Original Article

In-vitro Comparative Evaluation for the Surface Properties and Impact Strength of CAD/CAM Milled, 3D Printed, and Polyamide Denture Base Resins

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ABSTRACT

Objective: There is a paucity of data regarding the effect of fabrication techniques and compositions of CAD/CAM milled, 3D-printed, and polyamide flexible denture base resin materials (DBRMs) on the surface roughness (SR), surface hardness (SH), and impact strength (IS). Therefore, this study aimed to evaluate the SR, SH, and IS of CAD/CAM milled, 3D-printed, and polyamide flexible DBRMs. **Materials and Methods:** Ninety specimens were constructed from different DBRMs and divided into three groups (CAD/CAM, 3D-printed, and polyamide DBRMs; n = 30) with specific measurements: 15 × 10 × 2.5 mm for SR and H tests and 80 × 10 × 4 mm notched specimen for IS test. SR meter and Vickers micro SH tester were used to measure SR and SH, respectively, whereas the IS was evaluated using Charpy's impact testing machine. Data were collected and statistically analyzed using one-way analysis of variance and *post hoc* Tukey's tests (α=0.05). **Results:** There were significant differences between the tested materials (P< 0.05). The CAD/CAM milled showed lowest SR when compared with 3D-printed resin and polyamide flexible resin (P< 0.05); however, there was a significant increase in SH of CAD/CAM milled and 3D-printed DBRMs when compared with polyamide materials (P< 0.05). There was a significant increase in IS of polyamide and CAD/CAM milled resins when compared with 3D-printed DBRMs (P < 0.05). **Conclusion:** CAD/CAM milled resins showed high IS and SH with lower SR.

KEYWORDS: *3D printing, CAD/CAM, flexible resin, hardness, impact strength, surface roughness*

INTRODUCTION

The majority of denture bases are made of polymethylmethacrylate (PMMA) that are expected to be constructed within the standards of professionally acceptable prosthetic care. The most frequently utilized DBRM for a removable prosthesis is heat-polymerized PMMA. Despite numerous benefits such as aesthetics, stability, biocompatibility, simplicity of production, and repairability, some mechanical characteristics are less effective, resulting in denture base fractures.[1] Furthermore, searches for alternative resin materials with superior mechanical strength than PMMA are continuing. Also, alternative polymer systems to PMMA like polyamide have been tried. Polyamides are thermoplastic polymers produced when a diamine and a dibasic acid combine to form a polymer.[2]

Many researches have been conducted in the hopes of developing new or improved DBRMs. The application of CAD/CAM technology is increased with the

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milling and 3D printing of dentures; the denture might be constructed using subtractive or additive manufacturing techniques. Subtractive technique can be done by milling to remove sections of DBRMs; however, the additive technique includes adding layers of DBRMs using 3D printing to create denture base.[3,4]

There are several advantages for digital denture production: decrease in the number of visits, improvement in denture strength and fit, reduction in the hazard of microbial colonization on denture surfaces, advancements in standardizing for clinical studies on removable prostheses, easier denture reproduction and fabrication of a trial denture utilizing saved digital data, and improved process control by clinicians and technicians.[5]

Impact strength (IS) is a measure of energy absorbed by a material when it is broken by sudden blow; also it is the measure of brittleness of the material which is used as an indicator of the ability of dental structure to resist fracture when dropped or given a sudden shock, usually outside the patient's mouth.[6] A material may have reasonably high static strength values, such as tensile, compressive, and shear strengths, and even reasonable elongation, but may fail when load is applied under impact.[7] Surface roughness (SR) is an important property of acrylic resin as a rough surface may affect tissues' health due to microorganism adhesion and growth.[8] Therefore, smooth and highly polished denture surfaces are essential for aesthetical outcomes, good oral health and low plaque retention, preventing oral diseases, comfort of patients, and denture durability.[9,10] Surface hardness (SH) is described as the material's surface resistance to indentation or penetration. The correlation between material's mechanical properties and surface hardness was reported.[9] For example, the susceptibility of acrylic-polymer to surface deterioration makes it susceptible to fracture and increases the risk of plaque and microorganism and putting the denture base's lifespan at risk.[10]

There is lack of data regarding the effect of manufacturing process and compositions of different DBRMs on the surface properties and IS. So, this study was aimed to investigate SR, SH, and IS of CAD/CAM milled, 3D-printed, and injection molding DBRMs. The null hypothesis of this study was that the differences between the properties (SR, SH, and IS) of CAD/CAM milled, 3D-printed, and flexible DBRMs would be insignificant.

Materials and Methods

Specimen's fabrication

Specimen's dimensions and grouping

For this study, 90 specimens were fabricated and divided according to the fabrication methods into three groups; (GI): CAD/CAM prepolymerized acrylic denture base (n = 30), (GII) 3D-printed denture base resin (n = 30), and (GIII): flexible denture base resin (n = 30). Specimens were prepared in dimensions according to the test specifications: 15 × 10 × 2.5 mm for SR and SH, 80 mm × 10 mm × 4 mm with notch of 1 mm depth at the middle of specimen for IS test according to the ISO standard (20795-1:2013).[11]

CAD/CAM specimens

Rectangular specimens were designed using ExoCad software (ExoCad, ExoCad GmbH, Darmstadt, Germany) producing standard tessellation language (STL) file.

CAD/CAM milled specimens

The STL files of designed specimens were uploaded to the CAM program (Roland mind) in order to execute the cutting process command given to the milling machine (Ceramill Motion 2; Amann Girrbach, Austria). Then a computer-aided machine automatically milled those designed specimens from prepolymerized CAD/CAM resin discs (100% by weight, PMMA) (Polident d.o.o. VolčjaDraga 42, VolčjaDraga, Slovenia) using subtractive technique. During the milling process, burs with a maximum diameter of 2.5 mm and a minimum diameter of 1 mm and 5-axis were utilized to produce more fine details accurately and in wet condition to avoid overheating according to the manufacturer.

CAD/CAM 3D-printed specimens

GII; the STL files of the designed specimen were exported to a 3D printer (Phrozen Shuffle, Phrozen, Hsincu City, Taiwan) with UV light source and have wavelength ranging from 380 to 420 N m. The specimens were printed using acrylate ester-based resin using additive technique. The photopolymerized 3D-printed liquid (NextDent, Soesterberg, The Netherlands) was shaken for approximately 5 min before pouring into the 3D printer supply chamber. The printing parameters were 100 μm layer thickness and 45° printing orientations. Isopropyl alcohol (99% concentration) was used to rinse printed specimens to remove unpolymerized acrylic resin followed by post-polymerization by placing specimens in the UV light curing unit (bre.Lux Power Unit; bredent GmbH & Co., Senden, Germany) for 15 min.

Polyamide specimens' preparation

GIII; for polyamide specimens fabrication, stainless steel dies with 15 × 10 × 2.5 mm (for SR and hardness tests) and 80 mm ×10 mm × 4 mm with notch of 1 mm depth at mid-span (for impact test) were made. Metal dies and sprue former were invested in dental stone and then carefully removed leaving mold spaces and channels for material flow. According to the

manufacturer recommendations for injection molding technique, the polyamide resin (BreFlex Second Edition, Germany) was heated at 220–265°C for 15 min and injected by a multipress machine (MULTIPRESS, Moulding Machine, Roko, Poland).

Specimens finishing and polishing

Acrylic bur (Edenta, ISO No. 685 104 273 533 100) was used for specimens' finishing, followed by a 400 grit size silicon carbide paper. The standardized polishing method was applied using soft brush, wet pumice, and then rouge. One operator performed specimens' polishing. Specimens with proper dimensions were preserved in distilled water at 37 ± 1°C for 48 ± 2 h.

Testing procedure

Surface roughness test (μm)

The SR test was done using a surface roughness meter (SJ-210 Surface Roughness-Tester, Mitutoyo, Japan). It is made up of a Stylus that was mechanically moved across the surface of the specimen by the drive unit. It also moved in the vertical direction up to the specimen surface as it ascends or descends over the irregularities of the specimen. This movement was converted to a corresponding electrical signal. A high cut-off value was selected to measure all micro- and macro-irregularities. Three SR measurements were carried out for each specimen, and mean average Ra values were utilized for the statistical analysis.

Hardness test (VHN)

All specimens were tested for Vickers hardness number (VHN) immediately after they were removed from the distilled water to evaluate the SH. The VHN tester (Tukon 1102 Wilson Microhardness Tester, Buehler, Germany) was utilized. In the Vickers test, 300 gf load is gently applied for 15 s.[9] After the load was eliminated, the magnifying eye piece was used to focus the indentation and two impression diagonals were measured, usually to the nearest 0.1 μm with a filar micrometer, and averaged. The Vickers hardness (HV) is determined using the following formula:

$$VHN = 1854.4L/d^2,$$

where L is the load (gf) and d is the average diagonal (μm).

Impact strength test (kJ/mm²)

The testing of IS was done using a Charpy's impact testing machine (ZWICK/ROELL HIT 50P, Germany). Then the test was conducted, the specimens were supported horizontally at its ends on Charpy's type impact tester, and struck at the mid-span by a pendulum which dropped from a specified height in the middle. The force was applied to the specimen from the unnotched side. A 15 J pendulum was utilized for testing. The impact energy absorbed to breaking the specimen was recorded by a scale in joules. The Charpy IS was determined in kJ/mm² and calculated using the following formula:

$$\text{Impact strength} = \frac{E}{bd} \times 10^3$$

where E is the absorbed energy (J), b the specimens' width (10 mm), and d is the specimen thickness (4 mm).

Statistical analysis

IBM® SPSS® Statistics Version 20 (SPSS, Inc., IBM Corporation, NY, USA) was used for statistical analysis of collected data. One-way analysis of variance (ANOVA) was used to determine whether significant differences existed among the means of the studied groups or not. Tukey's pairwise *post hoc* test was used for pairwise comparison at the chosen level of probability ($P < 0.05$).

Results

The informative statistical analysis shows mean values and standard deviation (SD) of SR measured in μm, SH measured in gf/μm², and IS results measured in kJ/mm² for all tested groups [Table 1].

The statistical analysis of SR, SH, and IS of all tested groups showed that the difference among all tested groups was statistically significant as indicated by one-way ANOVA test: (F=26.689, $P < 0.0001$) for the Ra test, (F=953.56, $P< 0.0001$) for the SH test, and (F=156.628, $P< 0.0001$) for the IS test.

For all tested properties, Tukey's pairwise *post hoc* test showed statistically significant difference ($P< 0.001$) among all the tested groups. The CAD/CAM milled showed lowest SR when compared with 3D printing and polyamide flexible resins ($P < 0.05$); however, there was a significant increase in SH of both CAD/CAM milled and 3D-printed denture base resins when compared with the polyamide material ($P < 0.05$). There was a significant increase in IS of both CAD/CAM milled and polyamide flexible resins when compared with 3D-printed denture base resins ($P < 0.05$).

Discussion

DBRMs with superior surface properties accompanied with high IS are required for denture longevity. In the present study, newly introduced CAD/CAM DBRMs were investigated in terms of SR, SH, and IS. According to the results of this study, there were significant differences between DBRMs for all tested properties, so that the null hypothesis of this study was rejected.

Table 1: Comparison of SR test, hardness test, and IS test results between all tested groups

	Measurements mean (mm) ± SD	ANOVA test *P*-value (*F*-value)	Tukey's *post hoc* test Pairwise comparisons	*P*-value
Surface roughness (μm)				
GI. CAD/CAM	0.2445^{C}±0.32	$P < 0.0001$** (F=26.69)	G1-vs.-G2	$P < 0.0001$*
GII. 3D-printed	1.0003^{A}±0.24		G1-vs.-G3	$P < 0.0001$*
GIII. Flexible	0.7417^{B}±0.30		G2-vs.-G3	0.047*
Hardness (gf/μm^2)				
GI. CAD/CAM	26.14^{A}±0.39	$P < 0.0001$** (F=953.56)	G1-vs.-G2	$P < 0.0001$*
GII. 3D-printed	20.06^{B}±0.39		G1-vs.-G3	$P < 0.0001$*
GIII. Flexible	14.39^{C}±0.89		G2-vs.-G3	$P < 0.0001$*
Impact strength (kJ/mm^2)				
GI. CAD/CAM	25.88^{B}±0.54	$P < 0.0001$** (F=156.63)	G1-vs.-G2	$P < 0.001$*
GII. 3D-printed	23.18^{C}±0.71		G1-vs.-G3	$P < 0.001$*
GIII. Flexible	30.30^{A}±0.83		G2-vs.-G3	$P < 0.001$*

*Significant ($P < 0.05$)

The results of this study showed that CAD/CAM milled resins showed a significant decrease in SR value when compared with flexible resin and 3D-printed resin, respectively. This decrease may be due to the fabrication process of prepolymerized resin blocks, where it is fabricated at high temperature and under appropriate pressure which improved degree of conversion and lesser residual monomer. Therefore, the prepolymerized resin exhibited little shrinkage, porosity, or free monomers,[12] similar to Rosca *et al.* findings.[13]

Moreover, polyamide flexible resins showed a lower mean SR value when compared with 3D-printed resin because polymerization of thermoplastic resin at high pressure lowered its monomer contents, which in turn decrease its porosity due to monomer evaporation.[14] Furthermore, the pressure exerted by injection of the acrylic resin during injection molding processing technique of the thermoplastic resin prevents creation of any air bubbles and compensates for any polymerization shrinkage which resulted in the polymer causing surface porosity of the resulted prosthesis.[15,16]

However, in the present study, the 3D printing exhibited significantly higher SR values among the tested processing methods; this may be because of the lower degree of polymerization and leakage of excessive monomer, causing excessive surface porosity and hence raised its SR.[17] In addition to the layering technique and printing orientation (45°), this results in stepwise edges between successive layers,[18,19] which come in agreement with Gad *et al.*[19]

The previous clinical study by Kim *et al.*[20] suggested that a threshold level of SR of hard surfaces in the oral condition after polishing should not exceed 0.2 μm; SR below the previous level has no significant decrease in bacterial growth. In this study, although SR threshold for CAD/CAM milling is slightly higher, it was considered appropriate to be accepted clinically.

In the current study, CAD/CAM milled resin demonstrated highest SH, which might be attributed to reduced residual monomers with subsequent plasticizing action and therefore enhanced CAD/CAM milled DBRM hardness.[9] In contrast, 3D-printed resin showed a statistically significant lower SH when compared with CAD/CAM milled resin. This may be because of the 3D-printed resin having a weak double-bond conversion,[18] or may be because of the material constitution, printing layering, and water sorption with thermal stressing as reported in the previous study.[19] The obtained SH results showed a marked significant decrease in SH of polyamide resins than that of CAD/CAM milled and 3D-printed resins; this can be understood according to the resins' internal structures. The polyamide resin has a reduced number of cross-linking agents, which can affect the surface hardness.[21] Thermoplastic flexible polyamide resins are formed of linear chain (hexamethylenediamine and carboxylic acid forming polyamide bonds); the polyamide linear bonds cannot withstand and resist the effect of solvent and decrease the resistance to surface pressure.[22] The SH, rigidity, and abrasion resistance of flexible thermoplastic polyamides are affected by its crystalline degree.[21]

Polyamide resins showed higher IS when compared with CAD/CAM resins; this increase might be attributed to the high pressure exerted by injection of the acrylic resin during the injection molding processing technique of thermoplastic resin specimens, or maybe the polyamide resin has a lower amount of cross-linking agents which increases its flexibility and a relatively higher amount of residual monomer which acts as a plasticizer when compared

with CAD/CAM resin, which explains its higher toughness.[23] The polymerization at pressure enhanced the average molecular weight and the polymerization rates of MMA (prevents air bubbles formation and compensates polymerization shrinkage) and hence improved IS.[20] Additionally, the used Breflex thermoplastic resin in the present study was a polyamide-based thermoplastic resin; the polyamide resin, characterized by its lower crystallinity and its linear nature of polymer chains, decrease its brittleness nature.[19] Moreover, the flexibility of the polyamide denture base is beneficial in increasing the absorbed energy before fracture.[23] This could explain the resulted significant increase in IS of flexible resin in this study when compared with other groups. This finding is in agreement with a previous study by Arita *et al.*[16]

The low mechanical properties of 3D-printed resins limit its use, and the literatures recommended further investigations to overcome their drawbacks.[19,24] Although low number of studies has been investigated, the properties of 3D-printed DBRM and IS were neglected. Subsequently, the comparison of the IS results of this study with previous studies was difficult. When comparing IS of CAD/CAM resins, milled resin was significantly higher than 3D printing resin. This could be related to that of the aforementioned advantages of prepolymerized milled resins such as high homogeneous quality, decreased amount of residual monomer, and free of porosities or air bubbles. These enhance the fracture resistance and increase the lifetime of the denture for the patient.[12] In contrast, the inferior IS of 3D-printed resin could be related to the 3D printing processing technique (printed resin, light polymerization and printing method, and parameters), which could result in improper bonding between the successive polymerized layers, a high amount of residual monomer, and air bubbles, which is a matter of concern as it affects the physical properties of the resulted denture.[24-27]

Clinically, the reduction of possibilities of microorganisms adhesion at DBRMs increases the fracture resistance, and the lifetime of denture for the patient should be considered as the main factors during DBRM selection. Hence, the fabrication process of DBRMs affected the SR, SH, and IS; CAD/CAM milled is considered the most suitable material in terms of improved SR and SH as well as IS, which falls in the clinical acceptable value according to literatures. Meanwhile, 3D-printed resin still showed low performances for all tested properties; therefore, further investigations are required to get the benefits of additive technology for denture base fabrication with appropriate properties and long-term clinical use. Although three different denture base materials are investigated in the present study, lack of oral conditions simulation or dynamic loading and the absence of thermocycling were considered as limitations. So, this report suggested studying the physical properties of DBRMs in conditions mimicking the oral environment for further investigation.

Conclusions

1- The CAD/CAM milled denture base resin showed lowest surface roughness when compared with 3D-printed and polyamide flexible denture base resins.
2- The CAD/CAM milled and polyamide flexible denture base resins showed higher IS when compared with 3D-printed denture base resins.
3- The CAD/CAM milled and 3D-printed denture base resin showed higher hardness when evaluated with polyamide-pressed materials.

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Conflicts of interest

None.

Authors' contributions

Not applicable.

Ethical policy and institutional review board statement

Not applicable.

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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