The Impact of Oxygen Concentration on the Postcuring of 3D-Printed Dental Resin

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Purpose: This study investigated the impact of reducing the oxygen concentration via nitrogen injection during the postcuring process of 3D-printed dental materials. ***Materials and Methods:*** Resin specimens for dental crown and bridge (15-mm diameter, both 1-mm and 2-mm heights) were 3D-printed and rinsed. Subsequently, the postcuring process was conducted on nine groups categorized according to atmospheric conditions within the curing device (20% [control], 10%, and 5% oxygen) and curing times (10, 15, and 20 minutes). Surface roughness was measured using a gloss meter. Surface polymerization was confirmed through Fourier-transform infrared spectroscopy (FT-IR) analysis, and the flexural strength and elastic modulus of the specimens were measured using a universal testing machine. Water absorption and solubility were determined according to Inernational Organization for Standardization (ISO) standards. All evaluation criteria were statistically analyzed using one-way ANOVA and Tukey's post hoc test based on oxygen concentration. ***Results:*** The elastic modulus did not show statistically significant differences in all groups. However, compared to the control group, the flexural strength, degree of conversion, and gloss significantly increased in the groups with decreased oxygen concentrations. Conversely, water solubility and water absorption significantly decreased in a few groups with reduced oxygen concentration. ***Conclusions:*** Reducing oxygen concentration through nitrogen injection during the postcuring process of 3D printing enhances the suitability of the dental prosthetic materials. The significant increase in flexural strength can particularly enhance the utility of these materials in dental prosthetics. *Int J Prosthodont 2024;37(suppl):s151–s158. doi: 10.11607/ijp.8919*

According to the Gartner's hype cycle for 3D-printing technology,[1] 3D printing of dental devices has passed through the trough of disillusionment and has entered the slope of enlightenment. This progression has been among the fastest in all medical areas, and 3D-printing technology is already used extensively in the field of dentistry.[2] Surgical implant guides enable predictable surgeries, and 3D-printed prosthetics allow for the rapid and easy creation of esthetically pleasing outputs within short period of time. The use of 3D-printing models also enhances spatial efficiency by enabling the retrieval of cloud data as needed. Consequently, 3D printing is becoming a powerful driver of convenience and efficiency in dental practice.

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Table 1 Details of the Products Used

Product	Manufacturer	Features
Resin: TC-80DP	Graphy	Shade: A2 Component: methacrylate oligomer based on polyurethane resin, phosphine oxides, pigment Purpose: crown and bridge
Printer: Slash 2	UNIZ	3D printer powered by 8.9-in 4K ultra HD LCD light engine 6-core CPU 4-GB RAM Dual-layer resin tank
Postcuring device: Tera Harz Cure	Graphy	UV LED light source 405-nm UV wavelength 200-W output 280,000–mJ/cm^2 UV energy density 1,000–W/cm^2 UV irradiance

Table 2 Experimental Groups

Hardening condition	Postcuring time		
	10 min	15 min	20 min
5% oxygen	O_2 5%;10	O_2 5%;15	O_2 5%;20
10% oxygen	O_2 10%;10	O_2 10%;15	O_2 10%;20
20% oxygen	O_2 20%;10	O_2 20%;15	$O_2$20%;20

Groups were categorized by postcuring time and oxygen concentration.

Many dentists are aware of the diverse applications of 3D printing. However, the implementation of 3D printing is sometimes challenging because of its decreased accuracy relative to milling machines. Accuracy can be broadly categorized into two aspects: precision and trueness.[3] Inconsistencies between printing cycles would make individual products unsuitable for use in clinical dental practice. In the current advanced state of 3D-printing technology, many issues during the printing process have been improved. Therefore, considerations for postprocessing should now be prioritized, including washing, supporter removal, and postcuring.

There has been extensive research on the process of washing residual monomers after printing. Isopropanol (IPA) is commonly used for washing, and variations in washing times and washing equipment have been studied.[4,5] Variations associated with IPA concentrations (99%, 94%, and 92%) have also been reported.[6] Subsequently, nontoxic alternatives have been developed due to the hazards of alcohol and potential harms to human health, and the effectiveness of these new cleaning solutions has been investigated.[7]

Postcuring is also a crucial step in printing. Generally, the degree of curing for 3D-printing outputs is only around 60% to 70%.[8] Therefore, further increasing the degree of curing can significantly enhance strength and durability. Many postcuring techniques and products have been introduced to optimize the postcuring process.[9] To maximize the light for curing, powerful light sources are frequently utilized, or various devices are attached inside the curing chamber to reflect light. However, rapid curing of resin increases the stress due to polymerization shrinkage,[10] suggesting that such practices have a limitation.[10] Therefore, the present study considered an alternative method to increase postcuring performance rather than adjusting the intensity of light.

In other industrial applications, the degree of resin curing can be increased by blocking oxygen.[11] However, less is known about the impact of oxygen concentration in the postcuring process of 3D printing. Herein, it was reasoned that nitrogen injection into the postcuring machine's chamber will reduce the amount of oxygen and enhance efficiency of the postcuring process. Therefore, the present study aimed to determine changes in dental material performance associated with reduced oxygen within an appropriate range of postcuring times for 3D printing resin. The null hypothesis of this study was that the oxygen concentration in the atmosphere does not affect the postcuring of 3D-printed dental materials.

MATERIALS AND METHODS

Materials

This study used 3D-printing resin for crown and bridge applications (TC-80DP, shade A2, Graphy) as the sole material. Specimens were designed in ISO disc form 15-mm diameter; 1-mm or 2-mm height) and produced using a liquid crystal display (LCD) 3D printer (Slash 2, UNIZ). The 1-mm cylinders were used to determine flexural strength, elastic modulus, and degree of conversion, while the 2-mm cylinders were used to determine glossiness, water sorption, and water solubility. All samples were post-cured. All conditions for 3D printing were carried out according to the manufacturer's instructions, and details of the material, printer, and postcuring device are presented in Table 1.

Experimental Groups

Experimental groups were categorized according to the experimental objectives, considering postcuring times and the increased nitrogen concentration (Table 2). The time range included a minimum curing

time of 10 minutes and the recommended curing time of 20 minutes. The experimental procedures are detailed in Fig 1.

Specimen Preparation

The specimens were created as stereolithography (STL) files using computer-aided design (CAD) software (Solidworks) and manufactured as cylinders (15-mm diameter, 2-mm height) following ISO 10477:2020 specifications[12] (see Fig 1) and using an LCD-based 3D printing device. Subsequently, cleaning was performed by immersing the specimens in an ultrasonic cleaner containing 99% IPA for 30 seconds, followed by removing the residual resin using cotton swabs, and then further cleaning with an air gun. The supports were then removed, and the postcuring process was carried out for the nine groups of specimens, with oxygen concentrations of 5%, 10%, and 20% (control groups) and curing times of 10, 15, and 20 minutes, respectively. To prevent additional polymerization from natural light during various experiments, light-blocking opaque containers and aluminum foil were employed.

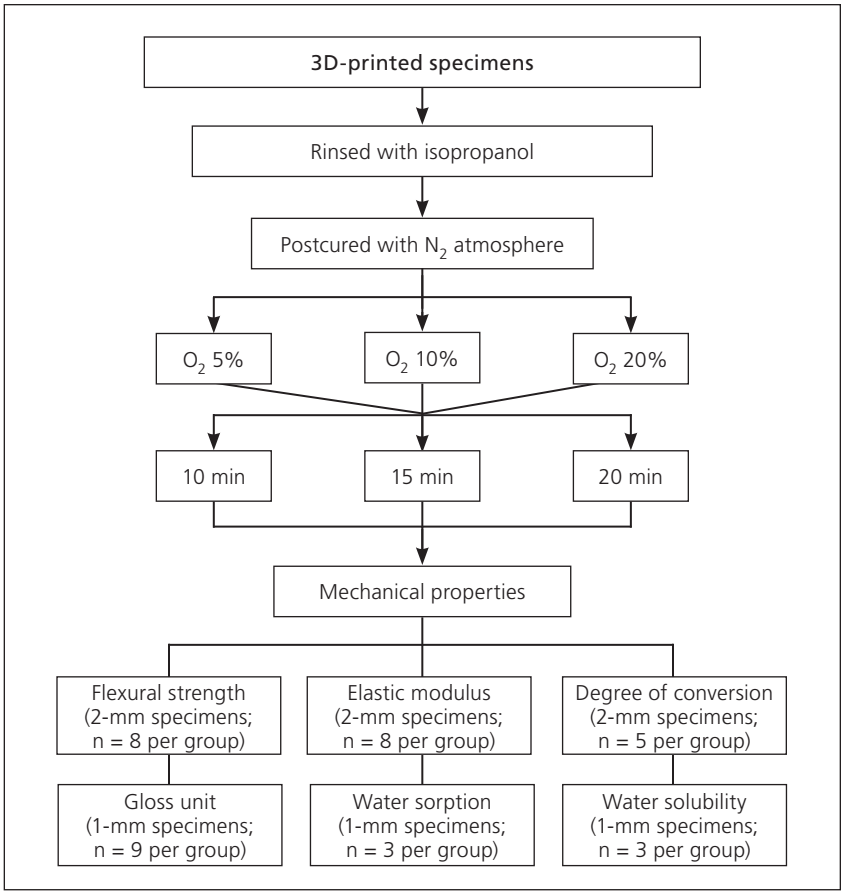

Fig 1 Flowchart of the experimental procedures.

Methods

Postcuring Process

For the postcuring process, five disc-shaped specimens were produced for each group (15-mm diameter, 1-mm and 2-mm heights). The testing procedure followed ISO 10477:2018 standards.[12] The specimens were placed in a desiccator containing dried calcium chloride and maintained at 37° ± 1°C for 24 hours. Then, they were transferred to and kept in another desiccator maintained at 24° ± 1°C for 1 hour, and the specimen weights were measured. Specimens were considered fully dried and ready for measurement when the weight difference was < 0.1 mg (mL). The specimens were then submerged in 15 mL of distilled water and maintained at 37°C ± 1°C for 24 hours. After, the specimens were wiped clean, their weights were measured, and they were placed back into distilled water. This process was repeated every 24 hours until a consistent weight (m2) was achieved, which took 7 days to achieve. Specimens were then removed from the distilled water and air-dried at 37°C ± 1°C until a consistent weight (m3) was attained.

Determining Material Properties

The flexural strength was measured on eight specimens from each group using a universal testing machine (Z010, ZwickRoell). Following ISO 10477:2020 standards,[12] a three-point flexural test was conducted. To determine the maximum flexural strength, a preload of 0.3 N was applied, and the test was carried out at a rate of 1 mm/minute from the center until fracture occurred.

Elastic modulus measurements were performed on 8 specimens per group using the universal testing machine (Z010). According to ISO 10477:2020 standards, a three-point flexural test was conducted with a preload of 0.3 N, and stress-to-strain ratios were calculated in the range of 0.05% to 0.25% strain.[13]

To assess the degree of surface polymerization (degree of conversion), an FT-IR spectrometer (Nicolet Summit, Thermo Fisher Scientific) was used to measure the peaks at 810 nm, which represented C=C bonds, and 1,730 nm, which represented C=O bonds. for the postcured specimens and 1,730 nm for the uncured specimens. These measurements were made on 5 specimens per group. These values were then used in the polymerization conversion formula (Fig 2) to calculate the surface polymerization conversion rate.[14]

$$X\,(\%) = \frac{(A_{810} / A_{1730})_0 - (A_{810} / A_{1730})_t}{(A_{810} / A_{1730})_0} \times 100\%$$

Where $(A_{810} / A_{1730})_0$ and $(A_{810} / A_{1730})_t$ are the relative absorbance of C = C bonds before curing and at a given curing time (t), repectively.

Fig 2 Calculation to determine the degree of conversion (DC).

$$W_{SP}\% = \frac{(m_2 - m_3)}{m_1} \times 100\% \qquad W_{SL}\% = \frac{(m_1 - m_3)}{m_1} \times 100\%$$

Fig 3 Calculations to determine water absorption *(left)* and water solubility *(right)*.

Water absorption and solubility were calculated using the formulas shown in Fig 3 using the m1, m2, and m3 values, which were determined from the experiments.[15]

The gloss units (GUs) were measured using a gloss meter (Novo-Curve, Rhopoint Instruments) with a 2 × 2–mm square measuring area at 60 degrees. Measurements were taken on each specimen, one at the central part of the surface and one at each lateral side, for a total of three measurements per specimen. From the five specimens per group, three specimens were measured for each of the nine groups, resulting in 81 data points for statistical analysis, and nine sets of gloss data (one set per group).

Statistical Analysis

All statistical analyses were performed using SPSS software (version 27, IBM). For flexural strength, elastic modulus, degree of conversion, water solubility, water absorption, and gloss, the assumption of homogeneity of variances was assessed using Levene's test. One-way ANOVA was conducted with postcoding through the Tukey honestly significant difference (HSD) test, using postcuring time (10, 15, and 20 minutes) as the independent variable. All results were tested for significance at the level of $\alpha < .05$.

RESULTS

Flexural Strength

On average, specimens that were postcured for 10 minutes exhibited a strength of 141.21 MPa after being cured in 5% oxygen, 143.73 MPa after being cured in 10% oxygen, and 117.06 MPa after being cured in 20% oxygen. For specimens postcured for 15 minutes, the mean strengths were 146.04 MPa, 145.15 MPa, and 126.21 MPa after being cured in 5%, 10%, and 20% oxygen, respectively. Specimens postcured for 20 minutes displayed mean strengths of 149.64 MPa, 141.30 MPa, and 114.68 MPa after being cured in 5%, 10%, and 20% oxygen, respectively.

When analyzing specimens with fixed curing times but varying oxygen concentrations, a one-way analysis revealed that specimens cured for 10 minutes had no significant difference between the 5% and 10% oxygen concentrations, but both groups differed significantly from the 20% oxygen concentration group. Similarly, for specimens cured for 15 minutes, there was no significant difference between the 5% and 10% oxygen concentrations, but both showed significant differences when compared with the 20% oxygen concentration group. This pattern was consistent for specimens cured for 20 minutes, the same pattern emerged, with no significant difference between the 5% and 10% oxygen concentrations but significant differences were observed when compared to the 20% oxygen concentration group (Fig 4).

Elastic Modulus

On average, specimens postcured for 10 minutes exhibited elastic moduli of 2393.86 MPa after being cured in 5% oxygen, 2255.99 MPa after being cured in 10% oxygen, and 2269.64 MPa after being cured in 20% oxygen. Specimens postcured for 15 minutes showed mean elastic moduli of 2358.04 MPa, 2535.11 MPa, and 2218.44 MPa after being cured in 5%, 10%, and 20% oxygen, respectively. Specimens postcured for 20 minutes displayed mean elastic moduli of 2324.50 MPa, 2216.09 MPa, and 2356.53 MPa after being cured in 5%, 10%, and 20% oxygen, respectively.

Upon performing a one-way analysis for each curing time (10, 15, and 20 minutes), grouping by the three distinct postcuring oxygen concentrations, no significant differences were noted across the groups (Fig 5).

Degree of Conversion

On average, specimens postcured for 10 minutes exhibited a degree of conversion (DC) of 98.68% after being cured in 5% oxygen, 96.19% after being cured in 10% oxygen, and 96.23% after being cured in 20% oxygen. Specimens postcured for 15 minutes showed mean DCs of 99.32%, 98.68%, and 98.43% after being cured in 5%,10%, and 20% oxygen, respectively. Specimens postcured for 20 minutes displayed mean DCs of 99.48%, 99.35%, and 98.82% after being cured in 5%, 10%, and 20% oxygen, respectively. One-way analysis indicated no significant differences in terms of DC between the 10% and 20% oxygen concentration groups for specimens postcured for 10 minutes. However, both of these groups showed a significant difference when compared with the 5% oxygen concentration group. For specimens postcured for 15 minutes, significant differences were observed between each of the oxygen concentration conditions,

with the 5% oxygen concentration group exhibiting the highest DC. For specimens postcured for 20 minutes, there were no significant differences between the 5% and 10% oxygen concentration groups. However, both groups showed significant differences from the 20% oxygen concentration group in the DC measurements (Fig 6).

Water Absorption

All nine experimental groups satisfied the specified requirements outlined in ISO 10477[12] (≤ 40 μg/mm^3). On average, specimens postcured for 10 minutes yielded water absorption measurements of 18.963 μg/mm^3 after being cured in 5% oxygen, 19.645 μg/mm^3 after being cured in 10% oxygen, and 19.829 μg/mm^3 after being cured in 20% oxygen. Specimens postcured for 15 minutes yielded mean measurements of 20.335 μg/mm^3, 20.534 μg/mm^3, and 20.421 μg/mm^3 after being cured in 5%, 10%, and 20% oxygen, respectively. Specimens postcured for 20 minutes yielded mean measurements of 20.019 μg/mm^3, 20.673 μg/mm^3, and 20.689 μg/mm^3 after being cured in 5%, 10%, and 20% oxygen, respectively. One-way analysis indicated that the only significant difference in water absorption was between the 5% and 20% oxygen groups cured for 10 minutes (Fig 7).

Water Solubility

Water solubility was measured for five specimens in each group, and all nine experimental groups satisfied the requirements specified in ISO 10477[12] (≤ 7.5 μg/mm^3). On average, specimens postcured for 10 minutes yielded water solubility measurements of 0.074 μg/mm^3 after being cured in 5% oxygen, 0.288 μg/mm^3 after being cured in 10% oxygen, and 0.656 μg/mm^3 after being cured in 20% oxygen. Specimens postcured for 15 minutes yielded mean measurements of 0.686 μg/mm^3, 0.998 μg/mm^3,

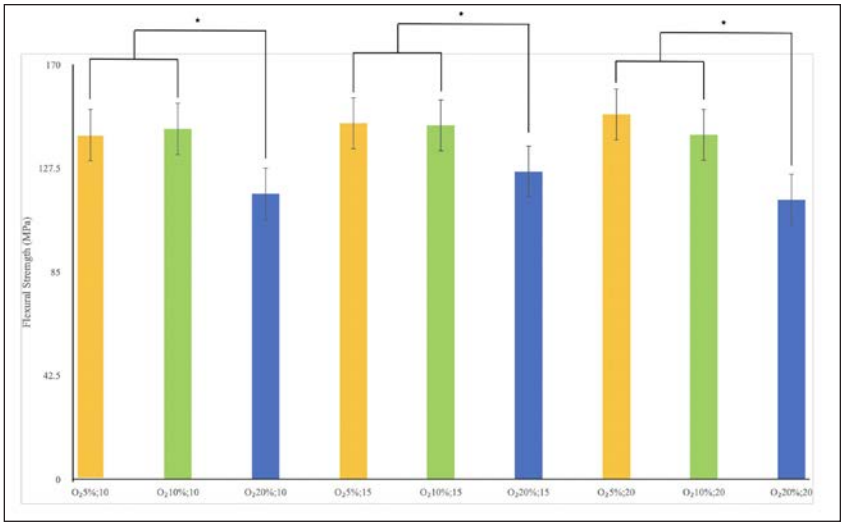

Fig 4 Flexural strength of each group and their statistical relation.

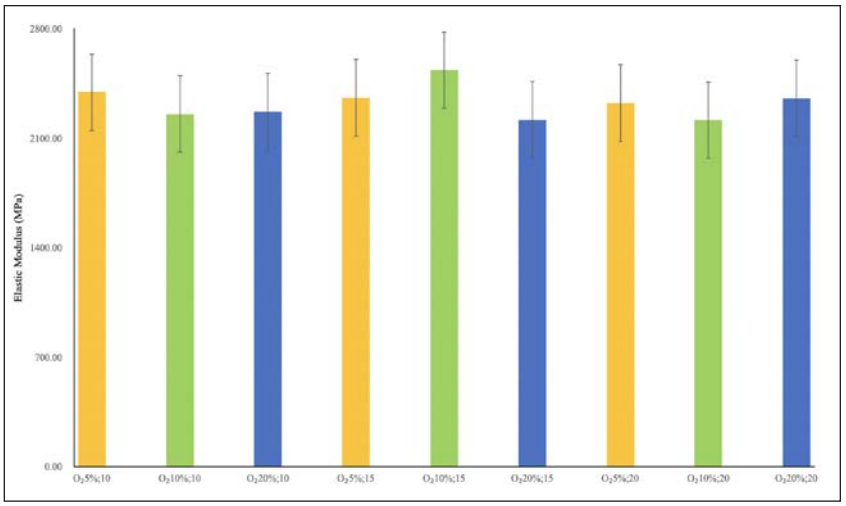

Fig 5 Elastic modulus of each group and their statistical relation.

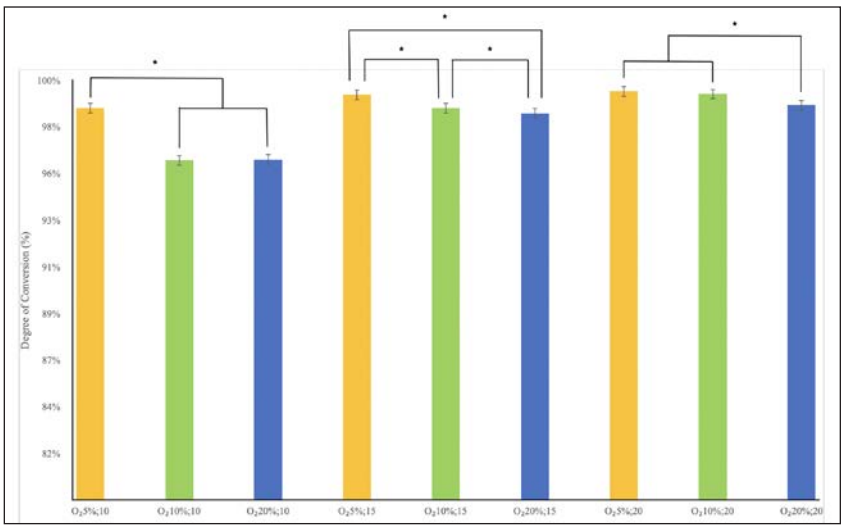

Fig 6 Degree of conversion for each group and their statistical relation.

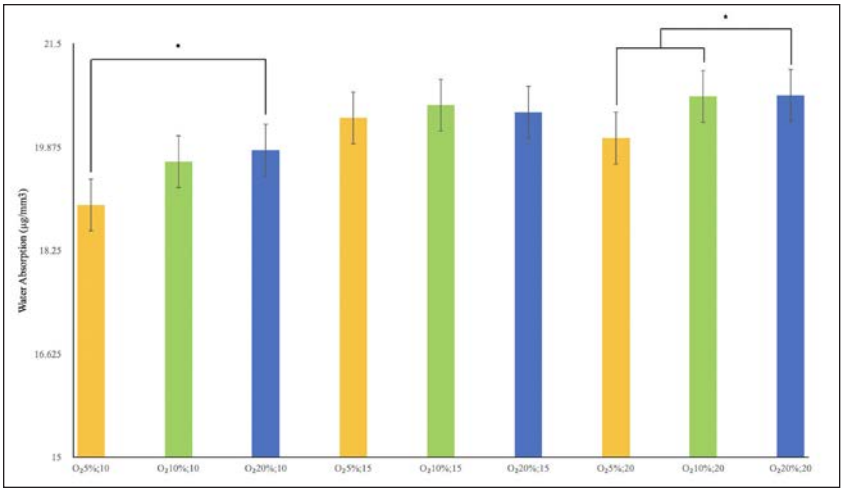

Fig 7 Water absorption of each group and their statistical relation.

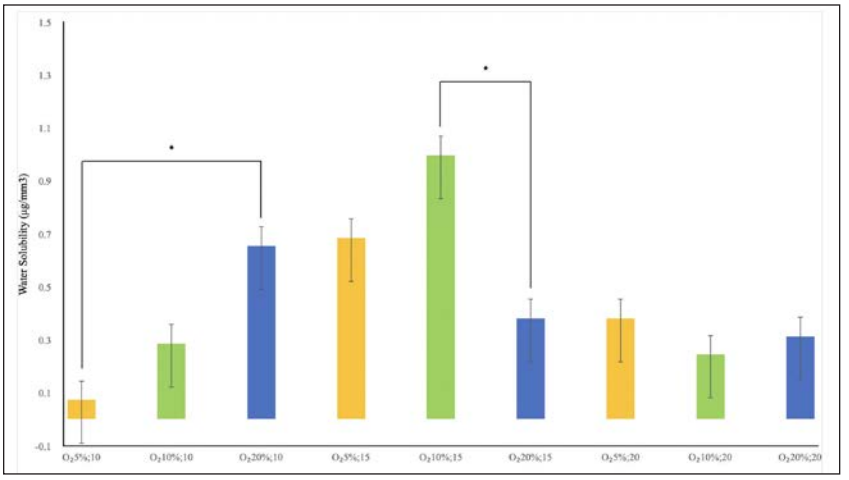

Fig 8 Water solubility of each group and their statistical relation.

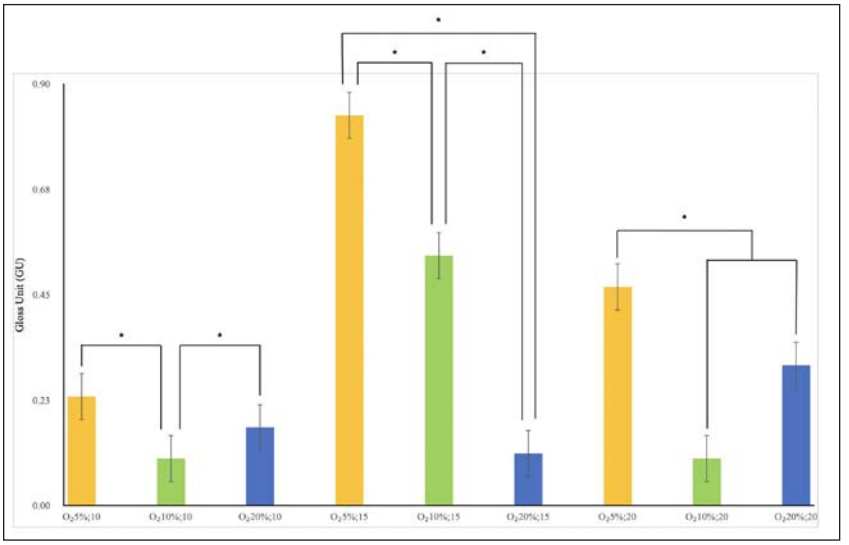

Fig 9 Gloss of each group and their statistical relation.

and 0.383 μg/mm^3 after being cured in 5%, 10%, and 20% oxygen, respectively. Specimens postcured for 20 minutes yielded mean measurements of 0.246 μg/mm^3, 0.246 μg/mm^3, and 0.315 μg/mm^3 after being cured in 5%,10%, and 20% oxygen, respectively. One-way analysis revealed significant differences between the 5% and 20% oxygen groups after 10 minutes of curing and between the 10% and 20% oxygen groups after 15 minutes of curing (Fig 8).

Gloss

On average, specimens postcured for 10 minutes yielded gloss measurements of 0.23 GU after being cured in 5% oxygen, 0.10 GU after being cured in 10% oxygen, and 0.17 GU after being cured in 20% oxygen. Specimens postcured for 15 minutes yielded mean gloss measurements of 0.83 GU, 0.53 GU, and 0.11 GU after being cured in 5%, 10%, and 20% oxygen, respectively. Specimens postcured for 20 minutes yielded mean gloss measurements of 0.47 GU, 0.10 GU, and 0.30 GU after being cured in 5%, 10%, and 20% oxygen, respectively.

One-way analysis indicated significant differences between the 5% and 10% oxygen groups, as well as between the 10% and 20% oxygen groups, when the curing time was 10 minutes. For specimens cured for 15 minutes with varying oxygen concentrations, significant differences were observed among all groups. For specimens cured for 20 minutes with varying oxygen concentrations, there was no significant difference between the 10% and 20% oxygen groups, but there were significant differences between the 5% group and the other groups (Fig 9).

DISCUSSION

This study aimed to investigate the association of oxygen concentration and curing time during the

postcuring of 3D-printed resin specimens as it relates to flexural strength, elastic modulus, degree of conversion, water absorption, water solubility, and glossiness. For the elastic modulus measurements, there was no statistically significant variation, and the null hypothesis was not rejected for this variable. However, the null hypothesis was rejected for the other experimental variables based on the following results: *(1)* flexural strength increased when the oxygen concentration was reduced; *(2)* the surface DC increased as the curing time was prolonged and as the oxygen concentration was decreased; *(3)* water absorption was lowest when the curing time was shortest and the oxygen concentration was lowest, with water solubility exhibiting a similar trend; and *(4)* gloss measurements were highest when the oxygen concentration was 5%.

On average, a person experiences over 11.7 minutes of tooth contact per day, and during a single meal, there can be approximately 600 to 800 occlusal contacts.[16] For teeth with crown resin applications, they are subjected to pressures from occlusion, bruxism, or habits like tooth grinding, which can lead to wear and damage.[17,18] Consequently, the expected lifespan of resin crowns averages around 6.8 years, with the actual duration depending on maintenance practices.[19] Therefore, it is advantageous for resin prosthetic materials to exhibit high flexural strength, which represents the force per unit area at which a specimen fractures when subjected to bending. Achieving the maximum flexural strength within the range below the flexural strength of natural teeth is desirable.[20] In this study, the specimens exhibited a mean flexural strength of 149.64 MPa, which is below the flexural strength range of natural teeth (160 ± 22 MPa). This suggests that the resin crowns can safely endure occlusal forces without fracturing.[21] Furthermore, because reduced oxygen concentrations during postcuring resulted in increased flexural strength, it is recommended to adjust the oxygen concentration to < 10% when using these materials in clinical practice. In contrast, the elastic modulus experiments did not reveal significant differences among the nine groups, regardless of variations in oxygen concentration or curing time. This aligns with previous research on 3D-printed occlusal splint resin using LCD and digital light processing printers, where varied postcuring oxygen concentration during postcuring yielded similar results.[22]

Prothetic resin materials for dental use rely on sufficient polymerization and minimal residual monomers to avoid allergic reactions or cytotoxicity.[20,23] Previous research, including that conducted by Reymus and others, has shown that under N2 conditions, postcuring polymerization conversion rates are more efficient compared with those achieved under atmospheric conditions when using ultraviolet (UV) or LED curing methods.[24] Therefore, the present study measured polymerization conversion rates to investigate how they change under varying postcuring conditions. It was observed that as postcuring time increased and oxygen concentration decreased, the polymerization conversion rate increased.

Water absorption and solubility are essential factors to consider when manufacturing 3D-printed resin prostheses. Prolonged water absorption by the resin can lead to internal stress formation and can generate cracks, potentially adversely affecting the properties of the prosthesis.[25] Additionally, when resin monomers dissolve in water, they can interact with fibroblasts and cause cytotoxicity, underscoring the importance of low solubility.[26] ISO 10477[12] provides guidelines for acceptable levels of water solubility and water absorption, and the 3D-printed resin used herein met these criteria.

One reason to choose resin crowns is their ability to closely match the color of natural teeth. Good esthetics are crucial, especially in the anterior region of the mouth. To optimize dental esthetics, considerations extend beyond selecting the appropriate color; factors such as gloss should also align with the natural dentition. Rocha et al used a natural tooth with a surface gloss of 80 GU as a reference and found that half of the observers in their study could perceive a gloss difference of 6.4 GU.[27] The present study examined the gloss of 3D-printed resin after postcuring, with conditions of 5% oxygen and 15 minutes of curing yielding the highest surface gloss. Notably, all specimens fell significantly below the 80 GU benchmark, with some values below 2.0 GU. As a result, future research should focus on conducting experiments based on increased gloss values achieved through short-duration polishing of 3D-printed resin.[28,29]

The present study aimed to investigate the impact of varying oxygen concentrations during postcuring on the properties of 3D-printed permanent resin crowns. The study holds significance, as it provided evidence for the use of 3D-printing device for dental purposes, which is currently a subject of active research and development in the field of dentistry. However, it is important to note certain limitations, such as the relatively small number of specimens, which may not have allowed for a definitive assessment of property differences. Additionally, 3D-printed prosthetics are intended for use within the oral environment, making it crucial to observe and analyze changes occurring in conditions similar to or mimicking the oral cavity. However, the present experiments employed water absorption and solubility tests, using water as a surrogate for saliva. Considering that the oral environment can be more complex than the experimental conditions (factors like pH, salivary flow rate, and viscosity), future studies should analyze clinical data from actual patients. Further, various types of resins used in 3D printing—particularly those employing UV-curable photoinitiators, such as BAPO (biacylphosphine oxide), TPO (monoacylphosphine oxide), CQ (camphorquinone),

and DMAEMA (2-[dimethylamino]ethyl methacrylate)—can have different effects on properties such as polymerization rate, degree of conversion, strength, and elasticity.[30–32] Therefore, additional research is needed to explore how different types of resins may affect each property under varying postcuring oxygen concentrations and times. Despite these limitations, the present study has demonstrated that different oxygen concentrations and postcuring times significantly influence the properties of 3D-printed resin, underscoring their meaningful potential for clinical use.

CONCLUSIONS

Within the limitations of this study, the following can be concluded: *(1)* The flexural strength of 3D-printed resin increases when the material is postcured with < 10% oxygen; and *(2)* postcuring with ≤ 5% oxygen increases the degree of conversion for 3D-printed resin.

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