Cumhuriyet Dental Journal: 2020; 23(4)
e-ISSN 2146-2852
Doi:10.7126/cumudj.798591

Original research

THE EFFECT OF AUTOCLAVE POLYMERIZATION ON THE TENSILE STRENGTH OF VARIOUS DENTURE BASE MATERIALS

*Gonca Deste Gökay[1]
Rukiye Durkan[2]
Perihan Oyar[2]

ABSTRACT

Objectives: The aim of this *in vitro* study was to evaluate the effect of autoclave polymerization method on the tensile strength of the polymethylmethacrylate (PMMA) acrylic denture base resins.

Materials and Methods: Total of 60 specimens were fabricated from four different denture base materials such as three heat polymerized denture resins Meliodent, Paladent, and QC-20 and one microwave polymerized resin Acron MC. Specimens were divided into control (heat and microwave polymerization), short (130 °C 10 min) and long (130 °C 20 min) autoclave polymerization groups according to polymerization techniques (n=5). The tensile strength tests were performed. Data were analyzed by ANOVA followed by the Duncan test (α=0.01).

Results: In control group, Acron-MC showed the highest tensile strength value, QC-20, Paladent and Meliodent were followed. Autoclave polymerization 20 minutes Acron MC group was significantly different from the other acrylic resins for the control and autoclave polymerization 10 minutes groups ($p<0.01$). Autoclave polymerized acrylic resin specimens showed higher tensile strength values than the control groups ($p<0.01$). There was no significant difference between the increasing time of autoclave polymerization methods 10 minutes and 20 minutes ($p>0.01$).

Conclusion: Within the limitations of the study, autoclave polymerization method may provide a stronger alternative to conventional polymerization methods. Autoclave polymerization method provides high temperature that the activation ratio of cross-linking agents (glycoldimethacrylate) can be increased.

Key words: denture base materials, acrylic resins, tensile strength, autoclave, polymerization.

ORCID IDs of the authors:
G.D.G. 0000-0002-5481-0063
R.D. 0000-0002-3381-4073
P.O. 0000-0003-3849-9153

[1] Bursa Uludağ University, Faculty of Dentistry, Department of Prosthodontics, Bursa, Turkey.
[2] Afyonkarahisar Health Sciences University, Faculty of Dentistry, Department of Prosthodontics, Afyonkarahisar, Turkey.
[3] Hacettepe University, Health Services Vocational High School, Department of Dental Prostheses Technology, Ankara, Turkey.

Received : 22.09.2020
Accepted : 17.11.2020

How to Cite: Deste Gökah G, Durkan R, Oyar P. The Effect of Autoclave Polymerization on the Tensile Strength of Various Denture Base Materials. Cumhuriyet Dent J 2020;23:4: 364-370.
***Corresponding Author:**
Bursa Uludağ University, Faculty of Dentistry, Department of Prosthodontics, Bursa, Turkey.
Phone: +90 553 431 30 40 **E-mail:** goncadeste@hotmail.com

INTRODUCTION

Poly (methyl methacrylate) (PMMA) polymers are the material of choice in dentistry to fabricate complete and partial removable dental prostheses, for use in provisional restorations and orthodontic applications and denture repair due to their low cost, relatively simple processing techniques with thermal energy, durability in oral fluids, the ease of manipulation and repair.[1]

However, PMMA has some disadvantages. During a three-year period, 68% failure was observed in removable prostheses, and 29% of them had fractures.[2] The most common problem for both patients and dentists is the fracture of denture bases.[1-3] The reason for denture fracture may be the denture function, fabrication process, handling, stress intensification, porosity, residual monomer, the presence of cracks, and poor adaptation.[4]

Fractures in dentures can be caused by impact and bending fatigue. Different methods have been tested to enhance the physical and mechanical features of PMMA, and alternative materials have been introduced. The addition of fibers (glass, polyamide, polyethylene, polypropylene), metal oxides (aluminum oxide, zirconia, titanium), noble metals (silver, nano gold, platinum, palladium), minerals (hydroxyapatite filler, silicon dioxide, silica-based filler, carbon-based filler, nanocarbon, nanodiamond) and hybrid boosters have been used to enhance the mechanical features of denture base resins.[5]

More attention has recently been paid to studies on microwave polymerization. Nevertheless, a lot of problems that should be explained for an effective application continue to exist.[6-8]

In case of their use as a denture base material, powder-like polymerized PMMA beads are mixed with methyl methacrylate liquid monomers that contain a small percentage of a dimethacrylate crosslinker. The polymerization of PMMA-powder and MMA-monomer liquid causes forming a semi-interpenetrating polymer (SIPN) network structure. The semi-IPN structure comprises one or more thermoplastic polymers that are mixed with a cross-linked polymer.[9]

A cross-linking agent represents a substance that promotes or irregulates intermolecular covalent bonding between polymer chains. The incorporation of cross-linking agents into the monomer leads to the formation of an insoluble cross-linked network in the course of polymerization and increases the glass transition of a polymer by introducing restrictions on the molecular motions of the chain. Therefore, many physical properties of the polymer may be improved. Furthermore, cross-linking agents, such as glycol methacrylate, provide increased resistance to crack formation and progression, impact and fatigue and create a stronger structure.[10]

An autoclave represents a pressurized device that is designed for heating aqueous solutions above their boiling point for the purpose of achieving sterilization and is commonly utilized in dentistry, microbiology, medicine, and metallurgy. Although the conventional method of heat polymerization is the most widely used method, different polymerization methods, such as injection molding, autopolymerization, light polymerization, microwave polymerization, and autoclave polymerization, have been used for the polymerization of denture bases. The autoclave polymerization method provides a high temperature at which the activation ratio of the cross-linking agent (glycol dimethacrylate) can be increased.[11,12]

Tensile strength, compressive strength, shear strength, impact strength, fatigue strength, proportional limit, flexural strength and modulus, and surface hardness methods can be used to investigate the mechanical properties of PMMA. Among the many desired mechanical properties of a denture base resin, high tensile strength is of particular practical importance to the final product. Tensile strength indicates the maximum tensile stress that can be applied in a uniform way over the cross-section of a test piece during stretching the test piece to failure.[12]

Various studies have reported the desirable features of polymer denture base materials, including biocompatibility, satisfactory durability in oral fluids, and the ease of handling.[7,13-15] Nevertheless, previous studies have not been

consistent concerning the mechanical features and behavior of the mentioned resins.[16,17] This *in vitro* study aimed to assess the impact of the autoclave polymerization method on the tensile strength of acrylic denture base resins. The null hypothesis stated that there would be a statistical difference when the tensile strength of the four denture base materials investigated was compared.

MATERIAL AND METHODS

Preparation of specimens

Four types of polymethyl methacrylate denture base materials were utilized in the research. The materials utilized in the present research are shown in Table 1.

Table 1. Denture base materials used in the present study

Materials	Polymerization Types	Chemical Compositions	
		Powder	Liquid
Meliodent	heat	Polymethylmethacrylate, ethyl hexyl acrylate, N-octyl methacrylate	methyl methacrylate, glycol dimethacrylate, dimethyl p-touludine
QC-20	heat	Copolymer (methyl-n-buthyl) methacrylate	methyl methacrylate, glycol dimethacrylate, N, N dimethyl p-touludine
Paladent	heat	Polymethylmethacryate, ethylmethacrytlate, N-octyl methacrylate	methyl methacrylate, glycol dimethacrylate, dimethyltouludine
Acron-MC	microwave	Polymethylmethacrylate, ethylmethacrytlate co-polymer	methyl methacrylate, glycol dimethacrylate, N-dimethyl p-touludine

Ethical approval was acquired from the Clinical Research Ethics Committee of Afyonkarahisar Health Science University (decision date: 3.04.2020, ID number: 2020/218). The samples that were prepared for tensile measurements were standard dumbbell-shaped test specimens in accordance with ASTM D 638M. A micrometer (Mitutoyo, Japan) was utilized for measuring the dimensions of the test specimens. A total of 60 specimens were fabricated from four different denture base materials: three heat-polymerized resins, Meliodent (Dental Bayer Ltd, Berkshire, UK), Paladent (Heraeus Kulzer GmbH & Co. KG, Wehrheim, Germany), and QC-20 (De Trey, Dentsply, Germany), and one microwave-polymerized resin Acron MC (GC Corp, Tokyo, Japan). The mixing of the powder (PMMA) and liquid (MMA) elements of the denture base materials was performed in a glass cup following the manufacturer's instructions for all groups. The mixture was allowed to stand until the formation of a dough stage, which was then packed into the flask and kept under pressure for a period of 10 min. The heat-polymerized resin specimens were manufactured by packing the acrylic resin into the stone molds present in denture flasks and curing for 30 min at 80 ºC in boiling water following the manufacturer's recommendations. For microwave-polymerized specimens, the dough formed from the powder/liquid mix was packed into the mold within a flask under pressure and subjected to microwave polymerization at 550 W for 3 min using a microwave oven (Vestel, Manisa, Turkey).

In accordance with polymerization techniques, specimens were divided into the control, short and long autoclave polymerization groups (n=5). Heat-polymerized acrylic resin specimens were processed by curing in a conventional water bath, while microwave-polymerized acrylic resins were processed in a microwave following the manufacturer's instructions for the control group. In the second group of acrylic resin specimens, a dough that was prepared from the powder/liquid mix was packed into the metal mold within the flask, and they were polymerized in an autoclave sterilization unit (OT 4060 Steam Sterilizer, Germany) at 60 °C for 30 minutes and at 130 °C for 10 minutes. In the third group, the resin specimens were polymerized

in the autoclave sterilization unit at 60 °C for 30 minutes and then at 130 °C for 20 minutes. There was no porosity problem. Then the flasks were cooled in the air for 30 min and then in the running water for 15 min. These temperature and time settings were identified according to a preliminary study.[18]

After polymerization, irregularities were removed from only one surface of each specimen by simulating oral conditions and utilizing a series of silicon carbide paper abrasive discs starting from 120 grit and ending with 320 grit for the purpose of acquiring a polished surface. The test specimens were stored in a water bath at a temperature of 37±2 °C for a period of 48 hours and kept in the open air for 1 hour before testing.

Tensile strength testing

The tensile test was conducted by utilizing the Llyod Universal Testing Machine (Llyod LRX, Llyod Instruments Ltd., Farehan, Hampshire, UK) at a crosshead speed of 1 mm/min. Tensile strength was calculated according to the following equation:

$Q=F/A$

Q: stress (N/mm^2)

F: maximum recorded force at failure (N)

A: the area of the specimen, expressed in mm^2

The data were obtained from the tests and analyzed by the analysis of variance (ANOVA). As a post hoc test, Duncan's HSD test was performed for the purpose of comparing the groups of means that could be attributed to the presence of interactions between the variables tested. All statistical testing was carried out at a 95% level of confidence.

RESULTS

Table 2 shows the mean and standard deviations of the tensile strength values of the four denture base materials tested.

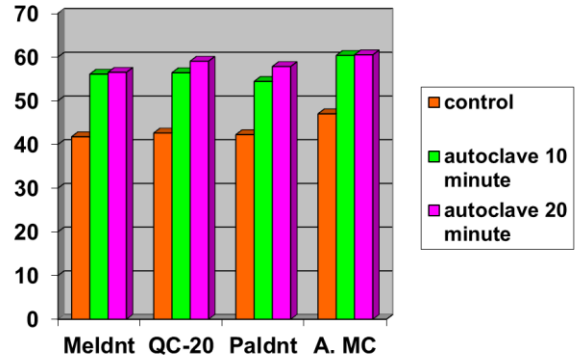
Table 2. Mean values and standart deviations of acrylic resins test specimens

Materials	Control groups	Autoclave polymerization 10 min	Autoclave polymerization 20 min
Meliodent	41.75 (1.17) **A**[a]	56.08 (2.99) **A**[b]	56.50 (2.96) **A**[b]
QC-20	42.61 (0.85) **A**[a]	56.40 (1.27) **A**[b]	59.04 (2.33) **A**[b]
Paladent	42.24 (2.04) **A**[a]	54.41 (0.73) **A**[b]	57.82 (1.43) **A**[b]
Acron- MC	47.00 (0.40) **B**[a]	60.36 (1.18) **B**[b]	60.50 (1.01) **A**[b]

Vertically, identical capital letters denote no significant differences among materials (p>0.01)
Horizontally, identical small letters denote no significant differences among materials (p>0.01)

Acron MC (47.0 MPa) exhibited the highest tensile strength value, followed by QC-20 (42.61 MPa), Paladent (42.24 MPa), and Meliodent (41.75 MPa) for the control group. Acron MC differed statistically significantly from the other acrylic resins for the control and 10-minute autoclave polymerization groups in terms of autoclave polymerization for 20 minutes (p<0.01).

Autoclave-polymerized acrylic resin specimens exhibited higher tensile strength values than the control group (p<0.01). No significant difference was found between the autoclave polymerization methods for 10 minutes and 20 minutes (p>0.01). No significant difference was determined between the tensile strength values of Meliodent, QC-20, and Paladent for three different polymerization methods (p>0.01). The increase in the tensile strength values of the autoclave polymerization method can be observed in the graph presented below (Graph 1).



Graphic 1. Tensile strength of different polymerization method and acrylic denture base resins

DISCUSSION

The complete polymerization of the polymer matrix is an important factor in the success of dentures. In the present study, the tensile strength of four different types of acrylic denture base resins was investigated using the autoclave polymerization method. The null hypothesis stated that there was a statistically significant difference among the groups.

The current *in vitro* study was designed for the purpose of comparing the tensile strength of four denture base resins obtained using a new curing method (autoclave polymerization method). Anusavice and Phillips[19] reported that dry heat-cured denture base PMMA exhibited a tensile strength of 52 MPa. The mentioned finding was not consistent with the current study, which revealed that Meliodent (56.08 MPa) and QC-20 (56.40 MPa) PMMA resin exhibited a higher tensile strength than Paladent 20 (54.41 MPa) under the 10 min autoclave polymerization condition.

In a study, Vertex acrylic resin specimens were divided into the heat, short and long autoclave polymerization groups in accordance with polymerization techniques. The groups were separated into three subgroups by weight based on the glass fiber (GF) concentration (0%, 2.5%, and 5% by weight). Autoclave polymerization caused a significant increase in the hardness of acrylic resin without the GF supplement, and it was reported that autoclave polymerization might be an alternative to the conventional heat polymerization method.[20] The results may differ from the present study due to the use of a reinforcement agent, different brands of PMMA, or different polymerization times.

Ayaz *et al*.[21] reported that autoclave polymerization increased the hardness of three acrylic resin groups (Meliodent, Paladent, and QC-20), but no significant differences were revealed between short (10 min) and long (20 min) autoclave polymerization. In the present study, while increasing the time of autoclave polymerization from 10 to 20 minutes caused no significant difference in tensile strength, it showed higher tensile strength values in autoclave polymerization compared to the heat-polymerized control groups. Similar to the findings of the current research, Gad *et al*.[22] assessed the impacts of autoclave polymerization on the elastic modulus and flexural strength of PMMA acrylic resins and reported no significant differences between the short and long-cycle autoclave-polymerized specimens. However, increased elastic modulus and flexural strength values were found compared to water bath-polymerized specimens.

Lai *et al.*[17] reported that microwave energy could efficiently polymerize denture base polymers. High statistical differences in mechanical properties were found in comparison with the water-bath method. The present research demonstrated that Acron MC resin exhibited the highest tensile strength in comparison with conventional heat curing denture base materials (Meliodent, Paladent, and QC-20). Moreover, autoclave polymerization was found to be stronger than microwave or heat curing methods. The underlying reason for this increase was composing a strong mesh structure with the autoclave polymerization method. Thus, it might be harder to break the connections of molecules between these materials. Therefore, owing to an ethylene glycoldimethacrylate structure in the composition of the microwave or heat-polymerized denture base material, they were readily curable by crosslinking at a high temperature and pressure, compared to the compression molding technique. However, the crosslinking of multifunctional monomers shows an abnormal kinetic behavior in the course of polymerization.[23]

Tensile strength indicates the maximum tensile stress that can be applied in a uniform way over the cross-section of a test piece during stretching the test piece to failure. Despite the fact that *in vitro* experiments may not always reflect intraoral conditions and predict clinical performance, they are valuable and applicable to clinical conditions.[24]

Factors such as the chemical composition of acrylic resin, polymerization time and type, the power of the microwave used in the studies should also be taken into consideration since they are

directly responsible for interchain force and polymer chain arrangements.[25]

Among the limitations of the present study, there are the usage of tensile strength testing in the air and testing only the denture base components of the Meliodent, Paladent, QC-20, and Acron-MC systems. Further studies are required to examine different resins, various polymerization durations, and temperature. Moreover, it is necessary to reveal the impacts of water absorption, staining, wear resistance, and other physical characteristics of the autoclave polymerization method and to find the possible alternative to the other methods used in prosthetic treatment.

CONCLUSIONS

The present study assessed the tensile strength of four different denture base materials: Meliodent, QC-20, Paladent, and Acron MC. When compared to the tensile strength of the four denture base resins, conventional heat curing, and microwave curing method, a statistically significant difference was found between Acron MC and the other acrylic denture base resins. Within the limitations of the current research, the microwave polymerization method showed significantly higher tensile strength when compared to the conventional water bath technique. The autoclave polymerization (130 ^{0}C, 20 min) method showed the highest tensile strength value, which was followed by the autoclave polymerization (130 ^{0}C, 10 min) and microwave polymerization method. The autoclave polymerization method may be a useful alternative to the conventional heat curing method.

ACKNOWLEDGEMENTS

There is no acknowledgement for this study.

CONFLICT OF INTEREST STATEMENT

There is no conflict of interest.

Otoklav Polimerizasyon Yönteminin Farklı Protez Kaide Materyallerinin Çekme Dayanımı Üzerine Etkisi

ÖZ

Amaç: *Bu in vitro çalışmanın amacı, otoklav polimerizasyon yönteminin polimetilmetakrilat (PMMA) protez kaide rezinlerinin çekme dayanımı üzerindeki etkisini değerlendirmektir.* ***Gereç ve Yöntemler:*** *Üç ısı ile polimerize olan akrilik rezin Meliodent, Paladent ve QC-20 ve bir mikrodalga polimerize rezin Acron MC olmak üzere dört farklı protez kaide materyalinden toplam 60 adet örnek üretildi. Örnekler, polimerizasyon tekniklerine göre kontrol (geleneksel ve mikrodalga polimerizasyon), kısa (130° C 10 dakika) ve uzun (130°C 20 dakika) otoklav polimerizasyon gruplarına ayrıldı (n=5). Çekme mukavemeti testleri yapıldı. Veriler ANOVA ve ardından Duncan testi (a=0,01) ile analiz edildi.* ***Bulgular:*** *Testler sonucunda çekme dayanımı değerleri sırasıyla en yüksek Acron MC, kontrol grubu için ise QC-20, Paladent ve Meliodent olarak sıralandı. Acron MC, kontrol ısı polimerizasyonu ve 10 dakika otoklav polimerizasyonu grupları için diğer akrilik rezinlerden önemli ölçüde farklı bulundu (p<0,01). Otoklavda polimerize akrilik rezin örnekleri, kontrol gruplarına göre daha yüksek çekme dayanımı değerleri gösterdi (p<0,01). Otoklav polimerizasyon yöntemlerinin artan süreleri arasında ise anlamlı bir fark bulunmadı (p>0,01).* ***Sonuç:*** *Çalışmanın sınırları dahilinde, otoklav polimerizasyon yöntemi geleneksel polimerizasyon yöntemlerine göre daha güçlü bir alternatif sağlayabilir. Otoklav polimerizasyon yöntemi, çapraz bağlama ajanlarının (glikoldimetakrilat) aktivasyon oranının artırılabileceği yüksek sıcaklık sağlar.* ***Anahtar kelimeler:*** *Protez kaideleri, akrilik rezinler, çekme dayanımı, polimerizasyon.*

REFERENCES

1. Lee SY, Lai YL, Hsu TS. Influence of polymerization conditions on monomer elution and microhardness of autopolymerized polymethyl methacrylate resin. Eur J Oral Sci 2002;110:179-183.

2. Agha H, Flinton R, Vaidyanathan T. Optimization of Fracture Resistance and Stiffness of Heat-Polymerized High Impact Acrylic Resin with Localized E-Glass FiBER FORCE® Reinforcement at Different Stress Points. J Prosthodont. 2016;25:647-655.

3. Seo´ RS, Vergani CE, Giampolo ET, Pavarina C, Nunes Reis JM, Machado AL. Effect of Disinfection by Microwave Irradiation on the Strength of Intact and Relined Denture Bases and the Water Sorption and Solubility of Denture Base and Reline Materials. J Appl Poly Sci. 2008;107:300-308.

4. Franklin P, Wood DJ, Bubb NL. Reinforcement of poly(methyl methacrylate) denture base with glass flake. Dent Mater 2006;21:365-370.

5. Jagger DC, Jagger RG, Allen SM, Harrison A. An investigation into the transverse and impact strength of "high strength" denture base acrylic resins. J Oral Rehabil. 2002;29:263-267.

6. Somkuwar S, Mishra SK, Agrawal B, Choure R. Comparison of the flexural strength of polymethyl methacrylate resin reinforced with multiwalled carbon nanotubes and processed by conventional water bath technique and microwave polymerization. J Indian Prosthodont Soc. 2017;17:332-339.

7. Palaskar JN, Singh S, Mittal S. Evaluation and comparison of different polymerization techniques, curing cycles, and thicknesses of two denture base materials. Indian J Dent Res. 2019;30:583-589.

8. Selecman AM, Brodine BA. Poly(methylmethacrylate) Microwave Processing: A Technique Paper. J Prosthodont. 2020;29:94-96.

9. Vuorinen AM, Dyer SR, Lassila LVJ, Vallittu PK. Effect of rigid rod polymer filler on mechanical properties of poly-methyl methacrylate denture base material. Dent Mater. 2008;24:708-713.

10. Saen-Isara T, Dechkunakorn S, Anuwongnukroh N, Srikhirin T, Tanodekaew S, Wichai W. Influence of the cross-linking agent on mechanical properties of PMMA powder with compromised particle morphology. Int Orthod. 2017;15:151-164.

11. Molinari JA, Runnells RR. Role of disinfectants in infection control. Dent Clin North Am. 1991;35:323-337.

12. Craig RG. Restorative dental materials. 9th ed. Mosby:St. Louis, 1993.

13. Zuo W, Feng D, Song A, Gong H, Zhu S. Effects of organic-inorganic hybrid coating on the color stability of denture base resins. J Prosthet Dent 2016;115:103-108.

14. Gungor H, Gundogdu M, Duymus ZY. Investigation of the effect of different polishing techniques on the surface roughness of denture base and repair materials. J Prosthet Dent 2014;112:1271-1277.

15. Sahin O, Koroglu A, Dede DÖ, Yilmaz B. Effect of surface sealant agents on the surface roughness and color stability of denture base materials. J Prosthet Dent 2016;116:610-616.

16. Alla R, Raghavendra KN, Vyas R, Konakanchi A. Conventional and contemporary polymers for the fabrication of denture prosthesis: part I – overview, composition and properties. Int J Appl Dent Sci. 2015;1:82-89.

17. Lai CP, Tsai MH, Chen M, Chang HS, Tay HH. Morphology and properties of denture acrylic resins cured by microwave energy and conventional water bath. Dent Mater. 2004;20:133-141.

18. Durkan R, Ozel MB, Bağiş B, Usanmaz A. In vitro comparison of autoclave polymerization on the transverse strength of denture base resins. Dent Mater J. 2008;27:640-642.

19. Anusavice KJ, Phillips RW. Phillips' science of dental materials. 11 th ed. St. Louis, Mo.: Saunders; 2003. Chapter 19 p. 474.

20. Gad MM, Rahoma A, Al-Thobity AM. Effect of polymerization technique and glass fiber addition on the surface roughness and hardness of PMMA denture base material. Dent Mater J. 2018;37:746-753.

21. Ayaz EA, Durkan R, Koroglu A, Bagis B. Comparative effect of different polymerization techniques on residual monomer and hardness properties of PMMA-based denture resins. J Appl Biomater Funct Mater. 2014;12:228-233.

22. Gad MM, Fouda SM, ArRejaie AS, Al-Thobity AM. Comparative Effect of Different Polymerization Techniques on the Flexural and Surface Properties of Acrylic Denture Bases. J Prosthodont. 2019;28:458-465.

23. Pavlinec J, Moszner N. Monomers for adhesive polymers, 8a crosslinking polymerization of selected N-substituted bis(acrylamide)s for dental filling materials. J Appl Poly Sci. 2009;113:3137–3145.

24. Johnston EP, Nicholls JI, Smith DE. Flexure fatigue of 10 commonly used denture base resins. J Prosthet Dent. 1981;46:478-483.

25. Faot F, Costa AM, Del Bel Cury A, Rodrigues Garcia CM. Impact strength and fracture morphology of denture acrylic resins. J Prosthet Dent. 2006;96:367-373.