Influence of the polymerization post-processing procedures on the accuracy of additively manufactured dental model material

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Conflict of Interest

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ABSTRACT
Purpose: To measure the influence of postpolymerization condition (dry or submerged in water) and time (2, 10, 20, and 40 minutes) on the accuracy of additively manufactured model material. Materials and Methods: A bar standard tessellation language file was used to manufacture the resin specimens (E-Model Light, EnvisionTEC) using a 3D printer (Vida HD, EnvisionTEC). Two groups were created based on the postpolymerization condition: dry (D group) or submerged in water (W group). Each group was divided into four subgroups (D1 to D4 and W1 to W4) depending on the postpolymerizing time (2, 10, 20, and 40 minutes; n = 20 each; N = 160). The specimen dimensions were measured using a low-force digital caliper (Absolute Low Force Caliper Series 573, Mitutoyo). The volume was calculated: \( V = \)
l × w × h. Shapiro-Wilk test revealed that the data were not normally distributed. Data were analyzed using Kruskal-Wallis and pairwise Mann-Whitney U tests (α = .05). **Results:** Significant differences in length, width, height, and volume values were found among the subgroups (P < .0018). In all groups, the width dimension (x-axis) presented the worst accuracy compared to height (z-axis) and length (y-axis) (P < .0018). The D2 and D4 subgroups obtained the closest dimensions to the virtual design; additionally, no significant differences were found between the two subgroups (P < .0018). Dry condition showed higher manufacturing accuracy compared to the water-submerged condition. In the water-submerged subgroups, the highest accuracy was obtained in the W2 and W4 subgroups (P < .0018). **Conclusion:** Postpolymerization conditions and time influenced the accuracy of the material tested. Dry postpolymerization condition with a time of 10 and 40 minutes obtained the highest accuracy. *Int J Prosthodont 2021. doi: 10.11607/ijp.7349*

**INTRODUCTION**

Vat-polymerization additive manufacturing (AM) technologies involves the fabrication of a 3D object in a layer-by-layer building process by using a photopolymerization procedure also known as photocuring or photo-cross-linking method. Vat-polymerization AM technologies use a vat of a photosensitive monomers and oligomers in a liquid state that can be photopolymerized upon exposure to light source of specific wavelength and form thermosets. This liquid to solid resin polymerization process normally results in material shrinkage.

The manufacturing workflow of a vat-polymerization printer is composed by 3 main steps namely data processing, manufacturing, and post-processing procedures. Data processing implicates the slicing of the standard tessellation language (STL) file and the definition of 3D printing parameters and support parameters. Manufacturing involves the
additive fabrication of an object using a 3D printer. The post-processing procedures include the processes that need to be performed on the AM object, such as removing the object from the building platform, cleaning the uncured resin, performing post-polymerization procedures, and removing the support structures.\textsuperscript{3,4}

Literature analyzing optimal post-polymerization conditions and post-polymerization time is scarce. Different post-polymerization conditions have been described namely dry and water-submerged conditions\textsuperscript{8-10}. Vat-polymerization technologies employ photosensitive polymers that are polymerized layer-by-layer using a laser, arc lamp, or a digital mirror device (DMD).\textsuperscript{1-4} The polymerization reaction is inhibited by oxygen diffusing from the ambient atmosphere into resin during the light polymerization procedure.\textsuperscript{9,11-13} Water-submerged polymerization conditions provide a reduced oxygen-free condition compared with air;\textsuperscript{9,12} however, the effect of performing post-polymerization procedures in a dry (ambient air) or water-submerged environment on the dimensional manufacturing accuracy remains unclear.

The manufacturing accuracy of object fabricated by vat-polymerization printers can be influenced by multiple factors namely vat-polymerization technology employed,\textsuperscript{14,15} printer calibration, ambient temperature where the 3D printer is located, power of the light source, support structures,\textsuperscript{14} platform positioning,\textsuperscript{14-21} material properties,\textsuperscript{15} slicer software,\textsuperscript{20} resin color,\textsuperscript{21,21} geometry of the object,\textsuperscript{16,22,23} photo-initiators, wavelength power, post-processing procedures,\textsuperscript{20-24} printing parameters,\textsuperscript{25-31} and printing angulation.\textsuperscript{14,16,18-20,26}

The accuracy of a 3D printer can be described by two factors namely trueness and precision.\textsuperscript{32} Trueness indicates to the ability of the printer to reproduce the virtual design contained in an STL file as close to its real form as possible without distortion, and precision stipulates the degree of identical objects manufactured under the same conditions.\textsuperscript{32}
Limited dental literature has evaluated the influence of post-processing procedures on manufacturing accuracy of an additively manufactured dental cast. Dental manufacturers of 3D print materials explicitly recommend a specific post-polymerization machine and post-polymerization protocol for their products to ensure the adequate manufacturing accuracy. However, the influence of the polymerization conditions and polymerization time on the manufacturing accuracy is scarce. The purpose of this in vitro study was to measure the influence of the post-polymerization conditions (dry and water-submerged) and post-polymerization times (2, 10, 20, and 40 minutes) on the dimensional manufacturing accuracy of additively manufactured dental model material. The null hypotheses were that there would be no significant dimensional differences in the additively manufactured dental model specimens among the dry and water-submerged post-polymerization conditions and that there would be no significant dimensional differences in the additively manufactured dental model specimens among the 2, 10, 20, and 40 minutes post-polymerization time.

**MATERIAL AND METHODS**

A digital design of a bar (15×4×3 mm) was created using an open source software program (Blender version 2.77a; The Blender Foundation). The standard tessellation language (STL) file was exported and used to manufacture all the specimens using a resin dental material (E-Model Light; Envisiontec) and a vat-polymerization 3D printer (VIDA HD; Envisiontec) following the printing parameters recommended by the manufacturer at a constant room temperature of 23 °C with a layer thickness of 25 μm. Moreover, the 3D printer was calibrated following the manufacturer’s recommendations. The manufacturer of the printer reports a resolution of 50 μm in the XY axes and between 25 μm to 150 μm in the z-axis.

Two groups were created based on the conditions of the post-processing procedures performed namely polymerization procedures in dry (D group) or submerged in container...
with room-temperature (23 ºC) water (W group) inside the chamber of the UV-polymerization machine (PCA-100; Envisiontec). Each group was further divided in 4 subgroups (D1 to D4 and W1 to W4) depending on the post-polymerizing time used. In order to standardize the manufacturing procedures, all the specimens of the same subgroup were manufactured at the same and in the same position on the build platform. All the post-processing procedures were performed using nitrile gloves and by the same dental technician with 6 years of previous experience in handling 3D polymer printers.

For D1 subgroup specimen’s fabrication, the manufacturing workflow of the manufacturer was followed. After printing, the specimens were carefully removed from the build platform using a spatula (Fig. 1A). Afterwards, specimens were fully submerged in an ultrasonic bath (TriClean Ultrasonic Cleaner U-10LHREC; BrandMax) with 99% isopropyl alcohol (IPA) (Isopropyl alcohol 99%; Cumberland Swan) for 3 minutes and subsequently in a second ultrasonic bath with a clean 99% IPA alcohol for 2 minutes (Fig. 1B). Specimens were rinsed with water and positioned in a paper towel and dried. Specimens were then polymerized in the UV-polymerization machine selected for 2 minutes. Twenty specimens per subgroup were manufactured and stored in a black container until measurements were completed.

In the D2, D3, and D4 subgroups, the same manufacturing procedures of D1 subgroup were followed except for post-polymerization time which varied among the groups. The polymerization time completed in the D2 subgroup was 10 minutes, 20 minutes in the D3 subgroup, and 40 minutes in the D4 subgroup. Twenty specimens per subgroup were manufactured and stored in a black container until measurements were completed.

In the W1, W2, W3, and W4 subgroups, the same manufacturing procedures of D1, D2, D3, and D4 subgroups respectively were followed except for post-polymerization conditions in which the specimens were submerged in a glass container with room
temperature water (23 °C) and placed inside the chamber of the same UV-polymerization unit. Twenty specimens per subgroup were manufactured and stored in a black container until measurements were completed.

The dimensions (length, width, and height) of all the specimens were measured using a low force digital caliper (Absolute Low Force Caliper Series 573; Mitutoyo) no later 24 hours of manufacturing the specimens (n=20, N=160). The manufacturer of this digital caliper reports an accuracy of 0.01 mm and its specific for plastics and resilient materials. Each measurement was performed 3 times and the mean value determined. The volume of each specimen was calculated using the formula \[ V = l \times w \times h, \] where “l” is the length, “w” the width, and “h” the height of the specimen.

The definition of trueness in the experiment was defined as the mean or median of the average absolute dimensional discrepancy between the virtual design dimensions and the specimens of each subgroup. Precision was defined as the standard deviation or interquartile range (IQR) of the dimensional discrepancies between the virtual design dimensions and the specimens of each subgroup.

Shapiro-Wilk test revealed that the data were not normally distributed. The length, width, and height data were analyzed using Kruskal-Wallis, followed by pairwise Mann-Whitney U tests. Significance level was adjusted using Bonferroni correction for multiple pairwise comparisons (\( P<0.05/28 \)). Data analysis were performed using a statistical software program (IBM SPSS Statistics, v24.0; IBM Corp.).

RESULTS

Post-polymerization conditions and post-polymerization time significantly influenced on the dimensional manufacturing accuracy. Kruskal-Wallis test revealed significant differences in length, width, height, and volume among the tested groups (\( P<0.0018 \)) (Fig. 2). The results
for Mann-Whitney U tests are summarized in the Table 1. None of the manufacturing workflows tested was able to manufacture a perfect match of the bar virtual design dimensions. The width dimension (y-axis) presented the worse dimensional reproduction in all groups compared with height (z-axis) and length (x-axis) \( (P<0.0018) \). The D2 and D4 subgroups obtained the closest dimensions compared with the virtual design dimensions.

The manufacturing dimensional discrepancies of the specimens compared with the virtual design dimensions are presented in Table 2 \( (P<0.0018) \). Dry post-polymerization condition obtained higher dimensional manufacturing accuracy compared with water-submerged post-polymerization condition, where the D2 and D4 subgroups obtained the smallest dimensional discrepancies. On the water-submerged post-polymerization conditions, the highest dimensional accuracy was obtained in the W2 and W4 subgroups. In both polymerization conditions tested, 10 and 40 minutes polymerization time obtained higher dimensional manufacturing accuracy compared with 2 and 20 minutes post-polymerization time.

**DISCUSSION**

Significant differences in length, width, height, and volume dimensions of the vat-polymerized dental model specimens were found among the different post-polymerization conditions and post-polymerization times tested; therefore, both null hypotheses were rejected. None of the manufacturing workflows tested was able to manufacture a perfect match of the bar virtual design dimensions; moreover, the D1 subgroup (control group) that corresponded with the post-processing protocol recommended by the manufacturer using their model resin, 3D printer, and UV-polymerization machine obtained significantly higher length and height dimensional discrepancies than the rest of the groups tested.
The dental application of the polymer selected in the present study is the additively manufactured dental casts including diagnostic and definitive casts. The clinician should understand that the manufacturing accuracy is dependent of the manufacturing trinominal namely AM technology, 3D printer, and resin selected. Furthermore, the manufacturing trinominal can be affected by the printing parameters and post-processing procedures. The manufacturing trinominal is specifically developed for a given dental application.

The reported clinically acceptable range of the AM casts varied from 100 to 300 µm.\textsuperscript{33-37} Based on the results of the present study, post processing procedures including curing times and conditions significantly influenced on the manufacturing accuracy. Furthermore, the post-processing procedures distortion obtained among the groups varied between 10 to 90 µm. The manufacturing discrepancy measured might not be clinically relevant to the restorative procedures when the additively manufactured cast is used as a diagnostic cast or to elaborate a dental device such a thermoplastic template. However, if the additively manufactured cast is used as a definitive working cast where a restoration is finished, this may require adjustments of the contact point and occlusal contacts on the delivery appointment. Further studies are needed to evaluate the clinical relevance of this post-processing manufacturing distortion.

Among the different post-polymerization conditions evaluated, the water-submerged condition obtained less manufacturing accuracy compared to non-water submerged post-polymerization conditions. Moreover, the y-axis values presented a significantly high interquartile range which represents a high variability on the data collected and a low precision of the manufacturing workflows tested. Therefore, water-submerged post-polymerization condition should be avoided. However, previous authors have reported a higher microhardness property under oxygen-protected polymerization conditions.\textsuperscript{5} Further
studies are recommended to completely understand the polymerization procedures of AM polymer objects.

Based on the results obtained in the present study, the 10 and 40 minutes dried-polymerized specimens achieved the highest manufacturing accuracy among the groups tested, while the 20 minutes dry-polymerized specimens presented lower manufacturing accuracy compared to the 10 and 40 minutes dried-polymerized groups. However, volume values data presented no significant difference among the 3 groups.

Overall, heigh measurements obtained higher manufacturing accuracy compared with width and length measurements. Considering the bar position on the build platform, the heigh corresponded to the z-axis resolution, while the length and width measurements evaluated the x- and y-axes. Therefore, the results of the present study can be explained by the higher resolution of the printer on the z-axis compared with the x- and y-axes.

Limited dental literature have reported the influence of the printing parameters such as layer thickness and build orientation in the manufacturing accuracy, surface roughness, and mechanical properties of the vat-polymerized dental devices.25-30 The studies present disparities on the research protocol which makes it difficult to draw comparison between them; furthermore, the majority of the studies evaluated multiple parameters at the same time, which make it difficult to see the clear effect of one parameter on the results presented. However, in the present study, in order to standardize the manufacturing procedures, all the specimens were printed at 0-degrees build orientation and all the specimens of each subgroup were printed at the same time and at the same position in the build platform of the 3D printer.

Previous authors have evaluated the impact of the post-processing procedures on the manufacturing accuracy and mechanical properties of the vat-polymerized dental devices, however, none of those reports provide information regarding different post-polymerization condition.20,21,23 Reymus et al20 reported significant fracture load values discrepancies among
the interim dental materials that were post-polymerized using 3 different UV-polymerization machines. Unkovskiy et al\textsuperscript{21} did not find a correlation between the post-polymerization procedures and the flexural strength, flexural modulus, and dimensional accuracy of the AM specimens. Reymus et al\textsuperscript{30} in another study demonstrated that the post-polymerization procedures influence the degree of conversion of the AM interim dental materials tested. In the present study, all the specimens were post-polymerized using the same UV-polymerization machine in which the only variables evaluated were polymerization-condition and polymerization-time.

The manual method using a low force digital caliper aimed to avoid the distortions due to pressure variations while measuring the specimens. As an alternative, a digital analysis could have been completed. However, there is no previous study that compared the accuracy of both measurement methods. Further studies are recommended to further analyze the different measurement methods available.

The generalization of the results obtained in the present study should be avoided as the results may vary if changing the vat-polymerization technology, 3D printer, printing parameters, rinsing procedures, UV-polymerization machine, polymer resin selected, and measurement method selected. The present study presents limitations such as the number of dental model resin evaluated, only one UV-polymerization machine was selected, the polymerization times tested were limited, and the manual measurement method used. Further studies are needed to understand the influence of the post-polymerization procedures on the manufacturing accuracy of vat-polymerization 3D printers and standardized the optimal manufacturing procedure for each AM technology, 3D printer, and resin specifically for a dental application.

CONCLUSIONS
With the limitations of this in vitro study, the following conclusions were drawn:

- None of the manufacturing workflows tested was able to manufacture a perfect match of the bar virtual design. Moreover, the width dimension (x-axis) presented the worse dimensional reproduction compared with height (z-axis) and length (y-axis).

- Post-polymerization conditions and post-polymerization time evaluated demonstrated a significant effect on the manufacturing accuracy of the vat-polymerized dental model specimens.

- Dry post-polymerization condition obtained the highest dimensional manufacturing accuracy compared to water-submerged post-polymerization condition.

- Dry post-polymerization condition with a polymerization time of 10 and 40 minutes obtained the highest dimensional manufacturing accuracy compared to water-submerged post-polymerization condition.
REFERENCES


Last accessed 08/01/2019


### TABLES

Table 1. Length (x-axis), width (y-axis), and height (z-axis) values obtained for each group tested. Data is provided in millimeters (mm). D, Dry or non-water submerged polymerization conditions; W, Water-submerged polymerization conditions

<table>
<thead>
<tr>
<th>GROUP</th>
<th>LENGTH Median ±IQR</th>
<th>WIDTH Median ±IQR</th>
<th>HEIGHT Median ±IQR</th>
<th>VOLUME Median ±IQR</th>
</tr>
</thead>
<tbody>
<tr>
<td>D1 (Control)</td>
<td>15.08 ±0.05 (^a)</td>
<td>4.07 ±0.04 (^a)</td>
<td>3.02 ±0.01 (^a)</td>
<td>185.11 ±2.29 (^a)</td>
</tr>
<tr>
<td>D2</td>
<td>15.02 ±0.09 (^b, c)</td>
<td>4.06 ±0.05 (^a)</td>
<td>3.00 ±0.01 (^b)</td>
<td>183.07 ±2.22 (^b, c, d)</td>
</tr>
<tr>
<td>D3</td>
<td>15.00 ±0.07 (^b, c)</td>
<td>4.07 ±0.04 (^a)</td>
<td>3.01 ±0.01 (^a)</td>
<td>183.67 ±1.50 (^b)</td>
</tr>
<tr>
<td>D4</td>
<td>15.02 ±0.09 (^b, c)</td>
<td>4.07 ±0.04 (^a)</td>
<td>3.00 ±0.01 (^b)</td>
<td>183.37 ±2.37 (^b)</td>
</tr>
<tr>
<td>W1</td>
<td>15.04 ±0.08 (^a)</td>
<td>4.10 ±0.04 (^b)</td>
<td>2.97 ±0.02 (^d)</td>
<td>183.18 ±2.27 (^b, c, d)</td>
</tr>
<tr>
<td>W2</td>
<td>15.02 ±0.08 (^b, c)</td>
<td>4.08 ±0.04 (^a, b)</td>
<td>3.00 ±0.01 (^e)</td>
<td>183.83 ±2.25 (^b)</td>
</tr>
<tr>
<td>W3</td>
<td>15.01 ±0.06 (^b, c)</td>
<td>4.06 ±0.38 (^a)</td>
<td>2.99 ±0.01 (^f)</td>
<td>182.38 ±1.93 (^c, d)</td>
</tr>
<tr>
<td>W4</td>
<td>14.99 ±0.07 (^b, c)</td>
<td>4.06 ±0.3 (^a)</td>
<td>2.99 ±0.01 (^e)</td>
<td>182.66 ±1.48 (^d)</td>
</tr>
</tbody>
</table>

There were no statistically significant differences (\( \alpha = .0018 \)) between groups with the same superscript. Table is designed to be read column wise and letters are unique to each element.

IQR, Interquartile range.
Table 2. Length (x-axis), width (y-axis), and height (z-axis) dimensional changes of the specimens of each group compared with the virtual design of the STL file. Data is presented in microns (µm). D: Dry or non-water submerged polymerization conditions; W: Water-submerged polymerization conditions

<table>
<thead>
<tr>
<th>GROUP</th>
<th>LENGTH</th>
<th>WIDTH</th>
<th>HEIGHT</th>
<th>VOLUME</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Median (trueness) ±IQR (precision)</td>
<td>Median (trueness) ±IQR (precision)</td>
<td>Median (trueness) ±IQR (precision)</td>
<td>Median (trueness) ±IQR (precision)</td>
</tr>
<tr>
<td>D1 (Control)</td>
<td>80 ±50 a</td>
<td>70 ±40 a</td>
<td>20 ±10 a</td>
<td>5.11 ±2.29 a</td>
</tr>
<tr>
<td>D2</td>
<td>20 ±90 b,c</td>
<td>60 ±50 a</td>
<td>0 ±10 b</td>
<td>3.07 ±2.22 b,c,d</td>
</tr>
<tr>
<td>D3</td>
<td>0 ±70 b,c</td>
<td>70 ±40 a</td>
<td>10 ±10 a</td>
<td>3.67 ±1.50 b</td>
</tr>
<tr>
<td>D4</td>
<td>20 ±90 b,c</td>
<td>70 ±40 a</td>
<td>0 ±10 b</td>
<td>3.37 ±2.37 b</td>
</tr>
<tr>
<td>W1</td>
<td>40 ±80 a</td>
<td>100 ±40 a</td>
<td>-30 ±20 d</td>
<td>3.18 ±2.27 b,c,d</td>
</tr>
<tr>
<td>W2</td>
<td>20 ±80 b,c</td>
<td>80 ±40 a,b</td>
<td>0 ±10 e</td>
<td>3.83 ±2.25 b</td>
</tr>
<tr>
<td>W3</td>
<td>10 ±60 b,c</td>
<td>60 ±380 a</td>
<td>-10 ±10 f</td>
<td>2.38 ±1.93 c,d</td>
</tr>
<tr>
<td>W4</td>
<td>-10 ±70 b,c</td>
<td>60 ±300 a</td>
<td>-10 ±10 g</td>
<td>2.66 ±1.48 d</td>
</tr>
</tbody>
</table>

There were no statistically significant differences (α=.0018) between groups with the same superscript. Table is designed to be read column wise and letters are unique to each element.

IQR, Interquartile range.
FIGURES

Figure 1. Post-processing workflow performed in all the groups before post-polymerization procedures. A, Specimens were removed from the build platform using a spatula. B, Ultrasonic bath (TriClean Ultrasonic Cleaner U-10LHREC; BrandMax) with 99% isopropyl alcohol (IPA) (Isopropyl alcohol 99%; Cumberland Swan).

Figure 2. Box plot of the specimen dimensions (mm) obtained in the different groups tested. A, Length. B, Width. C, Height. D, Volume. D: Dry or non-water submerged polymerization conditions; W: Water-submerged polymerization conditions.