

Effect of polymerization cycles on flexural strengths and microhardness of different denture base materials

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The purpose of this study was to evaluate the effect of different polymerization cycles on the flexural strengths and microhardness of two denture base materials (Meliodent and Paladent). Heat-polymerized acrylic resin specimens (65.0 mm long×10.0 mm wide×2.5 mm in height) were prepared using different short and long polymerization cycles. After the specimens had been polymerized, they were stored in distilled water at 37±1°C for 24 h. Flexural strength test was performed at a cross-head speed of 5 mm/min and Vickers microhardness was measured. Data were analyzed with a 1-way analysis of variance followed by Tukey test, and Student *t*-test ($\alpha=0.05$). The flexural strengths and microhardness were significantly different between Meliodent and Paladent ($p<0.05$). Significant differences were found among the polymerization cycles in terms of flexural strengths and microhardness ($p<0.05$). Polymerization with G cycle may be suggested for Meliodent and H cycle may be suggested for Paladent.

Keywords: Denture base material, Flexural strength, Microhardness, Polymerization cycle

INTRODUCTION

The aim of prosthetic rehabilitation is to improve masticatory function, esthetics, and speech. Although different materials have been used for denture base materials, poly(methyl methacrylate) (PMMA) has been extensively used since 1937^{1,2}. PMMA has the advantages of a lifelike appearance and stability in the oral environment, and it is easy to process, repair, and polish^{1,3,4}. However, PMMA also has several disadvantages, including allergic reactions to the residual monomer, insufficient surface hardness, poor wear resistance, and polymerization shrinkage^{1,5,6}. Additionally, PMMA does not have optimum physical properties of flexural and impact strength^{7,8}.

Fracture of the denture base materials constitutes a challenge and remains an unresolved problem. The fracture of the denture base usually occurs at the midline of the dental prosthesis due to flexural fatigue failure from repeated masticatory forces caused by cyclic deformation of denture base or high impact forces that occur as a result of dropping the prosthesis^{3,9,10}. Three-point bend test more closely simulated the type of stress that is applied to the prosthesis during mastication^{10,11}. Therefore, the flexural strength of denture base materials is widely evaluated by this test.

Hardness is also an essential physical property of denture base materials and indicates its resistance to plastic deformation^{12,13}. Hardness influences ease of cutting, finishing, and polishing which reduces scratches that can compromise fatigue strength and lead to premature failure. Finishing and polishing are extremely important to achieve ideal esthetics and good oral hygiene^{4,12}. The surface microhardness of acrylic resins is directly related to the longevity of the prosthesis in which the greater the microhardness, the greater its

resistance to abrasion and fracture of the denture base material^{13,14}. In addition, the microhardness is related to wear of dental materials. It may be more appropriate for the evaluation of physico-mechanical properties and is considered to be a parameter for the residual monomer content. Therefore, the Vickers hardness test is a simple, effective way to assess the degree of conversion of monomer to polymer during polymerization¹³⁻¹⁵.

The polymerization reaction of acrylic resins has been initiated by heat, light, auto, microwave, or injection molding¹⁶⁻¹⁸. Heat polymerization is the most widely used polymerization method of acrylic resin denture base materials and is usually carried out in a heated water bath¹⁹. During the polymerization reaction of acrylic resins, the conversion of monomers to polymers is not accomplished and varying amounts of free or unreacted monomer, called residual monomers, are retained in the denture base material¹⁵. In addition, porosity may occur in the denture base material after polymerization²⁰. The residual monomer content and porosity affect the physical and mechanical properties of the acrylic resins^{15,20}. It has been stated that the residual monomer content and porosity may be reduced by the polymerization method and cycle^{15,19-21}. Bural *et al.*¹⁹ have investigated that effect of polymerization cycles on residual monomer content and *in vitro* cytotoxicity of denture base materials. Several studies have evaluated that the properties of denture base materials polymerized with conventional water bath, microwave energy, and light^{17,20,21}. There are limited reports in the literature evaluating the effect of polymerization cycles on the physico-mechanical properties of acrylic resins. Therefore, the purpose of the present investigation was to evaluate the effect of different polymerization cycles on flexural strengths and microhardness of two different acrylic resin denture base materials. The first null

hypothesis is that polymerization cycles are not affect the flexural strengths and microhardness of denture base materials. The second null hypothesis is that the flexural strengths and microhardness are not different according to the denture base materials.

MATERIALS AND METHODS

Two types of commercially available heat-polymerized acrylic resin denture base materials were tested in the present study (Table 1). Eighty rectangular specimens (65.0 mm long×10.0 mm wide×2.5 mm in height) of each material were prepared according to American Dental Association (ADA) Specification No. 12²²⁾ using 10 different polymerization cycles (Table 2). Heat polymerization was performed in a thermostatically controlled water bath (PolyScience, Niles, IL, USA), and then the flasks were allowed to slowly cool down in the water bath. After the polymerization process was completed, residual acrylic resin was carefully removed with a tungsten carbide bur at low speed. The specimens were then stored in distilled water at 37±1°C for 24 h before testing.

A three-point bend test was performed immediately after removing the specimens from the distilled water without drying the specimens. This test was carried out on a universal testing machine (Model 2519-106, Instron, Norwood, MA, USA). A custom-made stainless steel device with a 50 mm span between the two supports was used, and the crosshead speed was set at 5 mm/min. A load was applied to the center of the specimens. The load was increased until the specimens fractured at which time the maximum fracture load was recorded.

The flexural strengths of each specimen were calculated using the following formula: $S=3WL/2bd^2$, where S is the flexural strength (in MPa), W is the maximum fracture load (in Newtons), L is the distance between the supports (50 mm), b is the specimen width (10 mm), and d is the specimen thickness (2.5 mm).

The Vickers hardness test was employed to measure surface hardness by using an indenter point in the shape of a square-based pyramid. The test was performed using a microhardness tester (HV-1000B, TMTeck Manufacturing Limited, Beijing, China) with an applied load of 2.942 N at a 15-s dwell time at room temperature. For each specimen, three Vickers hardness

Table 1 Denture base materials used in present study

Denture base materials	Product name	Manufacturer	Chemical composition	Batch number
Heat-polymerized acrylic resin	Meliodent	Heraeus Kulzer, Hanau, Germany	Powder: Methyl methacrylate, Ethyl hexyl acrylate, N-octyl methacrylate Liquid: Methyl methacrylate, glycol dimethacrylate, dimethyl p-toluidine	Powder: 10MAR048 Liquid: 012330
Heat-polymerized acrylic resin	Paladent 20	Heraeus Kulzer	Powder: Methyl methacrylate, Ethyl hexyl acrylate, N-octyl methacrylate Liquid: Methyl methacrylate, glycol dimethacrylate, dimethyl p-toluidine	Powder:* Liquid: 012337

*Manufacturer does not provide batch number.

Table 2 Polymerization cycles (polymerization temperatures and polymerization times)

Code	Polymerization cycle
A	9 h at 74°C
B	9 h at 74°C and then 100°C boiling water for 3 h
C	9 h at 74°C and then 100°C boiling water for 30 min
D	3 h at 74°C and then 100°C boiling water for 1 h
E	90 min at 74°C
F	90 min at 74°C and then 100°C boiling water for 30 min
G	30 min at 74°C and then 100°C boiling water for 30 min
H	100°C boiling water for 3 h
I	100°C boiling water for 1 h
J	100°C boiling water for 30 min

indentations were made at different points along the specimen. The mean hardness was calculated and used for the statistical analysis.

Data from flexural strengths and microhardness were analyzed using one-way analysis of variance (ANOVA) and Tukey's HSD test with a confidence level of 0.05 to determine the mean differences. Student *t*-test was used to compare differences between polymerization cycles for Meliodent and Paladent ($\alpha=0.05$). These analyses were performed with SPSS statistical software (SPSS v16.0, SPSS, Chicago, IL, USA).

RESULTS

The mean and standard deviation of the flexural strengths for each of the experimental groups are presented in Table 3. The flexural strengths for the polymerization cycle in the Meliodent group can be arranged as G>D>F>J>B>H>A>C>I>E. The specimens that were polymerized with the E cycle were significantly lower in flexural strengths than those of other cycles ($p<0.05$). No significant differences were noted among those polymerized with the I, C, A, H, and B; or the A, H, B, J, F, D, and G cycles ($p>0.05$). However, the I and C cycles had significantly lower flexural strengths than those of the J, F, D, and G cycles ($p<0.05$).

The flexural strengths for the polymerization cycle in the Paladent group can be arranged as H>I>F>G>D>B>J>C>A>E. The specimens that were polymerized with the E cycle were significantly lower in flexural strengths than those of other cycles ($p<0.05$). No significant differences were noted among those

polymerized with the H, I, F, G, D, and B; the I, F, G, D, B, J, and C; or the B, J, C, and A cycles ($p>0.05$). However, the H cycle had significantly higher flexural strengths than those of J, C, A, and E cycles ($p<0.05$).

The Meliodent specimens that were polymerized with A, D, G, and J cycles had significantly higher flexural strengths than those of the Paladent specimens ($p<0.05$). The Meliodent specimens that were polymerized I cycle had significantly lower flexural strengths than those of the Paladent specimens ($p<0.05$).

The mean and standard deviation of the microhardness for each of the experimental groups are presented in Table 4. The microhardness for polymerization cycle in the Meliodent group can be arranged as C>H>B>I>G>D>F>J>A>E. The specimens polymerized with the A and E cycles had significantly lower microhardness than those polymerized with the other cycles ($p<0.05$) and a significant difference was found between the A and E cycles ($p<0.05$). The specimens polymerized with the C cycle had significantly higher microhardness than those polymerized with the other cycles ($p<0.05$), except for the H, B, and I cycles ($p>0.05$). In addition, no significant differences were noted among the specimens polymerized with the H, B, I, G, D, and F; and G, D, F, and J cycles ($p>0.05$).

The microhardness for polymerization cycle in the Paladent group can be arranged as C>I>B>D>H>F>G>J>A>E. The specimens polymerized with the A and E cycles had significantly lower microhardness than those polymerized with the other cycles ($p<0.05$) and a significant difference was found between the A and E cycles ($p<0.05$). The specimens

Table 3 Mean (SD) flexural strengths (MPa) of denture base materials polymerized different cycles

Polymerization cycles	Denture base materials	
	Meliodent	Paladent
A	105.99 (7.67) ^{ab,A}	84.19 (15.28) ^{a,B}
B	112.18 (17.05) ^{abc,A}	103.41 (14.10) ^{abc,A}
C	95.96 (12.77) ^{b,A}	93.84 (14.66) ^{ab,A}
D	127.75 (14.42) ^{a,A}	108.38 (17.10) ^{bc,B}
E	58.19 (11.03) ^{c,A}	56.89 (5.90) ^{d,A}
F	126.75 (18.15) ^{a,A}	112.49 (17.36) ^{bc,A}
G	128.60 (15.57) ^{a,A}	109.54 (5.60) ^{bc,B}
H	110.96 (15.41) ^{ab,A}	124.09 (18.89) ^{c,A}
I	90.58 (16.55) ^{b,A}	115.34 (15.48) ^{bc,B}
J	126.68 (15.29) ^{a,A}	100.14 (11.39) ^{ab,B}

A: 9 h at 74°C; B: 9 h at 74°C and then 100°C boiling water for 3 h; C: 9 h at 74°C and then 100°C boiling water for 30 min; D: 3 h at 74°C and then 100°C boiling water for 1 h; E: 90 min at 74°C; F: 90 min at 74°C and then 100°C boiling water for 30 min; G: 30 min at 74°C and then 100°C boiling water for 30 min; H: 100°C boiling water for 3 h; I: 100°C boiling water for 1 h; J: 100°C boiling water for 30 min.

Vertically, significant difference between means are characterized by different lowercase letters; horizontally, significant difference between means are characterized by different uppercase letters.

Table 4 Mean (SD) microhardness (HV) of denture base materials polymerized different cycles

Polymerization cycles	Denture base materials	
	Meliudent	Paladent
A	21.53 (3.97) ^{a,A}	24.25 (2.76) ^{a,A}
B	29.33 (0.82) ^{cd,A}	30.17 (1.19) ^{cd,A}
C	31.99 (1.16) ^{d,A}	30.89 (1.95) ^{d,A}
D	27.48 (1.01) ^{bc,A}	29.03 (1.12) ^{bcd,B}
E	7.92 (0.80) ^{e,A}	10.14 (1.10) ^{e,B}
F	26.51 (1.99) ^{bc,A}	27.33 (1.02) ^{b,A}
G	28.03 (2.08) ^{bc,A}	27.14 (0.87) ^{b,A}
H	29.43 (2.05) ^{cd,A}	27.90 (0.97) ^{bc,A}
I	29.13 (0.83) ^{cd,A}	30.58 (1.71) ^{d,A}
J	25.81 (1.00) ^{b,A}	27.04 (1.34) ^{b,A}

A: 9 h at 74°C; B: 9 h at 74°C and then 100°C boiling water for 3 h; C: 9 h at 74°C and then 100°C boiling water for 30 min; D: 3 h at 74°C and then 100°C boiling water for 1 h; E: 90 min at 74°C; F: 90 min at 74°C and then 100°C boiling water for 30 min; G: 30 min at 74°C and then 100°C boiling water for 30 min; H: 100°C boiling water for 3 h; I: 100°C boiling water for 1 h; J: 100°C boiling water for 30 min.

Vertically, significant difference between means are characterized by different lowercase letters; horizontally, significant difference between means are characterized by different uppercase letters.

polymerized with the C cycle had significantly higher microhardness than those polymerized with the other cycles ($p < 0.05$), except for the I, B, and D cycles ($p > 0.05$). In addition, no significant differences were noted among the specimens polymerized with the B, D, and H; and D, H, F, G, and J cycles ($p > 0.05$).

The Paladent specimens that were polymerized with D and E cycles had significantly higher microhardness than those of the Meliudent specimens ($p < 0.05$). No significant differences were noted between the Meliudent and Paladent specimens that were polymerized with A, B, C, F, G, H, I, and J cycles ($p > 0.05$).

DISCUSSION

The present study evaluated the effect of different polymerization cycles on the flexural strengths and microhardness of acrylic resin denture base materials. The first null hypothesis was rejected because the data showed that the polymerization cycles significantly affected the flexural strengths and microhardness of denture base materials ($p < 0.05$). The flexural strengths in the A, D, G, J, and I cycles and microhardness in the D and E cycles were significantly different according to the denture base materials ($p < 0.05$). Thus, the second null hypothesis was partially rejected.

Seo *et al.*²³ reported that flexural strengths of heat-polymerized acrylic resin, when polymerized for 90 min at 73°C and then 100°C boiling water for 30 min (short cycle), were higher than when it was polymerized for 9 h at 71°C (long cycle). This finding is in accordance with the present study in which the specimens polymerized with

the F cycle (90 min at 74°C and then 100°C boiling water for 30 min) had higher flexural strengths than with the A cycle (9 h at 74°C). The physico-mechanical properties of polymers are determined by the molecular weight of the mer (unpolymerized molecule) and by the residual monomer levels, degree of chain branching, polymer chain length, cross-linking and cross-link density within the molecule, and the presence of plasticizers and/or fillers. Plasticizers are added to produce a softer, more resilient polymer and to lower the glass-transition temperature (T_g) of the polymer, so a material that is normally rigid at a particular temperature may become more flexible. The T_g is the temperature at which a resin ceases to be glassy and brittle and becomes rubberlike^{16,24}. In addition, Azzarri *et al.*²⁵ reported that the residual monomers act as plasticizers which reduce the polymer interchain forces; thus, this affects the physico-mechanical properties and biocompatibility of the acrylic resins. However, Urban *et al.*²⁶ reported that the residual monomer content of heat-polymerized acrylic resin, when polymerized for 9 h at 73°C, was higher than that of acrylic resin polymerized for 90 min at 73°C and then boiling water for 30 min because of the relatively low polymerization temperature, which was below T_g . Therefore, it is likely that the monomer had a poorer ability to polymerize due to lower molecular chain motions and immobilization of the monomer in the glassy polymer. Consequently, the differences among polymerization cycles of denture base materials may be explained by the increase of polymerization temperature. This increased may be caused enhanced the degree of conversion of monomer to polymer resulting in a lower

level of residual monomer and increased T_g .

The Meliodent specimens polymerized with G cycle exhibited the highest flexural strengths, while the Paladent specimens polymerized with H cycle exhibited the highest flexural strengths. In addition, the flexural strengths were significantly different between Meliodent and Paladent specimens polymerized with A, D, G, I, and J ($p < 0.05$). Pure polymers, such as PMMA, are clear and are adaptable to a wide range of pigmentation. Colorants are added to obtain the various tissue-like shades, and zinc or titanium oxides are used as opacifiers¹²). Thus, it may be that the differences between the denture base materials are probably due to the colorants or opacifiers. According to International Standards Organization (ISO) Specification number 20795-1, the required flexural strength for the heat-polymerized acrylic resin denture base materials should be not less than 65 MPa²⁷). In the present study, the denture base materials polymerized with different cycles had flexural strengths greater than 65 MPa, except with the E cycle.

Seo *et al.*²³) noted that no significant difference was found between the heat-polymerized acrylic resin specimens polymerized with short or long cycles for microhardness ($p > 0.05$). In contrast, in the present study, the microhardness for specimens polymerized with the F cycle were significantly higher than those polymerized with the A cycle ($p < 0.05$). These differences may be due to variation of time and temperature during polymerization which can affect the residual monomer content of the specimens. The residual monomer content in the polymerized acrylic resin can be decrease by diffusion into water and the leaching of residual monomer is a temperature-dependent process, thus increasing the temperature enhances the diffusion. In addition, the residual monomer content reduces due to additional polymerization at the sites of active radicals, and at higher temperatures, monomer molecules should diffuse more rapidly to these active sites and the residual monomer content should decrease. Another factor that affect the residual monomer content is its hydrolysis to methacrylic acid^{15,26}).

Ayaz *et al.*¹⁵) investigated microhardness of three different heat-polymerized acrylic resins (Meliodent, Paladent and QC) polymerized with conventional water bath (100°C boiling water for 30 min) and autoclave techniques. They demonstrated that QC had significantly higher microhardness than Meliodent and Paladent, and no significant differences were noted between Meliodent and Paladent for the conventional water bath polymerization. These results are in accordance with the present study in which no significant difference was found between the Meliodent and Paladent specimens polymerized with J cycle (100°C boiling water for 30 min) for microhardness ($p > 0.05$). In addition, the microhardness were significantly different between Meliodent and Paladent specimens polymerized with D and E cycles ($p < 0.05$). This result may be due to the colorants or opacifiers as discussed previously.

In the present study, the most efficient cycle was different between the flexural strengths and

microhardness. Nunes de Mello *et al.*²⁸) reported that an additional microwave or water bath polymerization caused higher internal microhardness at greater specimen depths. Therefore, this difference may be that the polymerization was more effective in the interior of the specimen than in the superficial layers.

The present investigation had a number of limitations. Only two denture base materials were tested. The use of rectangular specimens instead of more complex denture shapes and the *in vitro* nature of this investigation may not account for changes inherent in the materials after long periods of use under oral fluid conditions. Further *in vitro* studies and clinical research are necessary to investigate the different physico-mechanical properties of the denture base materials over a longer usage period of the dental prosthesis.

CONCLUSIONS

Within the limitations of the present investigation, the following conclusions were drawn:

1. The flexural strengths and microhardness of denture base materials varied depending on the polymerization cycles.
2. The G cycle was the most effective polymerization cycle in the Meliodent group in terms of the flexural strengths, while the H cycle was the most effective in the Paladent group.
3. The specimens polymerized with the C cycle had the highest microhardness in the Meliodent and Paladent groups, although this cycle was not better than the G and H cycles in terms of the flexural strengths.
4. The denture base materials that were polymerized with short and long cycles at 74°C did not have enough flexural strengths and microhardness. Therefore, they need to be polymerized at 100°C for at least 30 min.

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