THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Evaluating the conversion degree of interim restorative materials produced by different 3-dimensional printer technologies

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ABSTRACT

Statement of problem. Three-dimensional (3D) printers are a relatively new technology, but the degree of conversion (DC) of the resin specimens produced by using this method is currently unknown. However, the DC of resin interim restorative materials is critical for their biocompatibility and physical properties.

Purpose. The purpose of this in vitro study was to evaluate the DC of interim restorative materials produced by using different 3D printer technologies and compare them with conventionally manufactured polymethyl methacrylate.

Material and methods. Stereolithography, digital light processing, and liquid crystal display 3D printers were used as experimental groups, and a conventional (C) method was used as the control. Five different 3D printers (DWS Systems, Formlabs [FL], Asiga, Mega, and Vega) were included. The 3D printed specimens were designed in a rectangular prism geometry (10×4×2.5 mm) by using a computer-aided design software program (Materialise 3-matic) and printed with a layer thickness of 50 μm in the horizontal direction (n=15). Fourier transform infrared spectroscopy (FT-IR) spectra were measured in 3 steps: the liquid state of the resins, after washing with 99% isopropanol, and after final polymerization. For the C method, FT-IR spectra were assessed in 2 steps: immediately after mixing the liquid and powder and after polymerization. Statistical analysis of the data was performed with 1-way ANOVA followed by the post hoc Tukey honestly significant difference (HSD) test (α=.05).

Results. There was no statistically significant difference in DC values between the 3D printed groups ($P>.05$). There was a statistically significant difference only between FL and the C in terms of DC ($P=.042$).

Conclusions. Three-dimensionally printed interim resin materials found comparable results with those of the C group. The DC was not affected by different 3D printing technologies. (J Prosthet Dent 2023;130:654.e1-e6)

Although frequently used, manufacturing interim restorations conventionally is technique-sensitive, and errors may adversely affect the restoration.[1] Therefore, the use of digital fabrication with computer-aided design and computer-aided manufacturing (CAD-CAM) has become widespread and is a promising alternative to conventionally manufactured interim prostheses.[2,3] CAD-CAM technology can make the procedure shorter and more comfortable.[4,5]

The digital workflow typically consists of data collection, design, and manufacturing. Intraoral data is digitized by using either an intraoral scanner or indirectly with a laboratory scanner, and the prosthesis is designed with a dental CAD software program.[6,7] The restoration is manufactured by either subtractive milling[8–10] or additive manufacturing (AM),[11–13] a promising contemporary production method in dentistry.[13] The American Society of Testing and Materials (ASTM) defines AM as the process of creating the final object from a 3D design by

Supported by the Çukurova University Scientific Investigation Project (project TDH-2020-13272), Adana, Turkey as a specialty thesis in Prosthodontics.
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
Presented as an oral presentation at the International Dental Congress on Additive Manufacturing (IDCAM) (Çukurova University, Faculty of Dentistry), Adana, Turkey, March 18 to 19, 2022.
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Clinical Implications

Low DC indicates high residual monomer, which leads to decreased biocompatibility, Therefore, evaluating the DC of different interim restorative materials is important. The DC of 3D printing technologies is at least as reliable as the conventional method for manufacturing interim restorations.

adding one layer on top of another.[14] When AM is used, waste material is reduced compared with subtractive manufacturing. The final object is fabricated with a high precision regardless of the object geometry[15–17] and interim restorations are typically produced from liquid polymeric resins via vat photopolymerization.[18]

Vat photopolymerization technologies include stereolithography (SLA), digital light processing (DLP), and liquid crystal display (LCD).[19] In the SLA system, a laser beam is reflected by micromirrors called galvanometers, and the entire layer is scanned point by point. When the scanning of a layer is finished, the production platform moves up by 1 layer thickness. The same laser scanning process is repeated with point irradiation for the next layer, and it is repeated hundreds of times until the final object is completed.[20–22] DLP uses a projector instead of a laser beam to polymerize the liquid resin. The light is simultaneously reflected by thousands of micromirrors to the entire layer of interest which is polymerized in a single irradiation step. Because the entire surface is polymerized simultaneously, the production time is shorter than with SLA.[23,24] Similar to DLP technology, LCD polymerizes the entire layer in a single irradiation step. The main difference between DLP and LCD is that the light is reflected from an LCD screen instead of a projector.[25]

Regardless of the technology used, the 3D-printed objects are subjected to 2-stage postprocessing as recommended by the manufacturers: washing the 3D-printed object with pure alcohol to remove the residual unpolymerized resin immediately after printing and by using heat and ultraviolet (UV) light to complete the polymerization process and achieve the final mechanical and biological properties.[26]

The common feature of SLA, DLP, and LCD technologies is the use of UV light-sensitive liquid polymeric resin. The chemical composition of these photopolymers is predominantly methacrylate monomer, similar to the interim fixed prosthodontic materials used in the conventional (C) method.[27] In addition to methacrylate monomers, photoinitiators, inhibitors, and inorganic fillers (nanoceramic particles and pigmentation agents) are included.[28] The liquid resin contains nanoceramic particles such as silica, alumina, zirconia, and hydroxyapatite.[29] After the irradiation, these materials form cross-linked polymeric systems by photopolymerization free radical reactions to transform from a liquid to a solid state. Polymerization can be quantitatively expressed as the degree of conversion (DC), the ability of a monomer to transform into a polymer.[30] A high DC corresponds to a decreased amount of residual monomer, provides higher biocompatibility, increased physical properties including higher mechanical properties, and reduced water absorption and color change.[28–31]

The DC of 3D-printed objects depends on factors that include the photosensitive resin composition, the layer thickness, the printing time, the speed, the light source used in the final polymerization, and the final polymerization temperature and time.[28] Studies investigating factors affecting DC are sparse, and these studies have been limited to the effect of postprocessing procedures on the final product.[32–36] The authors are unaware of a previous study investigating the effect of different 3D printing technologies on DC.

The purpose of this in vitro study was to evaluate the DC of interim restorative materials produced with different 3D printer technologies and compare them with the C method. The null hypothesis was that the use of different 3D printer technologies would not affect the DC of interim restorative materials.

MATERIAL AND METHODS

Rectangular 10×4×2.5-mm prism specimens were designed with a CAD software program (Materialise 3-matic; Materialise) and printed with 5 different 3D printers by using SLA (XFAB 2500PD; DWS Systems [DWS], Form 3B; Formlabs [FL]), DLP (Max UV; Asiga [AS], Mega [MG]; Dentafab), and LCD (Vega; Dentafab) technologies. The control group was specimens made with autopolymerizing polymethyl methacrylate (PMMA) (Imident; Imicryl) (Table 1). Standards for evaluating the DC of polymer-based fixed prosthodontic materials have not been formulated by the International Organization for Standardization or by ASTM International. Therefore, the specimens were designed according to a previous study.[32]

The sample size (n=15) was determined from a power analysis with an effect size of .05, (α=.05, and power=0.95) by using a software program (G*Power; Heinrich Heine University Düsseldorf). The 3D-printed specimens (n=15) were manufactured in the horizontal direction (0 degree) with a layer thickness of 50 μm based on a pilot study and considering that horizontally manufactured specimens have higher mechanical strength than vertically manufactured ones.[37] The 3D printed specimens were washed with 99% isopropanol as suggested by the manufacturers. Fourier transform infrared spectroscopy (FT-IR) (FT/IR 6700; Jasco) measurements were made 24 hours after

Table 1. Printer technologies and printing resins used

Technology	Manufacturer	Light Source	Model	Material
Stereolithography	DWS Systems	Laser: Solid State BlueEdge	XFAB 2500PD	Temporis
Stereolithography	Formlabs	Laser: 250 mW	Form 3	Temporary C&B
Digital Light Processing	Asiga	Projector: 385 nm	Max	GC Temp Print
Digital Light Processing	Dentafab Mega	Projector: 385 nm	Mega	Power Resin Temp
Liquid Crystal Display	Dentafab Vega	LCD Led: 385 nm	Vega	Power Resin Temp
Conventional	Imicryl	Autopolymerized	Imident	Imident

specimen production. To prevent additional polymerization, the specimens were stored in dry and opaque containers.

The control autopolymerizing PMMA (Imident; Imicryl) specimens were made from printed castable wax resin (Castable wax, Photopolymer resin; Form 3B; FL) patterns printed from the same CAD data. The castable wax resin specimens were embedded in a silicone impression material (Presigum, President Dental) to prepare a mold. Imident powder (24 g) was mixed with the liquid (10 mL) for 1 minute in accordance with the manufacturer's instructions, and the mixture was poured into the mold. Finger pressure was applied to a glass cover during the polymerization.

The order of the tests for all specimens was randomized with a software program (Research Randomizer; Social Psychology Network) to avoid bias. DC measurements were made by using FT-IR with the attenuated total reflection (ATR) accessory. FT-IR spectra were recorded in transmittance mode with 16 times scanning at 4 cm^{-1} resolution and wavenumber range of 4000 to 450 cm^{-1}. The peak areas of vibration bands of C═C bonds observed at 1640 cm^{-1} and the peak areas of vibrational bands of the carbonyl group (C═O), observed at 1725 cm^{-1}, were calculated. The following formula was used to measure the DC:

$$DC(\%) = 100 \times \left[1 - \frac{(1640cm^{-1} \div 1725cm^{-1})polymerized}{(1640cm^{-1} \div 1725cm^{-1})unpolymerized}\right].$$

Measurements for calculating the DC of each 3D-printed group were made in 3 stages. The unpolymerized liquid resin was made by dropping it onto the ATR crystal (n=15). This spectrum was used in the formula to calculate the DC print and DC values. Then, spectra of all 3D-printed specimens were obtained after washing with 99% isopropanol, and DC print values were calculated. The DC print value represents the initial polymerization inside the 3D printer. Finally, the spectra were obtained after postpolymerization and DC values were calculated. The DC value refers to the final polymerization achieved after the postpolymerization procedures. ΔDC values were calculated based on the difference between DC and DC print values (Fig. 1). In this way, only the effect of the postpolymerization process on the DC of the final product was determined.

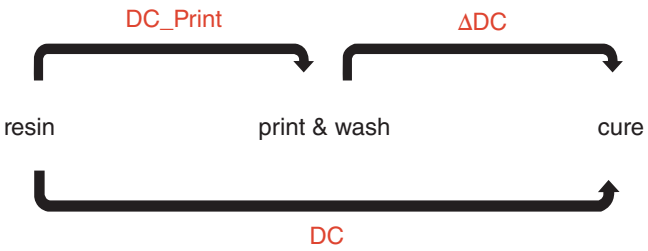

Figure 1. Explanation chart for degree of conversion (DC) values.

Measurements of the control group were made in 2 stages. The prepolymerization spectra of the autopolymerizing PMMA materials were made immediately after mixing the powder and liquid for 1 minute. Then, the spectra of the solid specimens were obtained, and DC values were calculated.

Statistical analysis was performed with 1-way ANOVA followed by the post hoc Tukey HSD test (α=.05). All statistical tests were performed with a statistical software program (IBM SPSS Statistics, v25.0.0.1; IBM Corp).

RESULTS

DC print, DC, and ΔDC values are given in Table 2. The highest DC print value was 64.8% in AS, and the lowest was 45.9% in MG. In general, SLA printers resulted in a higher DC print value. DWS and FL in terms of DC print were statistically similar (P=.811). DC print values of the 2 different DLP printers, AS, and MG were significantly different (P=.001) (Fig. 2A).

The highest DC value was 86.9% in FL, and the lowest was 76.1% in the C group. The DC values were statistically similar between the 3D printed groups (P>.05). In general, specimens obtained with 3D printers found a DC value comparable with that of the C method, with a statistically significant difference only between FL and C in terms of DC value (P=.042) (Fig. 2B).

The highest ΔDC value was 37.4% in MG, and the lowest was 17.9% in AS. The ΔDC between both MG and DWS (P=.001) and MG and FL (P=.002) was statistically different. The ΔDC between MG and Vega (VG) was statistically similar (P=.754) (Fig. 2C).

Table 2. Mean ±standard deviation values of groups

Technology	Manufacturer	DC Print	DC	ΔDC
Stereolithography	DWS Systems	60.9 ±7.8^{a}	81.5 ±7.8^{AB}	20.6 ±12.8^{x}
Stereolithography	Formlabs	63.7 ±4.3^{a}	86.9 ±7.1^{A}	23.1 ±7.2^{xy}
Digital Light Processing	Asiga	64.8 ±4^{a}	82.7 ±10.8^{AB}	17.9 ±10^{x}
Digital Light Processing	Dentafab Mega	45.9 ±5.9^{b}	83.3 ±5.7^{AB}	37.4 ±7.2^{z}
Liquid Crystal Display	Dentafab Vega	49.3 ±8.6^{a}	81.9 ±4.4^{AB}	32.6 ±11^{yz}
Conventional	Imicryl		76.1 ±9.9^{B}	

DC, degree of conversion. *Different letters show significant differences between groups ($P<.05$).

DISCUSSION

This in vitro study was carried out to assess the DC of interim restorative materials produced by using different 3D printing technologies (SLA, DLP, and LCD). The null hypothesis that by using various 3D printing methods would not affect the DC of interim restorative materials was not rejected.

DC has typically been calculated by comparing the peak sizes of the vibrational bands of the respective bonds in the FT-IR spectra obtained pre- and postpolymerization of the materials. As the blocks used in the subtractive milling method are provided as polymerized, prepolymerization could not be measured. Therefore, the subtractive milling process was excluded from the study.

The study was strengthened by using 3 different 3D printing (SLA, DLP, and LCD) technologies. In this way, the study aimed to investigate the DC between different brands that use the same technology. The statistically significant difference between the DC print values of DLP 3D printers shows that DC is affected not only by the technology but also by the parameters that are specific to the 3D printers.

All measurements of the DC in this in vitro study were made by using FT-IR spectroscopy with an ATR accessory, which has been commonly used for calculating the DC of polymerizable dental materials.[30,38] Raman spectroscopy, which is based on the scattering principle of the laser beam, has also been recently used in DC calculations.[32,39] Because the peaks of the bonds used in the DC calculations show nonpolar properties, it could be observed intensely with Raman spectroscopy[39]; however, the method is expensive. As FT-IR spectroscopy has been commonly preferred for calculating DC, it was selected to compare the results with those of previous studies.

The peaks associated with the aromatic phenyl ring at 1610 cm^{-1} could not be observed in the spectroscopy for all groups evaluated. Therefore, peaks of C═O carbonyl bonds at 1725 cm^{-1} were used as a reference in DC calculation. The same peak areas have been used previously to calculate the DC of polymeric materials.[40–42]

Isopropanol has been the most commonly used alcohol for washing 3D printed objects. All the specimens in the present study were washed with 99% isopropanol, which is suitable for 3D printing.

The DC print value represents the initial polymerization that occurs within the 3D printer. The DC print values obtained in the present study were relatively higher in the SLA printers (DWS and FL), possibly because polymerization in SLA printers occurs by reflecting the laser beam pointwise by using a galvanometer. Although AS has DLP technology, it has shown a DC print value comparable with those of SLA printers. This similarity could be ascribed to the power of the projector used in AS or the photo-initiator in the resin. Although AS and MG use the same technology, the difference between DC print values might be because of differences in the projectors used by the printers or the use of different resins. As the projectors of DLP printers require periodic maintenance, this might have been neglected in the MG. Another possibility is that the projector in MG may not be powerful enough to activate the initiator in the photosensitive liquid resin used. MG and VG found comparable DC print values. Although the technologies of these 2 printers differ, the similar DC print values may have been because the same resin was used. However, the DC print values of these 2 groups were relatively lower than those of the other groups.

DC value represents the final polymerization achieved after postpolymerization procedures involving washing and polymerization. For the material to be stable in the oral environment and exhibit sufficient mechanical properties, a high DC value is required. Ferracane et al[43] assessed the FT-IR analysis for restorative materials and concluded that a DC value of at least 55% is clinically acceptable. In terms of DC, 3D printing technologies found comparable values with those of a C method used routinely in clinical practice for many years. The DC values obtained in the current study found that the evaluated materials used for the 3D printers are suitable for clinical practice.

The ΔDC value is the difference between DC print and DC values, expressing the effect of the postpolymerization process on the polymerization degree of the final product (Fig. 1). In the current in vitro study, MG and VG were produced by using the same resin and postpolymerization method. The lack of a statistically significant difference in the ΔDC values between these 2 groups can be attributed to the fact that when the postpolymerization method and the resin are the same,

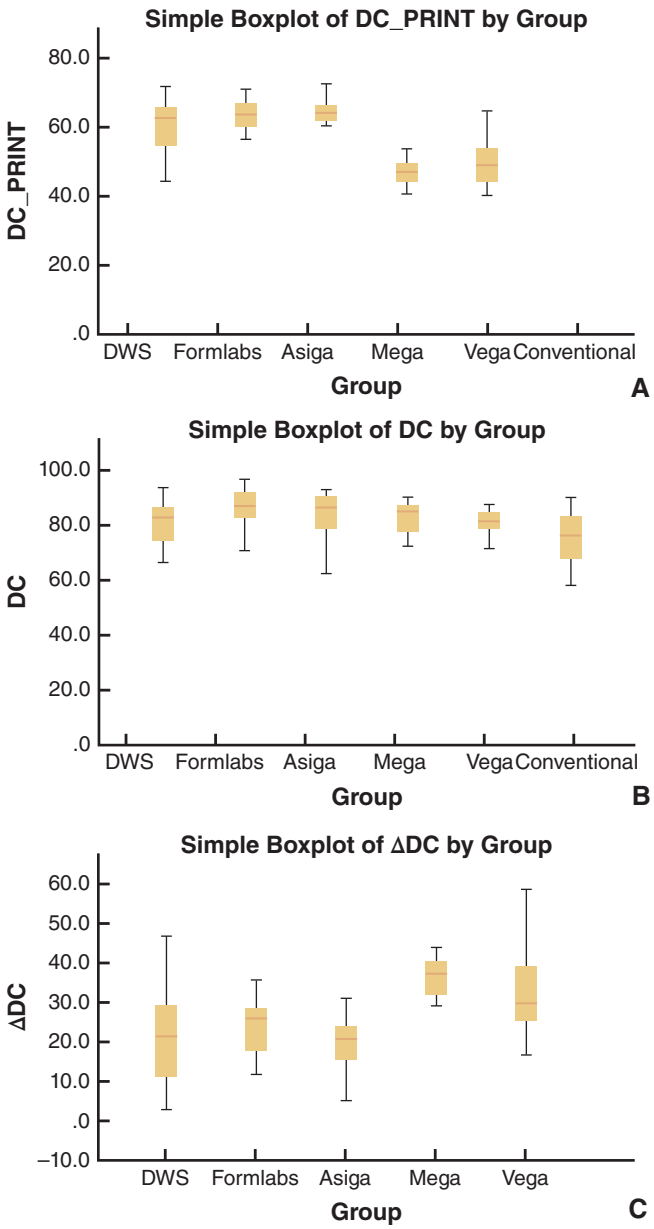

Figure 2. Conversion degree values of groups. A, DC Print values of groups. B, DC values of groups. C, ΔDC values of groups. DC, degree of conversion; ΔDC, difference between DC and DC print values.

the technology used has less effect on the final polymerization. Reymus et al[32] reported that the final polymerization method and the print layer thickness made a difference in ΔDC.

In the present study, the ΔDC value was found to be low in groups with high DC print value and vice versa. However, the final DC was not affected by these factors, and the revealed outcomes did not significantly differ. Wu et al[40] reported that the materials with low DC print value will have a higher ΔDC value, which causes increased polymerization shrinkage from the printed wash stage to the polymerization stage. These data show that the small ΔDC value is important for the dimensional accuracy of the final product. However, Wu et al made the postpolymerization irradiation from a single direction and perpendicular to the printing direction. The UV-induced bending in the final product may be because of a nonuniform stress field in the material because the postpolymerization was done from a single direction. Postpolymerization irradiation should be performed on all surfaces of the final product, and future research should validate the effect of postpolymerization on dimensional accuracy.

The final polymerization procedures used in the present study were made according to each manufacturer's instructions. Similarly, because of the closed system of 3D printers, the photosensitive liquid resin used in all groups was not constant. Although this situation does not make a difference between DC values, which parameter causes the difference between the ΔDC values requires investigation.

Limitations of the study included the impossibility of using the same photosensitive liquid resin in all groups because of the closed system of 3D printers. In addition, the final polymerization processes were carried out in accordance with the manufacturer's instructions. This prevented a standard procedure from being applied in the final polymerization process. Studies investigating the effect of final polymerization procedures on the DC found their importance to DC.

Tahayeri et al[44] evaluated the DC of the specimens produced by using interim restorative materials manufactured with only one 3D printer. The spectrum was taken at every 50 μm, and a heterogeneous polymerization pattern was observed throughout the specimen. DC measurements by ATR-FT-IR spectroscopy were limited to specimen surfaces. The homogeneity of DC throughout the final product is critical for adequate biological and mechanical properties. Further research should be conducted to determine whether the DC is homogeneous in all layers of the final product.

CONCLUSIONS

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

1. Interim fixed prosthodontic materials manufactured by using different 3D printing technologies are suitable for clinical use.
2. Three-dimensional printers have shown similar results to those of the C method that has been used for many years.
3. Final polymerization is necessary because the polymerization inside 3D printers does not always achieve sufficient conversion.

4. Although the DC was not affected by different 3D printing technologies, the DC print and the ΔDC values had significant differences.
5. It is impossible to disentangle the effects of the method of printing and the manufacturer.

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Ebru Sürer: Validation, Investigation, Resources, Data curation, Writing – original draft, Funding acquisition. **Mehmet Ünal:** Investigation, Resources, Data curation. **Esma Başak Gül Aygün:** Conceptualization, Methodology, Writing – review and editing, Visualization, Supervision, Project administration. **Yurdanur Ucar:** Conceptualization, Methodology, Formal analysis, Writing – review and editing, Visualization, Supervision, Funding acquisition.

https://doi.org/10.1016/j.prosdent.2023.07.001