

Effect of Various Pressure Cooker Curing conditions on Flexural Strength of Denture Hard Relining Materials

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Abstract

The aim of this study was to evaluate the effect of various curing conditions in a pressure cooker on the flexural strength of hard chairside relining resins. One hundred and forty hard chairside relining resin (Unifast™ Trad and Tokuyama® rebase II Fast) specimens were prepared per ISO 20795-1 (2013) and divided into 14 groups. Each material was cured following the manufacturer's instructions as a control group and six experimental groups: cured under 1,500 mmHg air or nitrogen compressed pressure cooker at 55°C for 10, 15 or 20 minutes. The specimens were stored in water at 37±1 °C for 50±2 hours before testing. The three-point bending test was performed using a universal testing machine at a cross-head speed of 5 mm/min. One-way ANOVA and post hoc Tukey's analysis at a 95% confidence level were used to statistically compare the mean flexural strengths of the groups. For each material, the flexural strength of the air and nitrogen compressed groups were significantly higher compared with the control group ($P<0.05$). The flexural strength of the 10-min nitrogen group was significantly higher compared with the 10-min air group ($P<0.05$). There was no significant difference in flexural strength between the 15-min nitrogen and 15-min air groups ($P>0.05$). However, the flexural strength of the 20-min nitrogen group was significantly higher compared with the 20-min air group ($P<0.05$). The flexural strength in the 10, 15, and 20 min curing time groups of each relining material with the same curing environment in the pressure cooker were not significantly different ($P>0.05$). Under the same curing conditions, Unifast™ Trad had significantly higher flexural strength compared with Tokuyama® rebase II ($P<0.05$). Curing in the pressure cooker increased the flexural strength of the hard chairside relining resins. Moreover, using nitrogen gas pressure with satisfactory curing duration increased the flexural strength compared with using air pressure.

Keywords: Acrylic resin, Flexural strength, Hard relining resin, Nitrogen gas, Pressure cooker

Received date:

Revised date:

Accepted date:

Doi:

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Introduction

Following tooth extraction, the residual alveolar ridge undergoes bone remodeling that results in bone resorption. The residual alveolar ridge reduction occurs rapidly and continuously during the first six months to two years after tooth extraction.¹ Bone resorption leads to a poor fit of the removable denture base that is used to replace the extracted teeth, and also adversely affects the patient's speech and mastication. When the residual ridge is reduced, the denture base needs to be relined to restore its fit to the residual ridge, and to improve the support, retention and stability of the denture base.^{2,3} When using the direct relining technique, an auto polymerized hard relining resin is directly relined on the denture base in the mouth. This technique is inexpensive, easy to perform, not time consuming and can be done in a single visit.² In addition, auto polymerized hard relining resins have demonstrated adequate physical and mechanical properties. The indications for using an auto polymerized hard relining resin are a poorly adapted prosthesis, poor retention and stability at delivery and after use, and found that the denture loss their properties.⁴

Hard chairside relining resins are classified as type 2 (auto polymerized resin) class 1 (powder and liquid) denture base polymer. Their polymerization initiates with a chemical reaction and do not require persistence of temperatures above 65°C to complete polymerization.⁵ The initiation reaction is a redox reaction. The benzoyl peroxide initiator is activated by a reducing agent such as dimethyl-p-toluidine producing a reactive center (or free radical). In the present study, the flexural strength of hard chairside relining resins were investigated using two brands which are commercially available in Thailand, Unifast™ Trad (MMA based) and Tokuyama® rebase II Fast (non-MMA based).

Auto polymerized hard relining resins can be divided into two groups based on the composition of the main liquid constituents: MMA based and non-MMA based relining materials. The MMA based materials which are commercially available include Unifast™ Trad (GC Corp., Tokyo, Japan), Probase Cold® (Ivoclar, Liechtenstein)

and Palapress Vario® (Heraeus Kulzer, Wehrheim, Germany). MMA based relining materials have better adhesion to denture bases due to MMA monomer that dissolve and penetrate into the denture base forming the interpenetrating polymer networks (IPN) which bonds the two layers of materials.^{6,7} There is also higher flexural strength compared with non-MMA based relining materials.⁸ Despite these advantages, MMA-based hard relines can irritate the oral mucosa due to residual MMA or exothermic heat during polymerization.⁹ These problems have been resolved with the introduction of non-MMA based hard relining materials. The non-MMA based materials available on the market include Kooliner® (Coe Laboratories, Chicago, USA), Ufi gel hard® (Voco, Cuxhaven, Germany) and Tokuso Rebase II® (Tokuyama Dental Corp, Tsukuba, Japan). Non-MMA based materials contain high molecular weight methacrylate, such as β -methacryloyl oxyethyl propionate (MAOP) and 1,6-Hexanedial dimethacrylate (1,6-HDMA monomers). The molecular weights of the MAOP and 1,6-HDMA monomers are 186 g/mol and 254 g/mol, respectively. These monomers are almost twice the molecular weight of the MMA monomer (approximately 100 g/mol). The usage of these monomers in the relining resin improve the material by reducing tissue irritation and heat generation during manipulation.¹⁰ In addition, they contain cross-linking agents in their liquid constituents, which improve their transverse bending strength.¹¹

Many studies found that one of the disadvantages of auto polymerized acrylic resin is residual monomer or unreacted methyl methacrylate (MMA). Residual monomer can affect on their mechanical properties such as flexural strength¹², reduce the glass transition temperature¹³, increase the possibility of deformation of the material¹⁴, limit tensile strength, increase water absorption^{15,16} and also cause an allergic reaction¹⁷, irritation and inflammation of the oral tissues.⁴ Thus, various methods have been used to decrease the amount of residual monomer, such as immersing the relined denture in hot water (50-55°C)¹⁸, immersing in water for 24 hours after complete polymerization¹⁹, microwaving post-polymerized radiation²⁰,

and ultrasonic immersion in water²¹⁻²³ or ethanol solution.^{24,25} Furthermore, some mechanical properties of auto polymerized hard reline resins can be increased by applying pressure to the curing environment.²⁶ The use of a pressure cooker can reduce the dimensional change and altered occlusion that occurs during auto polymerized acrylic resin polymerization.²⁷ In addition, less porosity and higher flexural strength of auto polymerized hard reline resin was observed after curing in a pressurized environment.²⁸

Free radical addition polymerization is inhibited or retarded by higher oxygen concentrations.²⁹ Some studies also found that oxygen inhibits the polymerization of acrylic resin.³⁰ However, a comparative study of the effect of using nitrogen gas in a pressure cooker curing method on the flexural strength of auto polymerized hard reline materials has not yet been reported.

The aim of this study was to evaluate the effect of various curing conditions in a pressure cooker on the flexural strength of hard chairside reline resins. The first null hypothesis was that there is no significant difference in the flexural strength between the groups of hard chairside reline resins cured in a pressure cooker and the groups cured in room atmosphere. The second null hypothesis was that the flexural strength between the groups of hard chairside reline resins cured in a nitrogen pressure cooker and groups cured in an air pressure cooker are not significantly different. The third null hypothesis was that variation in the curing time in the pressure cooker does not significantly affect the flexural strength of hard chairside reline resins. The fourth null hypothesis was that the flexural strengths of the various hard chairside reline resins cured at the same condition are not significantly different.

Materials and methods

Seventy specimens were prepared from Unifast™ Trad in a stainless-steel mold (Fig. 1) with dimension of 64x10x3.3 mm (Fig. 2) following the manufacturer's instructions (Table 1) using seven different curing conditions. For the control groups, the specimens were cured at

room temperature (25±1°C) and pressure (760 mmHg). For the air groups, a pressure cooker (Fig. 3) (IMT; Pressurepotter 003, Inmotion technology limited, Thailand) was connected to an air pump. (Fig. 4(a)) The pressure cooker was filled with water below the specimen level and the temperature control panel was adjusted to 55°C. The stainless-steel mold containing the material was placed on a stand inside the pressure cooker and the lid was closed tightly, and compressed with 1,500 mmHg air (2 bar). The specimens were cured for 10, 15 or 20 minutes. For the nitrogen groups, the pressure cooker was connected to a nitrogen tank via a polyurethane tube. (Fig. 4 (b)) The tip of the polyurethane tube was placed under the water level. After adjusting the temperature to 55°C, the nitrogen valve was opened to let nitrogen gas flow into the pressure cooker. The pressure release valve of the pressure cooker was opened simultaneously to purge the air with nitrogen gas for five seconds. The pressure release valve was closed to rise the pressure to 1,500 mmHg (2 bar). The specimens were cured under nitrogen gas pressure for 10, 15 or 20 minutes. Another 70 specimens were prepared from Tokuyama® rebase II using the same procedures as described for Unifast™ Trad. The cured Tokuyama® rebase II specimens were soaked in a Hardener® water solution (40-60°C) for three minutes then rinsed and dried. The 140 specimens were distributed into 14 groups (n=10) based on their curing condition.

The specimens were polished with metallographic grinding paper (P500, TOA, Thailand), on a polishing machine (NANO2000, Pace Technologies, USA) by wet grinding on both sides to a 3.3 mm thickness. The specimens were stored in water at 37±1°C for 50±2 hours prior to flexural strength testing. The specimens were removed from water storage and immediately subjected to the flexural strength test, following ISO 20795-1:20135, using a 3-point loading universal testing machine (SHIMADZU; EZ-S, SHIMADZU, JAPAN) at a cross-head speed of 5 mm/min, a span of 50 mm, and 500 N load cell until the specimen broke. (Fig. 5) The flexural strength (MPa) was calculated using the following equation:

$$\sigma = \frac{3Fl}{2bh^2}$$

- σ = the load (N) at fracture
- l = the distance between supports (mm)
- b = mean specimen width (mm)
- h = mean specimen height (mm)

The normality of the flexural strength data of each group was determined by using the One-Sample Kolmogorov-Smirnov test and the variance was evaluated

using the Levene test. If the data had a normal distribution with equal variance, two-way analysis of variance (Types of reline materials, and pressure cooker curing conditions), with a 95% confidence level, was used to determine the significance. If the results did not conform to the assumptions of two-way ANOVA that the data had to be statistically independent and with an equal number of observations, one-way analysis of variance (post hoc Tukey with a 95% confidence level), was used to determine the significance.

Table 1 Chemical composition and manufacturer of the materials.

Material	Major ingredients	Mixing time	Working time	Powder-liquid ratio	Manufacturer
Unifast™ Trad	Powder: PMMA, MMA&EMA copolymer Liquid: MMA monomer, dimethyl-p-toluidine	10-15 sec	2 mins	1.0g / 0.5mL	GC Corporation, Tokyo, Japan
Tokuyama® Rebase II Fast	Powder: PEMA Liquid :1,9-NDMA, AAEMA Hardener®: Sodium bicarbonate, Sodium sulphite	5-10 sec	20 -60 sec	2.40g / 1.0mL	Tokuyama dental corporation, Tokyo, Japan

PMMA, Poly (methyl methacrylate); MMA&EMA copolymer, Methyl methacrylate & Ethyl methacrylate copolymer; PEMA, Poly (ethyl methacrylate); 1,9NDMA, 1,9nonanedioldimethacrylate; AAEMA, 2-(acetoacetoxy) ethyl methacrylate.



Figure 1 Stainless steel mold with loaded material

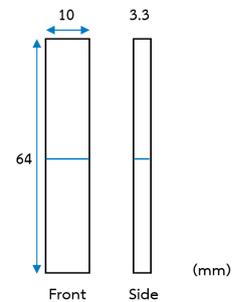


Figure 2 Illustration of the 64x10x3.3 mm specimens

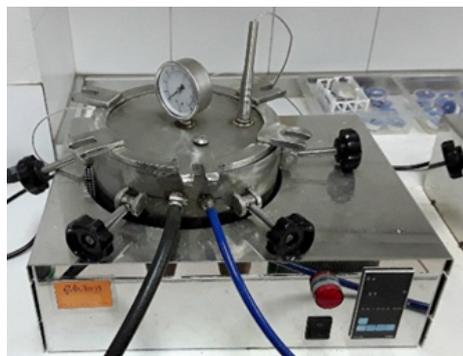


Figure 3 Pressure cooker with pressure release valve on the top surface of the lid. The temperature control panel is on the front side of the set up.

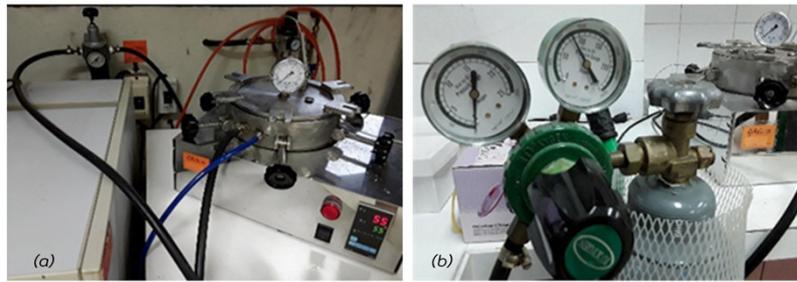


Figure 4 (a) pressure cooker connected with air pump, (b) pressure cooker connected with nitrogen gas.

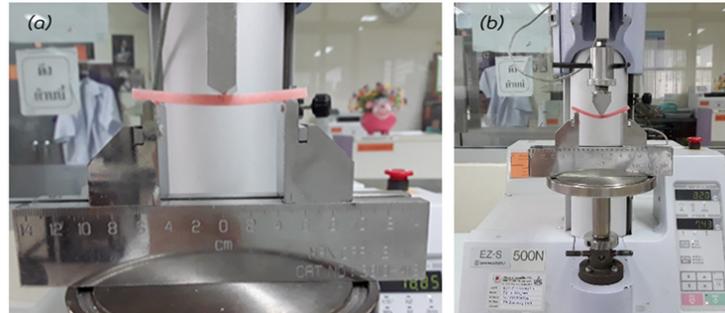


Figure 5 (a) lay the flat surface symmetrically on the supports of the flexural test rig, (b) flexural strength test, using 3-point loading universal testing machine.

Results

The data were analyzed using the Kolmogorov-Smirnov test to determine data distribution. The results indicated that the data were normally distributed in all groups ($P > 0.05$). The results did not conform to the assumptions of two-way ANOVA that the data had to be statistically independent and with an equal number

of observations (Table 2). The interaction effect between the two factors (types of reline materials and pressure cooker curing conditions) is defined as one in which the effect of one factor depends on the level of the other factor. Thus, the results were statistically analyzed by one-way ANOVA and Tukey HSD test.

Table 2 Two-way ANOVA analysis of the mean flexural strength.

Source	Type III Sum of Squares	df	Mean Square	F	P
Corrected Model	20704.682 ^a	3	6901.561	1065.669	.000
Intercept	473425.350	1	473425.350	73101.526	.000
product	20248.033	1	20248.033	3126.495	.000
atmosphere	406.014	1	406.014	62.693	.000
product * atmosphere	50.635	1	50.635	7.819	.006
Error	751.248	116	6.476		
Total	494881.280	120			
Corrected Total	21455.930	119			

The mean flexural strength and standard deviation of each group is presented in Table 3 and Figure 6. For each hard reline material, the flexural strength of the pressure

cooker cured groups was significantly higher compared with the control group ($P < 0.05$). For each hard reline material cured in the same environment in the pressure

cooker, the flexural strengths of the 10, 15, and 20 min curing time groups were not significantly different ($P>0.05$). Comparing the groups by curing time, the flexural strength of the 10-min nitrogen group was significantly higher than that of the 10-min air group ($P<0.05$). In contrast, no significant difference was found in flexural strength

between the 15-min nitrogen and 15-min air group ($P>0.05$). However, the flexural strength of the 20-min nitrogen group was significantly higher compared with the 20-min air group ($P<0.05$). In addition, at the same curing conditions, Unifast™ Trad demonstrated a significantly higher flexural strength compared with Tokuyama® rebase II ($P<0.05$).

Table 3 Mean flexural strength with standard deviation of the different groups.

Curing conditions				UT	TR+H
Pressure (mmHg)	Temperature (°C)	Curing environment	Curing time (min)		
760	25	air	2(UT),5.5(TR)	66.40 (5.47) ^{A, a}	45.98 (1.52) ^{A, b}
1,500	55	air	10	72.42 (2.13) ^{B, a}	48.35 (1.41) ^{B, b}
1,500	55	air	15	73.16 (3.94) ^{BC, a}	48.80 (1.85) ^{BC, b}
1,500	55	air	20	73.93 (4.23) ^{BC, a}	48.74 (1.81) ^{BC, b}
1,500	55	N ₂	10	77.92(2.72) ^{CD, a}	50.69 (1.28) ^{CD, b}
1,500	55	N ₂	15	77.71 (2.82) ^{CD, a}	50.89 (1.76) ^{CD, b}
1,500	55	N ₂	20	79.24 (4.27) ^{D, a}	51.45 (1.29) ^{D, b}

UT, Unifast™ Trad, TR+H, Tokuyama® Rebase II Fast with Hardener

Same uppercase letter indicates no significant difference between the groups in each column ($p>0.05$)

Same lowercase letter indicates no significant difference between the groups in each row ($p>0.05$)

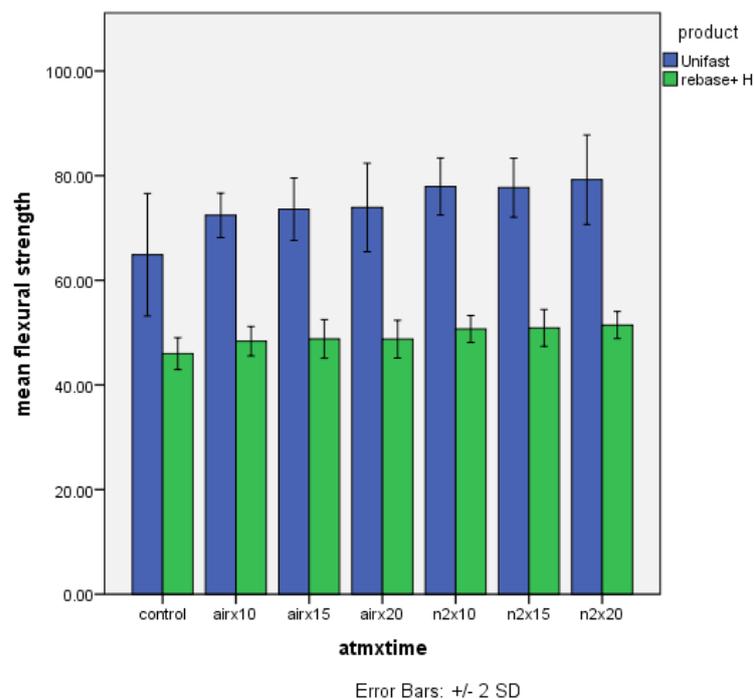


Figure 6 The mean flexural strength (bars) of Unifast™ Trad and Tokuyama® Rebase II Fast with Hardener at various conditions (MPa) and ± standard deviation (vertical lines) are given.

Table 4 P-values of One-way ANOVA analysis of UT and TR+H.

Materials	Group comparison	Sig.
Unifast™ Trad	Control-Air 10 min	0.004
	Control-Air 15 min	0.002
	Control-Air 20 min	0.002
	Control-Nitrogen 10 min	0.000
	Control- Nitrogen 15 min	0.000
	Control- Nitrogen 20 min	0.000
	Air 10 min- Air 15 min	1.000
	Air 10 min- Air 20 min	1.000
	Air 10 min- Nitrogen 10 min	0.037
	Air 10 min- Nitrogen 15 min	0.032
	Air 10 min- Nitrogen 20 min	0.023
	Air 15 min- Air 20 min	1.000
	Air 15 min- Nitrogen 10 min	0.074
	Air 15 min- Nitrogen 15 min	0.064
	Air 15 min- Nitrogen 20 min	0.048
	Air 20 min- Nitrogen 10 min	0.075
	Air 20 min- Nitrogen 15 min	0.065
	Air 20min- Nitrogen 20 min	0.048
	Nitrogen 10 min- Nitrogen 15 min	1.000
	Nitrogen 10 min- Nitrogen 20 min	1.000
Nitrogen 15 min- Nitrogen 20 min	1.000	
Tokuyama® rebase II Fast	Control-Air 10 min	0.021
	Control-Air 15 min	0.003
	Control-Air 20 min	0.004
	Control-Nitrogen 10 min	0.000
	Control- Nitrogen 15 min	0.000
	Control- Nitrogen 20 min	0.000
	Air 10 min- Air 15 min	0.995
	Air 10 min- Air 20 min	0.998
	Air 10 min- Nitrogen 10 min	0.023
	Air 10 min- Nitrogen 15 min	0.011
	Air 10 min- Nitrogen 20 min	0.001
	Air 15 min- Air 20 min	1.000
	Air 15 min- Nitrogen 10 min	0.120
	Air 15 min- Nitrogen 15 min	0.062
	Air 15 min- Nitrogen 20 min	0.007
	Air 20 min- Nitrogen 10 min	0.100
	Air 20 min- Nitrogen 15 min	0.051
	Air 20 min- Nitrogen 20 min	0.005
	Nitrogen 10 min- Nitrogen 15 min	1.000
	Nitrogen 10 min- Nitrogen 20 min	0.933
Nitrogen 15 min- Nitrogen 20 min	0.984	

Discussion

This study was designed to determine how various pressure cooker curing conditions affect the flexural strength of denture hard relining materials. There are two main variables which directly relates to the flexural strength, curing conditions and reline materials. Curing conditions refers to three factors, pressure cooker, curing environment and curing time.

For each hard reline material, the flexural strength of the pressure cooker cured groups was significantly higher than that of the control group because of two factors, curing temperature and curing pressure. The curing temperature's effect on the rate and degree of polymerization is of prime importance in determining the manner of performing polymerization. Increasing the curing temperature usually increases the polymerization rate and decreases the percentage of residual monomer.³¹ The effect of pressure on polymerization is important from the practical viewpoint because several monomers are polymerized at pressures that are above atmospheric pressure. High pressure can have appreciable effects on polymerization rates and polymer molecular weights. Increased pressure usually results in increased polymerization rates and molecular weight.³¹ Increased pressure improved the flexural strength of the hard chairside reline resins when compressed with either air or nitrogen gas. Our results are consistent with those of previous studies that reported that auto polymerizing acrylic resin cured under pressure demonstrated decreased porosity and increase flexural strength.^{28,32} The pressurized environment may prevent monomer evaporation during the initial stage of polymerization, thus, minimizing the pore formation and improving flexural strength.³³ The first hypothesis was rejected.

Because of the inert characteristics of nitrogen gas, it does not undergo chemical reactions. So, it is a consideration to use nitrogen as purging gas. Purging the pressure cooker and replacing with nitrogen gas may help increase the flexural strength of the hard chairside

reline resins because oxygen exposure was eliminated. Oxygen is a powerful inhibitor, as demonstrated by the very large inhibition constant values (ratio of the rate constants for inhibition and propagation). This value of MMA polymerization is 33,000.³¹ Oxygen reacts with radicals to form the relatively unreactive peroxy radical that reacts with itself or another propagating radical by coupling and disproportionation reactions to form inactive products (probably peroxides and hydroperoxides). Consistent with a study that reported that an excluded air curing environment decreased residual monomer.³⁰ Radical-chain polymerization can be inhibited by oxygen which reacts with free radicals. Large amounts of oxygen will compete with MMA for free radicals and inhibit polymerization. Oxygen has some characteristic like an unpaired electron, which can react with a free radical initiator or during propagation of polymer chain. Thus, the degree of inhibition is proportional to the concentration of oxygen.^{31,33,34} However, a study found of unpolymerized layer of hard chairside reline resin after curing auto-polymerizing acrylic resin under pressure. It was assumed that higher air pressure might provide more oxygen to the resin surface and retard polymer chain growth and affect surface hardness.³⁵ The second hypothesis was rejected.

For each hard reline material cured at the same environment in the pressure cooker, the flexural strength of the 10, 15, and 20 min curing time groups were not significantly different. This may be explained by the termination of the degree of polymerization. The respective manufacturers recommend that the Unifast™ Trad setting time is two minutes, while that of Tokuyama® rebase II Fast setting time is 6-8 minutes. Thus, varying the curing time above these amounts might not affect flexural strength. Therefore, the present study assumes that curing under pressure, compressed with either air or nitrogen gas, at 1,500 mmHg 55°C for ten minutes results in a flexural strength equal to that of curing for 15 or 20 minutes. In addition, it should be noted that

handing that follows the manufacturers recommendation results in lower flexural strength due to less polymerization with room temperature, lower pressure, and oxygen exposure. The third hypothesis was accepted.

Under the same curing conditions, Unifast™ Trad had a significantly higher flexural strength compared with Tokuyama® rebase II. These results agree with those of previous studies.^{8,36} The difference in flexural strength of the two types of reline materials might be due to the different molecular structures and mechanical properties of the polymerized materials. The Unifast™ Trad powder is composed of PMMA, while Tokuyama® rebase II mainly consists of PEMA. The Tokuyama® rebase II liquid consists of 59 % acetoacetoxy ethyl methacrylate (AAEM) monomer and 39 % 1,9-nonanediol dimethacrylate (1,9-NDMA) as the cross-linking agent.³⁷ The molecular weight of the AAEM monomer is 214.22 g/mol, and the cross-linking agent 1,9-NDMA has a higher molecular weight of 296.4 g/mol.³⁷ Unifast™ Trad predominantly consists of MMA monomer and dimethyl-p-toluidine, and the molecular weight of MMA is 100 g/mol.³⁸ In addition, because it is MMA-based, Unifast™ Trad has a higher exothermic behavior.³⁹ During polymerization proceeds, the carbon-carbon double bonds (C=C) are converted to carbon-carbon single bonds (C-C). The difference in energy between the two bonds may emit as heat.⁴⁰ The emitted heat increased the curing temperature and polymerization reaction. This resulted in higher flexural strength of Unifast™ Trad. Thus, the fourth hypothesis was rejected.

Conclusion

Within the limitations in this study, it can be concluded that curing in a pressure cooker significantly increases the flexural strength of auto polymerized hard reline resins. When performing curing in a pressure cooker, using nitrogen instead of air with appropriate curing time also significantly increases the flexural strength.

Acknowledgements

This study was financially supported by the Graduate student fund GCUGR1225621026M of the Graduate School, Chulalongkorn University. We are grateful for their support. In addition, we thank Dr. Kevin Tompkins, Faculty of Dentistry, Chulalongkorn University for his critical review of this manuscript.

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