



Effect of Repair and Thermal Cycling on The Flexural Strength of Denture Base Materials Fabricated from Different Methods

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Research Article

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ABSTRACT

Objectives: The aim of this study was to evaluate the flexural strength of the denture base materials produced by different methods after repair and was to evaluate effect of thermal aging.

Materials and Methods: A total of 120 specimens were fabricated by conventional, CAD/CAM milled, and 3D-printed denture base materials for this in vitro study. Specimens were divided into four groups; non-repaired groups (N), repaired groups (R), non-repaired-aged groups (N-aged), and repaired-aged (R-aged) groups. In N groups, specimens were stored in the water bath for 24 h before the flexural strength test. In N-aged groups, samples were subjected to thermal aging for 5000 cycles, then tested. For repaired R groups, samples were repaired with auto-polymerized acrylic resin and storage in the water bath for 24 h then tested. For the R-aged groups, samples were repaired with auto-polymerized acrylic resin, aged for 5000 cycles, then tested. Specimens were performed a three-point loading test using a universal testing machine. Data were analyzed using Wilcoxon and Kruskal-Wallis tests.

Results: When the groups were compared with each other, the difference between all groups was found to be statistically significant ($p < 0.05$). Regardless of the thermal cycling process, in N groups, the highest flexural strength was recorded in N-CAD group (92.53 ± 6.52 MPa), the lowest flexural strength was recorded in N-3D group (33.72 ± 2.74 MPa). Regardless of the thermal cycling process, in R groups, the highest flexural strength was recorded in R-C groups (31.80 ± 5.86 MPa) and the lowest flexural strength was recorded in R-3D groups (8.37 ± 1.20 MPa). Thermal cycling and repair process showed a decreasing effect on flexural strength in all groups.

Conclusions: The ideal flexural strength among denture base materials was found in CAD/CAM milled denture base materials.

Keywords: Denture base resin, CAD/CAM, 3D-printed, repair.

Tamir ve Termal Döngünün Farklı Yöntemlerle Üretilen Protez Kaide Materyallerinde Bükülme Dayanımına Etkisi

Süreç

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Öz

Amaç: Bu çalışmanın amacı farklı yöntemlerle üretilen kaide materyallerinin tamir sonrası bükülme dayanımına ve termal yaşlandırmanın etkisini değerlendirmektir.

Gereç ve Yöntemler: Bu çalışmada konvansiyonel, CAD/CAM kazıma ve 3D yazıcı ile toplamda 120 örnek üretildi. Örnekler dört gruba bölündü; Tamir edilmemiş grup (N), tamir edilmiş grup (R), tamir edilmemiş-yaşlandırılmış grup (N-aged), tamir edilmiş-yaşlandırılmış grup (R-aged). N gruptaki örnekler eğme testinden önce 24 saat su banyosunda bekletildi. N-aged gruptaki örnekler termal yaşlandırma için 5000 döngü yapıldı ve sonrasında test edildi. R gruptaki örnekler, otopolimerizan akrilik ile tamir edildi. Sonrasında 24 saat su banyosunda bekletildikten sonra test edildi. R-aged grupta ise örnekler otopolimerizan akrilik ile tamir edilip termal yaşlandırma için 5000 döngü yapıldıktan sonra test edildi. Örnekler Universal test cihazında üç nokta eğme testi yapıldı. Verilerin değerlendirilmesinde Wilcoxon ve Kruskal-Wallis testi kullanıldı.

Bulgular: Gruplar birbirleri ile karşılaştırıldığında tüm gruplar arası fark istatistiksel olarak anlamlı bulundu ($p < 0,05$). Termak siklus işlemine bakılmaksızın N grupları içerisinde en yüksek bükülme dayanımının N-CAD grubunda ($92,53 \pm 6,52$ MPa), en düşük bükülme dayanımının ise N-3D grupta ($33,72 \pm 2,74$ MPa) olduğu görüldü. Termal siklus işlemine bakılmaksızın R gruplarında en yüksek bükülme dayanımının R-C grupta ($31,80 \pm 5,86$ MPa) olduğu, en düşük bükülme dayanımının ise R-3D grupta ($8,37 \pm 1,20$ MPa) olduğu görüldü. Termal siklus ve tamir işlemi tüm gruplarda bükülme dayanımını azalttığı görüldü.

Sonuçlar: Protez kaide malzemeleri arasında ideal bükülme mukavemeti, CAD/CAM kazıma ile elde edilen protez kaide materyalinde bulunmuştur.

Anahtar Kelimeler: Protez kaide reçinesi, CAD/CAM, 3D yazıcı, tamir.

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Introduction

Conventional complete dentures and partial dentures are the treatment option frequently used in prosthodontics to replace missing teeth.¹ When fabrication of removable prostheses (complete or partial denture bases), a synthetic polymer, polymethyl methacrylate (PMMA) mostly preferred.^{1,2} Being cheap, biocompatible, easy to apply and repair, stable in the oral environment, satisfactory aesthetic, and mechanical properties are suitable and can be counted as advantages of PMMA.^{2,3,4} Besides these advantages, PMMA has low dimensional stability and low impact, and flexural strength. Low dimensional stability leads to crack propagation and the formation of microporosities which may cause prosthesis fracture in the oral environment under different thermal temperatures.^{1,3} These fractures can happen inside the patient's mouth fatigue failure in the course of service or outside the patient's mouth because of an accidental fall.^{1,4} Repeated denture flexure by occlusal forces causes stress accumulation and fatigue failure.^{1,4,5} Today, the most common reason for the failure of the removable prosthesis is fracturing of the denture bases.¹ Denture repair is preferred to new denture fabrication because the repair is less time-consuming and decreases the cost.⁴ Denture repair material should be the same color as the denture base, easy and quickly perform, and improves the strength to prevent further fracture.^{4,5,6} Auto-polymerized acrylic resin, visible light polymerized acrylic resin, heat polymerized acrylic resin, and microwave polymerized acrylic resins have been used to repair fractured denture bases.^{4,7} Auto-polymerized acrylic resin is used for denture base repairs commonly because of ease of manipulation, being less time-consuming and economic.^{1,4}

Recently, with improvements in digital technologies computer-aided design/computer-aided manufacturing (CAD/CAM) method and three-dimensional (3D) printed technologies started to use for denture base production.^{2,8,9} Digital methods have faster denture fabrication and decrease the number of clinical visits.^{2,9} Allowing visualization of residual ridge and dentures to be made from different aspects, permitting digital record keeping, and improving patient satisfaction by better speech and higher comfort are other advantages of the digital process.⁸

CAD/CAM milled resin blocs that are processed at high temperature and pressure, this minimizes shrinkage, voids, and residual monomer.⁹ As a result, one block denture base is produced by providing process control developed by clinicians and technicians.^{2,10} This technique improves denture strength and adaptation while reducing microbial adhesion on denture surfaces.^{2,9} 3D printing enables concurrent manufacturing of many products and economical than milling due to material and tool conservation.^{8,9} When comparing conventional denture base resin, CAD/CAM-milled resins have superior surface characteristics, flexural strength, elastic modulus, and 3D-printed materials have superior color stability and fracture toughness.¹¹

The thermal aging process is important to simulate intraoral conditions. While water absorption, water molecules interfere with polymeric chains and affect the physical properties of the denture base resin by causing excessive dimensional instability in the resulting polymer.¹²

The mechanical properties of conventionally polymerized denture base acrylics have been the subject of much research to date. But there is a lack of consensus on the comparison of mechanical properties between the fabrication of CAD-CAM milled, 3D-printed, and conventional denture base resins.^{8,13,14} The objective of this study was to evaluate the flexural strength of denture base materials fabricated by conventional method and digital technologies (CAD/CAM milled and 3D-printed) and to examine the effect of repair and thermal cycling. The null hypotheses of this study were that the thermal cycling process and repair would not affect the flexural strength of denture base materials produced by CAD/CAM milled, 3D-printed, and conventional methods.

Materials and Methods

Three different methods were used for fabricating the specimens; the conventional, CAD/CAM milled, and 3D-printed methods in this study (Table 1). A total of 120 specimens were tested for flexural strength and each group was divided into two subgroups (n=60); 1. No-repaired (N) group with no fracture 2. Repaired (R) group (Table 2). Half of each subgroup was subjected to thermal cycling, and the other half of the specimens were not subjected to thermal cycling.

Fabrication of Conventional Specimens

For conventional groups (C), a total of forty specimens were fabricated following the conventional method. Firstly, 65 mm x 10 mm x 2.5 mm stainless-steel molds were fabricated and wax samples were obtained from these molds (Figure 1). After these wax samples were muffled, acrylic samples were obtained. In the polymerization of acrylic, the muffle was placed in boiling water. The heat source was turned off and left for 15 minutes. It was then boiled for 20 minutes and allowed to cool slowly in a water bath.

After finishing the specimens with 600-grit silicon carbide paper, half of them were stored at 37°C temperature in water bath for 24 hours. Other half of the specimens were thermal aged before the flexural strength test.

For conventional repaired (R-C) groups, a vertical line was drawn with a marker pen to divide the prepared specimen into 2 equal parts. The prepared specimens were cut vertically with a gap of 3 mm between two parts by using a micromotor handpiece and fissure carbide. In order to hold two pieces of acrylic denture bases together, a stainless-steel mold was used. The consistent space of repair of 3 mm was sustained between segments, and repaired with auto-polymerized acrylic resin. The repaired

specimens were finished with 600-grit silicon carbide paper.

Half of the specimens were stored at 37°C temperature in a water bath for 24 hours. Other half of the specimens were thermal aged before the flexural strength test.

Fabrication of CAD/CAM Milled Specimens

A rectangular block with the dimensions of 65 mm x10 mm x 2.5 mm was designed using software (Solid Works 2022, Dassault Systems S.A, Service pack 5.0, France) and saved as a standard tessellation language (STL) file. For the fabrication of the designed specimens, the STL files were uploaded to the CAM program (Work, NC). Then a computer-aided machine (Redon, Istanbul, Turkey) milled the specimens from pre-polymerized CAD/CAM resin discs (Yamahachi Dental MFG, Aichi-Pref, Japan) using a subtractive technique.

For CAD/CAM milled no-repaired (N-CAD) groups, specimens from polymethyl methacrylate denture base resin, with the dimensions of 65 mm x 10 mm x 2.5 mm, were fabricated using CAD/CAM technology as mentioned above. For the CAD/CAM milled repaired group (R-CAD), specimens from polymethyl methacrylate denture base resin, with dimensions of 31 mm x10 mm x 2.5 mm, were fabricated using CAD/CAM technology. Then, the repairing process was done as mentioned in the R-C group.

Half of the specimens were stored at 37°C temperature in a water bath for 24 hours. Other half of the specimens were thermal aged before the flexural strength test.

Fabrication of 3D Printed Specimens

A rectangular block with the dimensions of 65 mm x10 mm x 2.5 mm was designed using software (AutoCAD 2018, Autodesk, California, United States) and saved as an STL file. The STL files of the designed specimen were exported to a 3D printer (Free Shape 120 Printer, Ackuretta, Chinese) with a UV light source. The specimens were printed using acrylate ester-based resin (MACK4D Resin, Dentona, Germany) with an additive technique. The printed specimens were rinsed with isopropyl alcohol (99% concentration) for 5 minutes to remove unpolymerized acrylic resin and then placed in an Ackuretta UV Box (Taiwan) for 3 minutes to complete post-polymerization.

For 3D-Printed no-repaired groups (N-3D), specimens from polymethyl methacrylate denture base resin, with 65 mm x10 mm x 2.5 mm dimensions, were printed as mentioned above using 3D printing technology. For 3D-Printed repaired groups (R-3D), specimens from polymethyl methacrylate denture base resin, with 31 mm x10 mm x 2.5 mm dimensions, were printed as mentioned above using 3D printing technology (Figure 2). Then, the repairing process was done as mentioned in the R-C group.

Half of the specimens were stored at 37°C temperature in a water bath for 48 hours. Another half of

the specimens were thermal aged before the flexural strength test.

Thermal Aging Process

Half of the specimens in all groups were thermocycled for 5000 cycles with a dwell time of 30 seconds in water at 5°C and 55°C.

Flexural Strength Test

The flexural strength was conducted using a universal testing machine (Lloyd LF Plus; Ametek Inc, Lloyd Instruments, Leicester, UK). Acrylic specimens were placed in a universal test machine. Three-point bending tests were performed on every specimen at a speed of 5 mm/min.

The maximum load at fracture was recorded for each specimen, and the flexural strength was calculated as follows:

$$FS = 3FL/2bd^2$$

FS = flexural strength in Mega Pascal (MPa), F = load at fracture in newtons (N), L = span between the supports (mm), and b and d = width and thickness of the specimen (mm).

Statistical Analysis

SPSS 22.0 program was used in this study. The parametric test assumptions could not be fulfilled in the evaluation of the data, so the Wilcoxon test was used. Kruskal-Wallis's test was used when comparing the measurements obtained from two independent groups. The Mann-Whitney U test was applied to find the groups that made a difference in the analysis result ($p = 0.05$).

Results

When the groups were compared with each other, the difference between all groups was found to be statistically significant ($p < 0.05$). Descriptive statistics are presented in Table 3, 4, 5.

Regardless of the thermal cycling process, in no-repaired groups, the highest flexural strength was recorded in N-CAD groups (92.53 ± 6.52 MPa), the lowest flexural strength was recorded in N-3D groups (33.72 ± 2.74 MPa) (Table 3). Regardless of the thermal cycling process, in repaired groups, the highest flexural strength was recorded in R-C groups (31.80 ± 5.86 MPa) and the lowest flexural strength was recorded in R-3D groups (8.37 ± 1.20 MPa) (Table 4).

Thermal cycling showed a decreasing effect on flexural strength in all groups. A statistically significant difference emerged between N-C, N-CAD, N-3D, and R-C flexural strength values. There was no statistical difference in R-CAD and R-3D groups.

Flexural strength values were decreased after the repair process in all groups (Table 5). The repair process has the most decreasing effect on flexural strength values in CAD/CAM groups (before and after thermal cycling, $p < 0.05$).

Table 1. Composition and manufacturer of materials used in this study

Brand	Material Type	Composition	Manufacturer
Meliodent	Heat-cured, conventionally manufactured denture base resin	Polymethylmethacrylate, benzoylperoxide, methyl methacrylate, ethylene glycol dimethacrylate	Heraeus Kulzer, Hanau, Germany
Yamahachi	CAD/CAM Milling Block	Polymethylmethacrylate, ferric oxide, titanium dioxide	Yamahachi Dental MFG, Aichi-Pref, Japan
MACK 4D Denture Light Pink	Additive manufactured denture base resin	Urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA)	Dentona, Germany
Blaucryl	Auto-polymerizing acrylic resin as repair material	Polymethylmethacrylate, benzoylperoxide, methyl methacrylate, hydroquinone, dimethyl-p-toluidine	Blaudent, UK

Table 2. Study Groups

Groups	Technique	Abbreviation	n
Conventional	No-repaired	N-C	20
Conventional	Repaired	R-C	20
CAD/CAM Milled	No-repaired	N-CAD	20
CAD/CAM Milled	Repaired	R-CAD	20
3D Printed	No-repaired	N-3D	20
3D Printed	Repaired	R-3D	20

Table 3. Flexural strength values (MPa) of no-repaired groups of each denture base before and after thermocycling

No-repaired group	Before thermocycling	After thermocycling	p-value
Conventional	78.13 (3.95) ^{aA}	66.16 (8.66) ^{aB}	p=0.013*
CAD/CAM milled	92.53 (6.52) ^{bC}	85.98 (3.92) ^{bD}	p=0.047*
3D Printed	45.17 (6.54) ^{cE}	33.72 (2.74) ^{cF}	p=0.007*
	KW=25,36 P=0.001*	KW=24,87 P=0.001*	

* The different superscript lower-case letters per column indicate statistically significant ($p < 0.05$); Different superscript upper-case letters indicate the statistically significant between each group before and after thermocycling ($p < 0.05$).

Table 4. Flexural strength values (MPa) of repaired groups of each denture base before and after thermocycling

Repaired group	Before thermocycling	After thermocycling	p-value
Conventional	31.80 (5.86) ^{aA}	22.76 (5.20) ^{aB}	p=0.012*
CAD/CAM milled	25.28 (3.33) ^{bC}	24.87 (2.42) ^{bC}	p=0.721
3D Printed	9.42 (1.51) ^{cD}	8.37 (1.20) ^{cD}	p=0.333
	KW=22.59 P=0.001*	KW=19.77 P=0.001*	

*The different superscript lower-case letters per column indicate statistically significant ($p < 0.05$); Different superscript upper-case letters indicate the statistically significant between each group before and after thermocycling ($p < 0.05$).

Table 5. Comparison of flexural strength of each base material

Groups		Before Thermocycling			After Thermocycling		
		Mean (MPa)	SD	p value	Mean (MPa)	SD	p value
Conventional	(No-repaired)	78.13	3.95	P=0.005*	66.16	8.66	P=0.004*
	(Repaired)	31.80	5.86		22.76	5.20	
CAD/CAM milled	(No-repaired)	92.53	6.52	P=0.004*	85.98	3.92	P=0.005*
	(Repaired)	25.28	2.42		24.87	3.33	
3D Printed	(No-repaired)	45.17	6.54	P=0.002*	33.72	2.74	P=0.003*
	(Repaired)	9.42	1.51		8.37	1.20	

*Statistically significant difference ($p < 0.05$).



Figure 1. Stainless steel mold used to repair specimens.



Figure 2. (a) Specimens fabricated from different techniques, (b) Specimens fabricated for repair

Discussion

This in vitro study investigated the repair and thermal cycling effect on the flexure strength of denture base materials fabricated with CAD/CAM milling and 3D-printed, compared with the conventional method. Statistical analysis indicated significant differences between groups for flexural strength thus the null hypotheses were rejected.

A complete denture is often the preferred treatment option for edentulous patients³. Complete and removable partial dentures and overdentures are most commonly made from PMMA.³ In prosthetic dentistry, fractures of PMMA denture base resin are common problems.¹ In many cases, repairing the denture base is a viable solution.¹ For repairing fractured denture bases, auto-polymerizing acrylic resin is the most common method.⁴ So, in this in vitro study, the auto-polymerized acrylic resin was used for repairing specimens. The repair area dimension was 3 mm for reducing the repair material and consequently, minimizing the polymerization shrinkage according to AlQahtani *et al.* and Deb *et al.*^{1,15} Flexural strength values decreased after the repairing process in all groups in this study. According to the author's current knowledge, there is no evidence for flexural strength values after repairing denture base materials fabricated with digital technologies. Current studies in the literature evaluated the flexural strength with surface treatment modification on the repaired areas.^{13,16}

The flexure strength is a material property that is defined as the amount of stress that exists in the material

just before it gives in a flexure test.^{2,8} This test simulates stress forces that are typically applied during the masticatory function to dentures, which can result with fracture.¹⁷

Two digital technologies, namely the CAD/CAM milled and 3D-printed methods, are frequently used today.¹³ There is no consensus on the flexural strength of CAD/CAM materials.² Freitas *et al.*¹⁷ evaluated the flexure strength of three different denture base materials and concluded CAD/CAM showed the highest flexural strength values while 3D-Printed lowest flexural strength.¹⁷ According to Ayman¹⁸ and Pacquet *et al.*¹⁹, heat-polymerized PMMA had higher flexural strength values than CAD/CAM milled denture bases. Ayman¹⁸, Pacquet *et al.*¹⁹, and Steinmassl *et al.*²⁰, results were in contrast to this in vitro study. Hence, CAD/CAM materials showed higher flexural strength values than conventionally fabricated denture base materials, in line with Aguirre *et al.* and Freitas *et al.*^{21,17} Differences between the flexural strength values of CAD/CAM and heat-polymerized denture base materials can explain by the use of different manufacturers and materials in the studies mention above.^{2,9,14,17}

The physical properties produced with 3D printers depend on the rate of monomer conversion formed by liquid photopolymer resins and light-induced polymerization.¹⁷ In this process, carbon double bonds are converted into single bonds.¹⁷ The higher the double bond conversion, the higher the polymer.¹⁷ If an unreacted monomer is present, the mechanical strength may decrease and cause soft tissue irritation.¹⁷ According to

ISO standards, a denture base resin must have a flexural strength value of 65 MPa.¹⁷ According to previous studies, 3D Printed denture base materials had lower flexural strength comparing CAD/CAM milled, conventionally fabricated, and polyamide denture bases.^{2,9} The results of this in vitro study corroborate Prpic and Zeidan's results. The variation in the values reported can be attributed to the material's brand and manufacturer.⁸ Further studies are needed to determine the mechanical properties of 3D-printed denture bases.⁸

A thermocycling cycle of 5000 cycles corresponds to six months of clinical use.¹³ A thermal cycle simulates the thermal changes in the oral environment during long-term use of dental materials.^{13,15} It is consistent with earlier research that shows reduced flexural strength after thermocycling.¹ This in vitro study corroborates with AlQahtani *et al.*, as reduced flexural strength after thermocycling for conventionally produced denture base materials.¹ According to Çakmak *et al.*, the thermal cycling process has decreasing effect on 3D-printed denture base resin, these results also corroborate with this in vitro study's results.¹⁴ Water absorption, thermal stress, and the presence of porous structures may be the cause of the reduction of fracture strength values.¹

According to the literature, no studies evaluate the effect of repair on flexural strength after the thermal cycling process. Repair and thermal cycling processes together have on decreasing effect on flexural strength in this study. Biocompatibility, microbial adhesion, manufacturing trueness, clinical fit, and color stability of the denture base resin materials should be investigated in further studies. According to the results of this study, new digitally produced denture base materials can be considered as an alternative.

Conclusions

Within the limitations of this study, it was possible to conclude that:

- 1- No-repaired specimens showed higher flexural strength than the repaired specimens.
- 2- Both before and after thermal cycling, 3D-printed specimens showed the lowest flexural strength values.
- 3- Thermal cycling process was found to have decreasing effect on flexural strength in three different denture base resins.

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