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## ARTICLE IN PRESS



# **RESEARCH AND EDUCATION**

# Effects of print orientation and artificial aging on the flexural strength and flexural modulus of 3D printed restorative resin materials

Shaymaa Mudhaffer,<sup>a</sup> Julfikar Haider,<sup>b</sup> Julian Satterthwaite,<sup>c</sup> and Nick Silikas<sup>d</sup>

# ABSTRACT

**Statement of problem.** The integration of computer-aided design and computer-aided manufacture (CAD-CAM) technology has revolutionized restorative dentistry, offering both additive and subtractive manufacturing methods. Despite extensive research on 3-dimensionally (3D) printed materials, uncertainties remain regarding the impact of print orientation on their mechanical properties, especially for definitive resin materials, necessitating further investigation to ensure clinical efficacy.

**Purpose.** The purpose of this in vitro study was to investigate the influence of print orientation and artificial aging on the flexural strength (FS) and flexural modulus (FM) of 3D printed resin materials indicated for definitive and interim restorations.

**Material and methods.** Specimens (2×2×25 mm) were additively manufactured in 3 orientations (0, 45, and 90 degrees) using five 3D printed resins: VarseoSmile Crownplus (VCP), Crowntec (CT), Nextdent CB MFH (ND), Dima CB temp (DT), and GC temp print (GC). A DLP 3D printer (ASIGA MAX UV) was used with postprocessing parameters as per the manufacturer recommendations. FS and FM were tested after storage in distilled water (DW) and artificial saliva (AS) for 24 hours, 1 month, and 3 months at 37 °C. Additional 2×2×16-mm specimens printed at 90 degrees were compared with the milled materials Lava Ultimate (LU) and Telio CAD (TC) after 24 hours of storage in AS at 37 °C (n=10). Measurements were conducted using a universal testing machine (Z020; Zwick/Roell) following the International Organization for Standardization (ISO) 4049 standard. Multiple way ANOVA, 1-way ANOVA, and Tukey HSD post hoc tests ( $\alpha$ =.05) were used to analyze the data.

**Results.** Print orientation significantly influenced the FS and FM of 3D printed resin materials, with the 90-degree orientation exhibiting superior mechanical properties (P<.05). Definitive resins (CT and VCP) exhibited higher FS and FM compared with interim resins (ND, DT, GC) at all time points (P<.001). LU had significantly higher FS and FM compared with other resins (P<.001), while TC had similar FS to definitive 3D printed resins. Aging time and media influenced FS and FM, with varying effects observed across different materials and time points. Strong positive correlations were found between filler weight and both FS (r=.83, P=.019) and FM. All materials met the minimum FS requirement of 80 MPa (ISO 4049) when printed at 90 degrees.

**Conclusions.** The 90-degree orientation produced specimens with higher FS than 0- and 45-degree orientations. CT recommended for definitive restorations displayed higher FS compared with VCP and those intended for interim use after 3 months of aging. LU exhibited higher FS and FM than 3D printed resins, while TC had similar FS and FM to the latter. Aging effects on 3D printed resins were minimal and were material specific. (J Prosthet Dent xxxx;xxx:xxx)

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The authors declare that they have no conflicts of interest related to the publication of this research.

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## **Clinical Implications**

The highest mean flexural strength values of 3D printed resins for fixed partial dentures were obtained by printing them vertically at a 90-degree angle to the build platform and by using 3D printed resins with high filler loading. The 3D printed definitive resins for fixed partial dentures performed similarly to the milled resins for interim restorations.

The implementation of computer-aided design and computer-aided manufacture (CAD-CAM) technology has significantly influenced restorative dentistry,<sup>1</sup> offering both additive manufacturing (AM) and subtractive manufacturing (SM) methods. SM has been popular in dentistry, involving the milling of solid materials. However, SM has limitations, including material waste, tool wear, accuracy constraints related to complex objects, and potential surface defects.<sup>2,3</sup>

In contrast, AM, or 3D printing, creates 3-dimensional (3D) objects layer by layer, allowing for the rapid production of custom prostheses, with minimal material waste and without tool wear,<sup>1</sup> leading to its increased popularity in dentistry as an alternative to SM.<sup>4–6</sup> VAT polymerization, including stereolithography (SLA) and digital light processing (DLP), is a commonly used AM technology in dentistry, where a light source polymerizes and solidifies photocurable polymers.<sup>7</sup> In SLA, an ultraviolet (UV) laser beam is used, while DLP uses a digital projector screen.<sup>8</sup> Three-dimensionally printed resins are used for surgical guides,<sup>9</sup> complete dentures,<sup>10,11</sup> occlusal devices,<sup>12</sup> as well as interim and, more recently, definitive dental restorations.<sup>13–16</sup>

The mechanical properties of 3D printed restorations are influenced by the material and the manufacturing process.<sup>1,17,18</sup> While the printing process is typically automated with preset parameters including printing velocity and laser intensity and speed, certain preprocessing parameters must be adjusted to achieve optimal outcomes. These include the build orientation, position on the build platform, support structures, and print layer thickness.<sup>1,18,19</sup> Postprocessing stages, such as washing and final polymerization, can also be adjusted and may affect the mechanical properties of the fabricated parts.<sup>20-23</sup> Most materials come with recommended pre- and postprocessing settings, but not all manufacturers provide guidance on the recommended print orientation, a parameter that influences print time, packing density, material consumption, accuracy, and mechanical strength.<sup>1,3,24-27</sup> The 3D printed parts are mechanically anisotropic, meaning mechanical properties can vary with different printing directions.<sup>28</sup> Therefore, understanding the effects of

print orientation on mechanical properties is essential for assessing restoration performance. However, published data on the impact of printing orientation on the mechanical properties of 3D printed restorative resin materials are conflicting, leaving uncertainty about which orientation yields favorable mechanical properties.<sup>1,18,19,24,29,30</sup>

While extensive research has been conducted on the mechanical properties of 3D printed interim resin restorative materials, <sup>3,16,19,24,31–35</sup> studies examining the mechanical properties of 3D printed definitive resin materials are sparse, <sup>15,36–38</sup> and some lack important information about the different printing parameters including orientation. <sup>13,39–43</sup>

The mechanical properties of composite resins are also influenced by factors such as the resin matrix,<sup>44</sup> filler load and morphology,<sup>45–47</sup> and the resulting features of the polymer network.<sup>48</sup> In wet environments, composite resins react through water sorption, water solubility, and filler particle exfoliation, affecting their strength.<sup>49–53</sup> Therefore, it is essential to investigate their behavior after artificial aging for clinically relevant testing.

The aim of this in vitro study was to evaluate the effect of print orientation on the flexural strength (FS) and flexural modulus (FM) of 3D printed composite resin materials indicated for definitive and interim restorations after aging in distilled water (DW) and artificial saliva (AS).

The null hypotheses were that no difference would exist in FS and FM between the different print orientations (0, 45 and 90 degrees) of 3D printed resins after aging in DW and AS, between the interim and definitive 3D printed resins after aging for 3 months in DW and AS, between the 3D printed and milled materials after storage in AS for 24 hours, and between the different storage durations (24 hours, 1 month, 3 months) and storage media (DW and AS) regarding the FS of the investigated 3D printed materials.

#### **MATERIAL AND METHODS**

Five resin materials for additive manufacturing and 2 for subtractive manufacturing were used in this study (Table 1). Specimens for the AM group were printed in 3 orientations (0, 45, and 90 degrees) with dimensions of  $2\times2\times25$  mm (N=180/material, 60/orientation). Measurements were recorded at 24 hours, 1 month, and 3 months after aging in DW and AS at 37 °C (n=10). Additional  $2\times2\times16$ -mm specimens were printed with a 90-degree orientation. FS measurements for these, in addition to the milled group, were recorded after 24 hours of storage in AS at 37 °C (n=10). The dimensions of the second set of specimens were dictated by the size restrictions of the milled blocks. The sample size (n=10)

Table 1. Manu	ifacturers' composit	ion informa	ition for investigated	materials				
	Material	Code	Manufacturer	Composition	wt%	Lot. #	Shade	Indications
3D printed	Varseosmile Crown <sup>plus</sup>	VCP	BEGO	Esterification products of 4.4'-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid Silanized dental glass (particle size 0.7 µm) Diphenyl (2.4,6-trimethylbenzoyl) phosphine oxide Methyl benzoviformate	5–75 30–50 <2.5	600414	A2	Definitive crowns, inlays, onlays, and veneers
	Crowntec	Ъ	Saremco Dental AG	Bis-EMA Trimethylbenzonyldiphenyl phosphine oxide Silanized dental glass, pyrogenic silica (particle size 0.7 µm)	50–75 0.1 - <1 30–50	D937	A2	Definitive crowns, inlays, onlays, veneers, denture teeth and interim fixed partial dentures
	NextDent C B MFH	QN	3D systems	7,7,9(or 7,9,9)-trimethyl–4,13-dioxo–3,14- dioxa–5,12-diazahexadecane–1,16-diyl bismethacrylate 2-hydroxyethyl methacrylate ( <i>HEMA</i> ) Ethoxylated bisphenol A dimethacrylate Ethylene dimethacrylate Silicon dioxide	50-75 <25 <10 1-5	WX495N02	L Z	Crowns and fixed partial dentures for long term interim use
				Uphenyi (2,4,5-trimetnyioenzoyi) prosprine oxue Mequinol: 4-methoxphenol; hydroquinone monomethyl ether Titanium dioxide	 			
	Dima CB temp	Ы	Kulzer GmbH	Esterification products of 4.4'-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid 7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12- diazahexadecane-1,16-diyl bismethacrylate Propylidynetrimethyl trimethacrylate Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide Mequinol	40-60 30-50 3-10 <1	CD21G06A35	A2	Interim crowns and fixed partial dentures up to 1 year
	GC temp print	09	GC dental	UDMA 2.2"-ethylenedioxydiethyl dimethacrylate Esterification products of 4,4"-isopropylidenediphenol, ethoxylated and 2-methylprop2-enoic acid Silicon dioxide (quartz) Diphenyl (2,4.6-trimethylbenzoyl) phosphine oxide 2-(2 1+benzotriazol-2-vl)-b-cresol	50–75 10- <25 2.5- <5 10- <25 <2.5 0.1-<0.2	2206101	A2	Long term interim crowns, fixed partial dentures, inlays, onlays, and veneers
Milled	Lava Ultimate	Э	3 M ESPE	BisGMA, UDMA, BisEMA, TEGDMA Silica nanomers (20 nm)Zirconia nanomers (4- 11 nm)Silica- zirconia nanoclusters (0.6–10 um)	20 80	NC95259	A2	Definitive inlays, onlays, and veneers
	Telio CAD	22	Ivoclar AG	Polymethyl methacrylate Pigments	99.5 <1	Z02TYX	A2	Interim crowns, interim fixed partial dentures, and implant-supported interim crowns

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Figure 1. Study design of additive and subtractive resin materials.

was based on previous studies,<sup>1,54</sup> and all specimens were allocated to their designated storage media/time using a simple computer randomization (IBM SPSS Statistics, v29.0; IBM Corp). The study design is described in Figure 1.

One specimen ( $2 \times 2 \times 25$  mm) was designed using an online software program (Tinkercad), saved as a standard tessellation language (STL) file, and imported into the CAM software program (Composer version 1.3.2, 2021; ASIGA). The print parameters were then selected; these included print orientation (0, 45, and 90 degrees) (Fig. 2A), specimen number (n=10/orientation), layer thickness (50 µm), and support design (automatically generated). Specimens were printed using an open system 3D printer (ASIGA MAX UV; ASIGA) that uses DLP technology and operates at a light wavelength of 385 nm.

The specimens were divided into 3 subgroups according to orientation. The 0-degree specimens were printed horizontally, perpendicular to the load direction; the 45-degree specimens were printed with an angle; and the 90-degree specimens were printed vertically, parallel to the load direction (Fig. 2B).

After printing, the specimens were cleaned in an automated wash device (Form Wash; Formlabs Inc) using an alcohol solution (96% ethanol; Sigma Aldrich) for 5-minutes to eliminate residual surface monomers. Supports were removed with a scalpel, and the specimens were postpolymerized following the manufacturers' recommendation for each material (Table 2) and manually abraded with a 320-grit silicon carbide paper (Metaserv 250 Grinder Polisher; Buehler Co) to remove any flash and smooth the edges. The dimensions of specimens were confirmed using digital calipers (PDC150M; Draper tools Ltd) following the International Organization for Standardization (ISO) 4049 standard.<sup>55</sup> Following postpolymerization, a noticeable bend was observed along the length of the specimens printed at 0-degrees, likely linked to polymerization shrinkage. This bend was absent in specimens printed at other orientations. A pilot study was therefore conducted to determine the most appropriate side for applying the force during flexural testing, and testing proceeded with the bend facing the applied force.

Specimens of subtractive CAD-CAM blocks were sectioned using a diamond blade (MK 303; MK Diamond) mounted on a saw (Isomet 1000 Precision Saw; Buehler Co) under constant water irrigation and then polished similarly to the 3D printed group. The dimensions ( $2 \times 2 \times 16$  mm) were confirmed using digital calipers to an accuracy of ±0.01 mm.

The inorganic filler content was determined by eliminating the organic component through a heating process known as the Ash technique (ISO 1172, 1999).<sup>56</sup> Disk specimens ( $\emptyset$ 12×2 mm) were printed in a 0-degree orientation (n=3), placed on a ceramic crucible, and heated in an electric furnace (Programat EP 5000; Ivoclar AG) to a temperature of 600 °C for 30 minutes. The specimens were weighed using an electronic scale with an accuracy of ±0.01 mg (Ohaus Analytical Plus; Ohaus Corp). The percentage of inorganic filler weight was calculated from:

Filler weight. 
$$\% = \frac{(w_3 - w_1)}{(w_2 - w_1)} \times 100,$$



**Figure 2.** A, Schematic representation of print design of bar-shaped specimens in 0, 45, and 90-degree print orientations. B, Direction of force on specimen surface during flexural strength test with respect to layer orientations.

where  $w_1$  is the initial mass of the dry crucible,  $w_2$  is the initial mass of dry crucible combined with the dried specimen, and  $w_3$  is the final mass of the crucible combined with the specimen residue.

To simulate chemical degradation, all specimens were placed inside glass vials filled with either DW or  $AS^{57,58}$  and placed in a  $CO_2$  incubator (function line BB 16; Heraeus Instruments) at 37 °C. The AS solution was prepared by dissolving sodium chloride (0.4 g), potassium chloride (0.4 g), calcium chloride (0.795 g), so-dium dihydrogen phosphate (0.69 g), and sodium sulfide

hydrate (0.005 g) in 1000 mL of distilled water.<sup>59,60</sup> The pH of AS was 5.52 as determined by a digital microprocessor pH meter (DELTA 340; Mettler Toledo Ltd).

Flexure testing has been recommended for evaluating dental composite resin, as it assesses the ability of the fixed dental prosthesis to resist plastic deformation when subjected to loads.<sup>61</sup> A universal testing machine (Z020; Zwick/Roell) with a 500-N cell load was used for the measurements. Each specimen was placed on 2 supporting rods mounted parallel with either a 20-mm ( $\pm$  0.1) (for the 25 mm specimens) or a 12-mm ( $\pm$  0.1) (for the 16-mm specimens) distance between them, with the third loading rod centered midway between the 2 supports. All specimens were subjected to a 3-point bend test under increasing load at a crosshead speed of 1 mm/minute until fracture as specified by the ISO 4049<sup>55</sup> and 10477 standards.<sup>62</sup> The flexural strength (MPa) was calculated from:

## $\sigma = 3 \mathrm{Fl} / (2bh^2),$

where F is the maximum load exerted on the specimen (N), 1 is the distance between the supports, b is the width of the specimen before water storage, and h is the height of the specimen before water storage (all mm). The flexural modulus was calculated from a tangent to the initial slope of the stress/strain curve.

One fractured specimen from each orientation was mounted on an aluminum stub, coated with gold, and examined with a scanning electron microscope (SEM) (JSM-6610 LV; JOEL Co). Images were captured at magnifications ranging from  $\times$ 500 (to assess for layer homogeneity) to  $\times$ 20 000 (to assess for resin matrix or filler degradation) using a secondary electron detector with an acceleration voltage of 10.0 kV.

The data were analyzed using a statistical software program (IBM SPSS Statistics, v29.0; IBM Corp). The results were tested for normal distribution and homogeneity of variance using the Shapiro-Wilk and Levene tests respectively. Multiple-way analysis of variance was performed to investigate the interactions between material group, build orientation, aging time, and aging media. Data within each measurement parameter were analyzed with 1-way ANOVA and a Tukey post hoc test. The *t* test was performed to investigate the interactions the difference

Table 2. Postpolymerization device parameters provided by their manufacturers

	Postpolymerization Device		
	Form Cure	Otoflash G171	Cara Print LED Cure
Manufacturer Technology Number of light sources Light intensity	Formlabs Ultraviolet light (UV) 13 39 Watt	NK-Optik Flashlight 2 200 Watt	Kulzer GmbH Light-emitting diode (LED) 10 15–150 W
Light spectrum (wavelength) Maximum temperature Materials and post-polymerization recommendation	405 nm 60–80 °C Nextdent CB MFH (60 °C for 30 min)	280–700 nm (peak 400–500 nm) n/a Varseosmile Crown <sup>plus</sup> (2×1500 flashes) Crowntec (2×2000 flashes) GC Temp Print (2×400 flashes)	370–470 nm (peak 397–450 nm) 30–80 °C Dima CB Temp (60 °C for 20 min)

Table	3. Mear	n ±standar	d deviation	values	for filler	content	wt%	of	all
studie	ed mater	ials measu	red using a	ash met	hod (n=3	3)			

Category	Material	Manufacturer Filler wt%	Measured Filler (Residue) wt%
Subtractive	LU	80	73.5 ±1.3 <sup>A</sup>
	TC	N/A	N/A
Additive	VCP	30–50	33.8 ±0.3 <sup>B</sup>
	CT	30–50	33.4 ±1.9 <sup>B</sup>
	ND	Not disclosed	7.4 ±0.1 <sup>D</sup>
	DT	Not disclosed	0.95 ±0.1 <sup>E</sup>
	GC	10–25	19.5 ±0.1 <sup>C</sup>

Different superscript letters denote significant variations between materials (P<.05).

between the aging media. Pearson correlation analysis was conducted to examine the relationship between filler weight and FS and FM ( $\alpha$ =.05 for all tests).

#### RESULTS

Filler wt% (Table 3) was found to be statistically different in the following sequence:  $LU > CT \ge VCP > GC > ND >$ DT (*P*<.05). The filler wt% of VCP and CT were similar (*P*=.9). TC is a PMMA material and does not contain any fillers.

Means and standard deviations for the FS and FM of the 3D printed resins are listed in Tables 4 and 5. The results indicated a significant main effect for material, print orientation, aging media, and aging time on FS and FM (*P*<.001). The parameter material exerted the highest influence on FS and FM (FS:  $\eta_p^2$ =.67, FM:  $\eta_p^2$ =.98) followed by the print orientation (FS:  $\eta_p^2$ =.46, FM:  $\eta_p^2$ =.34), aging time (FS:  $\eta_p^2$ =.15, FM:  $\eta_p^2$ =.14), and aging media (FS:  $\eta_p^2$ =.08, FM:  $\eta_p^2$ =.21). The interaction between the

Table 4. Means  $\pm$  standard deviations (MPa) for flexural strength of AM resins printed with three orientations (0, 45, and 90 degrees) and 2×2×25-mm specimen dimension after aging in distilled water and artificial saliva for 24 h, 1 m, and 3 m at 37 °C (n=10)

Category	Material	Orientation	Distilled Water			Artificial Saliva	Artificial Saliva			
		(Degrees)	24 h	1 m	3 m	24 h	1 m	3 m		
Definitive	VCP	0	87.3 ±3.7 <sup>a1</sup>	75.4 ±15.8 <sup>a1</sup>	84.6 ±9.9 <sup>a1</sup>	95.8 ±5.7 <sup>a1</sup>	64.0 ±3.9 <sup>a2</sup>	69.1 ±9.2 <sup>a2</sup>		
		45	100.7 ±5.0 <sup>b1</sup>	83.8 ±8.5 <sup>a2</sup>	93.8 ±10.5 <sup>ab3</sup>	86.1 ±7.1 <sup>b1</sup>	85.8 ±9.9 <sup>b1</sup>	86.4 ±7.2 <sup>b1</sup>		
		90	101.8 ±8.8 <sup>b1</sup>	98.9 ±7.2 <sup>b2</sup>	96.6 ±8.1 <sup>b2</sup>	103.2 ±8.3 <sup>a1</sup>	99.8 ±5.8 <sup>c1</sup>	100.5 ±6.7 <sup>c1</sup>		
	СТ	0	102.3 ±9.2 <sup>a1</sup>	100.2 ±9.8 <sup>a1</sup>	94.4 ±13.4 <sup>a1</sup>	111.9 ±8.8 <sup>a1</sup>	81.0 ±11.6 <sup>a2</sup>	88.6 ±9.7 <sup>a2</sup>		
		45	113.2 ±8.9 <sup>a1</sup>	113.2 ±9.7 <sup>b1</sup>	103.1 ±9.1 <sup>ab1</sup>	112.2 ±7.3 <sup>b1</sup>	95.6 ±6.8 <sup>b2</sup>	93.3 ±6.9 <sup>a2</sup>		
		90	125.9 ±11.5 <sup>b1*</sup>	121.3 ±9.5 <sup>b1</sup>	113.8 ±5.6 <sup>b1</sup>	115.9 ±5.5 <sup>c1*</sup>	116.9 ±9.4 <sup>c1</sup>	115.9 ±6.9 <sup>b1</sup>		
Interim	ND	0	90.5 ±4.1 <sup>a1</sup>	81.8 ±3.6 <sup>a2</sup>	87.6 ±4.8 <sup>a3</sup>	87.7 ±2.7 <sup>a1</sup>	72.9 ±1.4 <sup>a2</sup>	81.2 ±3.5 <sup>a3</sup>		
		45	102.6 ±5.6 <sup>b1</sup>	83.6 ±1.5 <sup>a2</sup>	89.6 ±1.7 <sup>a3</sup>	94.3 ±2.1 <sup>b1</sup>	77.5 ±1.2 <sup>b2</sup>	86.6 ±3.81 <sup>b3</sup>		
		90	106.2 ±4.9 <sup>b1*</sup>	83.2 ±1.5 <sup>a2*</sup>	89.9 ±1.9 <sup>a2</sup>	95.5 ±3.2 <sup>b1*</sup>	79.9 ±1.4 <sup>c2*</sup>	91.3 ±2.1 <sup>c3</sup>		
	DT	0	79.6 ±3.0 <sup>a1</sup>	78.3 ±8.6 <sup>a1</sup>	79.4 ±5.7 <sup>a1</sup>	$80.4 \pm 2.2^{a1}$	79.5 ±2.2 <sup>a1</sup>	78.1 ±7.0 <sup>a1</sup>		
		45	81.7 ±2.4 <sup>a1</sup>	87.5 ±10.0 <sup>b2</sup>	92.1 ±3.8 <sup>b2</sup>	80.0 ±4.2 <sup>a1</sup>	79.2 ±3.2 <sup>a1</sup>	87.7 ±3.2 <sup>ab2</sup>		
		90	87.7 ±2.1 <sup>b1</sup>	102.0 ±1.2 <sup>c2*</sup>	93.4 ±6.9 <sup>b3</sup>	86.8 ±1.1 <sup>b1</sup>	86.6 ±3.4 <sup>b1*</sup>	93.3 ±1.7 <sup>b2</sup>		
	GC	0	69.4 ±3.5 <sup>a1</sup>	82.6 ±3.3 <sup>a2</sup>	88.8 ±7.2 <sup>a2</sup>	74.4 ±4.0 <sup>a1</sup>	78.7 ±8.3 <sup>ab1</sup>	87.4 ±8.6 <sup>a2</sup>		
		45	74.9 ±3.3 <sup>b1</sup>	76.8 ±4.9 <sup>b1*</sup>	79.7 ±5.6 <sup>b1</sup>	85.4 ±5.5 <sup>b1</sup>	75.4 ±3.3 <sup>a2*</sup>	76.9 ±9.5 <sup>b2</sup>		
		90	82.4 ±3.4 <sup>c1</sup>	90.3 ±3.8 <sup>c2</sup>	89.7 ±5.8 <sup>a2</sup>	85.2 ±3.8 <sup>b1</sup>	82.9 ±4.3 <sup>a1</sup>	89.2 ±6.9 <sup>a2</sup>		

Values marked with same superscript letter or number not significantly different from each other (P>.05).

a,b,c Describe significant differences between orientations within one 3D printed material and aging level. 1,2,3 Describe significant differences between aging levels within one material and aging media. \* Indicates significant difference between aging media for same aging level.

Table 5. Means ±standard deviations (MPa) for flexural modulus of AM resins printed with three orientations (0, 45, and 90 degrees) and 2×2×25-mm specimen dimension after aging in distilled water and artificial saliva for 24 h, 1 m, and 30 m at 37 °C (n=10)

-			5 5						
	Category	Material	Orientation	Distilled Water			Artificial Saliva		
			(Degrees)	24 h	1 m	3 m	24 h	1 m	3 m
	Definitive	VCP	0	2976.1 ±86.9 <sup>a1</sup>	2965.7 ±75.2 <sup>a1</sup>	3083.8 ±53.4 <sup>a2</sup>	2989.9 ±134.3 <sup>a1</sup>	3013.9 ±80.3 <sup>a1</sup>	3453.9 ±94.6 <sup>a2</sup>
			45	3668.9 ±58.3 <sup>b1</sup>	2858.0 ±80.8 <sup>b2</sup>	3022.7 ±75.9 <sup>a3</sup>	2907.2 ±58.7 <sup>b1</sup>	3016.6 ±61.8 <sup>a1</sup>	3193.8 ±67.4 <sup>b2</sup>
			90	3730.0 ±155.3 <sup>b1</sup>	3161.4 ±105.8 <sup>c2</sup>	3102.7 ±168.6 <sup>a2</sup>	3186.0 ±37.9 <sup>b1</sup>	3264.4 ±85.2 <sup>b1</sup>	3565.9 ±72.2 <sup>c2</sup>
		CT	0	3357.9 ±215.2 <sup>a12</sup>	3434.7 ±105.9 <sup>a1</sup>	3362.5 ±240.1 <sup>a2</sup>	3328.7 ±98.8 <sup>a1</sup>	3222.8 ±79.5 <sup>a1</sup>	3508.8 ±101.7 <sup>a2</sup>
			45	3981.8 ±41.3 <sup>b1</sup>	3304.1 ±52.7 <sup>a2</sup>	3165.3 ±131.0 <sup>b2</sup>	3124.7 ±75.4 <sup>a1</sup>	3238.5 ±63.2 <sup>a1</sup>	3402.3 ±76.9 <sup>a2</sup>
			90	3706.6 ±65.0 <sup>b1*</sup>	3784.8 ±165.1 <sup>b1</sup>	3104.6 ±65.2 <sup>ab2</sup>	3181.4 ±63.1 <sup>a1*</sup>	3360.1 ±99.0 <sup>b1</sup>	3536.7 ±52.7 <sup>b2</sup>
	Interim	ND	0	1997.7 ±106.9 <sup>a1</sup>	2077.6 ±81.7 <sup>a2</sup>	2107.0 ±67.9 <sup>ab2</sup>	1779.5 ±50.3 <sup>a1</sup>	1754.5 ±39.9 <sup>a1</sup>	2045.4 ±88.7 <sup>a2</sup>
			45	2630.1 ±112.6 <sup>b1</sup>	1886.5 ±73.4 <sup>b2</sup>	2063.9 ±117.4 <sup>a3</sup>	1912.8 ±26.9 <sup>b1</sup>	1827.1 ±42.5 <sup>b2</sup>	2062.9 ±94.8 <sup>a3</sup>
			90	2765.7 ±165.8 <sup>b1*</sup>	1984.2 ±76.4 <sup>c2*</sup>	2186.1 ±24.8 <sup>b3</sup>	1925.5 ±71.8 <sup>b1*</sup>	1862.2 ±34.0 <sup>b1*</sup>	2196.5 ±51.0 <sup>b2</sup>
		DT	0	1575.1 ±62.6 <sup>a1</sup>	1849.6 ±82.6 <sup>a2</sup>	1735.6 ±69.4 <sup>a3</sup>	1667.5 ±81.4 <sup>a1</sup>	1693.6 ±64.2 <sup>a1</sup>	1746.8 ±74.0 <sup>a2</sup>
			45	1581.9 ±62.0 <sup>a1</sup>	1942.1 ±88.4 <sup>b2</sup>	1772.7 ±34.6 <sup>a3</sup>	1648.6 ±29.1 <sup>a1</sup>	1714.0 ±67.4 <sup>a2</sup>	1773.8 ±44.1 <sup>a3</sup>
			90	1678.5 ±44.1 <sup>b1</sup>	2098.3 ±40.2 <sup>c2*</sup>	1793.6 ±56.0 <sup>a3</sup>	1627.1 ±37.0 <sup>a1</sup>	1685.7 ±46.8 <sup>a1*</sup>	1780.4 31.3 <sup>a2</sup>
		GC	0	1786.6 ±43.2 <sup>a1</sup>	2137.8 ±63.1 <sup>a2</sup>	2310.6 ±111.1 <sup>a3</sup>	1903.3 ±87.5 <sup>a1</sup>	2183.9 ±66.3 <sup>a2</sup>	2377.8 ±69.0 <sup>a3</sup>
			45	1966.1 ±85.7 <sup>b1</sup>	2125.3 ±105.6 <sup>a12</sup>	2125.3 ±105.6 <sup>b2</sup>	2088.9 ±105.7 <sup>b1</sup>	2054.1 ±61.8 <sup>b1</sup>	2141.2 ±72.7 <sup>b1</sup>
			90	2017.6 ±86.6 <sup>b1</sup>	2285.6 ±79.6 <sup>b2*</sup>	2376.5 ±103.8 <sup>a2</sup>	2236.3 ±42.3 <sup>c1</sup>	2159.6 ±104.0 <sup>a2*</sup>	2356.0 ±66.1 <sup>a3</sup>

Values marked with same superscript letter or number not significantly different from each other (P>.05).

a,b,c Describe significant differences between orientations within one 3D printed material and aging level. 1,2,3 Describe significant differences between the aging levels within one material and aging media. \* Indicates significant difference between aging media for same aging level.



Figure 3. Flexural strength of 3-dimensionally printed resin materials (90-degree) with specimen dimension of 2×2×25 mm after aging in distilled water and artificial saliva for 24 h, 1 month, and 3 months.

parameters material and print orientation, material and aging media, and material and aging time were also significant (P<.001). A strong positive correlation was found between filler weight and FS (r=.83, P=.019) and between filler weight and FM (r=.96, P=.001).

Significant differences were found in FS and FM among the 3 orientations for all 3D printed materials (P < .05). A trend was observed at 24 hours in the specimens printed at 90-degrees, as they exhibited significantly higher FS and FM than the 0-degree specimens (P<.05), while the FS and FM for the 45degree specimens varied across materials and did not follow a specific trend. However, CT in AS did not show a significant difference in FS between orientations (P=.4), and DT in AS did not show any statistical differences in FM between orientations throughout the entire aging period (P=.2). Similar trends were observed at 1 month, when the 90-degree specimens maintained the highest FS and FM for all materials, except for ND in DW (P=.2). At 3 months, the FS of the 90-degree specimens for all materials remained statistically higher than those of the 0-degree specimens (P < .05), except for ND in DW (P=.3) and GC in both storage media (P=.9). Although the 90-degree specimens had higher FM measurements, the statistical differences varied among orientations.

Since the 90-degree specimens exhibited higher FS than the other orientations, this print orientation was selected for all comparisons of the remaining parameters

(materials, aging time, and aging media). Differences were observed in FS and FM between the definitive and interim 3D printed resins (P<.001) (Fig. 3). The definitive resin CT exhibited the highest FS and FM, followed by VCP, with a statistical difference in FS between them (P<.001) while having similar FM (P=.78). The interim 3D printed resins exhibited the lowest FS and FM with statistical differences between some of them initially (P<.001), but no statistical differences were found between them after 3 months (P≥.204); their FM was significantly different (P<.001).

Differences in flexural strength (Fig. 4) and flexural modulus (Table 6) were observed between milled and 3D printed materials (P<.001). LU had higher FS and FM compared with all other materials. TC had similar FS to the definitive 3D printed resins (P=.9), as well as DT (P>.999) and ND (P=.05), while having similar FM to the interim 3D printed resins (P≥.7).

At 24 hours and 1 month, significant differences were found between DW and AS for all materials except VCP, where FS and FM measurements in DW were higher than those in AS. However, after 3 months, no significant differences were observed between the 2 aging media (Tables 4 and 5).

The behavior of 3D printed materials varied throughout the aging period. For VCP, CT, and ND, the FS decreased after 3 months compared with 24 hours (P<.05), although this reduction was not statistically significant for VCP (in both aging media) and CT (in



**Figure 4.** Flexural strength of 3-dimensionally printed and milled resin materials with specimen dimension of  $2 \times 2 \times 16$  mm after storage in artificial saliva for 24 h.

**Table 6.** Mean ±standard deviation (MPa) for flexural strength and flexural modulus of milled and 3D printed resins with  $2\times2\times16$ -mm specimen dimension after storage in artificial saliva for 24 h at 37 °C (n=10)

	Material	Flexural Strength	Flexural Modulus
	LU	139.8 ±13.3 <sup>a</sup>	7874.4 ±628.9 <sup>a</sup>
	TC	105.1 ±3.1 <sup>b</sup>	1626.6 ±98.1 <sup>d</sup>
Definitive	VCP	101.8 ±7.6 <sup>b</sup>	2742.7 ±79.9 <sup>b</sup>
	СТ	105.8 ±5.9 <sup>b</sup>	3044.3 ±91.7 <sup>b</sup>
Interim	ND	95.9 ±2.4 <sup>d</sup>	1484.8 ±33.4 <sup>d</sup>
	DT	87.9 ±5.3 <sup>d</sup>	1453.9 ±28.6 <sup>d</sup>
	GC	89.9 ±3.3 <sup>d</sup>	1652.4 ±33.8 <sup>d</sup>
	Definitive Interim	Material LU TC Definitive VCP CT Interim ND DT GC	Material         Flexural Strength           LU         139.8 ±13.3°           TC         105.1 ±3.1°           Definitive         VCP           VCP         101.8 ±7.6°           CT         105.8 ±5.9°           Interim         ND           DT         87.9 ±5.3°           GC         89.9 ±3.3°

Values with same superscript letters in column represent non-significant difference between materials (P>.05)

AS). FS for DT and GC (in DW) slightly increased after 3 months compared with 24 hours (*P*<.05). The FM of all materials increased after 3 months compared with 24 hours, except for CT, VCP, and ND in DW, where the FM decreased.

SEM images of 3D printed specimens are shown in Figures 5 and 6. At 24 hours, no discernible 50–µm layering was observed in any of the 3 orientations. At 3 months, all materials exhibited signs of voids resulting from filler detachment, except for DT. Filler particle clustering was prominently observed in the ND and GC specimens. DT images displayed an absence of filler particles and indicated signs of peeling.

#### DISCUSSION

Print orientation had a significant impact on the FS and FM of 3D printed resin materials after aging for 3 months. Thus, the null hypothesis that no difference would exist in FS and FM among the different print orientations (0, 45, and 90 degrees) of 3D printed resins was rejected. Significant differences in FS and FM were identified between the definitive and interim 3D printed materials and between the 3D printed and milled

materials. Therefore, the null hypotheses that no difference would exist in FS and FM between the interim and definitive 3D printed resins after aging for 3 months in DW and AS and between the 3D printed and milled materials after storage in AS for 24 hours were rejected. Significant differences were identified between storage times and storage media; thus, the null hypothesis that no difference would exist between the different storage durations (24 hours, 1 month, 3 months) and storage media (DW and AS) regarding the FS of the investigated 3D printed materials was rejected.

Within the oral cavity, diverse stresses, including compressive, tensile, and shear, exert pressure on a fixed dental prosthesis, potentially leading to structural failure.<sup>52,53</sup> Therefore, fixed dental prostheses must meet specific standards in their mechanical strength to withstand deformation and fracture under various intraoral forces. According to the ISO 4049 standard,<sup>55</sup> polymer base materials should have a minimum FS of 80 MPa.

In the present study, the force was applied parallel to the layer orientation in the 90-degree printed specimens and perpendicular to the 0-degree printed specimens (Fig. 2B). It was assumed that the 90-degree specimens would result in lower values based on the assumption that the strength between successive layers was weaker than within individual layers.<sup>1,29,30</sup> This assumption was supported by other research findings<sup>19,25</sup> which indicated that the FS of 0-degree specimens exceeded that of 90-degree specimens. However, the present study contradicted these assumptions, revealing that the 90degree specimens exhibited higher FS and FM than the 0-degree specimens and, occasionally, the 45-degree specimens, consistent with other studies.<sup>17,24,34</sup> These findings could be attributed to the strong adhesion between layers, making strength differences negligible.<sup>10,18,27</sup> The SEM images (Figs. 5 and 6) showed no distinct layers at the fracture sites, confirming homogeneity, consistent with other studies.<sup>10,35</sup> Another factor could be the different degree of conversion during polymerization. Specifically, the 0-degree orientation required 86 layers per specimen, while the 90-degree orientation required 546 layers, resulting in increased light exposure and potentially influencing the degree of conversion.<sup>10</sup> Notably, at the 90-degree orientation, all materials met the minimum FS requirement of 80 MPa (ISO 4049), unlike the 0- and 45-degree orientations for some materials (VCP, GC, DT).

Among the tested 3D printed materials, CT and VCP had the highest filler loads at 33%, followed by GC at 19.45%. ND and DT had lower filler loads, at 7.43% and 0.95%, respectively. Higher filler loads can enhance mechanical properties,<sup>63</sup> explaining why CT and VCP showed greater FS than other materials. Yet, GC, despite its higher filler load than ND and DT, exhibited similar FS but higher FM than ND and DT after 3 months. The



Figure 5. Fractured surface morphology of definitive 3-dimensionally printed materials with different print orientations at 24 h (original magnification ×500) and 3 months (original magnification ×20 000) in AS. *Yellow arrows* indicate spherical voids from filler detachment.

similar performance could be associated with increased filler particle size and reduced particle-matrix adhesion, causing the detachment and exfoliation of filler particles due to chemical degradation, which reduced the flexural strength.<sup>49–51</sup> The SEM images of GC and ND specimens (Fig. 6) showed this effect, while SEM images of DT images did not show any fillers, suggesting that the measured filler weight consists of only the pigments.

Monomers such as bisphenol A ethoxylate dimethacrylate (BisEMA), present in CT, have been reported to enhance the toughness and impact resistance of a resin because of their high molecular weight.<sup>64</sup> The high molecular weight allows the material to withstand bending forces without fracture and reduces water sorption because of the hydrophobic nature,<sup>64</sup> explaining the higher FS and FM observed in CT compared with interim 3D printed materials. GC contains a 50% to 75% monomer composition of urethane dimethacrylate (UDMA), which forms a flexible backbone with weak hydrogen bonding from the urethane groups, making it prone to water sorption and hydrolytic degradation, thereby reducing its strength.<sup>52</sup> In addition to the observed filler exfoliation in the SEM images, the composition explains why GC, despite having a higher filler load than ND and DT, exhibited the lowest FS after 3 months of aging. Monomers such as 2-hydroxyethyl methacrylate (HEMA) in ND absorb water because of their hydrophilic nature, leading to decreased FS and FM over time, as observed in this study and consistent with other findings.<sup>19,22</sup>

The variations observed among the 3D printed materials can also be attributed to the specific postpolymerization devices used. Studies have highlighted the significant impact of these devices and the duration of their use on the mechanical properties of dental resins, a relationship directly linked to the degree of conversion.<sup>23,32,65</sup> This study used 3 different postpolymerization devices as recommended by each material's manufacturer (Table 2). Generally, specimens postpolymerized in Ottoflash (CT, VCP) exhibited



**Figure 6.** Fractured surface morphology of interim 3-dimensionally printed materials with different print orientations at 24 h (original magnification  $\times$ 500) and at 3 months (original magnification  $\times$ 10 000 to  $\times$ 20 000) in AS. *Yellow arrows* indicate spherical voids from filler detachment. *Red arrows* indicate filler particles and clusters. *Orange arrows* indicate signs of peeling.

higher FS than those postpolymerized in Form Cure (ND) and Cara Print LED Cure (DT). This finding was consistent with previous research indicating that specimens postpolymerized in Ottoflash exhibited superior

fracture load,<sup>20</sup> elastic modulus, and degree of conversion.<sup>21</sup> The broad light spectrum and concentrated flashes of Ottoflash likely resulted in higher energy and temperature, expediting polymerization.<sup>66</sup>

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Reymus et al<sup>67</sup> reported that light emitting diode (LED) postpolymerization devices yielded inferior mechanical properties compared with Ottoflash and UV light devices, likely because of an inadequate degree of conversion from the LED light sources. In this study, DT, the only material postpolymerized with the LED unit, showed the lowest FS and FM, possibly because of the lack of fillers or inadequate postpolymerization time or a mismatch between the photoinitiator system and the LED wavelength, leading to incomplete polymerization.<sup>68</sup>

Regarding artificial aging, FS and FM measurements in DW were initially higher than those in AS. However, after 3 months, no significant difference in FS between DW and AS was observed (*P*>.05), suggesting that AS is a more deteriorating medium for evaluating resin materials than DW over a short period. Over a longer aging period, resin materials reacted similarly to both DW and AS. Thus, DW and AS have similar long-term effects, consistent with another study.<sup>69</sup> The greater impact of AS is because of its lower pH (5.3) compared with DW (6.5), which can leach residual monomers and damage inorganic fillers, reducing the FS.<sup>70</sup> Therefore, comparisons between milled and 3D printed materials were conducted only in AS.

The behavior of the 90-degree specimens varied over time and was material specific. ND showed a decline in FS and FM, while GC and DT increased after 3 months compared with 24 hours. The FS of VCP and CT in AS remained relatively unaffected by aging. The decline in mechanical properties can be attributed to solvent penetration into the resin matrix, causing swelling and plasticization, filler dislodgment, and the release of nonreacted components, which reduce the mechanical properties.<sup>19,54,71</sup> Multiple voids resulting from filler dislodgment are visible in the SEM images after 3 months (Fig. 6). Conversely, a warm storage medium might induce additional crosslinking, increasing the mechanical properties.<sup>20,53</sup>

The FS of GC and DT increased after 3 months compared with 24 hours but decreased compared with 1 month, showing the impact of extended storage time on material properties. These changes are linked to water absorption, which is influenced by the resin matrix composition and filler loading.<sup>52,71</sup> Despite this increase, their mean FS remained the lowest among the tested materials, similar to ND. The SEM images of aged GC and ND support these low measurements, showing voids from filler detachment, while DT showed peeling, possibly due to its methacrylate monomer composition and lack of fillers.

All 3D printed materials demonstrated lower FS and FM compared with the milled LU, which was expected considering it has the highest filler load (74%). LU incorporates nanoclusters of nonaggregated, nonagglomerated silica and zirconia nanoparticles, which have been linked to a higher elastic modulus than spherical fillers.<sup>45,72,73</sup> Additionally, the monomer composition of LU, which includes bisphenol A

glycerolate dimethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), Bis-EMA, and UDMA, forms a densely crosslinked network, enhancing durability and minimizing softening.<sup>74</sup> In contrast, 3D printed materials face viscosity constraints that hinder filler incorporation, leading to lower FS and FM<sup>49,75,76</sup> and explaining the comparatively lower FS and FM observed in 3D printed materials compared with milled composite resin LU, similar to the findings of Prause et al.<sup>38</sup> Other studies comparing milled definitive crowns with 3D printed definitive crowns using VCP or CT reported similar or lower fracture resistance for 3D printed crowns.<sup>13,15,37,39,41</sup> However, direct comparisons are challenging because of variations in specimen geometry, printing parameters, and control materials.

TC exhibited FS similar to both definitive and interim 3D printed materials despite lacking fillers while maintaining a FM similar to that of interim ones. This high FS may be because of the high-pressure and high-temperature polymerization process in an industrial setting, which enhances the degree of conversion and mechanical properties, resulting in a more robust polymer network.<sup>67,77–79</sup> The higher FS of milled PMMA materials compared with 3D printed ones for interim restorations has been reported,<sup>17,24,43</sup> though another study<sup>33</sup> reported no significant difference.

Based on the current findings, all tested materials printed at a 90-degree orientation met the ISO 4049 standard specifications, and their performance remained largely unaffected after storage in media simulating their environment. Limitations of this study included the in vitro design that only simulated the chemical aspect of aging and therefore may not have accurately predicted clinical performance. Consequently, 3D printed definitive resins might not perform as well clinically in the long term as milled FDPs. Future investigations should assess the impact of aging induced by mastication, examine material stability under varying temperature conditions, and explore the effects of different solvents encountered in the oral environment. Using specimen shapes that closely resemble those in clinical applications would offer a more accurate representation of material performance in clinical scenarios. The effects of printing specimens horizontally but with the force applied on the edge should be investigated.

#### CONCLUSIONS

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

- 1. The 90-degree print orientation produced specimens with higher flexural strength than 0-degree and 45-degree print orientations.
- 2. 3D printed resin CT recommended for definitive restorations displayed higher FS compared with

VCP and those intended for interim use after 3 months of aging.

- 3. Milled composite resin LU exhibited higher FS and FM than 3D printed resins, while milled PMMA resin TC had similar FS and FM to 3D printed resin.
- 4. AS was a more deteriorating medium initially but, after 3 months, DW and AS had similar effects, while the effect of aging time was material specific.

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Shaymaa Mudhaffer: Conceptualisation, Methodology, Software, Formal analysis, Investigation, Resources, Data curation, Writing-original draft preparation, Visualisation, Funding acquisition. Julfikar Haider: Validation, Writing-review and editing, Supervision. Julian Satterthwaite: Methodology, Validation, Writing-review and editing, Supervision, Project administration. Nick Silikas: Validation, Writing-review and editing, Supervision, Project administration.

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