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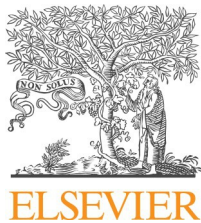
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Assessing the physical and mechanical properties of 3D printed acrylic material for denture base application

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ABSTRACT

Objective: Three-dimensional (3D) printing is increasingly being utilised in the dental field because of its time-saving potential and cost effectiveness. It enables dental practitioners to eliminate several fabrication steps, achieve higher precision, and attain consistency in complex prosthetic models. The properties of 3D-printed resin materials can be affected by many factors, including the printing orientation (PO) and insufficient post-curing time (CT). This study aimed to investigate the effect of PO and CT on the mechanical and physical properties of a 3D-printed denture base resin (NextDent).

Methods: 3D-printed specimens were fabricated in 0°, 45°, and 90° POs, followed by three CTs (20, 30, and 50 min). The microhardness was tested using a Vickers hardness test, while the flexural property was evaluated using a three-point bending test. Sorption and solubility were measured after the specimens had been stored in an artificial saliva for 42 days, and the degree of conversion during polymerisation was analysed using Fourier Transform Infra-red (FTIR) spectroscopy.

Results: The flexural strength of the material significantly increased ($p < 0.05$) when the printing orientation was changed from 0° to 90°. A similar increase was observed in the hardness, degree of conversion, and water sorption results. In general, no significant difference ($p > 0.05$) in any of the tested properties was found when the post-curing times were increased from 20 to 50 min.

Significance: The highest physical and mechanical properties of the 3D-printed denture base resin can be obtained by printing vertically (90° angle to the platform base). The

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minimal post-curing time to achieve ideal results is 30 min, as further curing will have no significant effect on the properties of the material.

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1. Introduction

The fabrication of denture was revolutionised by the introduction of clinical acrylic resin [1], a material based on polymethyl methacrylate (PMMA) that was first developed in 1936 [2]. Since 1948, 98 % of the dentures have been fabricated from PMMA and copolymers [3], and PMMA has become the ideal material for the denture bases due to its good aesthetic characteristics, biocompatibility with oral tissues, light weightiness, low cost, and ease of processing and handling [1]. However, it also poses some disadvantages that need to be addressed, such as dimensional inaccuracy, insufficient mechanical properties, including flexural and impact strength, and polymerisation shrinkage [4].

Recent advances in technology have shown potential in changing the conventional denture fabrication practice. In particular, three-dimensional (3D) printing, which first appeared in 1983, is advancing rapidly. This is a computer-controlled digital manufacturing technique that employs 3D model data to create both simple and complex objects. Its operating principle can be represented as opposite to subtractive manufacturing and sometimes named as rapid prototyping or additive manufacturing [5,6]. 3D printing technology produces complex models using a layer by layer build-up principle [7]. In addition, 3D printing has the capability to precisely print parts or products in a cost effective manner with less material waste [8].

More recently, several studies have reported 3D printing of photo-polymerised PMMA denture base resins with an aim to achieve similar mechanical properties of conventional PMMA [9–11]. Various factors related to resin material composition and chemistry, printer type and its operating principle, and post-curing process can affect the properties of the printed product, and one of the most important factors is printing orientation [12]. Among the literature, there are different opinions regarding the effect of layer orientation on the mechanical properties of the printed object, where some authors claimed that the horizontal printing orientation (0°) along the build platform has the highest flexural strength compared to the vertical printing orientation (90°). Weaker bonds between successive layers compared to the bond forces within the layer itself was suggested as the possible cause [13–15]. Others claimed [16,17] that a vertical layer orientation produced the highest flexural strength compared to the horizontal printing orientation, and they explained that there was no difference between the bond strength between successive layers and the bond within an individual layer itself.

Unlike the conventional heat-cured PMMA used for the denture base fabrication, 3D printed resin is a photo-polymerised material, and post-curing time is an essential process that affects the performance of the material. The curing

process occurs partially during the printing via the printer's laser or light projector, and further curing is continued in a light cure unit as a post-curing step to complete the polymerisation process. One study reported that photo polymerised denture base material had superior mechanical properties compared to the conventionally polymerised counterparts [18], but the study did not test any type of 3D printed denture base materials. With the emergence of different 3D printing techniques and photo-polymerised materials, the manufacturers advised varying amount of post-curing times (ranging from 20 to 60 min) to complete polymerisation of the materials [19,20]. Some authors investigated the effect of post-curing times on the 3D printed resins, and they confirmed that it had a significant effect on the properties of the 3D printed resin [11,21–23]. However, the studies on the effect of post-curing time with different denture base resins are insufficient and hence demands further investigation. To the authors' best knowledge, no study showed results on the effect of both printing orientation and post-curing time with 3D printed NextDent denture base resin.

In this study, the aim was to investigate the effect of changing post-curing time and printing orientation on the mechanical and physical properties of 3D printed denture base resin material in term of micro hardness, flexural strength and modulus, sorption and solubility, and the DC. The null hypothesis was that (1) the post-curing time and (2) different printing orientations did not affect the mechanical, chemical and physical properties of 3D printed denture base resin.

2. Materials and methods

2.1. Resin material

The material used was commercial NextDent denture 3D+ light cured resin with light pink colour (3D systems, Soesterberg, Netherland) for denture base application. The properties for the NextDent material such as ultimate flexural strength (MPa), flexural modulus (MPa), sorption ($\mu\text{g}/\text{mm}^3$) and solubility ($\mu\text{g}/\text{mm}^3$) suggested by the material's manufacturer are 84, 2383, 28 and 0.1 respectively [20]. According to the material's safety data sheet, the composition (w/w%) of the resin as follows: Ethoxylated bisphenol A dimethacrylate (≥ 75); 7,7,9(or 7,9,9-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate (10–20); 2-hydroxyethyl methacrylate (5–10); Silicon dioxide (5–10); diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide (1–5) and Titanium dioxide (< 0.1).

2.2. 3D printing and post processing

Liquid resin was poured into the resin tank of Formlabs Form 2 printer (Formlabs, Somerville, USA), which worked based

on SLA technology with 405 nm laser wavelength and a layer thickness 50 μ m layers. After designing test specimens by an open source CAD software (Tinkercad) the final design was exported in STL format to be compatible for use with the printer's software. Preform software was used to open the STL file and to manipulate the CAD design with vertical (90°), horizontal (0°), or any angular positions. Support structures were printed automatically to support the specimen during printing of the specimens. Once printing was finished, all specimens were removed from the build platform, cleaned by eliminating support structures and submerged in a container (Form Wash) filled with ethanol 99.8 % (Formlabs, Somerville, USA) for 5 min to get rid of any excess resin without damaging the printed parts. Then, the specimens were left out to dry from any ethanol residues for 10 min. After cleaning and drying, specimens were placed in an ultraviolet (UV) light box (Formlabs, Somerville, USA) under a temperature of 60 °C, 405 nm LED wavelength and 39 W LED power to complete the polymerisation process.

2.3. Specimen grouping

Literature suggested that properties of the 3D printed resins could vary with the printing orientation (PO) and length of the curing time (CT) after printing [11,13,16,21,22]. Therefore, in this study, all the specimens were divided into nine groups based on the three POs and three CTs (0°/20, 45°/20, 90°/20, 0°/30, 45°/30, 90°/30, 0°/50, 45°/50, 90°/50). A total of 147 specimens were prepared with different dimensions and group sizes required for testing different physical and mechanical properties (Table 1). Based on the manufacturer's recommendation of curing time and shortest printing time, 0°/30 was considered as the control group.

2.4. Characterisation of 3D printed specimens

The Vickers hardness (VH) of the specimens were measured using a micro hardness testing machine (FM-700, Future Tech Corp, Tokyo, Japan). The test load was set to 50 g with a dwell time of 30 s. For each specimen, three indentations were made at different equidistant points in a straight line after polishing the surface. The distance between the points was calculated by multiplying the average indentation's diagonal length by four. The mean hardness were recorded under dry conditions [24,25] after 24 h of printing.

The flexural strength of the specimens was evaluated using a 3-point bending test in a universal testing machine (Zwick/Roell Z202 Leominster, UK) with a load cell of 500 N [9,10,26]. Fig. 1 shows different layer orientations with respect to the direction of applied load during the testing. According to the BS EN ISO 20795-1:2013 standard for Denture Base Polymers, the dimension of the specimen was 64 mm (length) \times 10.0 \pm 0.2 mm (width) \times 3.3 \pm 0.2 mm (thickness). Before testing, the edges and faces of all specimens were wet grinded smooth and flat using silicon carbide grinding papers at a grain size of approximately 30 μ m (P500), 18 μ m (P1000), and 15 μ m (P1200) sequentially to the required width and height. Digital calliper (Draper, Eastleigh, Hants, UK) was used to measure the specimens' height and width at the middle and peripheries to an accuracy of \pm 0.01 mm. The

Table 1 – Determination of sample number with different testing conditions.

Tests	Printing orientations (°)	Curing time (min)	Total number of specimens	Specimen dimension
Flexural strength/Flexural modulus	0, 45 and 90	20, 30 and 50	$^3 \times ^3 \times ^5 \times ^{10} = 90$ (n = 10)	65 \times 10 \times 3.3 mm ³
Hardness			$3 \times 3 \times 3 = 27$ (n = 3)	15 mm dia \times 2 mm thick
Sorption and solubility		30	$3 \times 1 \times 5 = 15$ (n = 5)	50 mm dia \times 0.5 mm thick
DC			$3 \times 1 \times 5 = 15$ (n = 5)	2 mm dia \times 2 mm thick

³3 (number of printing orientations) \times ³3 (curing times) \times ⁵10 (group size).

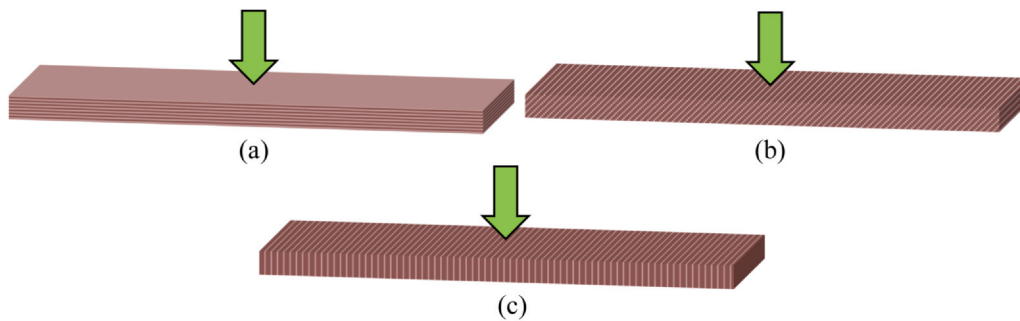


Fig. 1 – Direction of the force on the specimen surface during flexural strength test with respect to the layer orientations: (a) horizontal (0°), (b) angled (45°), and (c) vertical (90°).

specimens were stored in distilled water in an incubator at a temperature of 37 ± 1 °C for 50 ± 2 h. After that, each specimen was placed over a supporting jig separated by 50 ± 0.1 mm. The two polished cylindrical supports were 3.2 mm in diameter, and at least 10.5 mm long. The cross-head preload speed and the test speed were set to 5 mm/min. Eqs. (1) and (2) were used to calculate the flexural strength and flexural modulus respectively.

$$\sigma = \frac{3Fl}{2bh^2} \quad (1)$$

Where F is the maximum force applied in Newton, l is the distance between the supports in mm, b is the width of the specimen in mm, h is the height of the specimen in mm. The flexural modulus was determined from the slope of the linear portion of the stress strain curve for each test run.

$$E = \frac{F_1 l^3}{4bh^3d} \quad (2)$$

Where F_1 is the load, in Newtons, at a point in the straight line (with the maximum slope) of the load/deflection curve, b is the width of the specimen in mm, h is the height of the specimen in mm, d is the deflection in millimetre at load of F_1 .

Fourier Transform Infra-red spectroscopic (FTIR) was used to determine the DC of the 3D printed specimen using a Spotlight 200i FT-IR Microscope System with Spectrum Two (ALPHA II, Bruker, Massachusetts, USA). The parameters used to measure the FTIR spectra were a wavelength ranging from 4000 to 400 cm^{-1} , and a resolution of 4 cm^{-1} with an average of 32 scans at room temperature. A background spectrum was generated to calibrate the instrument. The 3D printed resin material was scanned in its liquid form as a baseline record, and then scanned again after the final polymerisation (after post-curing). To calculate the DC in percentage (Eq. (3)), the difference in the double carbon bond peaks ratio at two frequencies (stretch of aliphatic frequency at 1637 cm^{-1} against the reference aromatic frequency at 1608 cm^{-1}) was measured [27].

$$DC (\%) = \left(1 - \frac{\left(\frac{1637 \text{ cm}^{-1}}{1608 \text{ cm}^{-1}} \right) \text{peak heights after polymerization}}{\left(\frac{1637 \text{ cm}^{-1}}{1608 \text{ cm}^{-1}} \right) \text{peak heights before polymerization}} \right) \times 100 \quad (3)$$

Water sorption was carried out according to the BS EN ISO 20795-1:2013 standard for Denture Base Polymers. Five specimens used for each test group. Specimens were placed in a desiccator containing fresh dry silica gel at 37 ± 2 °C for 1 day, and then moved to room temperature in a second desiccator containing freshly dried silica gel for 1 h in order to be weighed. This process was repeated every day until the change between each successive weighing was not greater than 0.2 mg to gain the baseline mass (m_1). The volume (V) of each specimen was calculated by measuring the diameter and thickness using a digital calliper (Draper, Eastleigh, Hants, UK). Then, the specimens were immersed in artificial saliva at 37 ± 2 °C. Each specimen was dried after removal from the artificial saliva and continued re-weighing until the change between each successive weighing was not greater than 0.2 mg to achieve a constant mass (m_2). Subsequently, the specimens were reconditioned by placing them in a desiccator containing fresh dry silica gel at 37 ± 2 °C for 1 day, and then moved to room temperature in a second desiccator containing freshly dried silica gel for 1 h. The desorption process was carried out by continuing re-weighing until the change between each successive weighing was not greater than 0.2 mg to achieve a constant mass (m_3 , reconditioned mass). Finally, the sorption and solubility in $\mu\text{g}/\text{mm}^3$ were measured using Eqs. (4) and (5) respectively [28].

$$\text{Sorption} = \frac{m_2 - m_3}{v} \quad (4)$$

$$\text{Solubility} = \frac{m_1 - m_3}{v} \quad (5)$$

The volume of each specimen was calculated using Eq. (6).

$$\text{Volume} = 3.14 \times \left(\left(\frac{\text{mean diameter}}{2} \right)^2 \right) \times \text{mean thickness} \quad (6)$$

The percentage of mass change and mass loss during the sorption and solubility test were calculated using Eqs. (7) and (8) respectively.

$$\text{Change in mass, SP}(\%) = \left(\frac{m_2 - m_1}{m_1} \right) \times 100 \quad (7)$$

$$\text{Mass loss, SL}(\%) = \left(\frac{m_1 - m_3}{m_1} \right) \times 100 \quad (8)$$

The surface morphology of the as printed and polished specimens was studied using an optical microscope (Echo,

Revolve, California, USA) with a magnification of $\times 10$. The fractured surfaces of the specimens from the flexural strength test were studied using a scanning electron microscope equipped with an Energy Dispersive X-Ray Spectrometer (SEM-EDX, Carl Zeiss Ltd., 40 VP, Smart SEM, Cambridge, UK). The fractured specimen was mounted onto an aluminium stub and coated with a thin gold layer before it was loaded into the SEM chamber. The images were created using secondary electron detector at an acceleration voltage of 10.0 kV at various magnifications.

2.5. Statistical analysis

All data were statistically analysed using SPSS version 22 (IBM, New York, NY, USA). The normality of data distribution was determined using Shapiro-Wilk test, and the homogeneity of the data was confirmed by Levene test. The data were statistically compared using two-ways ANOVA for the mechanical tests (Vickers hardness, flexural strength and modulus), followed by Turkey's post-hoc statistical analysis according to a significance level set at $p \leq 0.05$. One-way ANOVA was used for the other tests (DC, sorption and solubility) as there was only one variance (layer orientation).

3. Results

3.1. Mechanical properties

Table 2 presents the mean hardness and the standard deviation for all three curing times and printing layer orientations. The results indicated no statistical difference between the mean values ($p > 0.05$) of the groups with different POs at a particular CT except for the samples prepared at 0° layer orientation and 20 min curing time ($0^\circ/20$ min), which showed significantly lower values compared to the other groups. On the other hand, the results did not show any statistical difference between the mean values ($p > 0.05$) of the groups with different CTs at a particular PO except for the $0^\circ/20$ min group, which showed significantly lower values compared to the other groups.

Table 2 – Hardness of 3D printed PMMA resin at different curing times and printing orientations.

Curing time (min)	Printing orientation		
	0° (^a)	45°	90°
20	10.74 (0.88) ^{Ab}	13.31 (0.20) ^{Ba}	14.08 (0.41) ^{Ba}
30 (^b)	12.99 (1.13) ^{Aa}	14.10 (0.12) ^{Aa}	14.63 (0.86) ^{Aa}
50	12.83 (1.04) ^{Aa}	14.14 (0.43) ^{Aa}	14.04 (0.46) ^{Aa}

^a Within a row, cells having similar (upper case) letters are not significantly different from the control (0° layer orientation).

^b Within a column, cells having similar (lower case) letters are not significantly different from the control (30 min curing time).

Table 3 – Flexural strength of 3D printed PMMA resin at different curing times and printing orientations.

Curing time (min)	Printing orientation		
	0° (^a)	45°	90°
20	55.3 (6.3) ^{Aa}	80.6 (2.8) ^{Ba}	88.9 (2.7) ^{Ba}
30 (^b)	58.9 (6.6) ^{Aa}	86.1 (2.1) ^{Ba}	88.4 (1.6) ^{Ba}
50	59.9 (5.5) ^{Aa}	81.5 (3.5) ^{Ba}	88.5 (2.8) ^{Ba}

^a Within a row, cells having similar (upper case) letters are not significantly different from the control (0° layer orientation).

^b Within a column, cells having similar (lower case) letters are not significantly different from the control (30 min curing time).

The means and the standard deviations of flexural strength for the groups were recorded and presented in Table 3. Two-way ANOVA of the flexural strength results indicated a statistical difference between the mean values ($p < 0.05$) of the groups and the Games-Howell's test showed a significant difference between the 0° PO group and the other two PO groups for each particular CTs, but no significant difference was found between the 45° and the 90° PO groups. On the other hand, no statistical difference between the mean values ($p < 0.05$) of the groups with different CTs at a particular PO was found.

The means and the standard deviations of flexural modulus for the groups were recorded and presented in Table 4. Two-way ANOVA showed a statistical difference between the groups' mean values. Games-Howell's test showed that at 30 min CT, 90° PO group was significantly different from the other two PO groups, where there was no statistical difference between the 0° and 45° PO groups. On the other hand, at 50 min CT, 0° PO group was significantly different from the other two PO groups, where there was no statistical difference between the 45° and 90° PO groups. At 45° PO, 30 min CT group showed statistically significant difference compared to the other two CT groups with no statistical difference between the 20 min and 50 min CT groups.

Fig. 2 shows graphical representations of the results on the mechanical properties at different CTs and POs.

3.2. Degree of conversion (DC) analysis

FTIR analysis was performed to assess the DC of the 3D printed resin and to reveal any difference between different the layer orientations and curing times. Fig. 3 shows a typical FTIR spectra of the specimen and the peak heights at the wave number of 1637 cm^{-1} and 1608 cm^{-1} was used to calculate the DC.

The mean and the standard deviation of DC were calculated to be $86.28\% \pm 0.05\%$, $83.27\% \pm 0.02\%$, and $87.79\% \pm 0.06\%$ for 20, 30, and 50 min respectively (Fig. 4a). The effect of post-curing time on DC was analysed, and One-way ANOVA indicated no statistical difference between the groups' mean values ($p < 0.05$).

DC mean values were recorded as $52.33\% \pm 0.02\%$, $67.88\% \pm 0.04\%$, and $83.02\% \pm 0.02\%$ for 0° , 45° , and 90° specimen groups respectively (Fig. 4b). One-way ANOVA

Table 4 – Flexural modulus of 3D printed PMMA resin at different curing times and printing orientations.

Curing time (min)	Printing orientation		
	0° (^a)	45°	90°
	Flexural modulus (MPa)		
20	2466.5 (97.8) ^{Aa}	2416.8 (70.6) ^{Ab}	2385.3 (16.5) ^{Aa}
30 (^b)	2571.0 (45.4) ^{Aa}	2610.9 (107.8) ^{Aa}	2380.3 (66.8) ^{Ba}
50	2554.3 (97.0) ^{Aa}	2386.3 (110.9) ^{Bb}	2374.2 (88.5) ^{Ba}

^a Within a row, cells having similar (upper case) letters are not significantly different from the control (0° layer orientation).
^b Within a column, cells having similar (lower case) letters are not significantly different from the control (30 min curing time).

indicated a statistical difference between the groups' mean values ($p < 0.05$). Tukey's test showed a significant difference between all the groups with different layer orientations.

3.3. Sorption and solubility analysis

The mechanical and structural test results indicated that 30 min curing could be optimum time as recommended by the manufacturers. Therefore, sorption and solubility analysis were carried out only for 30 min curing time but with different layer orientations.

The mean sorption and the standard deviation were calculated to be $31.2 \pm 1.3 \mu\text{g}/\text{mm}^3$, $30.6 \pm 0.5 \mu\text{g}/\text{mm}^3$, and $22.8 \pm 0.4 \mu\text{g}/\text{mm}^3$ for 0°, 45°, and 90° layer orientations respectively (Fig. 5a). One-way ANOVA indicated a statistical difference between the groups' mean values ($p < 0.05$), and the Tukey's test showed a significant difference between the 0° layer-oriented group and the other groups. No significant difference was reported between the 90° and the 45° layer orientation groups.

The mean solubility and the standard deviation for all three layer orientation groups were calculated to be $1.5 \pm 0.4 \mu\text{g}/\text{mm}^3$, $1.3 \pm 0.3 \mu\text{g}/\text{mm}^3$, and $1.4 \pm 0.5 \mu\text{g}/\text{mm}^3$ for 0°, 45°, and 90° layer orientation respectively (Fig. 5b). One-way ANOVA indicated no statistical difference between the groups' mean values ($p > 0.05$).

All experimental groups underwent mass change with time upon storage at artificial saliva. During the sorption process, the mass rapidly increased in the first 7 days, followed by a reduced rate of increase until the equilibrium was reached at day 42 (Fig. 6). Then, the mass was decreased with the desorption process steadily in the first 7 days, followed by a slow decrease until equilibrium was reached at day 21 of the desorption process. The highest rate of mass change was associated with the 0° orientation group, followed by 45° and 90° groups.

3.4. Morphology of the superficial and fractured surfaces

Layering structure was observed in the specimens prepared with all three orientations and with different curing times as shown in Fig. 7. However, after polishing to a depth of approximately 0.5 mm into the sample, the layering structure was disappeared in all cases. This indicated that the layering structure was only present at the superficial surface not within the main body of the specimens.

SEM images of the fractured surface are presented in Fig. 8 to illustrate the differences between different layer orientations. The microstructures in all the specimens showed globular-shaped flower type lamellar structure with clear separation boundaries. Long and narrow structures were also found in some cases. Random voids, white particles and cracks were also visible in the microstructure. However, after careful screening of the images, no clear differences were found among the specimens with respect to cracks direction, crack amount, or volume of voids.

4. Discussion

4.1. Summary of the study and hypothesis testing

Denture base is a polymeric material that is mainly composed of PMMA. The digital revolution of 3D printing technology introduced many advantages to the current manufacturing methods in dentistry [29,30]. Many factors could affect the properties of the printed PMMA for denture base application and these factors can be divided into uncontrollable factors such as material composition, light wavelength, and light power whereas the controllable factors like printing orientation, post-curing time, and post-curing temperature [14,16,23,31–34]. The aim of this study was to investigate the effects of printing orientation and post-curing time on the mechanical and physical properties of the 3D printed denture base resin. The results showed that the post-curing times and printing orientations considered in this study did affect properties of the printed NextDent PMMA resin. Therefore, the null hypothesis tested here should be rejected. The flexural strength and flexural modulus found in this study were higher than the ISO standard recommendation and the values provided by the manufacturer. One the other hand, the sorption was lower than the manufacturer's value, but the solubility was higher than the manufacturer's value but still within the ISO standard recommended range.

4.2. Analysis of the results and comparing with literature

4.2.1. Mechanical properties

During clinical functioning, a combination of compressive, tensile, and shear stresses are exerted on the denture base which could lead to failure of the prosthesis [35–38]. Hence, it is important to assure that the denture base meets certain standards in term of surface and core mechanical strength in

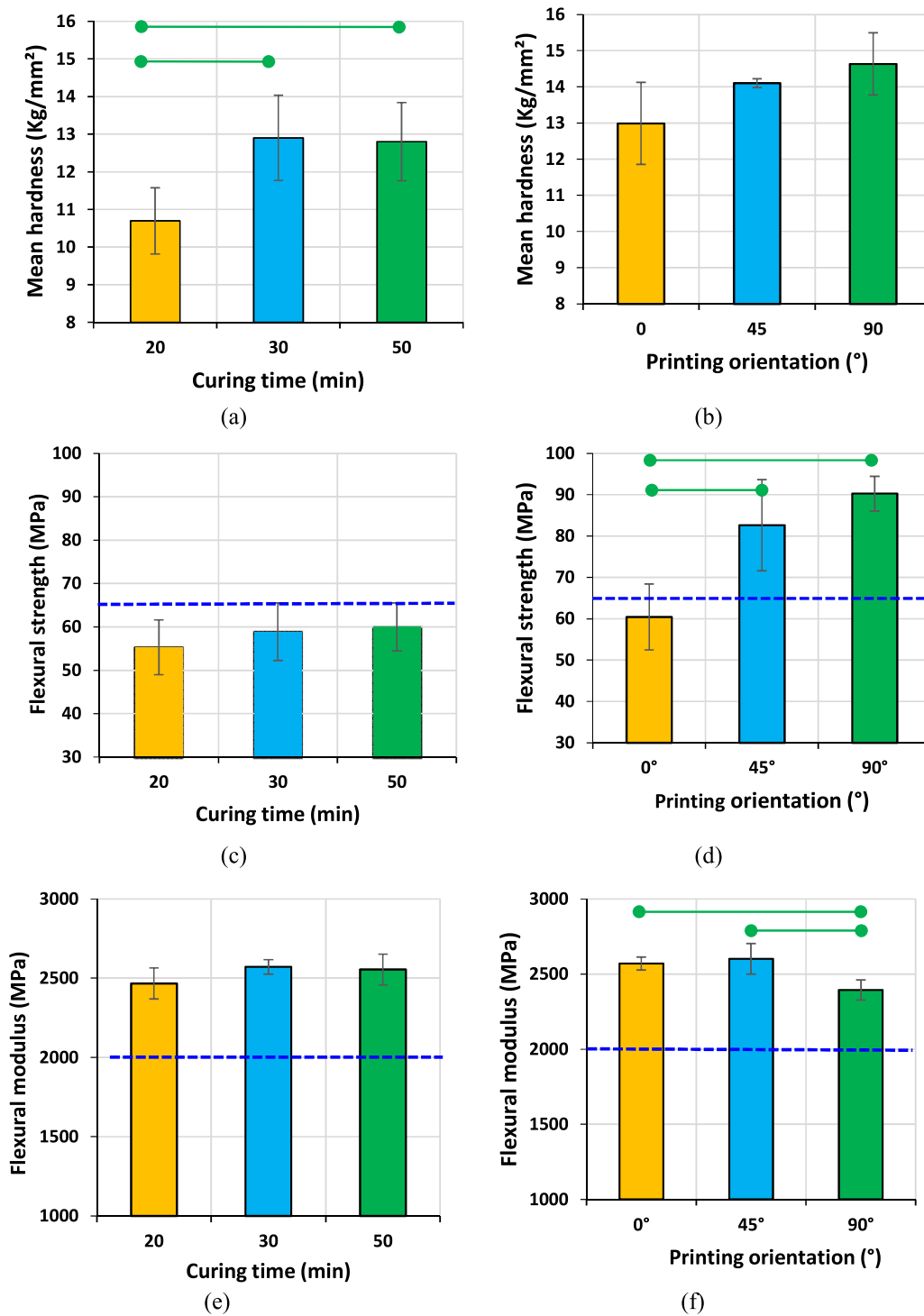


Fig. 2 – The effect of curing times and layer orientations on (a, b) Vickers hardness, (b, c) flexural strength, and (d, e) flexural modulus of 3D printed resin. Horizontal lines joining two points indicate statistically significant difference. Horizontal dotted lines indicate minimum requirements for denture base applications.

order to resist deformation and fracture under the mastication loads. Mechanical and physical properties of 3D printed objects differ than those made by subtractive manufacturing in which the subtractive manufactured blocks are cured in a high temperature and pressure environment, and their properties are well established before denture fabrication

[39]. 3D printing technology uses photopolymerised resin materials that are highly dependable on the parameter used during printing and post-curing processes [32,33,40].

VH represents abrasion and indentation of the prosthesis during function especially when chewing hard food [41]. Denture base materials should have high surface hardness in order

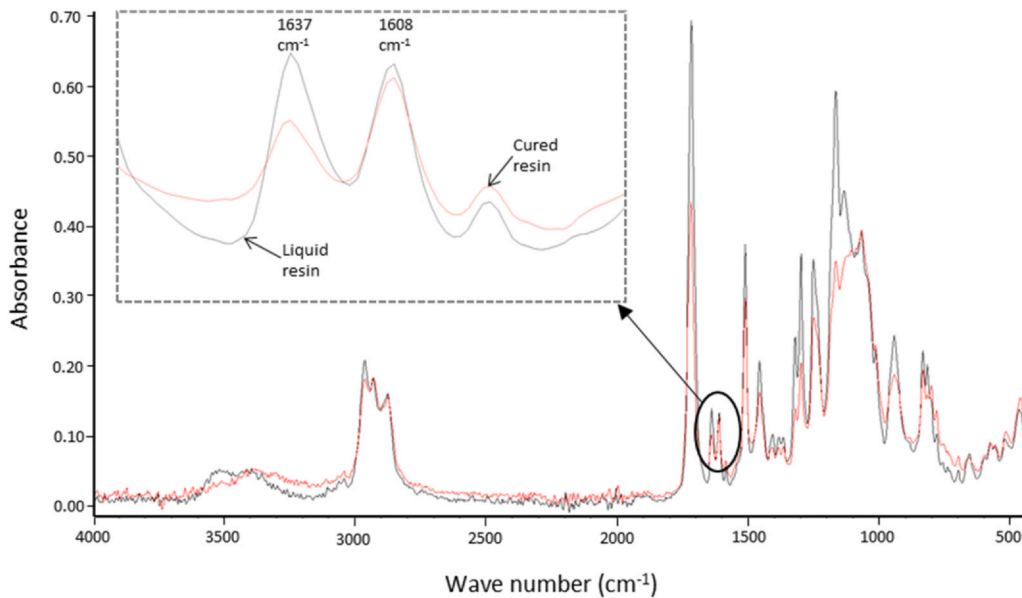


Fig. 3 – FTIR analysis for liquid and fully cured (30 min curing and 90° printing orientation) 3D printed resins.

to resist dimensional changes, surface damages, and scratches after using toothbrushes to clean the prosthesis [42,43]. VH results showed that the 0°/30 min group (control) did not show any significant difference compared to the other groups except 0°/20 min group, which showed significantly lower value. At 0° PO, an increase in curing time beyond 30 min did not show any further improvement in hardness. One study [23] found similar observations to this study as they disclosed an insignificant increase in the surface hardness when increasing CT beyond 15 min. Another study [11] revealed that the increase in CT had significantly improved the surface hardness of the denture base, but the comparison was made between specimens prepared with no post-curing (green state) and 20 min CT. However, generally the surface hardness showed an increasing trend with the POs in this study. The highest hardness was

achieved in the 90°/30 min group, which could be considered as the optimum combination of PO and CT.

Flexural strength and flexural modulus are important mechanical properties that determine the extent to which the denture base can resist plastic deformation under loading [44]. During flexural testing, the applied force was parallel to the layer orientation in a 90° PO specimen (Fig. 2) compared to the perpendicular to the layer orientation in a 0° PO specimen. Before carrying out the flexural test, it was assumed that the 90° orientation would produce the lowest values based on the assumption proposed by Shim et al. [13] and Alharbi et al. [14] that strength between the successive layers were weaker than the strength within individual layers. Surprisingly, the 90° PO group showed higher values compared to the 0° and 45° PO groups. This outcome agreed with

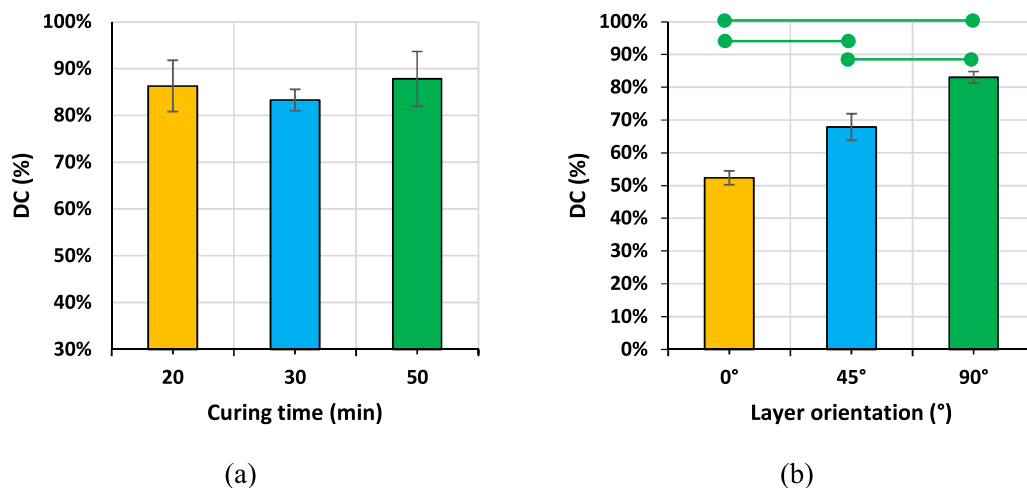


Fig. 4 – Degree of Conversion (DC) for the 3D printed resins at different (a) curing times and (b) layer orientations. Horizontal lines joining two points indicate statistically significant difference.

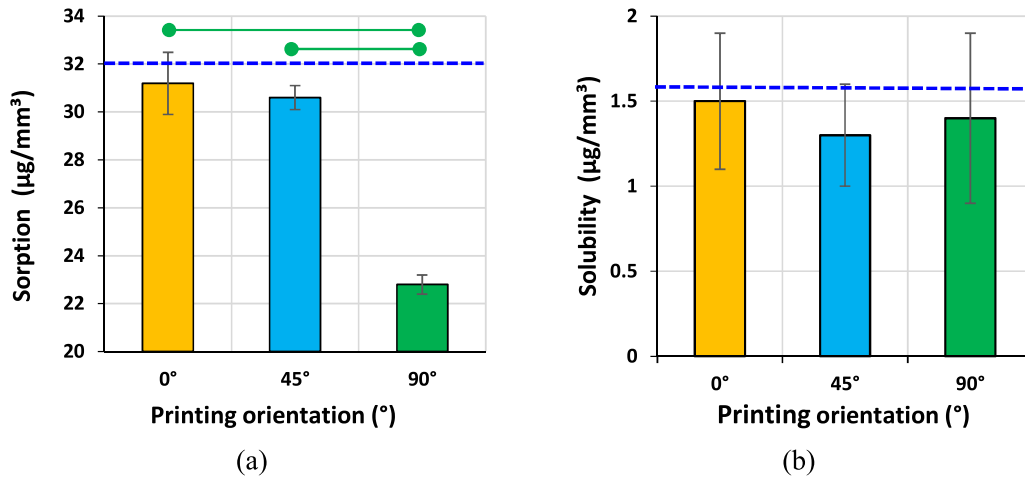


Fig. 5 – The effect layer orientation on artificial saliva (a) sorption and (b) solubility. Horizontal lines joining two points indicate statistically significant difference. Horizontal dotted lines indicate minimum requirements for denture base applications.

studies by Unkovskiy et al. [16] and Vayrynen et al. [17]. They hypothesised that there was no difference in strength between and within the resin layers. It should be noted that for all three curing times at 0° PO, the specimens failed to achieve the minimum required flexural strength of 65 MPa but the other two PO groups produced much higher flexural strengths than the recommended value, while the 90° PO groups produced the highest values (> 88 MPa). One of the

observations that could affect the results is the material's shrinkage behaviour, as the 0° PO specimens bent along the specimen, unlike to the others groups where the specimens were straight. The force was applied to the 0° PO specimens as the bending was facing downward (Appendix: Fig. A1). However, much longer time required for printing the 90° PO groups (~8 h) compared to the 0° PO groups (~2 h) might compromise the strength benefit to a certain extent.

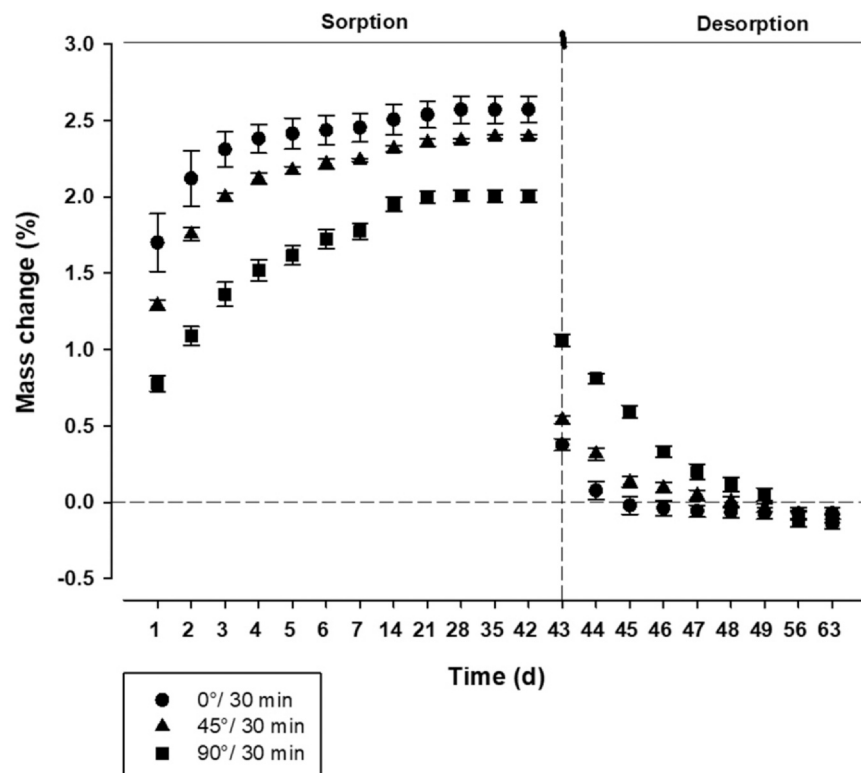


Fig. 6 – A graph illustrating the mass change of specimens immersed in artificial saliva over 63 days.

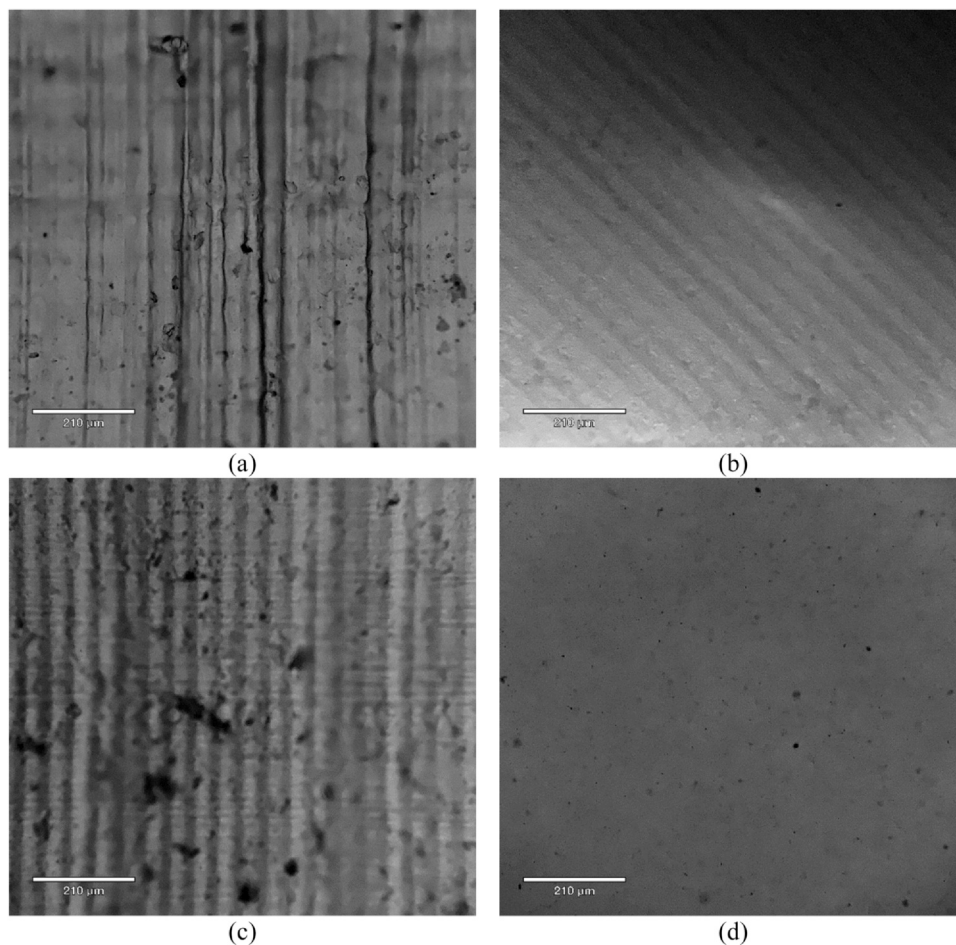


Fig. 7 – Optical microscope images of 3D printed denture base resin material cured for 30 mins showing the surface morphology across the thickness with different layer orientations: (a) 0° (b) 45° and (c) 90°. (d) Representative surface for all specimens after mechanical polishing.

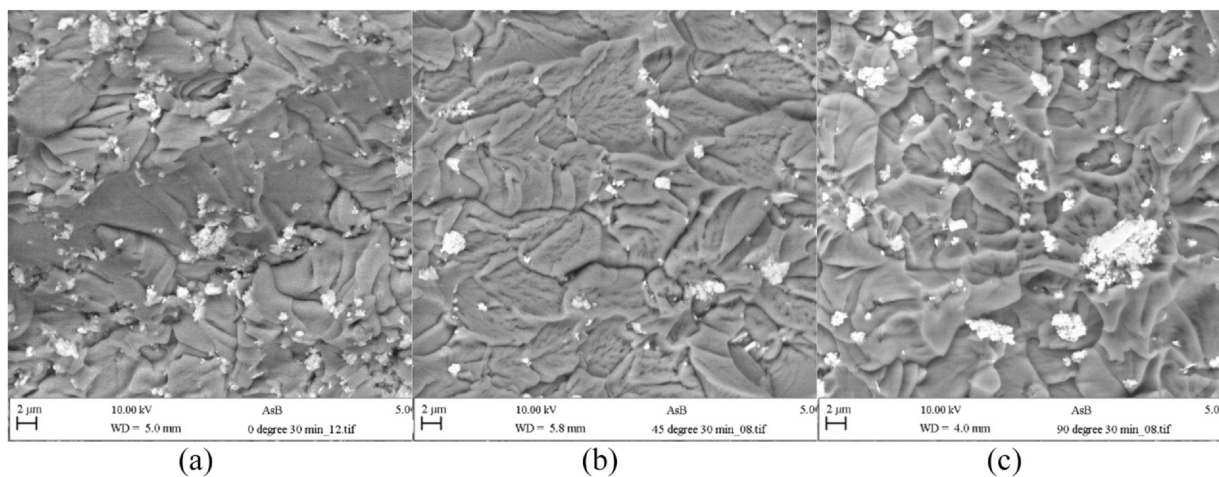


Fig. 8 – Fractured surface morphology of 3D printed denture base resin material with different layer orientations and 30 min curing times showing no layering at the internal structures: (a) 0°, (b) 45°, (c) 90°.

In this study, denture base resin material manufactured via 3D printing was evaluated based on three CTs. In general, 20 min curing time showed lower mechanical properties in this investigation. However, highest flexural strength and modulus were found with 50 min and 30 min curing times respectively. Bonada et al. [21] studied the effect of post-curing time on the flexural strength of a 3D printed resin, and found no significant difference between 20 and 40 min post-curing times (41.72 MPa and 40.36 MPa respectively). Katheng et al. [31] also studied the effect of CT and temperature on the printed part accuracy, fit, and DC and they concluded that the results were less likely to be affected by the CT. These two reported studies were in agreement with the current study. On the contrary, Kim et al. [23] found a significant difference in flexural strength of a 3D printed resin material when post-cured for 60 and 90 min (145.0 MPa and 150.0 MPa) compared to 15 and 30 min (around 121.9 MPa). These differences could be related to the difference in materials, printing technology, and the post-curing devices used. Aati et al. found a significant difference when compared mechanical and physical properties of 3D printed denture base of 0 and 5 min post-curing time groups to 20 min post-curing time group. They reported no significant difference between 10 and 20 min post-curing time in term of mechanical properties, DC, and water sorption and solubility, and 20 min was recommended to get the optimal results [11]. The current study did not include any groups that were cured beyond 50 min, as a prior pilot study found that 70 min post-curing time which did not result in any statistical difference in the properties of the material when compared to the 50 min group. The current study found that a curing time of 30 min represented the optimum duration to achieve the highest mechanical properties without wasting curing time.

For any combination of CTs and POs, the flexural modulus was found to be higher than the recommended value of 2000 MPa for the denture base, and the highest value was found for 45°/30 min group. Surprisingly the 0° PO group showed relatively higher flexural modulus compared to other PO groups. Unkovskiy et al. had similar results and they explained that this could be due to the reduction in the specimen thickness, which was inversely proportional to the flexural modulus by a power of 3. During printing, the Z axis of the printed object was reduced compared to the original dimension entered in the STL file [16]. Thus, the thickness of the 0° oriented specimen was reduced and subsequently increased the flexural modulus. Unkovskiy et al. claimed that if the thickness was controlled precisely between all PO groups, the 0° PO group would show the lowest flexural modulus value.

4.2.2. Degree of conversion

To investigate the effect of PO on the properties of the specimens, FTIR analysis was conducted to measure the DC. During printing, photo-initiators start to convert monomers into polymer in order to create bonded chains at the molecular level [45]. Increasing the DC could enhance the physical, mechanical, chemical, and biological properties of photopolymerised resin materials [46–50]. The layering technique used in additive manufacturing technology could lead to insufficient polymerisation through each added layer which could lower the degree of polymerisation. Hence, the

post-curing process was introduced to convert the unreacted monomers into polymers that could increase the DC [23]. Katheng et al. [31] reported a significant effect of curing time and temperature on DC of 3D printed resin. Also, Lowery et al. [51] found similar relationship between the post-curing environment and DC. In this study, although no significant difference in DC was found with different curing times, but the printing orientation was positively related with the DC. FTIR analysis confirmed that at 30 min CT, the DC of 90° PO group demonstrated significantly higher values compared to the other PO groups with 0° PO group showing the lowest DC values. To explain this effect of printing orientation, the number of layers required to form the flexural test specimens at each orientation were counted as an example. For the 0° PO group, 45 layers with a cross section of 65 mm × 10 mm were required to print one specimen, while 1297 layers per specimen was recorded for the 90° PO group having 10 mm × 3.3 mm cross-section. This extra light exposure and smaller cross-sectional area due to the higher number of layers during printing might have a direct influence on the DC as both groups underwent the same post-curing process to complete the polymerisation process. The DC's results could explain the significant improvement in the mechanical properties of the 90° PO group.

4.2.3. Sorption and solubility

Since the oral environment is the habitat of dentures, water sorption and solubility are very important factors that could affect the denture durability. Polymeric materials tend to absorb water due to their inherent polarity [52]. Water sorption is a process where water molecules are absorbed within the polymeric material due to its small size [53,54]. Water solubility represents the unreacted monomers and other soluble materials that was dissolved after water (or other solutions) penetrated the resin matrix causing leaching, weakening and plasticisation of the polymeric material [55,56]. Artificial saliva (AS) was used in this test as a storage medium in order to simulate the effect of oral fluids on the material properties. Sorption results only at 30 min CT with different POs showed that most of the mass changes at the specimens occurred within the first week, and the total equilibrium was reached after 2–4 weeks. Sorption progressively decreased with the increasing angles of the PO particularly at 90°. The sorption results were associated with the DC results, as the 90° oriented group showed highest DC compared to other groups, which led to a less AS uptake due to the higher monomer conversion rate. The mean values for sorption test also coincided with the hardness and flexural strength results as the higher values were associated with the 90° PO group. On the other hand, during desorption, the most mass changes occurred within the first 4 days, and the total equilibrium was reached after 14 days. 0° PO group showed slightly higher solubility as its DC was lower compared to the other groups. Therefore, more unreacted monomers were leaching out along with other additives [55].

4.2.4. Surface characteristics

Lines representing stacking of successive layers during printing were clearly visible along the width of 90° and 45° PO specimens and along thickness of 0° PO specimens' optical

microscope and the SEM images. After mechanical polishing, it was noticed that the layered structure disappeared, which indicated their presence only on the external surfaces of the specimens. SEM images of the fractured surfaces also confirmed this observation, as the layer separating lines could not be observed. This was in agreement with the observations by other studies [23,24].

4.3. Limitations and future studies

The complete specification of the commercial resin was not fully revealed by the material manufacturers due to some confidentiality reasons.

In some cases, the samples were printed from two different batches of the same resins due to one litre resin bottle. This could cause some variations in the sample properties.

Curing temperature was set to 60 °C as per the recommendations by the manufacturer. In general, the results demonstrated that the curing time did not cause any significant changes in the resin properties. However, variation in the combination of different curing times and curing temperatures could affect the resin properties and this could be a subject of future study. Other mechanical properties such as impact or fracture toughness and physical properties such as colour stability in food simulating fluids could be conducted in future.

4.4. Clinical significance

The 3D printed resin materials showed promises with improved mechanical and physical properties suitable for re-

placing conventional denture base fabrication technique in prosthetic dentistry.

5. Conclusions

In this study, NextDent resin denture base specimens were prepared using 3D printing at different printing orientations and curing times. Within the limitations of this study, the following conclusions can be derived.

- (1) The flexural strength of the 3D printed denture base resin was affected by the printing orientation but not the curing time, and the values increased with the increasing angles of the layer orientation from 0° to 90°.
- (2) Surface hardness was altered with the printing orientation and curing times, but in all cases the changes were not significant except for the 0°/20 min group which showed the lowest value.
- (3) An increasing trend of DC of the 3D printed resin with the printing orientation angles was found and the highest values were associated with 90° layer orientation.
- (4) At 30 min curing time, the 90° printed resin displayed significantly lower sorption compared to the other two orientations.
- (5) Overall, 90° printing orientations and 30 min curing times produced the highest mechanical and physical properties.

Acknowledgements

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Appendix

Deformation behaviour of the specimens printed at different orientation angles.

See Fig. A1.

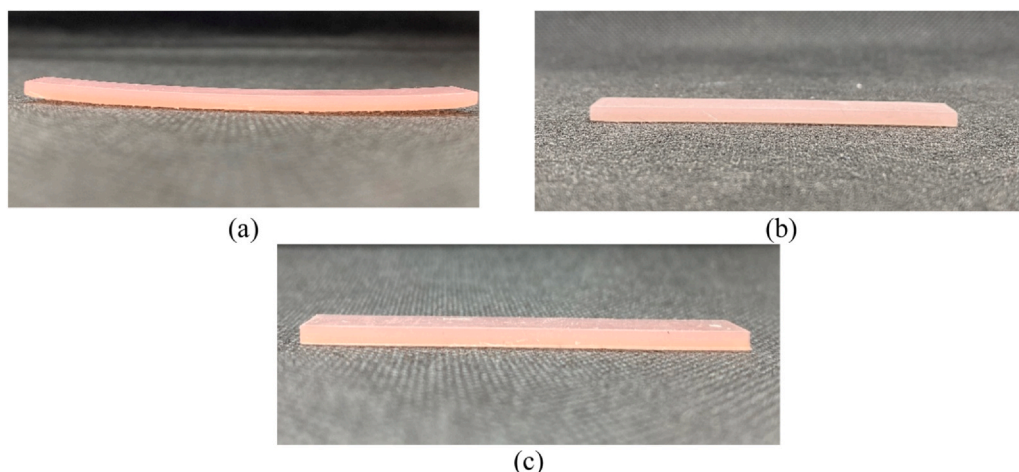


Fig. A1 – Images of 3D printed denture base resin material cured for 30 min showing the shrinkage behaviour among: (a) 0° (b) 45° and (c) 90°.

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