Original Article

Evaluation of Surface Properties and Elastic Modulus of CAD-CAM Milled, 3D Printed, and Compression Moulded Denture Base Resins: An *In Vitro* Study

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ABSTRACT

Objectives: This study evaluated the surface roughness, surface hardness, and elastic modulus of CAD-CAM (Computer-aided design/computer-aided manufacturing) milled, three-dimensional printed and conventional compression-moulded denture base resins. **Materials and Methods:** Thirty specimens (65*10*3 mm) were fabricated and divided into 3 groups (10 for each group) according to the type of denture base resin, Group I contained specimens of milled denture base resin, Group II contained specimens of 3-dimensional printed denture base resin, Group III contained specimens of polymethyl methacrylate heat cured denture base resin. The surface roughness of all specimens was evaluated using an atomic force microscope. Then by using the three-point bending test, the elastic modulus of the 30 specimens was evaluated. Finally, after fracturing the specimens from the bending test, the fractured specimens of the 3 groups were used to evaluate hardness using the Vickers hardness test. Data were analyzed using one-way ANOVA and Tukey's pair-wise post hoc tests. **Results:** There were significant differences between the tested groups ($P < 0.05$). The milled denture base resins showed the lowest surface roughness (27.46 ± 5.45 nm) when compared with printed (47 ± 7.01 nm) and conventional (39.72 ± 4.72 nm) denture base resins ($P < 0.05$); however, there was a significant increase in elastic modulus and hardness of milled (3240.06 ± 61.23 MPa and 29.18 ± 3.44 Vickers hardness number) and conventional (3017.16 ± 215.32 MPa and 22.44 ± 0.98 Vickers hardness number) denture base resins when compared with printed denture (576.65 ± 37.73 MPa and 2.64 ± 0.37 Vickers hardness number) base resins ($P < 0.05$). **Conclusions:** Milled denture base resins showed the lowest surface roughness, and highest hardness and elastic modulus among the three groups.

Keywords: *3D printing, additive technique, CAD-CAM, denture base resin, elastic modulus, hardness, roughness, subtractive technique, surface roughness*

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Introduction

Polymethylmethacrylate (PMMA) based heat-cured denture base resin (DBR) is the most widely used DBR material for complete denture (CD) manufacture in the field of prosthodontics. Its popularity derives from the material's attractive working characteristics,

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acceptable physical, mechanical, and esthetic attributes, and ease of fabrication with low-cost machinery.[1]

The surface qualities of materials used in denture fabrication affect the success of dentures. CD wearers' esthetic results and quality of life can be influenced by surface roughness.[2] Furthermore, the surface roughness of DBR affects the growth of germs, particularly candida albicans, which can cause denture stomatitis.[3,4]

Surface roughness (SR) can be assessed at the nanoscale using qualitative or quantitative approaches, such as scanning electron microscopy and profilometry.[5] Nowadays, atomic force microscopy (AFM) is used widely in dentistry to investigate the properties of various materials. AFM provides 3D imaging with nanometric resolution without the necessity of working in a vacuum or any specimen preparation. This technique has proven to be the most accurate in determining SR.[6]

The material's resistance to the indentation on its surface is defined as surface hardness (SH).[4] The vulnerability of DBR to surface distortion renders it prone to cracking or breaking, shortens the lifespan of the denture and increases the risk of plaque and microorganisms.[7]

Elastic modulus evaluates the stiffness and rigidity of a substance. The increased DBR flexibility results in an increase in the amount of absorbed energy and a decrease in the possibility of DBR breakage. The rigidity of the DBR framework is required to sustain intraorally under high functional loads during mastication and parafunction and to evenly transmit stresses to the underlying structures.[8,9]

The computer-aided design/computer-aided manufacturing (CAD-CAM) has been widely used in prosthodontics for manufacturing different removable or fixed dental prostheses due to its advantages of being highly productive, less time-consuming, more efficient and more accurate than conventional manner.[10,11]

Nowadays, DBR can be made digitally. Two digital processes are commonly used: the first is CAD-CAM, which uses a subtractive approach to mill prepolymerized resin pucks, and the second is three-dimensional (3D) printing, which uses an additive manufacturing technique to build the prosthesis layer by layer.[12]

The CAD-CAM approach has many advantages, such as faster manufacturing of prostheses, fewer laboratory procedures, and reduced probability of errors. Because the denture foundation is made of prepolymerized resin puck, it has better strength, fit, physical, mechanical, and surface properties and less bacterial adherence.[13,14] However, excessive waste during milling and expensive equipment maintenance prompted the development of 3D printing. In 3D printed denture manufacture, the denture base is formed from photopolymerized powder that is sintered together to form a photosensitive liquid polymer that is added progressively and cured layer by layer by ultraviolet light or a visible light source. This method allows for the creation of required prostheses and models with the least material amount possible. On the other hand, the bonding between layers and DBR shrinkage can affect the properties and DBR accuracy.[15-17]

The effect of fabrication techniques of different DBRs on some properties of digitally fabricated DBRs has been investigated[18-24] However, there are little data available in the literature concerning the effect of different fabrication techniques on the SR, SH, and elastic modulus of new digital DBR materials, so this study aimed to evaluate the effect of different fabrication techniques of CAD-CAM milled, 3D printed and heat-polymerized acrylics DBRs on the SR, SH, and elastic modulus.

The null hypothesis of this study was that the difference in the surface properties and modulus of elasticity between the different fabrication methods of DBRs would be insignificant.

Materials and Methods

The sample size was calculated using a freeware (G*Power3.1.9.3 for Mac OS X, Düsseldorf, Germany).[8,18,19] Thirty specimens with specific measurements (65 X 10 X 3 mm) were prepared. Those 30 specimens were divided into three groups equally (10 of each). Group-I (GI); contained the CAD-CAM milled DBR specimens, Group-II (GII); contained 3D printed DBR specimens, and Group-III (GIII); contained conventional compression-molded DBR specimens.

A specimen with a specific dimension that was previously mentioned was designed virtually in CAD software (ExoCad ChairsideCad 2.3 Matera, Germany) to produce a standard tessellation language (STL) file.

GI milled DBR specimens

The designed virtual specimen was exported to the milling machine (DENTSPLY Sirona In Lab MC X5 laboratory milling machine, Bensheim, Germany). Then, the CAD-CAM acrylic prepolymerized pucks (AvaDent, Digital Dental solutions HQ; Scottsdale; USA) had been milled into these specimens according to the previously mentioned dimension (65*10*3 mm).

Burs with 1: 2.5 mm diameter were used in the subtractive process. The milling process was carried out with 5-axis to produce a more accurate specimen and to prevent overheating the milling process was carried out in a wet condition.[20]

GII DBR specimens

Using the additive technique ten 3D printed DBR specimens were fabricated by the3D-printer (WANHAO-desktop 3D-printer; Zhejiang; China) as the following:[17,21]

The designed DBR specimen was exported on STL file to be sent to the 3D-printer, using Digital Light Projection technology and photopolymerized 3D-printed liquid (Harz-Labs, Moscow, Russia) the specimens were printed. The 3D-printer had a UV light full HD projector with 380–420 nm wavelengths.

The HARZ-Labs liquid was shaken manually for approximately 3min before printing the DBR specimens, then poured into the supply chamber of the 3D printer. The DBR specimens were printed in a 45º orientation with 100µ/layer thickness, the cured layers were successively bonded together to construct the printed DBR specimen. For additional polymerization, the final DBR specimens printed were placed in a UV-light curing box (Anycubic wash and cure light box; Shenzhen; China) for 15 min.

GIII DBR specimens

For this group, one milled specimen was used to create the stone mold for the fabrication of ten DBR specimens of heat-cured PMMA (Vertex-Dental-BV; Headquarters the Netherlands) using the conventional method of compression molded technique.[22,23]

The specimens were finished using tungsten carbide acrylic burs (Edenta AG, Au, Switzerland) and silicon carbide papers with 400 grit size and polished using rubber acrylic burs (Edenta), pumice (Shera, Lemfo¨rde, Germany), and rouge (Dialux, Lüdenscheid, Germany).[19,24]

Before testing, the specimens were stored in distilled water at 37 ± 1°C for 50+2 hours.[25]

SR evaluation

Thirty specimens were analyzed using AFM (Agilent 5600LS AFM, USA). AFM was operating in tapping mode. The used probes had spring constant and resonance frequency values of approximately 10 N/m and 250 kHz, respectively. Five standardized profilometric measurements of scanned area equal to 20 × 20 µm^2 were performed on each specimen and mean average Ra values were utilized for the statistical analysis.

The instrument was calibrated before the measurements using polyethylene spheres of known diameter. Five images were collected for each specimen. The scan processes were performed by one operator, who was blind towards the specimens and the processing method.[25]

AFM images [Figure 1] were analyzed using WSxM software to calculate the root mean square (RMS) of the average height of every specimen, which can be assumed as a reliable index of SR.[26]

Elastic modulus evaluation

The elastic modulus of the DBR specimens was evaluated using the three-point loading test. By the universal testing machine (Instron 3345; Buckinghamshire; England) to determine the elastic modulus while the distance between the two centers of support was set at 50 mm, and a load cell was applied at a midpoint of the specimen with a crosshead speed of 5 mm/min till the specimen fracture.

Elastic modulus (E) in mega Pascal (MPa) was calculated using the following equation:[27]

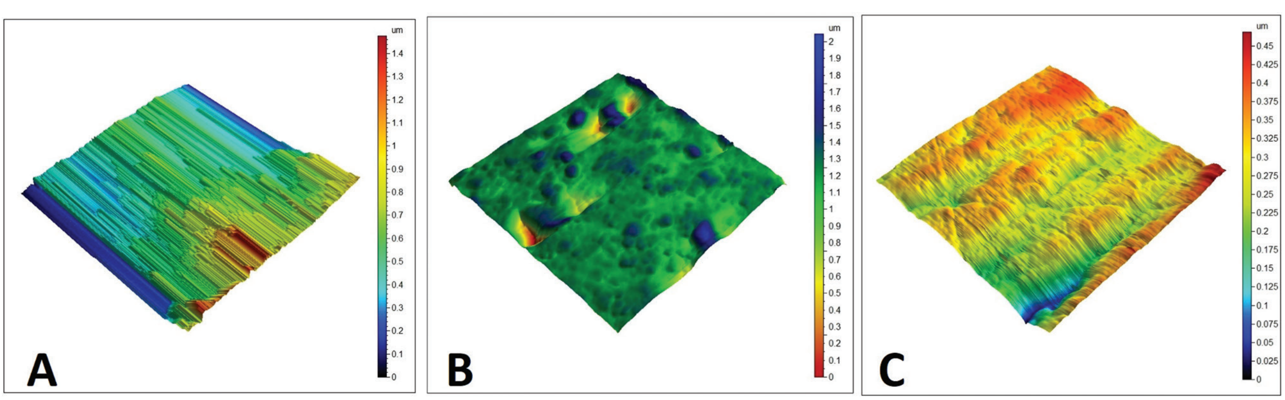

Figure 1: AFM images of the surface topography of denture base specimens with different manufacturing methods (scan of 20 x 20 µm). (A) CAD-CAM milled DBR specimen, (B) 3D printed DBR specimen, (C) compression-molded DBR specimen

$$E = \frac{FL^3}{4bh^3d}$$

where (F) is the load or force at which fracture occurred in newton (N), (L) is the span length of the specimen between the supports in millimeters (mm), (b) is the width (mm), and h is the thickness of the specimen (mm), and d is the deflection (mm).

Data were calculated and recorded using computer software (Bluehill Instron, England).

Vickers hardness evaluation

The Vickers hardness (VH) tester (Tukon 1102 Wilson hardness tester Buehler, Germany) was used for the evaluation VH of the specimens, for each specimen 300-gram load was applied smoothly, forcing the indenter into the test specimen. The indenter is held in place for (15) seconds. The specimen was subjected to this 300-gram load for 15 seconds at three different sites. After that load was removed, the indentation was focused with the magnifying eyepiece and the 2-impression diagonals were measured. The final VH value was arithmetically calculated by obtaining the mean of the 3 readings.[28]

The Vickers hardness (HV) is calculated using:

$$MVHN = 1854.4L/d^2$$

Where the VHN is the Vickers hardness number, L is the load in gf and d is the average diagonal in µm

Statistical analysis

Data were subjected to statistical analysis using 1-way-ANOVA and Tukey's pair-wise post-hoc tests at significant level (p-value< 0.05), by using IBM® SPSS® Statistics Version 20 (SPSS Inc; IBM Corporation; USA).

Results

The statistical analysis of SR values (Ra); the 1-way-ANOVA test revealed that there was a significant difference between all tested groups ($P < 0.05$). Where the GI showed, the lowest Ra mean value (27.46 ± 5.456 nm) of surface roughness, followed by the GIII (39.72 ± 4.725 nm). While the highest Ra mean value was recorded with the GII (47 ± 7.015 nm) as shown in [Tables 1 and 2]. Among the groups, Tukey's pair-wise post-hoc test showed significant differences between GI and GII, GI and GIII, and GII and GIII at a 95% confidence level ($P < 0.05$), as shown in [Table 3].

The statistical analysis of the elastic modulus, the 1-way-ANOVA test, revealed a significant difference between all tested groups ($P < 0.05$). The GI showed the highest mean value (3240.06 ± 61.23MPa) of elastic modulus, followed by the GIII denture bases (3017.16 ± 215.32MPa). While the lowest mean modulus of elasticity value was recorded with the GII (576.65 ± 37.73MPa), as shown in [Tables 4 and 5]. Among the groups, Tukey's pair-wise post-hoc test showed significant differences between GI and GII, GI and GIII, and GII and GIII at a 95% confidence level ($P < 0.05$), as shown in [Table 6].

The statistical analysis of Vickers hardness and the 1-way-ANOVA test revealed a significant difference between all tested groups ($P < 0.05$). The GI showed

Table 1: Descriptive statistics for the surface roughness (nm) for the different groups

Variables	N	Mean (nm) ± SD
GI	10	(27.46 ± 5.45) [a]
GII	10	(47 ± 7.01) [b]
GIII	10	(39.72 ± 4.72) [c]

Different superscript letter in the column denotes significant difference using Tukey`s post hoc test at 95% confidence level (p<0.05).

Table 2: One-way-ANOVA test of the surface roughness of the different groups

Variables	(p) value
GI	<0.00*
GII	
GIII	

*; denotes significant difference (p<0.05).

Table 3: Pair-wise comparisons of the surface roughness between different groups using Tukey post hoc test

Pair-wise Comparisons	(p) value	95 % CI
GI vs GII	<0.00*	-25.98 to -13.10
GI vs GIII	0.00*	-18.70 to -5.822
GII vs GIII	0.02*	0.8424 to 13.72

*; denotes significant difference at 95% confidence level (p<0.05).

Table 4: Descriptive statistics for the elastic modulus (MPa) for the different groups

Variables	N	Mean (MPa) ± SD
GI	10	(3240.06 ± 61.23) [a]
GII	10	(576.65 ± 37.73) [b]
GIII	10	(3017.16 ± 215.32) [c]

Different superscript letter in the column denotes significant difference using Tukey`s post hoc test at 95% confidence level (p<0.05).

Table 5: One-way-ANOVA test of the elastic modulus of the different groups

Variables	(p) value
GI	0*
GII	
GIII	

*; denotes significant (p<0.05).

Table 6: Pair-wise comparisons of the elastic modulus between different groups using Tukey post hoc test

Pair-wise Comparisons	(p) value	95 % CI
GI vs GII	0.00*	2518 to 2809
GI vs GIII	0.00*	77.57 to 368.2
GII vs GIII	0.00*	-2586 to -2295

*; denotes significant difference at 95% confidence level (p<0.05).

Table 7: Descriptive statistics for the vickers hardness (VHN) for the different groups

Variables	N	Mean (VHN) ± SD
GI	10	(29.18 ± 3.44) [a]
GII	10	(2.64 ± 0.37) [b]
GIII	10	(22.44 ± 0.98) [c]

Different superscript letter in the column denotes significant difference using Tukey`s post hoc test at 95% confidence level (p<0.05).

Table 8: One-way-ANOVA test of the Vickers hardness of the different groups

Variables	(p) value
GI	<0.00*
GII	
GIII	

*; denotes significant (p<0.05).

Table 9: Pair-wise comparisons of the vickers hardness between different groups using Tukey post hoc test

Pair-wise Comparisons	(p) value	95 % CI
GI vs GII	<0.00*	24.24 to 28.84
GI vs GIII	<0.00*	4.438 to 9.042
GII vs GIII	<0.00*	-22.10 to -17.50

*; denotes significant difference at 95% confidence level (p<0.05).

the highest mean value (29.18 ± 3.44VHN) of hardness, followed by the GIII denture bases (22.44 ± 0.98VHN). While the lowest mean hardness value was recorded with the GII (2.64 ± 0.37VHN), as shown in [Tables 7 and 8]. Among the groups, Tukey's pair-wise post-hoc test showed statistically significant differences between GI and GII, GI and GIII, and GII and GIII at a 95% confidence level ($P < 0.05$), as shown in [Table 9].

Discussion

The results revealed a significant difference in SR, SH, and elastic modulus between different groups, so the null hypothesis was rejected. The surface of the CAD-CAM DBRs milled group was relatively flat, smooth, and uniform in comparison with the DBRs of the other groups.

The quality and roughness of the mucosal denture surface are process specific and determined by the DBR material quality and, more importantly, by the effectiveness of the manufacturing system. In CAD-CAM milled dentures, the milling machine and milling burs affect the quality of the surface. In contrast, in the case of a conventional denture and a 3D printed denture, the quality of the printer and the master cast and the manufacturing protocol affect this property. The dental technician's manual finishing and polishing skills affect the quality of the oral surface of DBR.[29,30] So, in the present study, the finishing and polishing technique was standardized for all specimens and done by the same operator.

In the present study, we chose the Ra value as a reference measure for evaluating the SR of the three manufacturing methods. The denture with the smaller Ra values and deviations represents the denture with a smoother surface.[31,32]

The AFM images of the surface of the milled group revealed relatively few signs of serration, but it is regular, with little sharpness. In addition, the shape of the traces was also uniform. These results revealed that, all CAD-CAM DBRs had significantly smoother surfaces than the other groups with a Ra-value cut-off value of 27.46 nm.

The AFM images of the 3D printed group exhibited a relatively coarse and wrinkled surface pattern with some indentations. In addition, the overall deepness of the specimen appeared large and non-uniform in the 3D image. The results of the DBRs of the 3D-printing method exhibited a higher SR with a mean value of 47 nm more than that of the other groups.

The AFM images of the compression-molded group exhibited a smooth surface with surface heights which is regular in shape and distribution to some extent. In addition, the overall deepness of the specimen was not significant. The results of the conventional compression-molded dentures revealed a surface roughness mean value of 39.72 nm.

Thus, SR of the DBRs of milled group are significantly less than that of heat polymerized and 3D-printed DBRs ($P < 0.05$). However, according to the recommendations available in the literature, the precision level in surface qualities' reproduction should be between 0.5to 1 µm.[32] Therefore, all manufacturing methods tested in the present study consider within the acceptable clinical level.

Elastic modulus reflects the stiffness of a material and is influenced by the degree of polymerization reached. When comparing acrylic resin strengths, those with a lower degree of polymerization have less favorable mechanical characteristics.[33,34]

The mean elastic modulus values were the highest for the milled DBR (3240.06±61.23 MPa). The elastic modulus exhibits the degree of rigidity of a material, therefore, denture bases that were made from the milling technique showed the highest rigidity. However, the denture bases that were made from photopolymerized 3D printed manufacturing methods exhibited the least rigidity. The modulus of elasticity of the processed DBR should not be less than 2 GPa,[18] so the tested DBRS that were made from milled and compression-molded manufacturing methods in the current study are suitable for clinical use. However, the DBRs made from photopolymerized 3D-printed manufacturing methods are not suitable for clinical use.

The lower SR and higher elastic modulus of CAD-CAM milled DBR compared to those of 3D-printed and conventional DBRs are attributed to the fact that milled DBRs are fabricated from solid, pre-polymerized plates, which are manufactured under a high degree of pressure and condensation leading to a high degree of conversion with less residual monomer, porosities, and voids.[34]

The highest SR and lowest elastic modulus values of the DBRs made via the 3D-printing method may be due to the lower degree of polymerization and the leakage of excessive residual monomer. The residual monomer may increase surface porosity and hence increase its SR. It also acts as a plasticizer which decreases the modulus of elasticity of the fabricated denture.[35]

When comparing the compression molding fabricating method to the milling method, the higher SR and lower elastic modulus of the conventional heat-cured PMMA are due to voids and porosities that result from monomer vaporization during the heat-curing process and/or air trapped during mixing. This result supports the claim of manufacturers assigning mechanical favorability of CAD/CAM DBRs to the polymerization process of PMMA at high pressure and temperature.[33,36]

The current study's findings demonstrated that denture bases formed via compression molding had a much greater mean elastic modulus than those created by 3D printing. This could be explained by that the conventional approach achieves a higher degree of conversion and polymerization.[37]

In the current study, the CAD-CAM milled DBR demonstrated the highest SH among the three groups, this may be attributed to the reduced residual monomers with subsequent plasticizing action that would enhance hardness.[4] In contrast, the 3D-printed group showed the lowest SH among the three groups. This may be due to a weak double bond conversion,[38] printing layering and the material constitution of the 3D-printed DBRs, or due to water sorption with thermal stressing.[2,39]

The lack of simulation of oral condition and long-term water storage are the main limitations of the current research work, also the thermocycling was not performed, and different results may be shown with different types of printing resins and printers.

Conclusion

The CAD-CAM milled DBRs had the lowest surface roughness, highest surface hardness and highest elastic modulus than 3D printed and compression moulding DBRs. The 3D-printed DBRs group showed the highest surface roughness, lowest surface hardness and lowest elastic modulus among the three groups.

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Conflicts of interest

None.

Authors' contributions

Ahmed A. Zeidan conceived of and designed the study, collected and analyzed the data, wrote the manuscript, financially supported this research work and gave final approval for the manuscript. Mohamed Helal is the principal investigator, conceived of and designed the study, collected and analyzed the data, wrote the manuscript and gave it his final approval. Ramy A. Abdelrahim collected and analyzed the data, provided a critical revision and gave final approval for the manuscript. Nehad M. Harby undertook statistical analysis, provided a critical revision, financially supported this research work and gave final approval for the manuscript. Adel Fawzy Abdelhakim contributed in data collection and analysis, provided a critical revision, financially supported this research work and gave final approval for the manuscript.

Ethical policy and institutional review board statement

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Patient declaration of consent

Not applicable.

Data availability statement

The data set used in the current study can be made available on request.

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