

Original Article

Comparison of Mechanical and Dynamic Mechanical Behaviors of Different Dental Resins Polymerized by Different Polymerization Techniques

R Durkan, P Oyar¹

Department of Prosthodontics, Faculty of Dentistry, Afyon Kocatepe University, Afyonkarahisar, ¹Department of Dental Prosthesis Technology, Hacettepe University, Ankara, Turkey

ABSTRACT

Purpose: The aim of the present investigation was to evaluate the effect of autoclave polymerization method on the mechanical and dynamic mechanical properties of different polymethylmethacrylate denture base materials. **Materials and Methods:** Three different denture materials were used during the study, two of them were heat polymerizable denture base material (Meliodent and Paladent) and one was microwave polymerizable denture base material (Acron MC). Duncan test was used for the statistical analysis. Statistical analyses were completed using a two-way analysis of variance. Statistical analysis of test results was carried out with a 95% confidence level. **Results:** Tensile strength was increased with autoclave polymerization regardless of the denture base material type. Paladent specimens with autoclave polymerization (30 min at 60°C and 10 min at 130°C) have the highest average impact strength value. Acron MC specimens have the highest average flexural strength and modulus. Flexural strength improved with autoclave polymerization for both of 10 and 20 min polymerizations for each of Meliodent and Paladent specimens. **Conclusions:** Autoclave polymerization provided higher polymerization temperatures compared with the conventional heat polymerization. Autoclave-polymerized acrylic resin specimens showed higher tensile strength values; however, this was not the case for the impact test results. Flexural strength of specimens was improved with autoclave polymerization. Glass transition temperature was increased with autoclave polymerization.

KEYWORDS: Autoclave, dental polymers, denture base resin, mechanical properties

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INTRODUCTION

Polymethylmethacrylate (PMMA) is the most widely used dental material for removable dentures, implant-supported prostheses, and maxillofacial prostheses. Acrylic resins have many advantages of favorable working characteristics, ease of processing, good and accurate fit, polishability, processibility with inexpensive technical equipment, long-term stability in the oral environment, and esthetic appearance for long-term usage.^[1-5] However, there are some disadvantages of PMMA denture base materials such as fracture. Fractures in dentures result from different types of forces: flexural, fatigue, and impact.^[6] There are continuing efforts to

improve the mechanical and physical properties of the denture base materials. The performance could be explained as the ability of material to respond the different types of forces in both short-term and long-term time periods within the wide temperature range. Even though heat curing is the most commonly applied method (the conventional method) for polymerization of denture base materials, different techniques have been introduced such as injection molding, microwave polymerization,

Address for correspondence: Assoc. Prof. P Oyar, Department of Dental Prosthesis Technology, Hacettepe University, Ankara, Turkey.
E-mail: poyar73@gmail.com

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autoclave polymerization, and different types of polymers have been used in the dental applications.^[7-9]

The autoclave processing technique is a better substitute for water-bath technique^[10] and also one of the methods to improve physical and mechanical properties of high-impact acrylic resins. It is an easy method and requires less time compared to water-bath polymerization technique.^[11]

Mechanical properties of acrylic bases are much important. The impact strength and the flexural properties of denture base materials are of importance for predicting their clinical performance. The impact strength could be defined as the amount of energy required for fracturing a material under an impact force; in other words, it is a measure of the amount of energy that is absorbed by the material before fracture. Impact failures usually occur out of the mouth as a result of a sudden blow to the denture or by accidental dropping. The flexural strength of a material is a measure of stiffness and resistance to fracture. Tensile strength could be defined as the maximum tensile stress that can be applied uniformly over the cross-section of the material. Among many desired mechanical properties of a denture base resin, high tensile strength is particularly important for the materials to meet the required performance during their function. The flexural strength test, one of the mechanical strength tests, is especially useful in comparing denture base materials in which a stress is applied to the denture during mastication.^[10-12]

Dynamic mechanical analysis evaluates the behavior of the materials under dynamic loading.^[13,14] The storage modulus determines its rigidity and depends on its ability to store mechanical energy. The damping factor (loss factor) represents the temperature at which polymer chains acquire the ability to move freely within a plyometric mass. It is a measure of the energy dissipation of a material. A high damping factor indicates high molecular mobility in the material. As temperature increases, the material approaches the rubbery.^[15] The change of the dynamic mechanical properties of the denture base resins through autoclave polymerization has not been encountered in the literature. In addition, there are very limited studies in the literature about the autoclave polymerization of the denture base materials.

The aim of this *in vitro* study was to evaluate the effect of polymerization carried out in autoclave device on tensile strength, flexural strength, impact strength, and dynamic mechanical properties of the different acrylic denture base resins.

The hypothesis of the present investigation was that autoclave polymerization could improve the properties of PMMA denture base.

MATERIALS AND METHODS

Four different denture base materials were tested, three of them were heat-polymerized denture base material (Meliodent and Paladent) and one was microwave-polymerized denture base material (Acron MC). These are given in Table 1.

In preparing the test specimens, stainless steel mold was used. The standard wax specimens were obtained by pouring the melted pink plate waxes (Modeling Wax, De Trey S. A, Bios Colombes, France) into the isolated mold. The prepared wax specimens were taken into the mold, and these wax specimens were removed by melting them, and then, the molds were made ready for acrylic molding. Acrylic resin specimens were prepared at a powder/liquid ratio of 2.34 g/mL in accordance with the manufacturer's instructions. The acrylic resin was molded into the molds. After the molding procedure was executed, 5 min pressure was applied onto the molds under a hydraulic press (Rucker PHI, Birmingham, UK). The curing of heat-polymerized specimens was carried out for 30 min at 100°C for heat-polymerized control samples. Before deflasking, all the acrylic resin specimens were bench cooled. Test specimens were wet ground with silicone carbide grinding papers of 200, 400, and 600-grit sizes using an automatic polishing machine (Grin PO 2 V grinderpolisher; Metkon A. S, Bursa, Turkey).

Powder/liquid mix was packed into the mold and subjected to 550 W microwave irradiation for 3 min in microwave oven (EM-M 553 T, Sanyo) for microwave polymerization control specimens. All specimens (n:7) were prepared for all tests of tensile, flexural, dynamic mechanical tests (DMA), and impact.

The tensile strength was calculated by the following formula:

$$T. S = F (N)/A (\text{mm}^2)$$

T. S: Tensile strength (N/mm²), F: Peak load (N), A: Cross-sectional area (mm²)^[10]

Impact strength was calculated using the following formula:^[16]

$$IS = E/wt$$

Where IS is the impact strength, E is the energy, w is the width, and t is the thickness of the specimen.

Flexural strength was determined using the following formula:^[17]

$$FS = 3Fl/2bh^2$$

Where F is the maximum load applied (N), l is the distance between supports, b is the width of the test specimen, and h is the thickness of the specimen.

Flexural modulus was determined using the following formula:^[18]

$$Ef = FL^3/4 \delta wh^3 = mL^3/4wh^2$$

Ef: Flexural modulus (GPa), δ : The beam deflection when a force F is applied. F: The fracture load (N). L: The distance between the two supported points. w: The specimen width (mm). h: The specimen thickness (mm).

The specimens prepared for tensile tests were standard dumbbell-shaped test specimens (ISO 5271).^[19] Tensile tests were carried out for three groups. The first group, control group, consists of heat-polymerized acrylic resin specimens (Meliodent and Paladent) and microwave-polymerized specimens (Acron-MC). For the second group, acrylic resin specimens (Meliodent, Paladent, Acron-MC) were polymerized in the autoclave at 60°C for 30 min and followed by polymerization at 130°C for 10 min. The polymerization was achieved within the autoclave device (OT 4060 Steam Sterilizer, Nuve) which is a pressurized device designed to heat aqueous solutions above the normal boiling point of the aqueous solutions. The third group (Meliodent, Paladent, Acron-MC) was polymerized in autoclave sterilization unit at 60°C for 30 min and followed by polymerization at 130°C for 20 min. The polymerization was again achieved in the autoclave device. The temperature and time settings for the present investigation were determined according to a preliminary experimental study.^[11] Tensile and flexural tests were performed using Lloyd Universal Testing Machine (Lloyd Instruments, Model LRX) at a crosshead displacement of 10 mm/min. Impact strength tests were carried out using rectangular acrylic resin specimens measuring 60 mm × 7 mm × 4 mm according to ISO 1567:1998 standards. The impact strength test was performed using an impact test machine (Coesfeld GmbH and Co. KG, Pendulum Impact Tester) with a 40-mm opening between the two fixed supports. For dynamic mechanical testing (DMA) (Storage Modulus, Damping Factor, Glass Transition Temperature (T_g)), rectangular specimens measuring 60 mm × 5 mm × 3 mm were prepared, and dynamic mechanical analysis was carried out on Thermal Dynamic Mechanical Analyzer (Perkin Elmer Pyris Diamond DMA; Model 983 MA) under N₂ atmosphere, the temperature range of DMA tests was -100°C to 150°C with 10°C/min heat rate. All specimens were stored in a distilled water bath at 37°C for 48 h before testing.

Statistical analyses were completed using a two-way analysis of variance. Duncan test was used for the statistical analysis. Statistical analysis of test results was carried out with a 95% confidence level.

RESULTS

Dynamic mechanical tests - storage modulus

Change of storage modulus with polymerization method and denture base resin type is given in Figures 1 and 2. As can be seen from figures, the storage modulus values of Paladent denture base resin at -100°C with

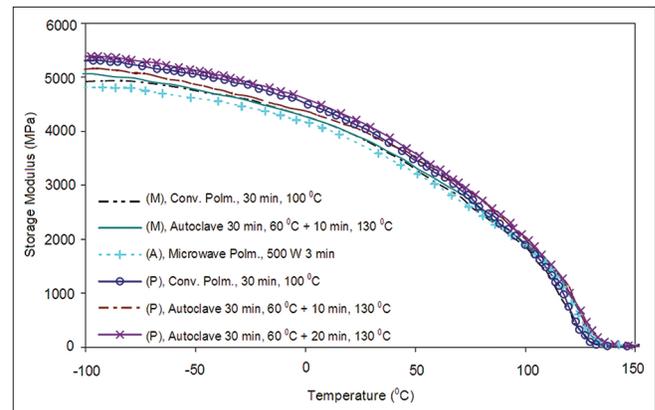


Figure 1: Change of storage modulus with polymerization method and denture base resin type 150°C–150°C (M: Meliodent, P: Paladent, A: Acron MC)

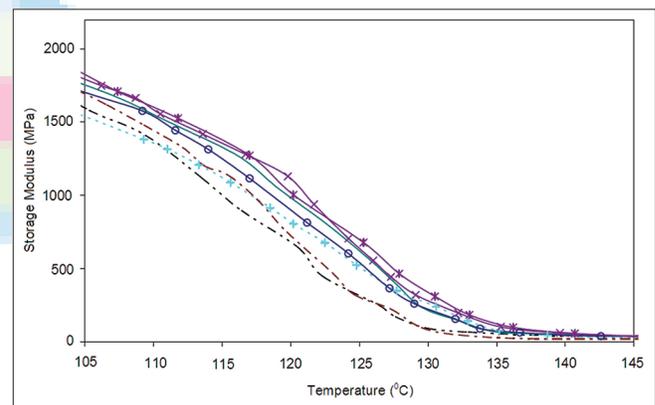


Figure 2: Change of storage modulus with polymerization method and denture base resin type 100°C–150°C (M: Meliodent, P: Paladent, A: Acron MC)

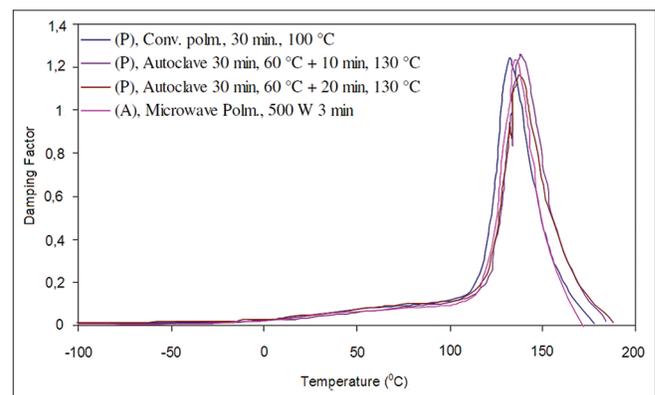


Figure 3: Change of damping factor with polymerization method (A: Acron MC, M: Meliodent, P: Paladent)

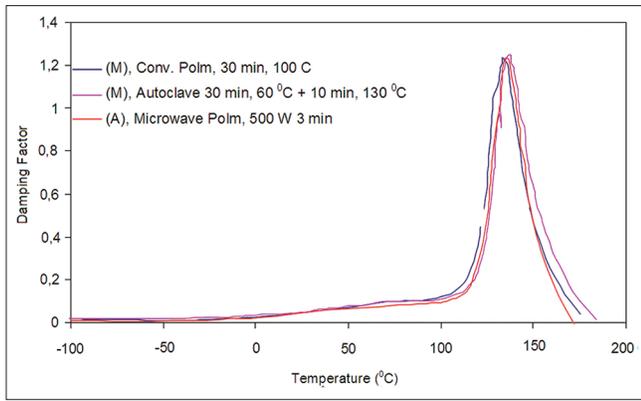


Figure 4: Change of damping factor with polymerization method (A: Acron MC, M: Meliodent, P: Paladent)

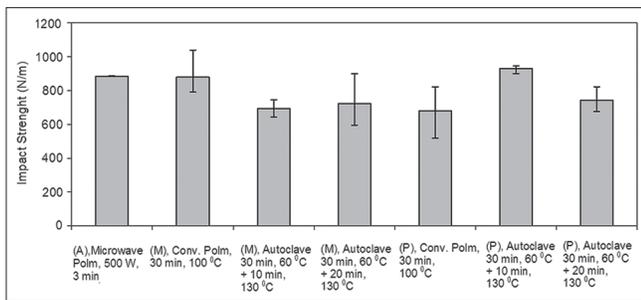


Figure 5: Change of impact strength with polymerization method and denture base resin type (A: Acron MC, M: Meliodent, P: Paladent)

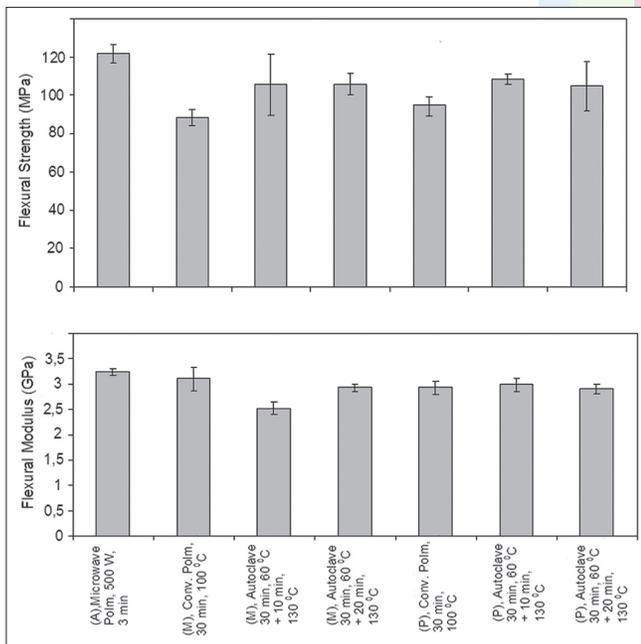


Figure 6: Change of flexural strength and modulus with polymerization method and denture base resin type (A: Acron MC, M: Meliodent, P: Paladent)

autoclave polymerized (30 min at 60°C and 10 min at 130°C), conventional heat polymerized, and autoclave polymerized (30 min at 60°C and 20 min at 130°C) were 5.15, 5.31, and 5.39 GPa, respectively. For

Table 1: Denture base materials used in the study

Materials	Manufacturer	Polymerization type
Meliodent	Dental Bayer Ltd	Heat
Paladent	Heraeus Kulzer GmbH and Co. KG	Heat
Acron-MC	GC Corp	Microwave

Table 2: Mean and standard deviations of tensile strength (MPa) values of specimens

Materials	Control group	Autoclave polymerization	
		30 min at 60°C and 10 min at 130°C	30 min at 60°C and 20 min at 130°C
Meliodent	41.75 (1.17) ^{A,a}	56.08 (2.99) ^{A,b}	56.50 (2.96) ^{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}

Same capital letters (A or B) in each column (vertical direction) denote statistically insignificant differences between tensile strength of materials. Same small letters (a or b) in each row (horizontal direction) denote statistically insignificant difference between tensile strength of materials

the Meliodent denture base resin case, conventional heat-polymerized and autoclave (30 min at 60°C and 10 min at 130°C)-polymerized specimens showed very similar storage modulus curve.

Dynamic mechanical tests - damping factor (loss factor)

Change of damping factor with polymerization method is shown in Figures 3 and 4. It is seen from Figure 3 that the glass transition temperature (T_g) of the denture base resin was improved with autoclave polymerization. T_g of the conventional heat-polymerized Paladent sample was 133°C and that of autoclave-polymerized samples was about 139°C as shown in Figure 3. T_g of the control sample of Meliodent was 133°C and that of autoclave (30 min at 60°C and 10 min at 130°C) polymerized sample was 138°C as shown in Figure 4.

Tensile strength

The means and standard deviations of tensile strength values of denture base materials tested are given in Table 2. Acrylic resin samples of both 10 and 20 min polymerized in autoclave device showed higher tensile strength values than the control groups. There was no statistically significant difference in between the increase of time of autoclave polymerization from 10 to 20 min. In addition, there was no statistically significant difference between the tensile strength values of Meliodent and Paladent samples for three different polymerization methods within each groups of control and autoclave polymerizations of 10 and 20 min. It was seen that tensile strength was increased with autoclave polymerization, regardless of the denture base material type.

Impact strength

Change of average impact strength values with polymerization method and denture base resin type is given in Figure 5. Paladent specimen with autoclave polymerization (30 min at 60°C and 10 min at 130°C) has the highest average impact strength value.

Flexural strength and modulus

Change of flexural strength and modulus with polymerization method and denture base resin type is given in Figure 4. Acron MC has the highest average flexural strength and modulus. Flexural strength improved with autoclave polymerization for both of 10 and 20 min polymerizations for each of Meliodent and Paladent specimens as can be seen from Figure 6.

DISCUSSION

The hypothesis that autoclave polymerization could improve the properties of PMMA denture base was accepted because autoclave polymerization had an effect on the mechanical and dynamic mechanical properties of different PMMA denture base materials.

Flexural strength improved with autoclave polymerization for each of Meliodent and Paladent specimens; however, this result was not parallel for flexural modulus results; this might probably be due to the difference in the composition and structure of different commercial denture base materials. The results of the present investigation were in agreement with Gad *et al.*^[9] who found autoclave polymerization significantly increased the flexural properties of PMMA denture bases; in addition, a nonsignificant difference in flexural strength between the short and long cycle of autoclave polymerization was found. The results of the present study were in disagreement with Abdulwahhab^[9] who found a nonsignificant difference in flexural strength between autoclave and water-bath polymerization methods; these conflicting results may be due to differences in the autoclave polymerization cycles or materials.

In the present study, a direct relation was not observed regarding the polymerization method and impact strength. Difference in the composition and structure of different commercial denture base materials might probably have resulted with irregularity in polymerization method and impact strength.

The autoclave polymerization was studied in 1977, and the best properties for the PMMA resin were achieved with the autoclave polymerization technique, and during that study, the tensile strength of the autoclave-polymerized resin was found as 61 MPa.^[20] It was seen that all of the postcuring methods studied,^[21] including autoclave curing for 15 min at 100°C, increased the hardness and tensile

strength of denture base material studied.^[22] In the current study, parallel finding was observed regarding the increase of the tensile strength with autoclave polymerization.

The glass transition temperature (T_g) of the denture base resin was improved with autoclave polymerization. It is clear from these results that the autoclave polymerization has an effect on the increase of glass transition temperature, thus showing the increase of mechanical properties. Storage modulus for Meliodent denture base resin with autoclave (30 min at 60°C and 20 min at 130°C) polymerization was lower than the conventional heat-polymerized and autoclave (30 min at 60°C and 10 min at 130°C)-polymerized specimen; this could be attributed to inversion of dynamic mechanical properties probably due to the deterioration of chain network with the increased period of heat transfer under pressurized environment. The storage modulus values have shown irregular characteristics for the relatively lower temperatures; in other words, a direct relation with the storage modulus and polymerization method was not observed. However, for relatively higher temperatures, a direct relation was observed. The storage modulus values for the conventional heat-polymerized denture base materials were lower than the autoclave-polymerized resins within the temperature range of 117°C–135°C.

There was no significant difference between the autoclave polymerization of the two cycles (long and short) and water bath curing methods regarding transverse strength test and tensile strength test.^[10] Regarding autoclave processing technique, the slow (long)-curing cycle provides better denture bases material including the tested physical and mechanical properties as compared with the fast (short)-curing cycle. The effect of autoclave processing on some properties of heat-cured denture base material has been investigated.^[9] For a long autoclave polymerization cycle, it could be used as an alternative to water-bath polymerization.^[8]

Effect of autoclave polymerization on the transverse strength of denture base polymers was studied.^[10,11] The results revealed that polymerization in an autoclave led to a statically significant increase in transverse strength when compared to the water bath. The autoclave curing resulted in better stability when compared with water bath; because autoclave provides even heat spreading and more cross-linking between the polymer chains with better opportunity for complete polymerization.^[23]

There is a continuing effort to improve the properties of denture base materials. Curing processes have been modified to improve the physical and mechanical properties of those materials.^[10,24] Autoclave

polymerization curing is suggested as a good alternative method for denture base resins.^[24-26]

The changes in the intraoral conditions can influence properties of acrylic denture base resin; for this reason, predicting the clinical behavior of denture base resin is difficult. Therefore, clinical and further studies are required.

CONCLUSIONS

Autoclave polymerization changed both the mechanical and dynamic mechanical properties of denture base materials. Higher tensile strength values were obtained when compared with the conventional water-bath technique for the heat-polymerized specimens. Flexural strength of the heat-curable denture base resins was improved regardless of the resin type with autoclave polymerization. In addition, T_g of the heat-curable denture base resin specimens were increased with the autoclave polymerization. Higher storage modulus for the autoclave-polymerized specimens at temperatures higher than the normal boiling temperature of the monomer supports the idea of the decrease of the residual monomer content of the denture base material polymerized with autoclave polymerization. It could be stated that autoclave polymerization method might be an alternative to conventional heat method.

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Conflicts of interest

There are no conflicts of interest.

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