (25×2×2 mm) were additively fabricated by using a polymer printer (Asiga Max) and an interim resin (Nexdent C&B MFH). Specimens were fabricated with the same printing parameters and postprocessing procedures, but with 7 different layer thickness: 50 (control or 50-G group), 10 (10-G group), 25 (25-G group), 75 (75-G group), 100 (100-G group), 125 (125-G group), and 150 μm (150-G group). Two subgroups were created: non-aged and aged subgroups (n=10). A universal testing machine was selected to measure flexural strength. Two-parameter Weibull distribution values were computed. Two-way ANOVA and Tukey tests were elected to examine the data (α=.05).

Results. Artificial aging methods (*P*<.001) were a significant predictor of the flexural strength computed. Aged specimens acquired less flexural strength than non-aged specimens. The Weibull distribution obtained the highest shape for non-aged 50-G and 75-G group specimens compared with those of other non-aged groups, while the Weibull distribution showed the highest shape for aged 125-G specimens.

Conclusions. The flexural strength of the additively fabricated interim material examined was not influenced by the layer thickness at which the specimens were fabricated; however, artificial aging techniques reduced its flexural strength. Aged specimens presented lower Weibull distribution values compared with non-aged specimens, except for the 125-G specimens.

Keywords: 3D printing; additive manufacturing technologies; interim dental prostheses; provisional dental material; vat-polymerization technologies

Digital light processing (DLP) procedures are considered vat-polymerization additive manufacturing (AM) techniques. DLP methods are clinically relevant methods to fabricate interim dental materials.[1,2] While the methods and manufacturing technologies used to fabricate them have been evaluated, the chemical composition, optimal printing and post-processing parameters, and mechanical characteristics of vat-polymerized interim dental materials are still uncertain.[3,4]

The characteristics and physical properties of AM interim materials[5-20] including manufacturing accuracy,[11,16] chemical composition,[9] color,[5,12] surface roughness,[9,11,14,20] marginal and internal fit,[13,16,19] mechanical properties,[5,6,8,10,11,15,18] adhesion of the microbiota,[11] wear,[7] and impact of accelerating aging techniques[5,6,19,10] have been analyzed in dental literature. An association have been recognized between the elected fabricating protocols (printing parameters and postprocessing methods) and the characteristics of the 3D printed devices including interim dental restoration.[4] Furthermore, there is a correlation among the additive technology, printer, and resin elected to process the dental device.[4] Dental literature has not been able yet to determine the optimal printing protocol based on the manufacturing trinomial, manufacturing protocol, and dental device.

Layer thickness has been recognized as one of the manufacturing variables that can affect the surface roughness, manufacturing accuracy, marginal and internal fit, fabricating time, and degree of conversion of 3D printed dental devices including interim dental restorations.[3,13,14,17,21] Furthermore, printed interim restorations exposed to aging techniques demonstrated lower flexural strength,[5,6,10] color stability,[5] and internal and marginal discrepancies[19] when compared to non-aged AM interim restorations. While many of the previously mentioned manufacturing variables have been evaluated in the scientific literature, the flexural strength and Weibull characteristics between aged and non-aged 3D printed interim materials fabricated with varying layer thickness remain unknown.

The goal of this investigation was to measure the flexural strength and Weibull characteristics of aged and non-aged AM interim dental material fabricated with varying layer thickness (10, 25, 50, 75, 100, 125, and 150 μm). The null hypotheses were that no significant discrepancy on the flexural strength and Weibull characteristics would be found among the specimens manufactured using different layer thicknesses and that no significant discrepancy on the flexural strength and Weibull characteristics would be found between aged and non-aged specimens.

Material and methods

A digital bar-shape (25×2×2 mm)[22] specimen was obtained by using a program (Blender, version 2.77a; The Blender Foundation). All the manufacturing methods were accomplished by a prosthodontist (M.S.) with more than 10 years of preceding experience managing vat-polymerization printers.

The virtual bar-shape design was used to additively fabricate all the interim bars by using a polymer vat-polymerization printer (Asiga Max; Asiga) and a resin designated for interim restorations (Nexdent C&B MFH Shade N1; 3D Systems). The printer was storage in a room with constant temperature of 23ºC and was calibrated accordingly to the manufacturer's calibration procedure prior producing any specimen. A fresh bottle of resin was obtained to manufacture all the specimens. Except for layer thickness, the bars of each group were processed all together with identical printing protocol including same location in the build platform, print orientation, and supportive material (Fig 1). All the bars were positioned in the build platform so that the layer was perpendicular to the load to be applied in the fracture resistance test.

Seven groups were produced depending on the layer thickness chosen to fabricate the specimens: 10 (10-G group), 25 (25-G group), 50 (control or 50-G group), 75 (75-G group), 100 (100-G group), 125 (125-G group), and 150 μm (150-G group). The 50-G group was treated as the control group because 50-μm is the layer thickness endorsed by the manufacturer (Table 1).

After completing the manufacturing procedures, the postprocessing rinsing and polymerization procedures were completed following the manufacturer's protocol. First, a spatula was used to remove the specimens from the build platform. Then, the specimens were washed in a 91% isopropyl alcohol (IPA) (Isopropyl alcohol 91%; Cumberland Swan) bath for 3 minutes, followed by a second 91% IPA clean bath for another 2 minutes. The specimens were dried on a paper towel and polymerization procedures were completed (LC-3DPrint Box; 3D Systems) at 300-550 nm for 30 minutes. Lastly, a removal tool was used to remove the supportive material. The bars were stored in a light-proof bottle until the tests were performed.

Twenty bars per group were obtained, and arbitrarily distributed into 2 subgroups by using a shuffled deck of cards depending on the artificial aging techniques: non-aged and aged subgroups (N=20, n=10). Sample size was stablished based on previous investigations.[6] In the aged subgroup, the bars were exposed to thermocycling methods which included 6,000 cycles of 3 successive sequences each: (1) 20 seconds (dwelling phase) at 5°C; (2) 5 seconds (transfer phase) at ambient air temperature at 23°C; and (3) 20 seconds (dwelling phase) at 55°C.

A universal testing machine (Universal Testing Machine; ZwickRoell) at a crosshead speed of 1mm/min on a 10-mm span was elected to measure flexural strength (MPa).[22] The bars were loaded to failure and fracture load (N) data were documented. The flexural strength (σ) was calculated using the 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, and d is the thickness of the bar.[17]

Weibull distribution maximum likelihood estimation without a correction factor was used including the Weibull modulus, scale (m), and shape (0) to interpret the predictability and reliability of the flexural strength tests (Minitab Software V.16; Minitab).[23]

The Shapiro-Wilk and Kolmogorov-Smirnov tests disclosed that the data presented a normal distribution (*P*<.05). Two-way ANOVA and post hoc multiple pairwise comparison

Tukey tests were elected to examine the data (α=.05) (IBM SPSS Statistics for Windows, v26; IBM Corp).

Results

Two-way ANOVA indicated that only the accelerating artificial aging methods (df=1, MS=238829, F=583.16, P<.001) was a significant predictor of the flexural strength measured (Fig 2a, Table 2). Additionally, the artificial aging techniques explains the 80.33% of difference in the flexural strength obtained, while the layer thicknesses evaluated explains the 1.49% of the disparity in the flexural strength computed.

With respect to the group factor, Tukey pairwise comparison revealed no significant flexural strength differences among the differing layer thicknesses tested. With respect to the subgroup predictor, Tukey pairwise comparison demonstrated significant flexural strength discrepancies between non-aged (mean of 289.77 MPa) and aged specimens (mean of 207.17 MPa) (Fig 2b).

The Weibull distribution obtained the highest shape for non-aged 50-G (control) (45.98) and 75-G group specimens (42.24) compared with those of other non-aged groups (11.36 to 27.12), while the Weibull distribution showed the highest shape for aged 125-G specimens (68.18) compared with those of other aged groups (10.62 to 44.58) (Fig 3).

Discussion

The results of this investigation demonstrated that the specimens fabricated with different layer thicknesses using the polymer vat-polymerization DLP 3D printer tested, with its specific manufacturing protocol described, obtained no significant discrepancies in the flexural strength computed. But accelerating artificial aging techniques caused in a significant reduction in the flexural strength mean values of the 3D printed interim material tested. Hence, only the second null hypothesis was rejected.

The differing layer thicknesses assessed on the specimens manufactured using the selected interim dental resin, DLP printer, and manufacturing protocol tested on this investigation did not significantly influence the flexural strength values obtained. Therefore, just considering the flexural strength characteristics, interim dental restorations could be manufactured using any of the layer thicknesses tested. However, dental literature has shown a correlation between layer thickness, surface roughness, manufacturing accuracy, fabricating time, and degree of conversion of AM dental devices.[13,14,17,21] Additionally, the generalization of the results is not recommended as the characteristics of the printed device are the result of multiple manufacturing variables such as technology, printer, and resin chosen, printing parameters, or postprocessing processes performed. Therefore, additional studies might be needed to assess the influence of the layer thickness on the mechanical properties of varying 3D printed devices before further conclusions can be done.

Different manufacturing variables can affect the characteristics of AM interim specimens.[5-21] To limit these factors, the specimens were fabricated from a single bottle of dental resin with the same printing parameters (except for the layer thickness), location in the build platform, and post-processing methods. Furthermore, the bar-shape specimens were fabricated following the ISO recommended dimensions[22] with the layer orientation positioned perpendicular to the load direction of the 3-bend test to optimize the flexural strength of the printed interim material.[15]

Limited studies have tested the flexural strength of interim materials processed using AM technologies.[5,6] Scotti et al[5] reported a flexural strength value of 105.10 ±9.80 MPa for the same interim material tested (Nexdent C&B MHF). However, the specimens had different dimensions (10×2×2 mm) and no details were provided regarding the printer, printing parameters, or postprocessing techniques selected to manufacture the specimens. Hence, comparisons with the data obtained in this investigation are difficult.

Scherer et al[6] assessed the influence of different post-polymerization times (25, 30, 35, 40, and 45 minutes) and conditions (dry, or inside a container containing water or glycerin) on the flexural strength of aged and non-aged bar-shape (25×2×2 mm).[22] The specimens were fabricated using a resin designated to fabricate interim restorations (Nexdent C&B MHF) and a polymer printer (Nexdent 5100; 3D Systems). In the present investigation, the same resin was used, but a different polymer printer was elected (Asiga Max; Asiga). Variations on the printing or supportive parameters can be expected between both studies; however, further interpretations of how these discrepancies might impact on the outcome of the printed specimens is unknown. Additionally, the postpolymerization methods were completed in dry conditions and performed for 30 minutes of time; thus, the results of the present investigation could be compared with the 30-minute and dry-condition post-polymerization groups. Scherer et al.[6] reported a mean flexural strength value of 274.85 ±15.64 MPa for non-aged samples and of 267.84 ±34.34 MPa for aged samples. Those results agree with the data obtained in this investigation.

Additional dental literature has evaluated the properties of printed interim restorations;[8,24] however, variations on the research methodology such as crown-shape specimens, fabricating protocols, and testing procedures make comparisons with the data obtained in this investigation challenging.

Thermal cycling techniques aim to reproduce the deterioration of the material in the oral environment.[25] The flexural strength of AM interim materials seems to be affected by artificial aging procedures.[5,6,10] The results of this investigation obtained a significant difference between non-aged (mean of 289.77 MPa) and aged specimens (mean of 207.17 MPa). While the phenomenon is multi-factor, water absorption and matrix degradation of the resin is the suggested cause for this finding.[26,27] However, all the bars tested achieved a clinically acceptable flexural strength.[28]

Artificial intelligence (AI) models have been reported for optimizing manufacturing procedures.[29,30] This might be a tool in the future for stablishing printing protocols based on the manufacturing trinomial and clinical application of the printed dental device. While the broad availability of printers and materials provide wider manufacturing options for dental professionals, there is a need of scientific literature that assess the properties of these new materials, as well as, stablishing the optimal manufacturing protocol.

This investigation has limitations such as reduced additive technologies, 3D printers, and materials examined. Studies are suggested to further assess the influence of the different printing parameters on the fabricating accuracy, marginal and internal discrepancies, and physical characteristics of printed interim dental restorations.

Conclusions

With the limitations of this in vitro investigation, the varying layer thickness tested did not influenced on the flexural strength and Weibull characteristics of the interim material selected manufactured with its described DLP printer and printing protocol. However, artificial aging techniques reduced the flexural strength of the printed interim material assessed. Additionally, the Weibull distribution of flexural strength values on aged specimens were lower than those on non-aged specimens, with the exception of the 125-G specimens.

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TABLES

Table 1. Characteristics of the groups assessed.

Group	Subgroup	Layer thickness	Aging methods
10-G	Aged	10 µm	Yes
	Non-aged		No
25-G	Aged	25 µm	Yes
	Non-aged		No
50-G	Aged	50 µm	Yes
	Non-aged		No
75-G	Aged	75 µm	Yes
	Non-aged		No
100-G	Aged	100 µm	Yes

	Non-aged		No
125-G	Aged	125 μm	Yes
	Non-aged		No
150-G	Aged	150 μm	Yes
	Non-aged		No

FIGURES

Figure 1. (a) Representative specimen orientation. (b) Representative AM specimen. AM, Additively manufactured

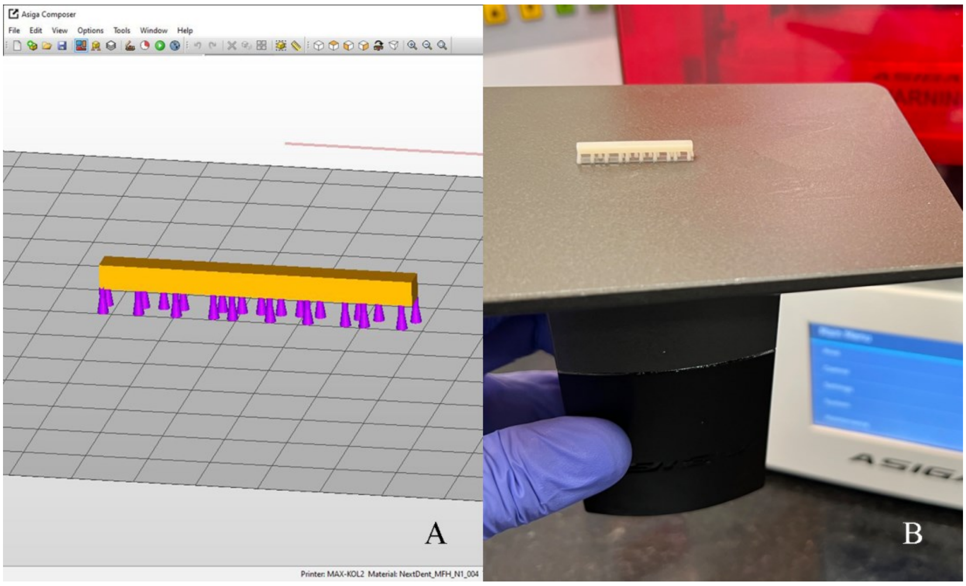
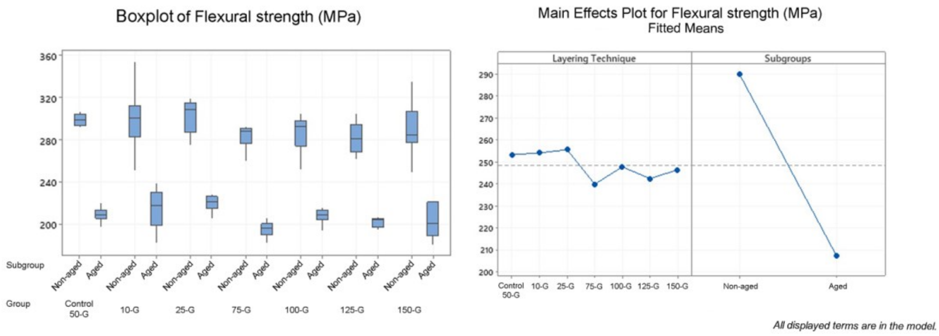


Figure 2. (a) Boxplot of flexural strength values obtained among the groups. (b) Main effects plot for flexural strength obtained among the groups.


Boxplot of Flexural strength (MPa)
360
320
280
240
200
Subgroup
Non-aged
Aged
Group
Control 50-G
10-G
25-G
75-G
100-G
125-G
150-G
Main Effects Plot for Flexural strength (MPa)
Fitted Means
Layering Technique
Subgroups
290
280
270
260
250
240
230
220
210
200
Control 50-G
10-G
25-G
75-G
100-G
125-G
150-G
Non-aged
Aged
All displayed terms are in the model.

A B

Figure 3. Weibull modulus for all the groups tested.