Flexural properties of ethyl or methyl methacrylate-UDMA blend polymers

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Light-curing polyethyl methacrylate (PEMA)-urethane dimethacrylate (UDMA) resins and polymethyl methacrylate (PMMA)-UDMA resins were prepared by two processes. For first step, PEMA or PMMA powders were fully dissolved in ethyl methacrylate (EMA) or methyl methacrylate (MMA) and then the PEMA-EMA/PMMA-MMA mixtures were mixed with UDMA. The flexural properties of cured PEMA-UDMA and PMMA-UDMA polymers were measured using two PEMA (Mw: 300,000–400,000 and 650,000–1,000,000) and three PMMA (Mw: 30,000–60,000, 350,000 and 650,000–1,000,000) powders with different molecular weight, four mixing ratios of PMMA-MMA, and three mixing ratios of PMMA-MMA mixture and UDMA oligomer. Polymers with PMMA(Mw: 350,000)-MMA=25/50, and with PMMA(Mw: 350,000)-MMA/UDMA=1/2 and =1/1, showed no-fracture in a flexural test at 1 mm/min and flexural strength and flexural modulus showed no significant difference compared with those of commercially available heat- and self-curing acrylic resins (p>0.01). Within limitation of this investigation, methyl methacrylate-UDMA blend polymer of this composition is available for denture base resin.

Keywords: Denture base resin, Urethane dimethacrylate, Flexural properties

INTRODUCTION

Clinical reports1,2) showed that midline fractures have commonly been caused in complete dentures. Intraoral repeated masticatory forces lead to fatigue fracture, while extraorally impact fracture may occur as a result of dropping the denture onto the hard surface. Many attempts to improve the mechanical properties of polymethyl methacrylate (PMMA) have included using alternate polymers such as polycarbonate3, nylon4,5, co-polymers6,7, and urethane dimethacrylate (UDMA)8,9, and incorporating of butadiene-styrene rubber into PMMA matrix10,11. As another attempt, various fiber or chip materials12-16 such as carbon, aramid and glass have been added into PMMA resin for reinforcing materials, and metal wires and meshes have ordinary been used for reinforcing materials to prevent the midline fracture of denture.

The light-curing UDMA denture base resin such as Triad and Eclipse (DENTSPLY, York, USA) were used because of easiness of manipulation and of a lack of mixing process in contrast to PMMA as conventional denture base resin. However, its application has been limited because of the material’s brittleness and low impact resistance. The materials fracture without plastic deformation17,18. In addition, Eclipse requires extra laboratory armamentarium and training are required19. Therefore, we designed a PMMA-UDMA blend polymer to make a trial denture base resin. As this method, it is also one of the alternatives to mix MMA with UDMA and polymerize the mixture. However, the viscosity of the mixture is too low for denture base materials to manipulate. Moreover, longer-chained UDMA does not interpenetrate into PEMA or PMMA powder and then methacrylate powder does not swell and dissolve in the UDMA. Therefore, we used two processes to mix the methacrylate powder into UDMA. For first step, the methacrylate powder was fully dissolved in methacrylate monomer and then the viscous polymer-monomer mixture was mixed with UDMA.

In this study, the flexural properties of light-cured ethyl or methyl methacrylate-UDMA blend polymers were measured using two polyethyl methacrylate (PEMA) powders and three PMMA powders with different molecular weight, four mixing ratios of PMMA and MMA, and three mixing ratios of PMMA-MMA mixture and UDMA oligomer.

MATERIALS AND METHODS

Materials
Two polyethyl methacrylate, three polymethyl methacrylate, one ethyl methacrylate (EMA) (Wako Pure Chemical Industries Ltd., Osaka, Japan), one methyl methacrylate (Wako Pure Chemical Industries Ltd., Osaka, Japan), and one urethane dimethacrylate oligomer were used in this investigation. Two commercially available denture base resins (PourResin and Acron) were used for control of flexural properties (Table 1).

Preparation of mixtures of PEMA/EMA or PMMA/ MMA and UDMA oligomer
1. Two PEMA and three PMMA powders
Two PEMA powders and EMA monomer, and three PMMA powders and MMA monomer were mixed in polypropylene cups with the mixing ratios shown in
Table 1 Materials used in this investigation

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Information</th>
<th>Mw*</th>
</tr>
</thead>
<tbody>
<tr>
<td>PourResin</td>
<td>SHOFU1</td>
<td>Acrylic self-curing resin</td>
<td>-</td>
</tr>
<tr>
<td>Acron</td>
<td>GC2</td>
<td>Acrylic heat-curing resin</td>
<td>-</td>
</tr>
<tr>
<td>M-5000</td>
<td>Negami Chemical Ind.3</td>
<td>Polyethyl methacrylate</td>
<td>700,000–1,300,000</td>
</tr>
<tr>
<td>M-5001</td>
<td>Negami Chemical Ind.</td>
<td>Polyethyl methacrylate</td>
<td>300,000–450,000</td>
</tr>
<tr>
<td>M-4003</td>
<td>Negami Chemical Ind.</td>
<td>Polymethyl methacrylate</td>
<td>650,000–1,000,000</td>
</tr>
<tr>
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<td>Negami Chemical Ind.</td>
<td>Polymethyl methacrylate</td>
<td>350,000</td>
</tr>
<tr>
<td>M-4006</td>
<td>Negami Chemical Ind.</td>
<td>Polymethyl methacrylate</td>
<td>30,000–60,000</td>
</tr>
<tr>
<td>SH-500B</td>
<td>Negami Chemical Ind.</td>
<td>Urethane dimethacrylate</td>
<td>600</td>
</tr>
</tbody>
</table>

1) Kyoto, Japan 2) Tokyo, Japan 3) Ishikawa, Japan

*Average molecular weight by manufacturer's information

Table 2 The mixing ratios of two PEMA powders and EMA monomer, and three PMMA powders and MMA monomer

<table>
<thead>
<tr>
<th>Code</th>
<th>Methacrylate</th>
<th>Mixing ratio</th>
<th>Mixing ratio*</th>
</tr>
</thead>
<tbody>
<tr>
<td>M-5000/E/U</td>
<td>M-5000/EMA</td>
<td>15/50</td>
<td>1/1</td>
</tr>
<tr>
<td>M-5001/E/U</td>
<td>M-5001/EMA</td>
<td>15/50</td>
<td>1/1</td>
</tr>
<tr>
<td>M-4003/M/U</td>
<td>M-4003/MMA</td>
<td>10/50</td>
<td>1/1</td>
</tr>
<tr>
<td>M-4005/M/U</td>
<td>M-4005/MMA</td>
<td>15/50</td>
<td>1/1</td>
</tr>
<tr>
<td>M-4006/M/U</td>
<td>M-4006/MMA</td>
<td>45/50</td>
<td>1/1</td>
</tr>
</tbody>
</table>

*Methacrylate/UDMA(SH-500B)

Table 3 Four mixing ratios of PMMA and MMA, and three mixing ratios of PMMA-MMA mixture and UDMA oligomer

<table>
<thead>
<tr>
<th>Code</th>
<th>Methacrylate</th>
<th>Mixing ratio</th>
<th>Mixing ratio*</th>
</tr>
</thead>
<tbody>
<tr>
<td>10/50-1/1</td>
<td>M-4005/MMA</td>
<td>10/50</td>
<td>1/1</td>
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<tr>
<td>15/50-1/1</td>
<td>M-4005/MMA</td>
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<td>M-4005/MMA</td>
<td>15/50</td>
<td>2/1</td>
</tr>
</tbody>
</table>

*Methacrylate/UDMA(SH-500B)

Table 2. The mixtures were oscillated on a vibrating shaker (Vibramax 100; Heidolph, Nurnberg, Germany) for 7 days until the PEMA and PMMA fully dissolves and the viscosity shows about 120 Pa·s at 25°C by viscometer (RheoStress 600; Haake, Karlsruhe, Germany). The viscosity of mixtures is important for a manipulation property on a plaster model and the viscosity of 120 Pa·s was decided for the period that PMMA clearly dissolves to MMA within 7 days. The dissolved mixtures were mixed with UDMA oligomer with the mixing ratio of 50/50 by weight. At the same time, 0.5% (wt) camphorquinone (Wako Pure Chemical Industries Ltd., Osaka, Japan) as a photosensitizer and 0.5% (wt) 2-dimethyl amino ethyl methacrylate (Wako Pure Chemical Industries Ltd., Osaka, Japan) as a reductant were added. A homogenizing machine (AR-100; Thinky, Tokyo, Japan) with high-speed rotation (800 rpm) and orbital motion (2,200 rpm) was used to mix the materials in a darkened room. The each mixing time was 30 s. The mixtures were stored in lightproof bottles. The mixture based on M-4006 was separated to an upper phase with low viscosity (-LV) and a lower phase with high viscosity (-HV) after a few days; therefore high viscous M-4006/E/U was held by the spatula and low viscous M-4006/E/U escaped to another cup by devoting cup, and two mixtures were used to make test specimens. PEMA-EMA and UDMA, and PMMA-MMA and UDMA mixtures were light cured using an irradiation unit (α-Light, Morita, Tokyo, Japan) for 3 min in a 3-mm-thick PTFE mold. The cured sheets (50×70×2.5 mm) were cut into about 7-mm-wide strips using a water-cooled diamond blade. Self- and heat-curing denture base resins and UDMA polymer (SH; SH-500B) were used as controls. The self-curing denture base resin, a PMMA-based, multiphase polymer (PourResin; PR), was mixed at a powder/liquid ratio of 1.8 g/mL. The PTFE mold was filled at 5 min after mixing and heated in an oven at 50°C for 90 min.
The heat-curing denture base resin, a PMMA-based (Acron; AC), was mixed in a powder/liquid ratio of 2.5 g/mL, and the mold was filled at 20 min after mixing; the mold was pressed at 5 MPa and heated in an oven at 100°C for 60 min.

2. Four mixing ratios of PMMA and MMA
The four mixtures of M-4005 and MMA monomer were prepared to measure the effect of the mixing ratio of PMMA and MMA. The mixing ratios were shown in Table 3. The mixtures and the test specimens were prepared by the same method as the former.

3. Three mixing ratios of PMMA-MMA mixture and UDMA oligomer
The three mixtures of M-4005, MMA and SH-500B were prepared to measure the effect of the mixing ratio of PMMA-MMA and UDMA. The mixing ratios were shown in Table 3. The mixtures and the test specimens were prepared by the same method as the former.

Measuring the flexural properties
All test specimens were polished with wet-and-dry polishing emery paper (#600) until they were 2.5-mm-thick and 7-mm-wide. A three-point flexural test was conducted with a universal testing machine (TGE-5kN; Minebea, Nagano, Japan) using a crosshead speed of 1 mm/min and a span length of 30 mm. The flexural strength and flexural modulus were calculated using formulas of previous report.13

SEM observation
The surfaces of fractured test specimens were examined with a scanning microscope (JSM-5510; JEOL, Tokyo, Japan) with an accelerating potential of 20 kV after Au evaporation.

Gel permeation chromatography
The mixture separated to two phases (M-4006/M/U-LV and -HV) was measured the molecular weight distribution using the gel permeation chromatography (GPC) (DS-4; Shodex, Tokyo, Japan) and data processing controller (SIC480II DataStation; System Instruments Co. Ltd., Tokyo, Japan). A sample (100 mg) of M-4006/M/U-LV and -HV before curing was stored in 5 mL tetrahydrofuran (THF) for more than 24 hr. The temperature of the chromatographic column (KF-803L+KF-805L; Shodex, Tokyo, Japan) was maintained at 40°C. The mobile phase was THF at a flow rate of 1 mL/min. The detection wavelength was 254 nm (UV) and the sample volume was 20 µL.

After flexural test, to measure the residual monomer or oligomer, six polymerized test specimens (M-5000/E/U, M-5001/E/U, M-4003/M/U, M-4005/M/U, M-4006/M/U) were crushed in the agate mortar. The crushed powder (100 mg) was stored in 5 mL tetrahydrofuran (THF) for more than 48 hr and the peaks of residual UDMA oligomer and EMA or MMA monomers were detected by GPC. The ratios of residue (%) were calculated from the height under the UDMA, EMA and MMA peaks before and after polymerization.

Statistical analysis
One-way analysis of variance (ANOVA) and Tukey's test were used to compare the differences in flexural strength, flexural modulus, and deflection determined in the three-point flexural test (n=5).

RESULTS

Typical Stress-deflection curves of each denture base resins (AC, PR, and experimental polymer of code 25/50-1/1) measured by flexural test were shown in Figure 1. The flexural strength, flexural modulus, and deflection at maximum stress of the blend polymers made from two PEMA and three PMMA powders with different molecular weight are shown in Figure 2 with controls. No significant difference in flexural strength is showed between two acrylic resins (PR and AC) and trial polymer based on M-5000 (M-5000/E/U), M-5001 (M-5001/E/U), and M-4005 (M-4005/M/U) (p>0.01). No significant difference in flexural modulus is showed between two acrylic resins (PR and AC) and polymer based on M-4005 (M-4005/M/U) (p>0.01). Flexural strength and flexural modulus of polymers added PMMA-MMA mixtures of M-4003 and M-4005 to UDMA oligomer (M-4003/M/U and M-4005/M/U) are increased compared with UDMA polymer (SH) (p<0.01); however, deflection is decreased, respectively. Flexural strength and flexural modulus of M-4005/M/U were larger than those of M-4003/M/U, and M-4006/M/U-LV and -HV (p<0.01). Flexural strength was significantly decreased by mixing UDMA with M-4006 (p>0.01). Deflection of M-5000/E/U, M-5001/E/U, M-4003/M/U, and M-4005/M/U increased compared with PR and AC (p<0.01). Flexural modulus in M-5000/E/U is smaller than that in M-4005/M/U; however deflection in M-5000/E/U is larger than that in M-4005/M/U (p<0.01). Flexural strength, flexural modulus, and deflection of the test specimen (10/50-1/1, 15/50-1/1, 20/50-1/1, and 25/50-1/1 for codes) made with four mixing ratios

![Fig. 1 Stress-deflection curves measured by flexural test.](PR: PourResin; AC: Acron; 25/50-1/1: PMMA/MMA=25/50 and PMMA-MMA/UDMA=50/50)
of PMMA/MMA are shown in Figure 3. All results obtained were not showed statistically significant difference ($p>0.01$).

Flexural strength, flexural modulus, and deflection of the test specimen (15/50-1/2, 15/50-1/1, and 15/50-2/1 for codes) made with three mixing ratios of PMMA-MMA/UDMA are shown in Figure 4. No significant difference in flexural strength and flexural modulus were found between code 15/50-1/2 and code 15/50-1/1 ($p>0.01$).

SEM images of test specimens fractured by flexural test were shown in Figure 5. The SEM images of PR
and AC recognized shapes or cracks caused by polymer particles (Figs. 5a and b). M-4006/M/U-HV showed the flat surface (Fig. 5c) and there was no sign of drawing. Fractured surfaces of 10/50-1/1 and 15/50-1/2 covered with continuous wrinkle and the image which is analogized the heterogeneous shape is not observed in all fractured test specimen surfaces (Figs. 5d and e).

The peak-height of M-4006/M/U-LV and -HV were 58.4 and 101.0 mV for UDMA, and 97.1 and 122.0 mV for MMA. The peak-height of M-4006/M/U-LV was 1.7-times larger than that of M-4006/M/U-HV in UDMA, and the peak-height of M-4006/M/U-HV was 1.3-times larger than that of M-4006/M/U-HV in MMA. The ratios of residue (%) of EMA, MMA, and UDMA in M-5000/E/U, M-5001/E/U, M-4003/M/U, M-4005/M/U, and M-4006/M/U-HV measured by GPC were shown in Table 4. The ratios of residue of M-4005/M/U were lowest among those of M-5000/E/U, M-5001/E/U, M-4003/M/U, and M-4006/M/U-LV and -HV. The ratios of residue in UDMA were under 5% except M-4006/M/U-LV and M-4006/M/U-HV. The ratios of residue (%) of oligomer in M-5000/E/U, M-5001/E/U, M-4003/M/U, M-4005/M/U, and M-4006/M/U-LV and -HV were lower than those of monomers.

### DISCUSSION

The denture fracture has been introduced by many factors of base materials, production process, and clinical using, and the fracture has been caused by porosity, microcrack, residual monomer, water sorption, or poor adaptation of the denture. There are several attempts\(^3\)-\(^{16}\) to increase the mechanical properties of denture base resins. One approach is to devise a reinforcement of denture base polymer with fibers, chips, or rods\(^12\)-\(^{16}\). This technique is effective to improve the mechanical properties; however it is very difficult to incorporate accurately reinforcing materials in the desired position of the denture base resin. Another approach is to apply other polymer materials\(^3\)-\(^5\) such as polycarbonate or nylon, to disperse a rubbery material\(^10\), such as butadiene, or to add a cross-linking agent\(^18\)-\(^20\), such as dimethacrylate. However, the flexural modulus and the flexural strength of nylon denture base resin were lower than those of acrylic denture base resins. Furthermore the addition of butadiene to MMA increased the impact strength but decreased the modulus, and the addition of ethylene

![Fig. 5 SEM images (×300 magnification) of fracture surfaces of test specimens after flexural test. (a) PourResin; (b) Acron; (c) M-4006/M/U-HV; (d) 10/50-1/1; (e) 15/50-1/2](image)

**Table 4** The ratios of residue (%) calculated from the height under the UDMA oligomer and EMA or MMA monomer peaks measured by GPC before and after polymerization

<table>
<thead>
<tr>
<th></th>
<th>UDMA</th>
<th>EMA</th>
<th>MMA</th>
</tr>
</thead>
<tbody>
<tr>
<td>M-5000/E/U</td>
<td>4.4</td>
<td>7.2</td>
<td>-</td>
</tr>
<tr>
<td>M-5001/E/U</td>
<td>1.1</td>
<td>6.4</td>
<td>-</td>
</tr>
<tr>
<td>M-4003/M/U</td>
<td>4.0</td>
<td>-</td>
<td>19.2</td>
</tr>
<tr>
<td>M-4005/M/U</td>
<td>0.7</td>
<td>-</td>
<td>4.3</td>
</tr>
<tr>
<td>M-4006/M/U-LV</td>
<td>7.3</td>
<td>-</td>
<td>14.6</td>
</tr>
<tr>
<td>M-4006/M/U-HV</td>
<td>13.6</td>
<td>-</td>
<td>16.2</td>
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</table>
glycol dimethacrylate to MMA could not induce the increase in impact strength and fracture toughness. In this investigation, we produced UDMA polymers in which PEMA or PMMA is dispersing. The result shows that flexural strength, flexural modulus, and deflection of M-4005/M/U were significantly same with those of PR and AC ($p>0.01$). Moreover, M-4005/M/U did not break at the test speed of this investigation, and was more resistant to fracture.

Practically acrylic dentures are produced from powder and liquid using the conventional polymer/monomer dough molding process. A cured polymer shows macroscopically homogeneous surface; however microscopically contains two phase in inner-section (Figs. 5a and b). One is PMMA particle based on PMMA powder and the other one is UDMA matrix polymerized from MMA monomer. Some problems should be caused by being left the shape of PMMA powder. Different molecular weight distribution of PMMA powder and PMMA matrix polymerized from MMA monomer exists in both inner-sections, even if the molecular weights of both PMMA are the same, morphology is different.$^{16,22}$ As a result, denture surfaces are roughed up suiting to PMMA powder shape by the denture brush or masticatory movement, and then more adherences of denture plaque are produced.$^{23,24}$

There is another problem caused by polymer/monomer dough molding process. The acrylic denture has the macro-interface in the inner-section. There are several studies$^{25-27}$ describing fatigue fractures of acrylic dentures. Vallittu$^{26}$ reported that the fracture propagation is designated as stable when it causes total energy to increase and it occurs under increasing loads from the SEM examination of fracture surfaces. Fujii$^{27}$ mentioned that the fatigue failure was induced by the development of cracks in the specimen surfaces and/or a flaw within the specimens. The existences of cracks, flaws, or interfaces introduce the stress concentration onto the surface or inner-section of denture bases and the denture fracture is propagated. Therefore, many attempts$^{27,28}$ have been performed for long periods to dissolve the morphology and microstructural behavior, surface defects, and fracture initiation. However, the mechanical properties of these materials are not ideal at present. The light-curing UDMA denture base polymers (Triad and Eclipse) contain the methacrylate polymers$^{29}$, too. Light-cured polymers should be produced the interface between the UDMA matrix and the methacrylate polymer particles$^{30}$. The blend polymers used in this investigation have no-macro-interface and excellent splinterless property (Figs. 1–4). It can be guessed from fractured surfaces of 20/50-1/1 and 15/50-1/2 covered with continuous wrinkle that the flexural stress disperses to whole matrix (Figs. 5c and d).

For first step, PEMA (M-5000 and M-5001) and PMMA (M-4003, M-4005, and M-4006) powders were fully dissolved in ethyl methacrylate (EMA) and methyl methacrylate (MMA) to make PEMA-UDMA blend polymers (M-5000/E/U and M-5001/E/U) or PMMA-UDMA blend polymers (M-4003/M/U, M-4005/M/U, and M-4006/M/U-LV and -HV). As PEMA or PMMA powders formed by the molecular entanglement and secondary bonds such as van der Waals forces are mixed with EMA or MMA, EMA or MMA permeates into PEMA or PMMA powder, and then swells/dissolves in EMA or MMA with the time. For second step, PEMA or PMMA disperses homogeneously in EMA or MMA, and those free-PEMAs or -PMMAs without intermolecular attractions and UDMA oligomer are mixed, and finally the free-PEMAs or -PMMAs would disperse in the UDMA. Furthermore investigation is necessary whether those free-PEMAs or -PMMAs and UDMA polymer make semi-interpenetrating polymer network (semi-IPN) or not, and whether semi-IPN would help the orientation of the polymer. M-4006 which is smaller than M-4003 and M-4005 in molecular weight, formed two phases (M-4006/M/U-LV and -HV). The GPC peaks of UDMA and MMA in M-4006/M/U-LV were 1.7- and 1.3-times larger than those in M-4006/M/U-HV. M-4006/M/U-HV is PMMA-rich. Low miscibility of M-4006/M/U-LV and -HV would be explained that PMMA content is larger; intermolecular chains are shorter, flexibility of molecules is larger than the other polymers (M-4003/M/U and M-4005/M/U).

Flexural strength and flexural modulus of M-4005/M/U are larger than those of M-4003/M/U, and M-4006/M/U-LV and -HV ($p<0.01$). The purpose of the addition of PMMA is to adjust the viscosity of the UDMA mixture before curing. As the volume of PMMA powder is increasing, the viscosity of PMMA-MMA mixture is increasing. As a result, the viscosity of PMMA-MMA-UDMA mixture is increased. The viscosity is important for this trial blend polymer, because this mixture is made in manufacturer’s factory and used without a mixing process in dental laboratory. There is no statistical difference in flexural strength and flexural modulus of four polymers (10/50-1/1, 15/50-1/1, 20/50-1/1, and 25/50-1/1) ($p>0.01$) (Fig. 3). Deflection of those polymers showed the increasing tendency with the PMMA increasing. The orientation of PMMAs dissolved in MMA would help the drawing of the polymer. In this investigation, we used the maximum mixing ratio of PMMA/MMA=25/50. As well known in dental technique, further volume of PMMA does not uniformly dissolve into MMA in room environment. The result of Fig. 4 indicates that the mixing ratio of UDMA polymer for a base matrix and PMMA for a dispersoid is important for flexural strength and flexural modulus. However, the detailed mechanism is not clear from this investigation.

The mixture based on PMMA of M-4006 was separated to an upper phase and a lower phase (M-4006/M/U-LV and -HV) for few days, and flexural strength of M-4006/M/U-LV and -HV are peculiarly small compared with the other compositions (Fig. 2). The fractured surface of M-4006/M/U-HV is flat (Fig. 5c) without sign of drawing. The residue of UDMA and
MMA in M-4006/M/U-LV and -HV would affect to those results. Excessive additive-rate of PMMA (M-4006) may not make homogeneous distributions of cross-linking by UDMA; however the reason in detail cannot make clear from this investigation.

Within limitation of this investigation, a PMMA-UDMA blend polymer with the mixing ratios of PMMA/MMA=25/50 indicated the excellent splinterless property; no-fracture at this testing speed, and flexural strength and flexural modulus of the material showed no significant difference compared with those of commercially available acrylic resins (PR and AC) \((p>0.01)\). Results indicate that PMMA-UDMA blend polymers showed low ratios of residues and then are available for a denture base resin without the interface of PMMA powder in the polymer matrix. Furthermore investigation using faster test speed, such as impact test, should be necessary.

ACKNOWLEDGMENTS

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REFERENCE