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# Ultrasonic Post-polymerization and Surface Treatments Increased the Flexural Strength of Denture Base Reline with Hard Lining Material

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## Abstract

**Objectives:** To evaluate the effect of ultrasonic post-polymerization and chemical surface treatment on the flexural strength of relined denture base.

**Methods:** Ninety heat-polymerized acrylic resin blocks (64x10x2 mm) were randomly divided into three groups by chemical surface treatments before relining; no treatment (N), applied with methyl methacrylate for180 seconds (MMA), and applied with methyl formate: methyl acetate (25:75% v/v (MF-MA)) for15 seconds. The samples were relined with auto-polymerized acrylic resin. Each relined sample was 64x10x3.3 mm. The relined groups were divided to three subgroups based on post-polymerization method: no post-polymerization (X), ultrasonic treatment in water (W), and ultrasonic treatment in 30% ethanol (E). The ultrasonic water bath was set at 40 kHz, 50°C, for 5 minutes. The samples were polished and stored in 37±2°C distilled water for 48±2 hours before undergoing a three-point bending test. The results were analyzed using two-way and one-way ANOVA.

**Results:** There was no interaction between ultrasonic treatment and chemical surface treatment. In each surface treatment group, W groups demonstrated a significantly higher flexural strength than X groups ($p<0.05$). E groups had a significantly higher flexural strength than W groups ($p<0.05$). In the same post-polymerization groups, N groups presented a significantly lower flexural strength than the MMA and MF-MA groups ($p<0.05$). The MMA and MF-MA groups were not significantly different ($p>0.05$).

**Conclusions:** ultrasonic treatment increased the flexural strength of relined denture base. MMA and MF-MA treatment increased the flexural strength of relined denture base.

**Keywords:** denture base, flexural strength, methyl acetate, methyl formate, ultrasonic treatment

## Introduction

Existing removable prostheses often require denture base relining to improve the fit against supporting tissues which beneath alveolar ridge contour gradually changes.[1,2] Relining technique can be performed directly in the patient's mouth at 'chairside' or in a laboratory. The laboratory relining involves an extra patient visit as well as a laboratory fee. Moreover, they have to be without their dentures for a while. The direct or 'chairside' re-lining benefits not only faster but can also reproduce the morphological features of supporting tissues directly on the denture base.[3,4] The hard chairside lining materials are more convenient.[5]

Auto-polymerized acrylic resins are commonly used as denture liners due to their acceptable cost and rapid processing. These acrylic resins are classified in the monomethacrylate group. The methyl methacrylate-based group (MMA-based) provides mechanical properties superior to the non-MMA-based group.[6,7] Moreover, MMA-based materials provide better bond strength to denture bases.[8] However, this material contains high amounts of residual monomer (RM) that can cause inflammation[9] and diminish its mechanical properties.[10-13]

Immersion in water is the simplest method to reduce RM. Immersion in 55°C water for 60 minutes reduced the cytotoxicity of the leached monomer.[14] Ethanol solution is another immersion medium for this purpose. 20-50% ethanol immersion (50°C, 10 minutes) reduced the RM content more than water and improved biocompatibility without mechanical property deterioration.[15] The study found that an ultrasonic bath, which is typically present in general dental offices, could be used to reduce RM in a very short time.[16] Radiation treatment, such as microwave and ultrasonic post-polymerization, is another method to reduce RM. However, Chia *et al.*[17] found that microwaving raised the temperature of acrylic resins over 100°C within 5 minutes, which might adversely affect the dentures. Notably, microwave post-polymerization had no effect on reducing denture base cytotoxicity.[14] Ultrasonic treatment is another option for post-polymerization. The cavitation bubbles generated by ultrasonic waves explode and generate high pressure and energy for mechanical and chemical effects.[18] Ultrasonic treatment in either water[16,19] or 30% ethanol[20] reduced the RM effectively in a short time. However, the effect of ultrasonic post-polymerization on the flexural strength of whole relined denture base has not been evaluated.

Chemical surface treatment is necessary for joining hard liners and denture base. Substances with solubility parameters similar to PMMA can swell the denture base surface and provide mechanical retention. Many substances can wet the surface of heat-polymerized denture base, such as methyl methacrylate (MMA)[21,22], chloroform[21] and acetic acid.[23] Vallittu *et al.*[24] found that wetting the denture base surface with MMA for 180 seconds before adding the auto-polymerized acrylic resin increased the flexural strength of repaired materials. Methyl formate (MF) and methyl acetate (MA) which have low toxicity, left no RM and provided high bond strength.[25] Other studies, wetting with a mixture of MF-MA 25:75% by volume for 15 seconds increased the bond strength of relined denture base.[26,27]

The aims of this study were to evaluate the effect of ultrasonic post-polymerization treatment and chemical surface treatment on the flexural strength of relined denture base. The first null hypothesis was that ultrasonic post-polymerization treatment did not affect on the flexural strength of relined denture base. The second null hypothesis was that chemical surface treatment did not affect on the flexural strength of relined denture base.

## Materials and Methods

Ninety 64x10x2 mm heat-polymerized acrylic resin (Meliodent®, Kulzer, Hanau, Germany) samples were prepared as recommended by the manufacturer. The sample size was determined based on ISO 20795-1.[28] Long cycle curing was performed in a water bath (70°C for 8 hours). The specimens were polished with 500, 1000, and 1200 grit silicon carbide paper using an automatic grinding and polishing unit (Minitech 233, Metallography India, Maharashtra, India) to within ±0.02 mm in all dimensions. The lining surface of the heat-polymerized specimen was polished with the same carbide paper using 54 Newtons at 450 rpm for 20 seconds. The samples were kept in 37±2°C distilled water for 48±2 hours. The samples were randomly divided into three surface treatment groups ($n$=30): non-treatment (N), applied with MMA for 180 seconds (MMA), or applied with MF-MA (25:75% volume (MF-MA)) for 15 seconds. The samples were placed in the split metal molds. The auto-polymerized acrylic resin (Unifast Trad®, GC, Tokyo, Japan) was mixed as recommended by the manufacturer and used to reline the prepared heat-polymerized specimens. The molds were covered with a 5-kilogram metal block and were polymerized for 8 minutes. The relined specimens were divided to 3 subgroups ($n$=10) based on their post-polymerization treatment; no post-polymerization (X), ultrasonic in water (W), ultrasonic in 30% ethanol (E) (Figure 1). The ultrasonic bath was set at 40 kHz, 50°C for 5 minutes. Materials used in this study were presented in the Table 1.

The relined specimens were finished with 500 grit silicon carbide paper. The relined specimens were 3.3±0.02 mm thick; each specimen was composed of 2 mm heat-polymerized resin and 1.3 mm auto-polymer-

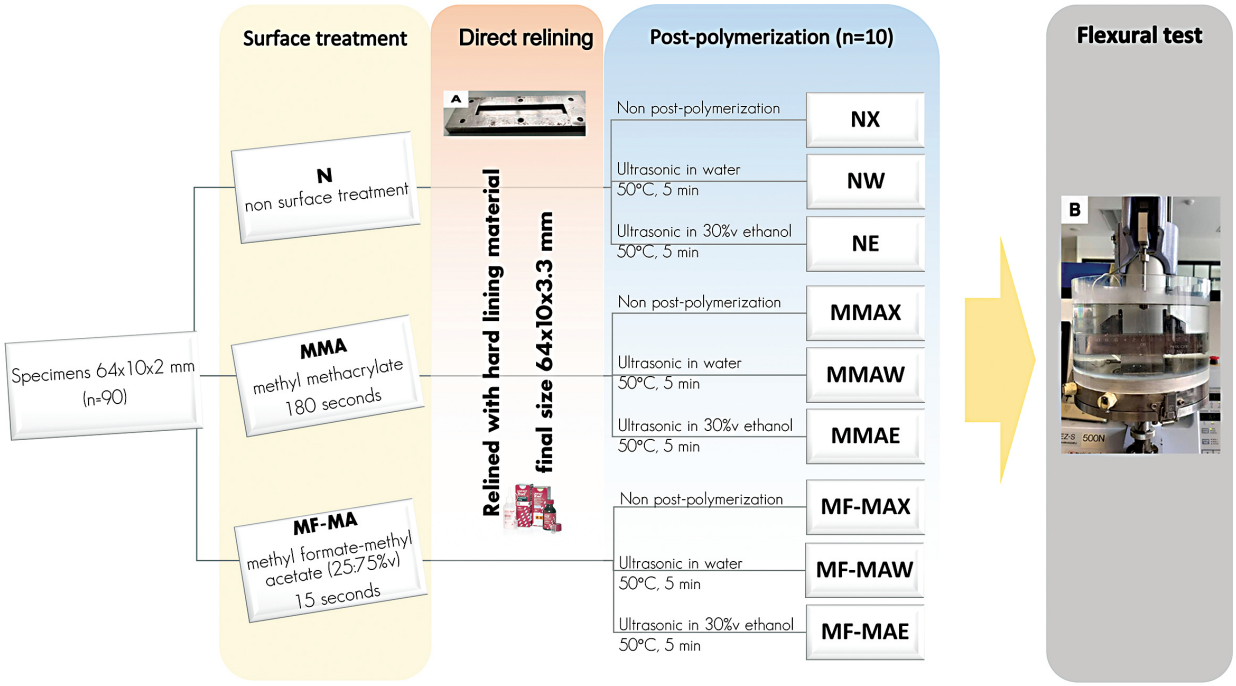


**Figure 1:** Description of all nine experimental groups (NX, NW, NE, MMAX, MMAW, MMAE, MF-MAX, MFMAW, MF-MAE) and specimens' preparation procedure. (A: a split metal mold for relining, B: a setting of three-point bending test). N: Non surface treatment, MMA: methyl methacrylate, MF-MA: methyl formate methyl acetate, X: non ultrasonic treatment, W: water ultrasonic treatment, E: 30%v ethanol ultrasonic treatment.

**Table 1:** The materials' names and manufacturers of samples used in this study.

| Product name | Material | Lot. number | Manufacturer | Instruction |
|---|---|---|---|---|
| Meliodent® | Powder: PMMA<br>Liquid: MMA | K010035 k010124 | Kulzer, Hanau, Germany | Heat-polymerized resin ratio: 35 g/14 ml |
| Unifast Trad® | Powder: PMMA<br>Liquid: MMA | 1907011 1811221 | GC, Tokyo, Japan | Auto-polymerized resin ratio: 2 g/1 ml |
| Methyl methacrylate | Surface treatment agent (Liquid of Unifast Trad®) | 1811221 | GC, Tokyo, Japan | |
| Methyl formate | Surface treatment agent | S6246689 111 | Merck Schuchardt OHG, Germany | |
| Methyl acetate | Surface treatment agent | S7082511 530 | Merck KGaA, USA | |
| Ethanol (LiChrosolv®) | Ultrasonic media | K45361627 409 | Merck KGaA, Germany | |

PMMA: Poly(methyl methacrylate), MMA: Methyl methacrylate

izing resin. The specimens were stored in 37±2°C water for 48±2 hours before testing. The flexural strength was measured by a universal testing machine (SHIMADZU, EZ-S 500N model, Japan) at a crosshead speed 5 mm/minutes within water bath 37±2°C. The distance between the two supporting bars was 50 mm. The flexural test was performed under water. (The samples were prepared and tested per ISO 20795-1)[28]

The morphological changes on the surfaces after surface treatment was analyzed by scanning electron microscope (SEM). SEM examination (SEM: FEI Quanta250, USA) was set at 20 kV. The images were developed with 1500x and 6000x magnification for visual inspection. The specimens were traced without gold particle coated. The untreated surface was used as a control.

Mode of failure was determined (cohesive, adhesive, mixed failure) along the fracture surface using a stereomicroscope (SZ61, Olympus Corp., China) at 35x magnification. Cohesive failure was defined when there was a line cutting through the relined specimen without step between heat-polymerized and auto-polymerized layer along the total fracture surface. Adhesive failure was determined when there was a step cutting between those two layers (interface fracture) then the heat-polymerized layer could split apart the auto-polymerized layer. Mixed failure was defined when there were a combine of interlayer-step and straight-line cutting within the whole fracture surface (Figure 2).

Statistical analysis was performed using IBM SPSS version 23.0 (IBM Corporation, Armonk, NY, USA) at a 95% confidence level. Normality of the data was tested using Kolmogorov-Smirnov test. Effects of ultrasonic treatment and chemical surface treatments on the flexural strength were performed using two-way ANOVA. Then, the effect of each factor on the flexural strength was determined using one-way ANOVA followed by Tukey's HSD *post-hoc* test.

## Results

The homogeneity of variance was considered equal. Two-way ANOVA analysis found no interaction between the two factors (ultrasonic treatment and chemical surface treatments) ($p>0.05$).

The results from one-way ANOVA (Table 2) indicated that that W groups increased their flexural strength compared with X groups ($p<0.05$). Moreover, E groups demonstrated a higher flexural strength than W groups ($p<0.05$). Within the same post-polymerization factor groups, the results revealed that the mean flexural strength of the relined denture bases applied with MMA or MF-MA as chemical surface treatments were significantly higher than the non-surface treatment groups (N groups) ($p<0.05$). The mean flexural strength of the MMA and MF-MA groups were not significantly different.

SEM examination presented the morphological changes on the treated surface. Both chemical surface treatments created different surface irregularities of heat-polymerized material (Figure 3). MMA generated delicately rough surface on heat-polymerized acrylic resin. MF-MA generated multiple size porosities with well-defined margin on the denture base surface. Failure type analysis revealed that there was no adhesive failure (Figure 4). There was the most mixed failure in N groups. Especially after ultrasonic post-polymerization, N group

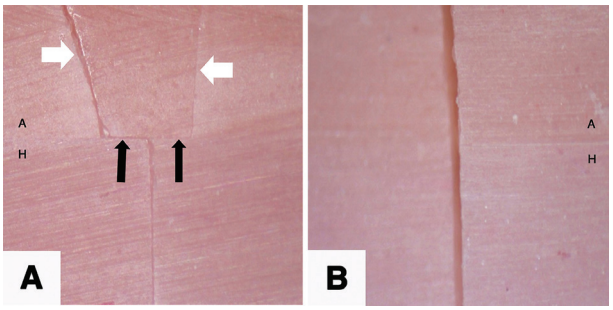


**Figure 2:** Stereomicroscope images at 35x magnification show fracture characteristics (A: auto-polymerized acrylic resin, H: heat-polymerized acrylic resin). (A) There is an interface fracture. Black arrows indicate interlayer fracture (steps between two layers). White arrows indicate a fracture within auto-polymerized acrylic resin, (B) There is a straight line cutting without any relining-interface fracture, referreing as a cohesive failure.

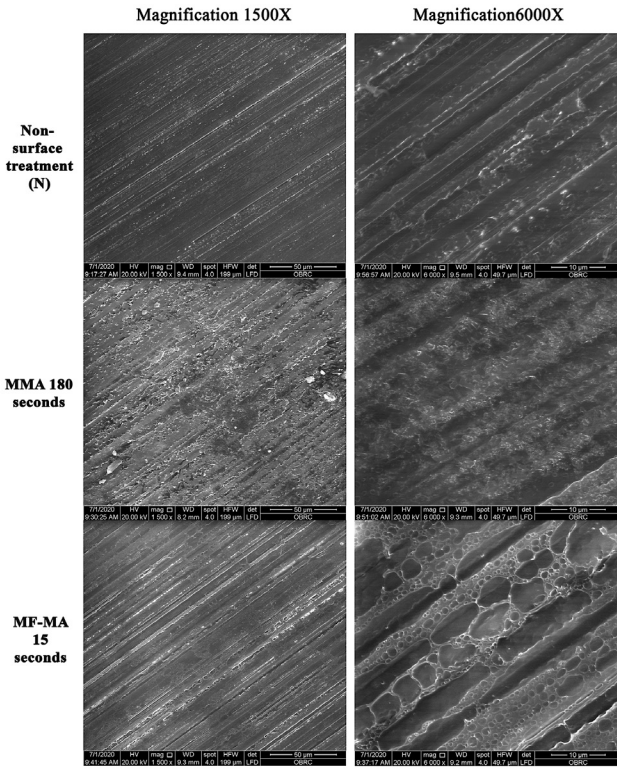


**Figure 3:** Scanning electron microscope images of the denture base after the different surface treatments. SEM was set at 20kV for both 1500x and 6000x magnifications. Chemical surface treatment created different surface irregularities on heat-polymerized material. MMA created the small irregular surface. MF-MA created a mix fine margin of small and large porosities on the heat-polymerized material, obviously.

**Table 2:** The mean transverse strength and standard deviation of each relined group presented in the table (MPa).

| | | Chemical surface treatment | | |
|---|---|---|---|---|
| | | Non (N) | MMA 180 seconds | MF-MA 15 seconds |
| Post-polymerization | No US (X) | 67.75 (1.05)$^{Aa}$ | 70.14 (1.29)$^{Ab}$ | 69.03 (1.12)$^{Ab}$ |
| | US water (W) | 69.51 (1.47)$^{Ba}$ | 72.11 (0.99)$^{Bb}$ | 72.16 (1.06)$^{Bb}$ |
| | US 30% ethanol (E) | 72.62 (1.58)$^{Ca}$ | 74.92 (1.87)$^{Cb}$ | 75.03 (1.89)$^{Cb}$ |

*Same uppercase latter indicated no significant difference between the groups in each column ($p$>0.05).
*Same lowercase latter indicated no significant difference between the groups in each row ($p$>0.05).

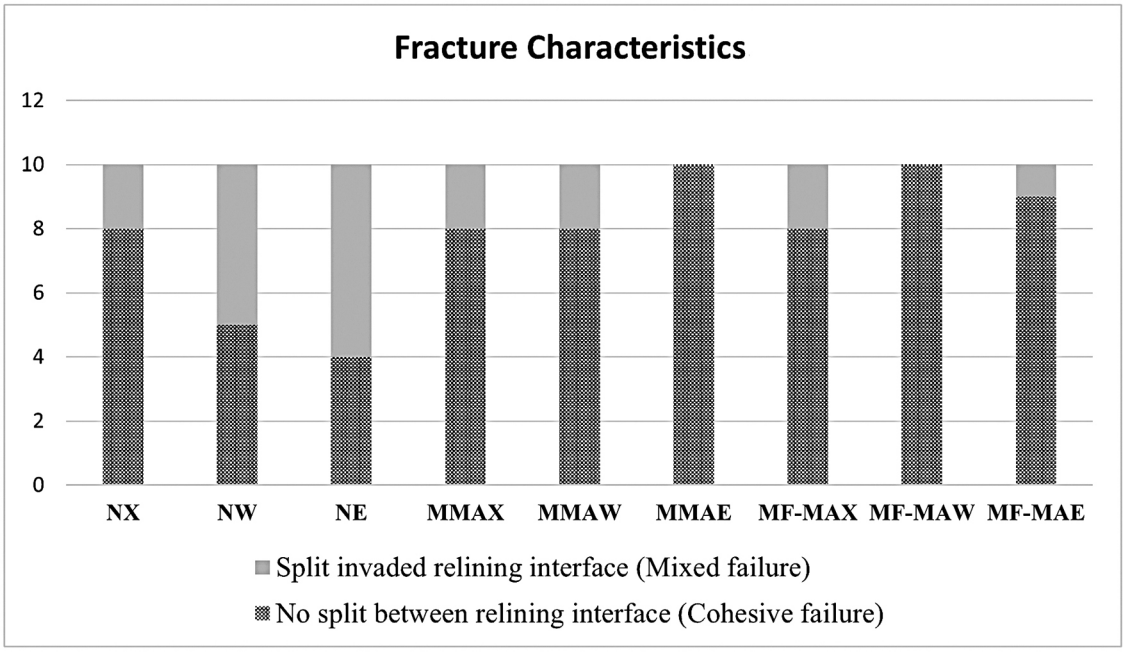


**Figure 4:** Number of specimens' fracture characteristics (pcs). Fracture surface of each specimen was determined using stereomicroscope (35x magnification).

presented more mixed failure. Cohesive failure found in both chemical surface treatment groups predominantly.

## Discussion

The present study evaluated the effect of ultrasonic post-polymerization treatment and chemical surface treatment of the flexural strength of relined denture base. We found that the flexural strength of relined denture base increased after ultrasonic treatment.

According to the post-polymerization results, the first null hypothesis was rejected. The post-polymerization process reduces RM.[10,29,30] RM causes a plasticizing effect that affects the mechanical properties of acrylic resins.[31] Thus, reducing RM improves denture base transverse strength. Studies reported that post-polymerizations improved the mechanical properties of acrylic resins.[10,32] The increased flexural strength in this study might be the result of liquid immersion and ultrasonic treatment. Residual monomer was leached out[33] by liquid immersion. Arriwiratchakun and Wiwatwarrapan[20] revealed that ultrasonic immersion reduced the leaching of unreacted monomer better compared with water immersion (50°C, 1 hour). High temperature immersion causes additional polymerization that consumed RM. Studies found that the elution level of RMs decreased ($p$<0.05) and the degree of conversion trended to increase after post-polymerization.[29,30] Another factor that influenced these results was ultrasonic energy. Our results indicated that ultrasonic treatment generated increased flexural strength within a short post-polymerization time. The cavitation bubbles generated by ultrasonic treatment affect the specimens via two mechanisms when they explode. Ultrasonic treatment

stimulated high velocity liquid-movement that increased RM diffusion from the material. Moreover, more energy was released from the process, which might accelerate further polymerization. Ultrasonic treatment generates the free radicals that initiate polymerizations.[18] Lamb *et al.*[33] found that active radicals for auto-polymerized acrylic resins were present for 60 minutes after polymerization finished. Notably, the specimens in our study were immersed in an ultrasonic bath immediately after relining, thus, active radicals may have been presented to induce additional polymerization. Thummawanich and Wiwatwarrapan[34] found that the flexural strength of auto-polymerized acrylic orthodontic plates significantly increased after ultrasonic treatment post-polymerization ($p<0.05$).

Ultrasonic treatment in 30% ethanol (NE, MMAE, MF-MAE) increased the flexural strength compared with ultrasonic in water among all surface treatment groups (NW, MMAW, MF-MAW) ($p<0.05$) (Table 2). Together when immersed in ethanol solution, RM was extracted more easily compared with water. This effect can be explained by the solubility parameter of the chemicals and the polymer's dissolving constant. The solubility parameter (Hildebrand parameter) and hydrogen-bonding capabilities of ethanol are closer to methyl methacrylate compared with water (26.0, 18.0 and 47.9 MPa1/2 respectively).[35] The ethanol concentration is related to the amount of MMA eluted.[36] Despite the low ethanol concentration, high elution occurred compared with water.[15,37] A dilute ethanol solution (<50%) did not cause significant adverse effects to material's properties, such as hardness[15,38], roughness[38] and flexural strength.[15,38] Moreover, the solvent temperature significantly affects the degree of plasticizing of a polymer. High temperature increases a materials' rigidity, which counteracts ethanol's plasticizing effect.[15] Moreover, in our study the specimens were stored in distilled water for 48±2 hours before testing. This might have reduced the plasticizing effect of residual ethanol. Therefore, type of ultrasonic media influenced the flexural strength of relined denture base.

In clinical situation, ultrasonic post-polymerization was a practical method for enhancing the flexural strength of relined denture base immediately. Moreover, ultrasonic treatment might cause auto-polymerized acrylic hard liner more biocompatible due to RM reduction.[16,19,20]

From the results, chemical surface treatments influenced the flexural strength of relined denture bases. The second hypothesis of no change flexural strength after applying chemical surface treatment was rejected. Hout *et al.*[7] found that the bond strength of hard liner to denture bases correlated with the flexural strength of the entire piece. The chemical surface treatments used in this study provided more micromechanical retention. The surface morphology of the control group (N) presented only scratches. Scanning electron microscopy revealed that there were irregularities on the surface of heat-polymerized acrylic resins (Figure 3). Based on the theory of solubility, the solvent having a solubility parameter similar to that of the denture base can dissolve and swell the denture base surface, generating surface irregularities. The solubility parameter of the denture base, MMA, MF, and MA were 18.3, 18.0, 20.9, and 19.6 MPa1/2, respectively.[35] The irregular surfaces generated by the solvent increase bonding areas between the denture base and reline material. The monomer of hard liner acrylic resins easily penetrates surface irregularities and polymerize, forming a hybrid layer that might increase the bond strength. MMA and MF-MA differentially swelled the denture base surface resulting in different appearances between the samples in the respective solvent groups. The changes in surface morphology in these groups likely contributed to their increase flexural strength compared with no treatment. However, MMA and MF-MA treatment showed no significant differences in flexural strength. The flexural strength of the relined specimens might have been influenced by the thickness of heat and auto-polymerized acrylic resin.[7] For bonding, material type influenced the bond strength.[8,39] Thus, in the same material testing, the bond strength between these chemical surface treatments might not directly generate different flexural strength. Furthermore, the fracture characteristic showed that chemical surface treatment decreased relining interface fracture or crazing (Figure 2A), especially after ultrasonic treatment (Figure 4). Factually, interlayer area was the weakest part of relined specimens. Poor interlayer bond might initiate interface debonding after loading. Even though ultrasonic provided a benefit, vibration generated from ultrasonic possibly attacked the fragile area of specimens. Thus, without chemical surface treatment, mixed failure presented predominantly after ultrasonic treatment (NW, NE groups).

In clinical situations, relined dentures are submitted in several oral environments. These materials endure both thermal and loading stress. Those stresses may affect bond strength between denture base and hard liner resins then debonding possibly occurs. A long term successful of relining relies on many factors including the bond strength of two layer.[40] Accordingly, chemical surface treatment was recommended before direct relining to reduce unacceptable failure from any clinical situations.

## Limitation

The study was performed *in vitro*. With fund and schedule limited, thermocycling and RM test was not performed. Thus, further investigation requires thermal and loading stress including RM amount as factors to confirm the evaluation.

## Conclusions

Within the *in vitro* study limitations, ultrasonic post-polymerization benefited auto-polymerized acrylic relining. Therefore, using ultrasonic in either water or 30% ethanol enhances the flexural strength of relined denture bases.

Either MMA or MF-MA can be used as a chemical surface treatment before relining. Chemical surface treatments reduce relining-interface fracture and improve the flexural strength of relined denture bases.

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## Conflicts of interest

The authors declare no conflicts of interest.

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