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Influence of composition and powder/liquid ratio on setting characteristics and mechanical properties of autopolymerized hard direct denture reline resins based on methyl methacrylate and ethylene glycol dimethacrylate

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We evaluated the influence of composition and powder/liquid (P/L) ratio on the setting characteristics and mechanical properties of autopolymerized hard direct denture reline resins composed of methyl methacrylate (MMA, monomethacrylate) and ethylene glycol dimethacrylate [EGDMA, dimethacrylate (cross-linking agent)], with poly (ethyl methacrylate) used as the powder, and a mixture of MMA and EGDMA containing p-tolyldiethanolamine as the monomer. Setting times were determined using an oscillating rheometer and mechanical properties were based on ISO specifications. Setting time increased exponentially with an increase in the ratio of EGDMA to MMA and decrease in P/L ratio. Materials with a liquid component of approximately 75–85 wt% EGDMA and a higher P/L ratio showed higher ultimate flexural strength and flexural modulus. Our results suggest that setting characteristics are more influenced by the ratio of monomethacrylate and cross-linking agent, whereas mechanical properties are more influenced by P/L ratio.

Keywords: Reline resin, Methyl methacrylate, Ethylene glycol dimethacrylate, Setting characteristics, Mechanical properties

INTRODUCTION

Reline of ill-fitting complete and removable partial dentures enhances their stability and retention by improving the adaptation between the denture intaglio surface and denture-bearing mucosa[1)]. In clinical situations, that reline procedure is usually performed using a direct (chair-side reline system) rather than indirect (laboratory-processed reline system) method, because the former is more convenient, inexpensive, and less time consuming[2,3)]. Although many kinds of autopolymerized hard direct denture reline resins are available for use with the direct method, they have some disadvantages such as unpleasant taste and odor, irritation to the oral mucosa by the monomer, and generation of heat during polymerization[1-6)]. Furthermore, the stiffness of autopolymerized hard direct denture reline resins is lower than that of heat-polymerized denture base resins[7-9)]. The mechanical properties of reline resins influence the longevity of the relined denture as well as the denture base resins.

Autopolymerized hard direct denture reline resins for chair-side use are generally supplied in powder and liquid forms. The powder mainly consists of poly (ethyl methacrylate), while poly(methyl methacrylate) and poly(methyl methacrylate/ethyl methacrylate) are also available[2)]. A peroxide initiator and pigment are also included. Commercially available liquids are classified into 3 groups; a monofunctional methacrylate (monomethacrylate) monomer, monofunctional methacrylate monomer and plasticizer, and monofunctional methacrylate monomer and cross-linking agents[2)]. A chemically activated accelerator such as tertiary amine is also added to the liquid[6)]. The differences in composition have great influence on mechanical properties and setting behavior, which are important factors in clinical situations.

Several studies have been conducted to seek improvement in the mechanical properties of autopolymerized hard direct denture reline resins. In our previous study, we evaluated the effects of addition of fluorinated monomer on the properties of reline resins based on various monomers[10)]. It has been found that the effects of an added fluorinated monomer on stiffness vary among the base monomers used, with changes in the mechanical properties of reline resins containing the fluorinated monomer over time smaller as compared to materials containing no fluorinated monomer. Thus, a reline resin based on a specific monomer may be stiffer, while that based on another may be more viscous with addition of a fluorinated monomer. In addition, the effects of cross-linking agents on the properties of autopolymerized hard direct denture reline resins have also been evaluated. Cross-linking agents have been added to liquids in denture base resins to improve the mechanical properties such as flexural strength, and resistance to temperature and solvent attack[11,12)], with ethylene glycol dimethacrylate (EGDMA) widely used[11, 13)]. It has also been shown that the solubility of the liquid components of autopolymerized hard direct denture reline resins drops with an increase in cross-linking agents over the whole range of concentrations in the liquids, though the influence on water absorption varies among types of cross-linking agents[11)], as some decrease and others increase water absorption with increasing concentration. Water absorbed by denture

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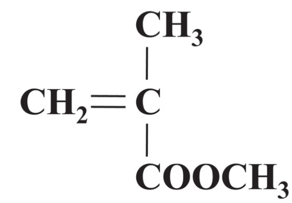
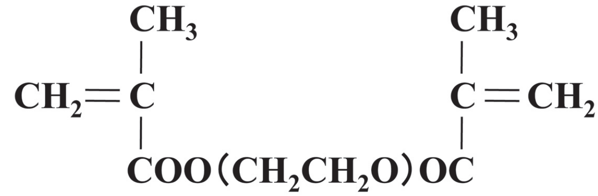
reline resins leads to deterioration such as a decrease in mechanical properties and distortion due to loosening or plasticization of the resin structure[11,14]. The solubility of the unreacted monomer, which acts as a plasticizer[11,15], also decreases the mechanical properties and irritates oral mucosa[16,17].

Properties such as setting behavior and mechanical properties are important for evaluation of autopolymerized hard direct denture reline resins, because these influence manipulation when used in a direct method and the service life of dentures, respectively. Many studies of such properties of commercial reline resins have been conducted[4,5,7,8,18,19]. However, scant information is available concerning the relationship between composition and these properties of autopolymerized hard direct denture reline resins.

In the present study, we evaluated the influence of the ratio of monomethacrylate and dimethacrylate (cross-linking agent) and powder/liquid (P/L) ratio on setting characteristics and the mechanical properties of flexural strength and flexural modulus of autopolymerized hard direct denture reline resins based on methyl methacrylate (MMA: monomethacrylate) and ethylene glycol dimethacrylate (EGDMA: dimethacrylate, a cross-linking agent). We hypothesized that setting time would decrease with an increase in P/L ratio and a decrease in the ratio of the cross-linking agent. It was also expected that the flexural strength and flexural modulus would increase with increases in the P/L ratio and ratio of the cross-linking agents.



methyl methacrylate (MMA)



ethylene glycol dimethacrylate (EGDMA)

Fig. 1 Chemical formulas of monomers used in this study.

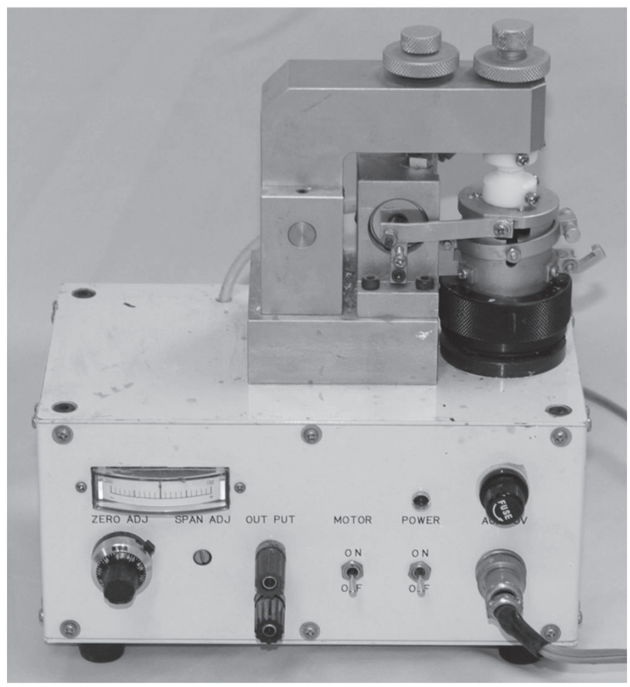


Fig. 2 Oscillating rheometer.

MATERIALS AND METHODS

Poly(ethyl methacrylate) (PEMA) polymer powders containing a trace of benzoyl peroxide were used during preparation of all specimens. The weight average molecular weight (M_w) was 50.0×10^4. Two monomers, methyl methacrylate (MMA) as a monomethacrylate and ethylene glycol dimethacrylate (EGDMA) as a dimethacrylate (cross-linking agent), were utilized in this study (Fig. 1). Four liquids were made by mixing these monomers; 37.5%MMA/62.5%EGDMA, 25.0%MMA/75.0%EGDMA, 12.5%MMA/87.5%EGDMA and 0%MMA/100.0%EGDMA (by weight). To produce autopolymerized materials, 1.0wt% p-tolyldiethanolamine, a tertiary amine, was added to the liquids as a chemical activator. The 4 powder/liquid (P/L) ratios utilized were 1.0, 1.2, 1.4, and 1.6, by weight.

Setting behavior

The method used for measuring the setting time of the autopolymerized material has been previously reported[10]. The apparatus was an oscillating rheometer (Seiki Co., Tokyo, Japan) (Fig. 2), which applies torsional force to the test material and measures the change in amplitude of the lower platen by viscosity build-up of the material over time. The upper and lower platens were fixed to the arm and the 3 sheet springs which were connected to the drive shaft, respectively. The diameter of the platens was 10 mm. The core of the transducer was attached to the side of the lower platen and the output of the transducer was fed to a hybrid recorder (AL3000, Chino Corp., Tokyo, Japan). Torsional deformation was applied to the material through the sheet springs by the motor at a speed of 10 rpm. The maximum oscillating movement of the lower platen when no material was set between the 2 platens was

1.45°. The amplitude of the rheometer trace decreased as the material became set.

After mixing the powder and liquid for 15 s at 23±2°C, the paste was placed on the lower platen of the rheometer and the upper platen was lowered into position 30 s after the start of mixing. The relative change in viscosity over time for the various combinations was recorded at 37°C. Five tests were conducted for each material. The so-called setting time was defined as the time required for a 75% reduction in width of the rheometer trace[10] (Fig. 3).

Mechanical properties

The ultimate flexural strength and flexural modulus were determined according to ISO specification 20795-1 (2008)[20]. Five specimens for each material were prepared in the form of rectangular blocks (64.0×10.0±0.2×3.3±0.2 mm) using a metal mold. The specimens were stored in distilled water at 37±1˚C for 50±2 h before measurement. Three-point bending tests were conducted using a universal testing machine (Model 5565, Instron Corp., Norwood, MA.) with a constant crosshead speed of 5 mm/min (Fig. 4). The distance between the centers of the supports was 50 mm.

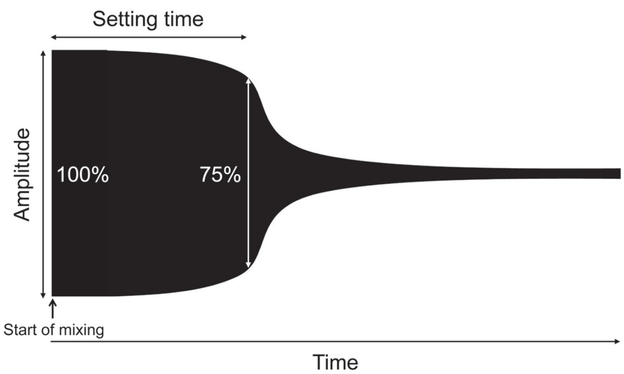


Fig. 3 Typical rheometer trace of an autopolymerized hard direct denture reline resin at 37˚C.

Ultimate flexural strength (σ) and flexural modulus (E) were calculated using the following equations[20];

$$\sigma = 3Fl/2bh^2$$
$$E = F_1 l^3/4bh^3 d$$

where F is the maximum load exerted on the specimen, l the distance between the supports, b the width of the specimen, h the height of the specimen, F_1 the load at a point in the straight-line portion (with the maximum slope) of the load/displacement curve, and d the deflection at load F_1.

Statistical analyses

Comparisons of setting time, ultimate flexural strength, and flexural modulus were subjected to 2-way analysis of variance (ANOVA), and the contribution ratios (ρ) of the ratio of monomethacrylate and dimethacrylate (cross-linking agent) and P/L ratio, and their interactions were determined. Regression analyses were also used to determine the correlation between 3 values (setting time, ultimate flexural strength, flexural modulus) and 2 factors (ratio of monomethacrylate and dimethacrylate, P/L ratio). For all statistical analyses, we used SPSS Statistics 17.0.

RESULTS

Results from 2-way ANOVA indicated significant effects for all factors on the setting time of autopolymerized hard direct denture reline resins based on methyl methacrylate (MMA) and ethylene glycol dimethacrylate (EGDMA) (p<0.0005) (Table 1). The ratio of

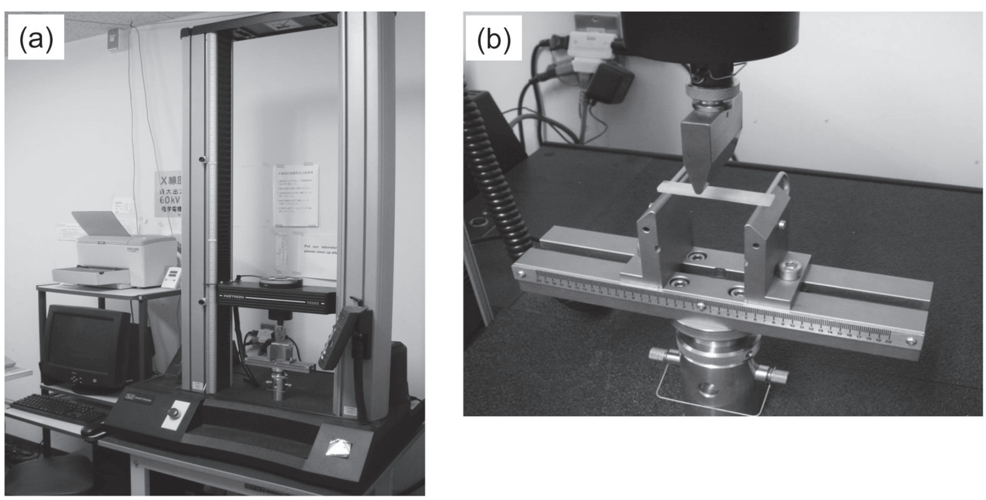


Fig. 4 Apparatus for measuring ultimate flexural strength and flexural modulus. (a) Universal testing machine, (b) Three-point flexure fixture.

monomethacrylate (MMA) and dimethacrylate (EGDMA) (cross-linking agent) (ρ=63.0%) had more influence on setting time than P/L ratio (ρ=28.9%). Figure 5 shows the relationship of setting time with the ratio of EGDMA to MMA content and P/L ratio. Setting time increased exponentially with increases in the ratio of EGDMA to MMA content and decreased exponentially with increases in the P/L ratio. A positive linear relationship was found between the logarithm of the setting time and EGDMA content (p<0.0005), while a negative linear relationship was noted between the logarithm of the setting time and P/L ratio (p<0.0005).

Our 2-way ANOVA findings also indicated significant effects for all factors on ultimate flexural strength of the autopolymerized hard direct denture reline resins based on MMA and EGDMA (p<0.0005) (Table 2). P/L ratio (ρ=47.9%) had a greater influence on ultimate flexural strength than ratio of monomethacrylate (MMA) and dimethacrylate (EGDMA) (ρ=12.3%). Figure 6 shows the relationship

Table 1 Two-way ANOVA for the setting time of the 16 tested materials

Source	df	Sum of squares	Mean square	F	Significance of F	Contribution ratio ρ (%)
Ratio of monomethacrylate and dimethacrylate (cross-linking agent)	3	40146.3	13382.1	925.4	0.000	63.0
P/L ratio	3	18606.6	6202.2	429.0	0.000	28.9
Ratio of monomethacrylate and dimethacrylate (cross-linking agent) × P/L ratio	9	4475.7	497.3	34.4	0.000	6.8
Residual	64	925.2	14.5	—	—	1.3
Total	79	64153.8	—	—	—	100.0

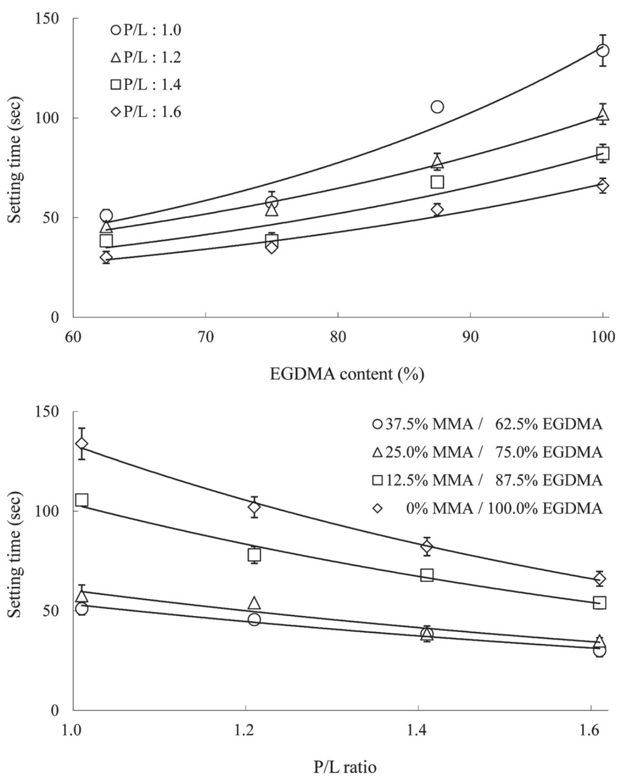


Fig. 5 Relationship between setting time of autopolymerized hard direct denture reline resins based on MMA and EGDMA and both the ratio of EGDMA to MMA content and P/L ratio.

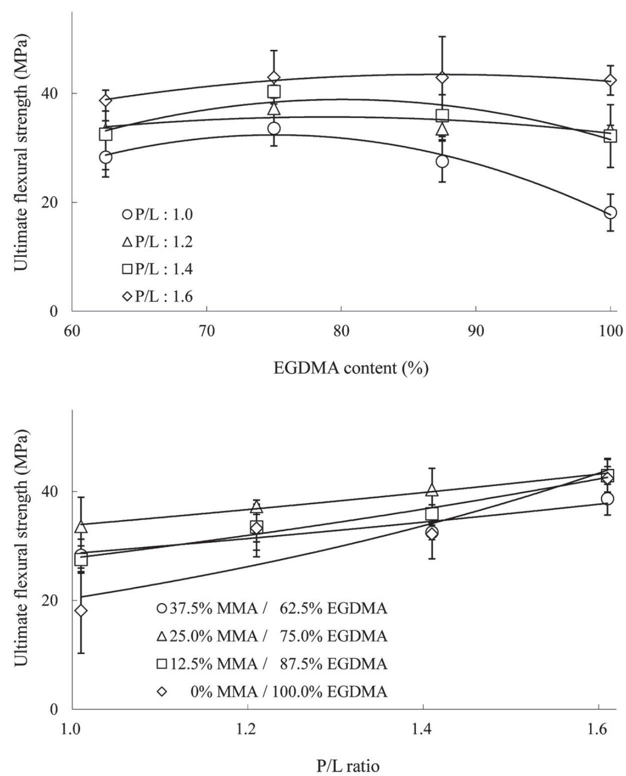


Fig. 6 Relationship between ultimate flexural strength of autopolymerized hard direct denture reline resins based on MMA and EGDMA and both the ratio of EGDMA to MMA content and P/L ratio.

Table 2 Two-way ANOVA for the ultimate flexural strength of the 16 tested materials

Source	df	Sum of squares	Mean square	F	Significance of F	Contribution ratio ρ (%)
Ratio of monomethacrylate and dimethacrylate (cross-linking agent)	3	396.0	132.0	8.1	0.000	12.3
P/L ratio	3	2444.6	814.9	50.1	0.000	47.9
Ratio of monomethacrylate and dimethacrylate (cross-linking agent) × P/L ratio	9	669.2	74.4	4.6	0.000	16.9
Residual	64	1041.8	16.3	—	—	22.9
Total	79	4551.6	—	—	—	100.0

Table 3 Two-way ANOVA for the flexural modulus of the 16 tested materials

Source	df	Sum of squares	Mean square	F	Significance of F	Contribution ratio ρ (%)
Ratio of monomethacrylate and dimethacrylate (cross-linking agent)	3	607933.1	202644.4	15.1	0.000	7.6
P/L ratio	3	2245642.5	748547.5	55.7	0.000	52.6
Ratio of monomethacrylate and dimethacrylate (cross-linking agent) × P/L ratio	9	900193.0	100021.5	7.4	0.000	11.5
Residual	64	860630.3	13447.3	—	—	28.3
Total	79	4614398.9	—	—	—	100.0

of ultimate flexural strength with the ratio of EGDMA to MMA content and P/L ratio. The values for ultimate flexural strength increased gradually with an increasing ratio of EGDMA and then decreased from approximately 75–85 wt% EGDMA. The ultimate flexural strength values increased with increases in P/L ratio, and a positive linear relationship was found between those values and P/L ratio (p<0.0005–p<0.001).

Our 2-way ANOVA results also indicated significant effects for all factors on flexural modulus of the reline resins (p<0.0005) (Table 3), with nearly the same tendency found for flexural modulus as seen with ultimate flexural strength. P/L ratio (ρ=52.6%) had a greater influence on the flexural modulus than the ratio of monomethacrylate (MMA) and dimethacrylate (EGDMA) (ρ=7.6%). The relationship of flexural modulus with the ratio of EGDMA to MMA content and P/L ratio is illustrated in Fig. 7. The values for flexural modulus increased gradually with increases in the ratio of EGDMA and then decreased from approximately 75–85 wt% EGDMA. These flexural modulus values increased with increases in the P/L ratio and a positive linear relationship was found between these values and P/L ratio (p<0.0005 – p<0.01).

DISCUSSION

The present findings confirmed our hypothesis that the setting time of autopolymerized hard direct denture reline resins would decrease with an increase in P/L ratio and decrease in the ratio of the cross-linking agent, while flexural strength and flexural modulus would increase with increased P/L ratio. However, flexural strength and flexural modulus did not increase with an increase in the ratio of the cross-linking agent, and materials with approximately 75–85 wt% ethylene glycol dimethacrylate (EGDMA, cross-linking agent) exhibited higher flexural strength and flexural modulus.

Although many types of monomethacrylate are commercially available for autopolymerized hard direct denture reline resins, methyl methacrylate (MMA) was chosen for the present study because it is widely used as liquid component in many denture reline resins. Some commercial products contain 100 wt% monomethacrylate, while cross-linking agents are added to improve the mechanical properties of denture base resins. As for heat-polymerized denture base resins, it was reported that the percentage of cross-linking agents in the liquid is not usually more than 15%[11, 12]. However, some autopolymerized hard direct denture reline resins contain more than 50% cross-

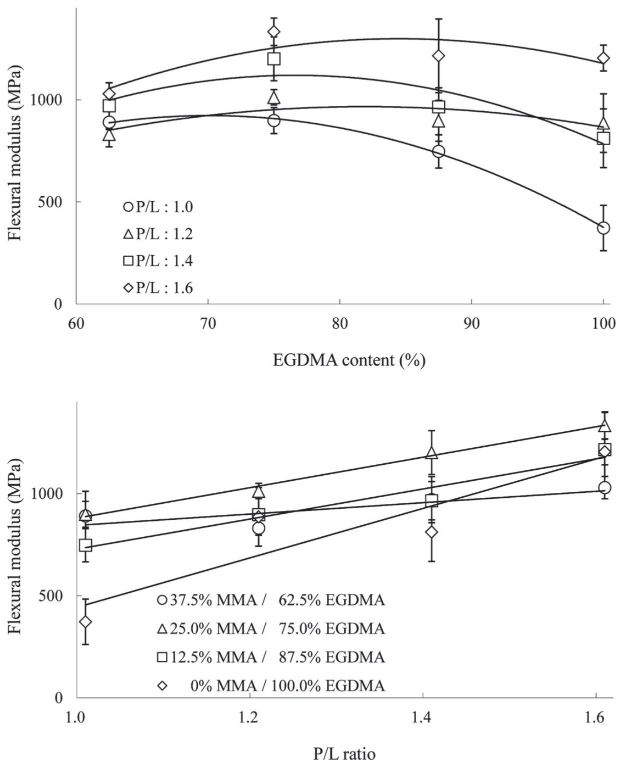


Fig. 7 Relationship between flexural modulus of autopolymerized hard direct denture reline resins based on MMA and EGDMA and both the ratio of EGDMA to MMA content and P/L ratio.

linking agents, such as 1,6-hexandiol dimethacrylate (1,6-HDMA) and trimethylolpropane trimethacrylate (TMPT), in the liquid component[2]. This difference may be due to many factors such as polymerization method, powder/liquid (P/L) ratio, and working time, though further studies are necessary.

In our previous study, we evaluated the effects of 6 different cross-linking agents on water absorption and solubility characteristics of autopolymerized hard direct denture reline resins based on poly (ethyl methacrylate) (PEMA) and MMA[11]. Those results showed that all cross-linking agents had a tendency for decreased solubility with increasing concentration. However, there were significant differences among the cross-linking agents tested. Triethylene glycol dimethacrylate (TEGDMA) and diethylene glycol dimethacrylate (DEGDMA) showed increased water absorption with increasing concentration, whereas 1,4-butanediol dimethacrylate (1,4-BDMA), 1,6-HDMA, TMPT, and EGDMA showed that to be decreased with increasing concentration. Moreover, EGDMA is widely used as a cross-linking agent for denture base resins. Thus, EGDMA was examied in the present study to evaluate the fundamental properties of cross-linking agents.

Setting behavior, flexural strength and flexural modulus were evaluated in the present study. The former is an important factor for evaluation of autopolymerized dental materials such as denture liners and impression materials. Initial properties of autopolymerized hard direct denture reline resins were previously determined using consistency and temperature measurements[5,21], which provided valuable information regarding setting characteristics after mixing powder and liquid. However, those cannot be used to evaluate setting time in a detailed manner, because the increase in hardness of the materials over time cannot be measured. Instruments known to be suitable for measurement of setting time include a displacement rheometer[22] and oscillating rheometer[23]. The former measures elastic recovery during setting and is utilized for determination of setting time of temporary soft denture liners[24] as part of the ISO specifications[25]. Although the setting time of autopolymerized hard direct denture reline resins can be measured with a displacement rheometer, it is very difficult to remove the materials from the plate of the apparatus after measurement, because the material becomes hard at the perforated steel plate. Thus, a displacement rheometer is suitable for measurement of the setting time of soft materials such as soft denture liners and impression materials[22,24,25]. On the other hand, an oscillating rheometer is suitable for hard materials such as autopolymerized hard direct denture reline resins as well as soft materials, because it is easy to remove the set materials from the Teflon platen. Therefore, an oscillating rheometer was employed in the present study. This type of rheometer, which gives torsional force to the material between the platens, measures change in amplitude of the lower platen by elasticity build-up of the tested material over time. The setting time was determined as the time required for a 75% reduction in width of the amplitude of the rheometer, as previously reported[23]. Autopolymerized hard direct denture reline resins, which reach that 75% reduction, are considered to become elastic enough to be removed from the mouth.

We used liquids containing more than 62.5 wt% EGDMA in the present study, as those containing less than 62.5 wt% EGDMA have been found to have a short setting time, thus no samples could be prepared in the preliminary experiment. Autopolymerized reline resins with less than 62.5 wt% EGDMA are difficult to manipulate in clinical situations before setting when the MMA monomer and PEMA powder used in the present study are applied. The higher P/L ratio and lower ratio of EGDMA to MMA content produced an exponentially shorter setting time. That higher P/L ratio may be associated with a greater entanglement of polymers, resulting in a shorter setting time. Although the P/L ratio was shown to have significant influence (contribution ratios (ρ)=28.9%), setting time was more greatly influenced by the ratio of EGDMA and MMA (ρ=63.0%). Polymerization of hard direct denture reline resins occurs following penetration and diffusion of monomers into the polymers. Thus, increases in the ratio of EGDMA, with a higher molecular weight (M_w:

198), to MMA, with a lower molecular weight (M_w: 100), would inhibit the penetration and diffusion of monomers into the polymers, leading to a longer setting time. The penetration and diffusion of monomers was found to have a greater influence on the setting speed of autopolymerized hard direct denture reline resins as compared to entanglement of polymers.

On the other hand, the influence of each factor on mechanical properties after setting was opposite of that on setting times. Ultimate flexural strength and flexural modulus of the reline resins were more greatly influenced by P/L ratio (ρ=47.9% and 52.6%, respectively) than ratio of EGDMA and MMA (ρ=12.3% and 7.6%, respectively). Higher P/L ratio led to higher values for ultimate flexural strength and flexural modulus, probably because that higher P/L ratio produced a closer three-dimensional network structure and decreased the quantities of the unreacted monomers. Although the influence of the ratio of EGDMA and MMA was not large as compared with the P/L ratio, a significant effect on these values was found. Also, stiffness, represented by ultimate flexural strength and flexural modulus, increased as the ratio of the cross-linking agent EGDMA increased up to 75–85 wt%, and then decreased with further addition. A higher concentration of cross-linking agent resulted in formation of a closer three-dimensional network structure[26], which might have led to stiffer reline resins. This type of reline resin will not be easily swollen by water absorption. However, reline resins with too high a concentration of cross-linking agent tended to exhibit lower ultimate flexural strength and flexural modulus, probably due to the presence of unreacted monomers including the cross-linking agent EGDMA in the set materials. These residual monomers will degrade mechanical properties, dimensional stability, and surface condition. Furthermore, the residual monomers will gradually leach out from the reline resins over time and cause mucosal damage, as shown by burning sensation and irritation[17].

From the standpoint of mechanical properties evaluated by 3-point bending test, the liquid component of 25.0% MMA/75.0% EGDMA and a P/L ratio of 1.6 is more suitable for an autopolymerized hard direct denture reline resin based on MMA and EGDMA when PEMA polymer powders used in this study are applied. The durability of the above reline resin over time is expected to be higher than that of other reline resins, because of the closed polymer network structure and lower amount of residual monomers. The present study focused on setting behavior and mechanical properties. For more rigorous and clinical evaluations, further studies such as adhesive bond strength to the denture base resins[5,27], color stability[5], and the influence of thermal cycling[19], and denture cleansers[28] on properties will be necessary.

The present results suggest that setting characteristics are more affected by the ratio of monomethacrylate and dimethacrylate (cross-linking agent), whereas mechanical properties are more affected by P/L ratio. Ideally, autopolymerized hard direct denture reline resins should have the same mechanical properties as heat-polymerized denture base resins[8,29], though those have not yet to be developed. Although further information such as the effects of other monomers including cross-linking agents, and type and molecular weight of polymer powders is necessary, the present findings should contribute to development of ideal autopolymerized hard direct denture reline resins.

CONCLUSION

Within the limitations of the present study, the following conclusions can be made:

1. The setting time of autopolymerized hard direct denture reline resins based on methyl methacrylate (MMA: monomethacrylate) and ethylene glycol dimethacrylate [EGDMA: dimethacrylate (cross-linking agent)] increased exponentially with increases in the ratio of EGDMA to MMA content and decreases in powder/liquid (P/L) ratio. Linear relationships were found between the logarithm of setting time and both EGDMA content and P/L ratio.
2. Ultimate flexural strength and flexural modulus of the reline resins increased with increases in P/L ratio, and positive linear relationships were found between those values and P/L ratio. Reline resins with a liquid component of approximately 75–85 wt% EGDMA tended to show higher ultimate flexural strength and flexural modulus.
3. The setting time of reline resins was found to be more greatly influenced by the ratio of EGDMA to MMA content than by P/L ratio. On the other hand, the mechanical properties after setting were greatly influenced by P/L ratio when compared to the ratio of EGDMA to MMA content.

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