Tensile Bond Strength between Three Hard Reline Materials and Denture Base Resin Influenced by Methyl Formate-Methyl Acetate

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Abstract

This study investigated the effect of methyl formate-methyl acetate (MF-MA) wetting times on the tensile bond strength (TBS) between 3 non-MMA based reline materials and denture base material. Four hundred heat-cured denture base resin (Meliodent®) were prepared and randomly divided into 3 groups according to hard reline resins (Kooliner®, Tokuyama® Rebase II and Ufi Gel Hard®). Each group of reline material consisted of 6 or 7 subgroups (n=10), based on their surface treatment; control, adhesive, MF-MA 15, 30, 60, 180 s and MMA 180 s. The TBS test was performed using a Universal testing machine. Data were analyzed using one-way ANOVA and post hoc Tukey's analysis at $p<0.05$. The means TBS of the treated groups were significantly higher compared with those of the control group ($p<0.05$). In the Kooliner® groups, there were no significant differences in TBS between the MF-MA and the MMA treatment groups ($p>0.05$). In the Tokuyama® Rebase II groups, application of MF-MA solutions for 180 s produced the highest TBS compared with the other groups ($p<0.05$). In the Ufi Gel Hard®, the groups of MF-MA 180 s and MMA 180 s groups demonstrated significantly higher TBS compared with the other groups ($p<0.05$). Surface treatment with MF-MA solutions significantly increases the TBS between denture base resin and non-MMA hard reline resins. This study suggests that an MF-MA wetting time of 15 s for Kooliner® and 180 s for Tokuyama® Rebase II and Ufi Gel Hard® is adequate for creating a strong bond.

Keywords: Chemical surface treatment, Denture base, Methyl acetate, Methyl formate, Reline material, Tensile bond strength

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Introduction

Denture bases are made from poly (methyl methacrylate) resins which are constructed by connection of monomers to form polymer chain.[1] Fabrication of denture base has to bring about mechanical and physical properties, although poor fitting of the prosthesis always occur with the passing of time. This is because alveolar bone resorption is a continuous process due to tooth loss, causing denture base to be less stable on the ridge.[2] Therefore, dental prosthesis should be examined periodically and re-established to increase their adaptation. Relining a denture base with the reline materials is a common procedure to reproduce the fit of the denture and to improve the masticatory function.[3] Two main types of denture lining materials, classified by consistency, are soft and hard liners.[4] Soft liners are intended to be used for reducing masticatory force to the residual ridge. These liners consist of plasticizers, which serve as 'stress absorber' between denture and the underlying tissue.[5] However, prolonged exposure to water produces significantly higher hardness values and lower bond strength values.[6] Hard reline materials contain methyl methacrylate (MMA) or other type of monomers.[1] MMA can dissolve and penetrate into the denture base forming an adhesion.[7] After the setting of reline materials, residual monomers still leach out for a month causing oral tissue inflammation by direct contact.[8,9] Non-MMA based reline materials have a large amount of cross-linking agents added to a liquid part, which promotes greater transverse bending strength.[10] The interface of reline material-denture base resin depends on the ability of the monomers in the reline resins to diffuse and penetrate into the denture base, forming Inter-penetrating polymer networks (IPN).[11] Failure of adhesion promotes microleakage which enhances staining and bacteria accumulation.[11,12] Thus, surface treatment has been suggested to revise poor bonding.[13,14] Some studies reported that chemical surface treatment increased the flexural strength, while mechanical surface treatment had no effect.[15] Application of chemical agents dissolves the surface of denture bases and improves the diffusion of reline resin monomers to the denture base.[7,14] A mixture of MF-MA solution has been investigated in recent years, as it provides a high bond strength similar to Methyl methacrylate (MMA).[16] Considering a mixture of MF-MA solutions, a ratio of 25:75 (CU Acrylic Bond, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand) significantly increases the bond strength between denture base resin and reline resin.[17] The effect of various MF-MA wetting times on the tensile bond strength between non-MMA based reline materials and denture base has not yet been studied.

The objective of this study was to evaluate the effect of various MF-MA wetting times on the tensile bond strength between three non-MMA based reline materials and a denture base resin. The first null hypothesis was that there were no significant tensile bond strength differences between non-MMA based reline materials and a denture base resin when different wetting times of MF-MA surface treatment were used. The second null hypothesis was that no significant variation would be noted in the tensile bond strength between non-MMA based reline materials and a denture base resin when different chemical surface treatments were used. The third null hypothesis was that types of non-MMA based reline materials did not significantly affect the tensile bond strength between non-MMA based reline materials and a denture base resin when the same chemical surface treatments were used.

Materials and Methods

The method of this study mainly followed ISO10139-2:2009(E).[18] Four hundred heat-cured acrylic resin (Meliodent®) plates were prepared (25 ± 3 mm^2 and 3 ± 0.5 mm thick) by investing in dental stone in dental flasks. The flasks were then pressed (2,000 kgf) for 1 hour. The specimens were polymerized at 74°C for 9

hours (as recommended by the manufacturer). The plates were finished with silicon carbide paper (P500, TOA, Thailand) using an automatic grinding and polishing unit (NANO2000, Pace Technologies, USA). A digital vernier caliper (500 series, Mitutoyo Corp., Japan) was used to verify samples' dimension after polishing. The plates were stored in a water bath (160M, Contherm Scientific Ltd., New Zealand) at 37±1°C for 28±2 days. The surface of each heat-cured acrylic plate was visualized using stereo microscope (SZ61, Olympus Corp., China) before receiving surface treatment. Next, the samples were randomly divided into three groups of hard reline materials [Group I: Kooliner® (n=60), Group II: Tokuyama® Rebase II fast (n=70), Group III: Ufi Gel Hard® (n=70)]. Each group consisted of six to seven subgroups (n=10), according to surface treatment (Fig. 1).

Table 1 *Trade name, manufacturer and chemical composition of the tested materials.*

Product name	Lot No. and Manufacturer	Composition		
		Powder	Liquid	Adhesive
Heat-activated acrylic resin (Meliodent®)	2018457, Tokuyama Dental Corp., Japan	PMMA	MMA	-
Self-cured hard reline (Kooliner®)	1211074, GC America, USA	PEMA	IBMA	-
Self-cured hard reline (Tokuyama® Rebase II Fast)	035EZ4, Tokuyama Dental Corp, Japan	PEMA	AAEMA 1,9 NDMA	Ethyl- ace-tate Acetone
Self-cured hard reline (Ufi Gel Hard®)	1511506, Voco, Germany	PEMA	1,6 HDMA	Acetone, 2-HEMA
Methyl Acetate	S6246689, Merck Schuchardt OHG, Germany	-	-	-
Methyl Formate	S6238911, Merck Schuchardt OHG, Germany	-	-	-

PEMA, poly(ethyl methacrylate; 1,6 HDMA, 1,6-hexanediol dimethacrylate
IBMA, isobutyl methacrylate; AAEMA, 2-(Acetoacetoxy) ethyl methacrylate
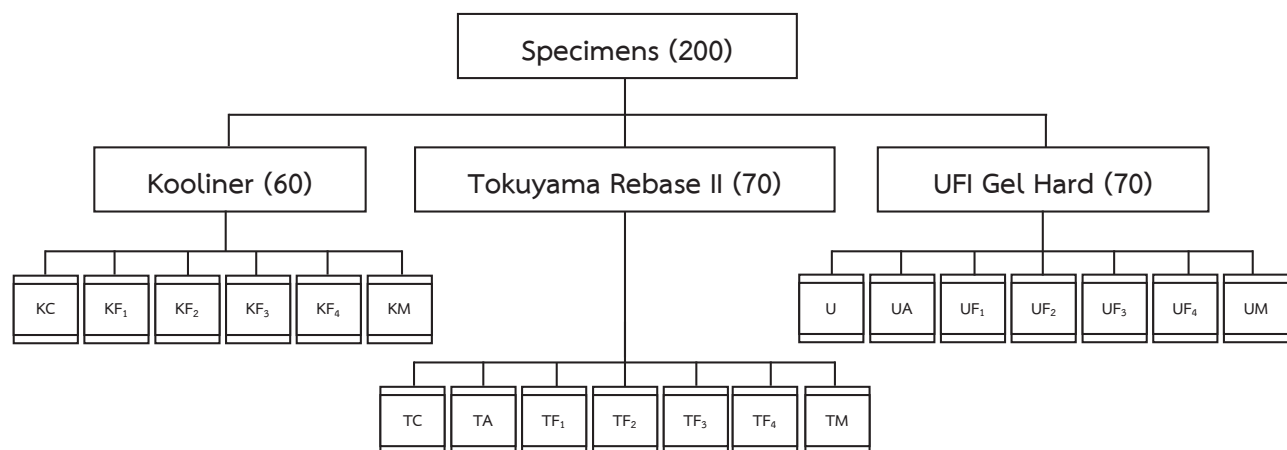1,9 NDMA, 1,9-Nonanediol dimethacrylate; 2-HEMA, 2-Hydroxyethyl methacrylate

Figure 1 *The distribution of the specimens from each material. "K": Kooliner®, "T": Tokuyama® Rebase II fast, "U": Ufi Gel Hard®. "C": negative control groups (3 groups)-not treated with any solution on the bonding surface, only lined with the three reline materials. "M": MMA groups (3 groups)-treated with monomer of Meliodent® (MMA) monomer for 180 s. "A": adhesive was used following the manufacturer's recommendation in Tokuyama® Rebase II fast (T) and Ufi Gel Hard® (U) groups. A single layer of adhesive bonding agent had been applied before the reline material was loaded. Kooliner® did not require an adhesive bonding agent. "F": application of MF-MA solution for varying wetting times, 15 s(1), 30 s(2), 60 s(3), and 180 s(4), before applying the reline material.*

The specimens were constructed in a metal split mold (Fig. 2[B]) at room temperature. A bond area was controlled by Teflon collar; 10 mm diameter and 3 mm in height. Two plates of heat-cured acrylic resin that were separated by self-cured acrylic resin were used to form one test specimen. The test specimen was pressed by a 4 kg metal pendulum, simulating complete denture maximum bite force.[19] After the hard reline had set, the test specimens (Fig. 2[C]) were placed in a water bath at 37±1°C for 23±1 hrs. Two hundred test specimens were evaluated using a tensile strength testing machine in a vertical alignment (Fig. 2[E]). The tensile bond strength was measured by a Universal testing machine (8872, Instron Co., UK) with crosshead speed at 10 mm/min. The maximum load was recorded during debonding and the bond strength was calculated according to the following equation.

$$B = F/A$$

Where B was the tensile bond strength in MPa, F was the maximum load in Newton before debonding occurred and A was the adhesive area (mm^2).

The mode of failure of the debonded surface was determined (cohesive, mixed or adhesive failure) using a stereomicroscope (SZ61, Olympus Corp., China) at 10x magnification. Cohesive failure was defined as a failure where there was more than 50 % of the reline material on the denture base surface. Adhesive failure was defined as a failure where there was no trace of reline material on the denture base surface. Mixed failure was defined as a failure where there was less than 50 % of the reline material on the denture base surface.

Differential scanning calorimetry was conducted using a differential scanning calorimeter (DSC 7, Perkin-Elmer, Waltham, USA) to determine the exothermic energy of autopolymerizing hard reline materials. Each specimen of the hard reline materials was placed into an aluminum pan and the test was performed under a nitrogen purge with a flow rate of 70 mL/min. The scan speed for thermal heating was 10°C/min and the temperature range was from 25–120°C.

Data were analyzed using SPSS for Windows 25.0 (SPSS Inc., Chicago, IL, USA). The Kolmogorov Smirnov test was used to determine the normal distribution of the results and the equality of variance was evaluated using the Levene's test. The results did not conform to the assumptions of the Two-Way ANOVA that the data had to be statistically independent and with an equal number of observations. There is an interaction effect on the tensile strength between the two factors of the hard reline materials and the surface treatments. The interaction effect between two factors is defined as one in which the effect of one factor depends on the level of the other factor.[20] Thus, the results were statistically analyzed by One-Way ANOVA and Tukey HSD test.
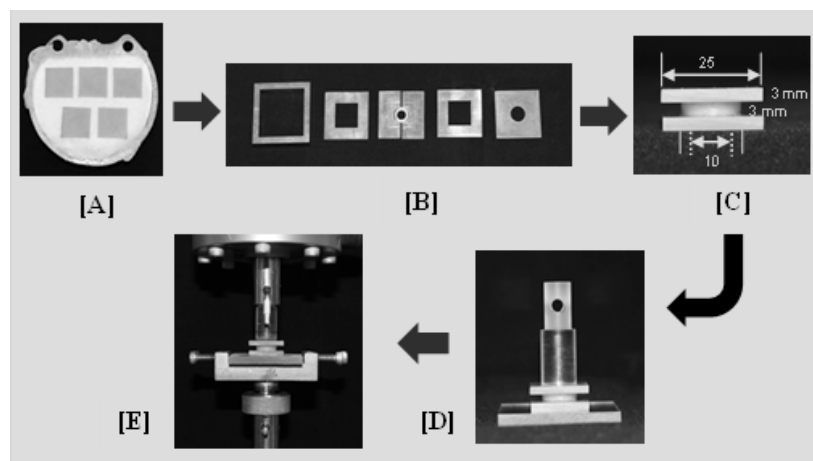

Figure 2   *Specimen preparation. [A] heat-cured denture base in a dental flask, [B] split metal mold, [C] test specimen, and [D,E] test specimen in a vertical alignment.*

Results

The mean tensile bond strength and standard deviation of each group and the percentage of each failure type were presented in Table 2. The mean tensile bond strength of the treated groups were significantly higher than those of their respective control groups ($p<0.05$). In the Kooliner groups, there were no significant differences in the tensile bond strength between the MF-MA solution wetting time groups and the MMA 180 s group ($p>0.05$). The tensile bond strength of the Tokuyama rebase II groups showed that the groups applied with MF-MA solution for 15, 30 and 60 s were not significantly different from that of the adhesive and the MMA 180 s group ($p>0.05$). In the Ufi Gel Hard groups, there were no significant differences in the tensile bond strength between the groups applied with MF-MA for 15, 30 and 60 s and the adhesive group ($p>0.05$). However, the mean tensile bond strength of the MF-MA 15 s, 30 s, 60 s and the adhesive groups were significantly lower than those of the MF-MA 180 s and the MMA 180 s groups ($p<0.05$). The mean tensile bond strength of the MF-MA 180 s group was not significantly different from that of the MMA 180 s group (MU) ($p>0.05$).

Failure type analysis demonstrated that all 3 reline materials in the control groups had 100 % adhesive failure. Most of the failures in MF-MA and MMA groups were mixed failures. The Tokuyama Rebase II and the Ufi Gel Hard MF-MA 180 s groups presented 40 % and 10 % cohesive failure, respectively, whereas the Ufi Gel Hard MMA 180 s group demonstrated 20 % cohesive failure. The percentage of the failure types in each group was shown in Table 3. The failure patterns by stereomicroscopy images were shown in Figure 3, 4, and 5.

Table 2 *The mean tensile bond strength of each reline material according to surface treatments*

Surface treatment	Kooliner	Tokuyama Rebase II fast	Ufi Gel Hard
control	4.94 ± 0.75 [B]	3.04 ± 0.72 [A]	3.53 ± 0.79 [A]
Adhesive	-	5.17 ± 0.61 [B,C]	5.21 ± 0.80 [B,C,D]
MF-MA 15 s	7.38 ± 0.40 [E, F, G]	5.81 ± 0.45 [B,C,D]	5.42 ± 0.77 [B,C,D]
MF-MA 30 s	7.82 ± 0.88 [G]	5.68 ± 0.52 [B,C,D]	6.19 ± 0.82 [C,D,E]
MF-MA 60 s	7.50 ± 0.64 [F,G]	5.28 ± 0.80 [B,C,D]	6.29 ± 0.70 [C,D,E]
MF-MA 180 s	7.98 ± 0.52 [G]	7.85 ± 0.79 [G]	7.83 ± 0.90 [G]
MMA 180 s	8.23 ± 0.53 [G]	6.40 ± 0.74 [D,E,F]	7.90 ± 0.72 [G]

The same superscript letter indicated no significant difference between groups (p>0.05).

Table 3 *The percentage of failure pattern of the three reline materials and different surface treatments*

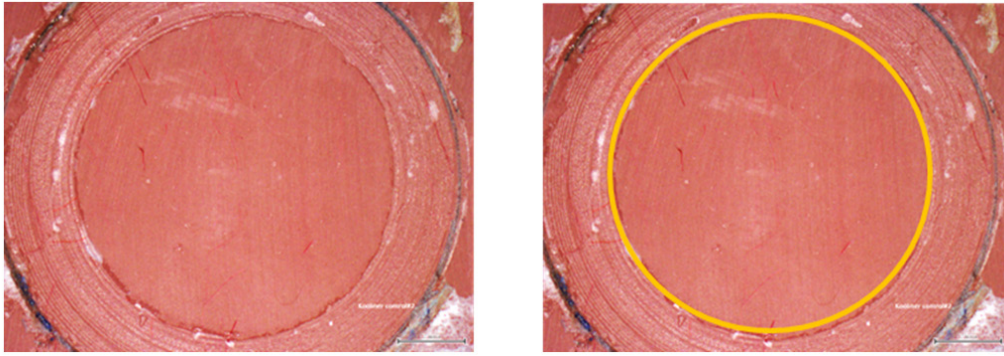| Surface treatment	Kooliner			Tokuyama Rebase II			Ufi Gel Hard		
	Co (%)	Mixed (%)	Ad (%)	Co (%)	mixed (%)	Ad (%)	Co (%)	Mixed (%)	Ad (%)
control	-	-	100	-	-	100	-	-	100
Adhesive	-	-	-	-	100	-	-	50	50
MF-MA 15 s	-	90	10	-	100	-	-	80	20
MF-MA 30 s	-	80	20	-	90	10	-	90	10
MF-MA 60 s	-	90	10	-	90	10	-	100	-
MF-MA180 s	-	90	10	40	60	-	10	90	-
MMA 180 s	-	80	20	-	100	-	20	80	-

Figure 3 *Adhesive failure, no reline material attached, at denture base surface using a stereomicroscope at 10x magnification.*
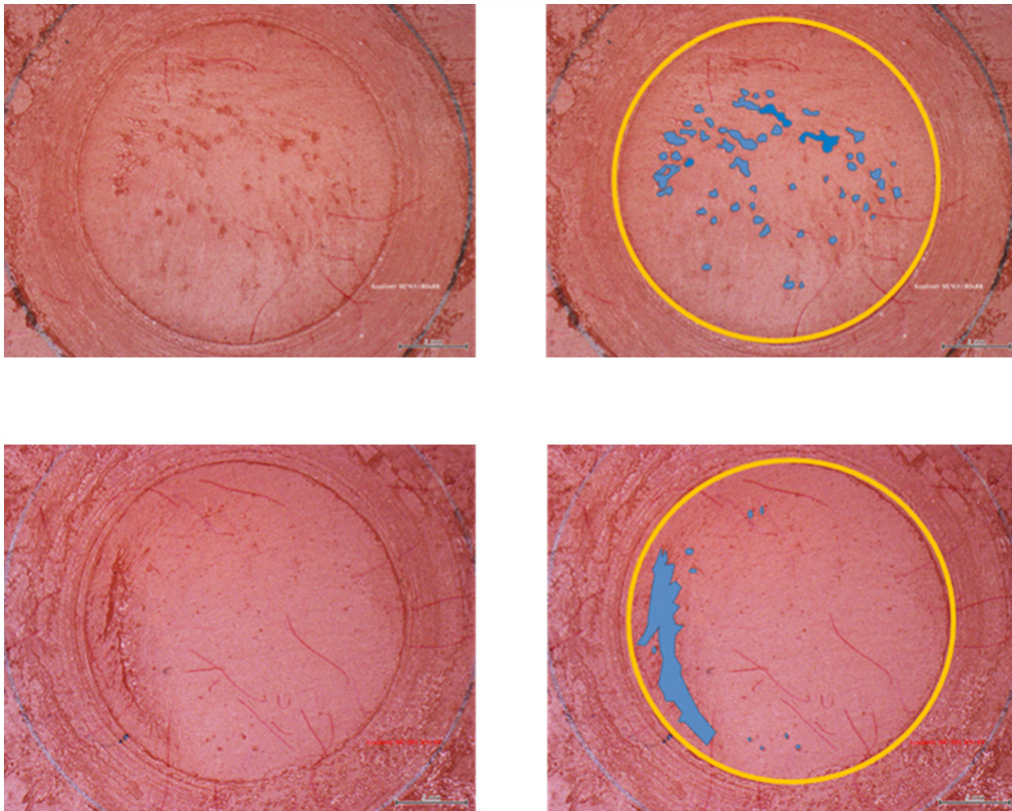

Figure 4 *Mixed failure showing the reline material (<50%) attached to the denture base surface using a stereomicroscope at 10x magnification. Blue area in the right representing the reline material.*
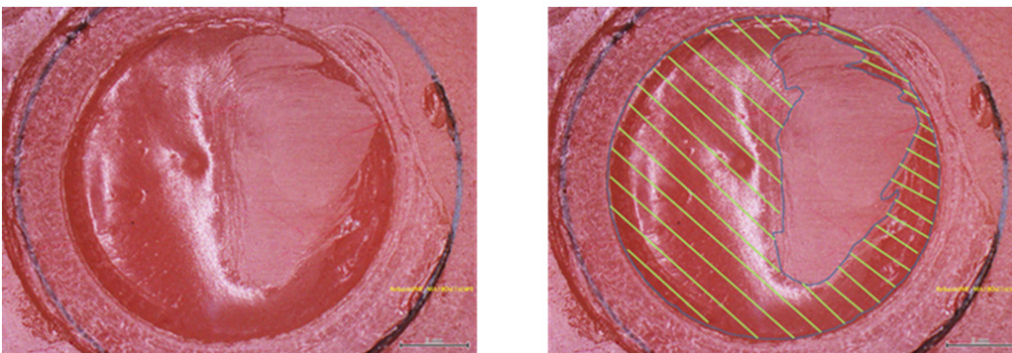

Figure 5 *Cohesive failure showing most of the reline material (>50%) attached to the denture base surface using a stereomicroscope at 10x magnification. Shading area in the right representing the reline material.*

SEM examination was used to observe the morphological changes on the denture base surface after surface treatment (Fig. 6). The untreated denture base surface, and the control group, exhibited scratch lines in a single direction with some acrylic debris from polishing (Fig. 6[A]). The surface of the denture resin treated with MF-MA for 15 and 30 s demonstrated numerous porosities with different sizes and patterns in the superficial layer, however, the deep layer still showed scratch lines (Fig. 6[B,C]). Denture base resin applied with MF-MA for 60 s showed the same surface pattern as the 15 and 30 s wetting times and with obscured scratch lines in the deep layer (Fig. 12[D]). The denture base resin treated with MF-MA for 180 s demonstrated a honeycomb appearance with 3-dimensional pores from the superficial into the deep layer (Fig. 6[E]). The denture base resin treated with MMA for 180 s had irregular scratch lines similar to the denture base resin applied with Tokuyama Rebase II adhesive (Fig. 6[F,G]). The Ufi Gel Hard adhesive created a smoother denture base surface (Fig. 6[H]).
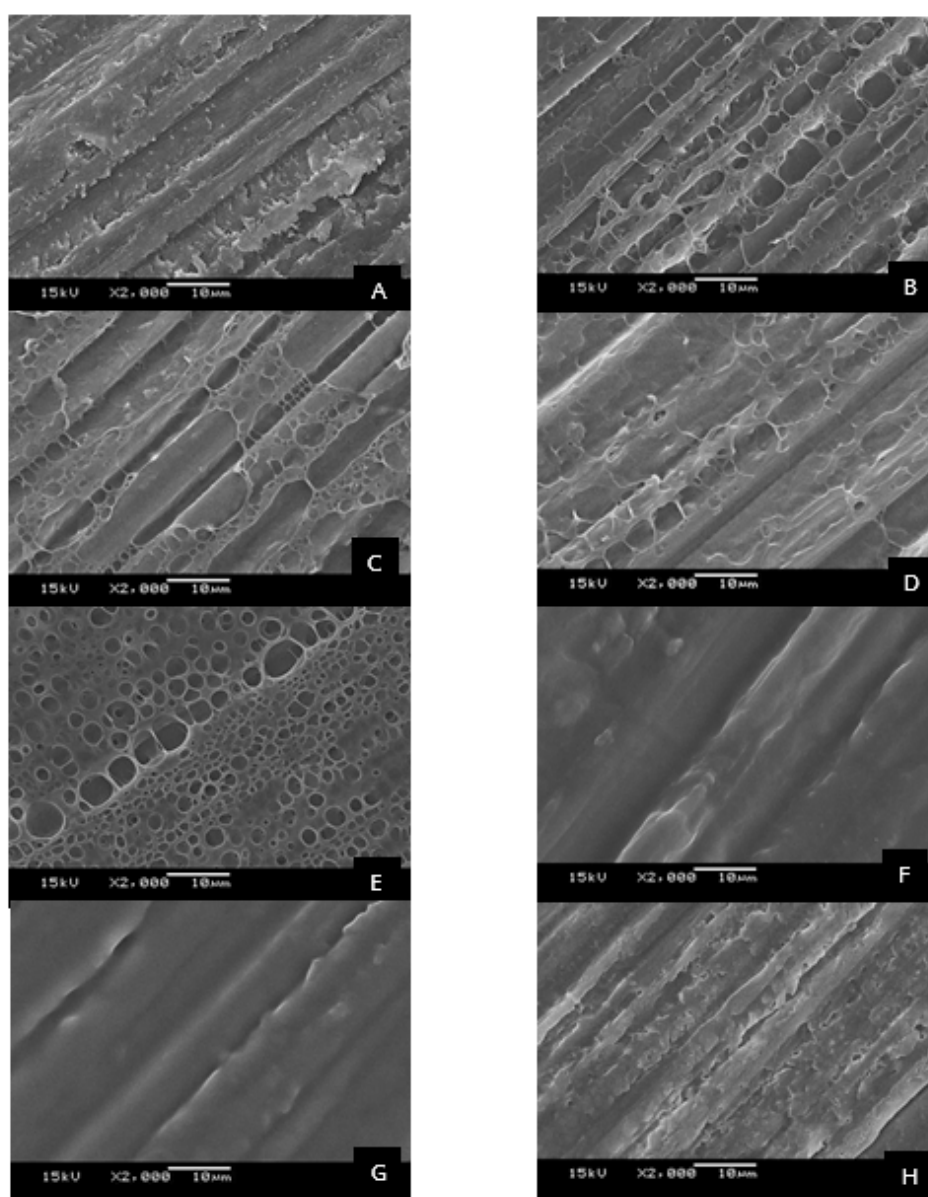

Figure 6 *SEM analysis of the morphological changes of heat-cured denture base surface treated with different surface treatments. [A] no treatment, [B] MF-MA solutions 15 s, [C] MF-MA solutions 30 s, [D] MF-MA solutions 60 s, [E] MF-MA solutions 180 s, [F] MMA 180 s, [G] Tokuyama Rebase II adhesive, [H] Ufi Gel Hard adhesive, respectively.*

Discussion

This study was designed to determine how various MF-MA wetting times affected the tensile bond strength between 3 non-MMA based reline materials and the denture base resin. The tensile bond strengths of specimens treated with MF-MA for 15, 30, 60, 180 s and no treatment were compared. These wetting times were selected based on a previous study that found that increased MMA wetting time caused an increased thickness of the swollen layer at the denture base surface.[21] Vallittu *et al.* concluded that an MMA wetting time of 180 s was sufficient to provide a strong bond.[7] Therefore, we used MF-MA wetting times ranging from 15-180 s to determine the optimum time for the highest tensile bond strength.

There are two main variables which directly relates to the tensile bond strength, reline materials and the surface treatment. Surface treatment refers to two factors, type of solvent and wetting time.

In the Kooliner groups, there were no significant differences in the tensile bond strength among the various MF-MA wetting times. However, the mean tensile bond strengths of the Tokuyama Rebase II and Ufi Gel Hard were significantly different in 180 s-MF-MA wetting time compared to those of the 15, 30 and 60 s-MF-MA groups. The mean tensile bond strengths of the Tokuyama Rebase II and Ufi Gel Hard of the 15, 30 and 60 s-MF-MA groups were not significantly different from each other. From the two-dimension appearance from 180 s-MF-MA SEM image (Fig. 6[E]), it was postulated that the monomer of Tokuyama Rebase II and Ufi Gel Hard could penetrate and form the better bond compared with 15, 30 and 60 s-MF-MA wetting time. The first null hypothesis was rejected.

Four solvents were used for denture base surface treatments (MF-MA, MMA, Ufi Gel Hard adhesive and Tokuyama Rebase II adhesive). The Ufi Gel Hard adhesive contains 2-HEMA and acetone, whereas the Tokuyama Rebase II adhesive includes ethyl acetate and acetone. The dissolution efficiency can be explained by the relative closeness of solubility parameters and polarities of PMMA and the solvents.[22] The solubility parameter of PMMA is 18.3 MPa1/2, while those of MF, MA, MMA, ethyl acetate and acetone are 20.9, 19.6, 18.0, 18.2 and 19.7 MPa1/2, respectively.[23] The solubility parameter of 2-HEMA (26.93 MPa1/2) is markedly different from that of PMMA. The MF, MA and MMA have similar polarities due to their methyl ester groups that enhance their ability to soften PMMA, while the other solvents have different functional groups. Acetone has ketone group. Ethyl acetate is ethyl ester. 2-HEMA contains ethyl ester and hydroxyl group. The dissimilar polarity of ethyl acetate, acetone and 2-HEMA to PMMA is likely to bring these compounds out of the range of effective solubility.[22]

The molecular weight of solvent has an effect on the softening efficacy, in which lower molecular weight promotes the faster kinetics of diffusion.[22] Acetone (58.08 Da) has a molecular weight close to MF (60.05 Da). The other four solvents have the higher molecular weight; MA (74.08 Da), ethyl acetate (88.11 Da), MMA (100.12 Da) and 2-HEMA (130.14 Da) than acetone and MF. Boiling point affects the bonding process in that lower boiling point of solvent causes an easier evaporation and takes less chair-time. Methyl formate (31.8°C) has the lowest boiling point compared to the other solvents. Methyl acetate (56.9°C) and acetone (57°C) have a similar boiling point. Ethyl acetate, MMA and 2-HEMA have a boiling point of 77.1°C, 101°C and 213°C, respectively.

As in the aforementioned, MF and MA have a low boiling point, 31.8°C and 56.9°C, respectively, compared to the other solvents.[24,25] This allows the solution to evaporate with no residual on the bonding surface after their application. The bond mechanism between 2 materials has two processes, diffusion and penetration.[26] First, the solvent diffuses and adheres to the denture base surface. This process is related to the size of the solvent molecules.[26] MF and MA have smaller molecules compared with MMA and the other two adhesives. The

second process is dissolution and penetration. MF-MA solution generates a swollen gel-like pattern on the denture base surface. This process depends on the solubility parameter, polarity and the concentration of the solvent in the polymer.[27] The similar solubility parameter and polarity of MF-MA compared to PMMA are one of the reasons for providing a good bond at the relined interface. The molecular structures of MF and MA also do not contain carbon–carbon double bonds (C=C) that might polymerize with the monomer of the autopolymerized reline materials. Thus, using MF-MA solution can create a proper bond area without any residual material that can block the bonding. The large amount of pores at the interface of the MF-MA treated relined denture base surfaces allow the monomer of the reline material to penetrate, and then polymerize to create a mechanical interlocking bond at the molecular level. Subsequently, an interpenetrating polymer network layer is formed between the denture base and the reline material.

Methyl methacrylate is a solvent commonly used for the surface treatment. This solvent has similar solubility parameter and polarity compared to PMMA. However, a higher molecular weight and boiling point of MMA might provide a lower solubility to the denture base material compared to MF-MA. Ethyl acetate and acetone have similar solubility parameter compared to PMMA, but they have different functional groups in their chemical structures. Besides, ethyl acetate has a higher molecular weight and boiling point compared to MF-MA and acetone. Acetone has many requirements to promote PMMA dissolution similar to MF-MA except the different functional group in chemical structure. 2-HEMA has not only a considerably higher molecular weight and boiling point compared to the other solvents, but also dissimilar solubility parameter and polarity. Thus, it would explain why 2-HEMA is not a good effective promotor to dissolve PMMA. The second null hypothesis was rejected.

The mean tensile bond strength of the Kooliner groups was significantly higher compared with those of the Tokuyama Rebase II and the Ufi Gel Hard groups.

The molecular weight of the liquid part of reline materials plays a role in its viscosity. The Tokuyama Rebase II liquid contains AAEMA (214.21 Da) and 1,9 NDMA (296.40 Da) that are higher in molecular weight compared with the IBMA (142.20 Da) in the Kooliner, or the 1,6 HDMA (254.32 Da) in the Ufi Gel Hard. According to the diffusion theory, the higher the viscosity, the slower the material moves.[28] The high molecular weight of the components of the liquid monomer of Tokuyama Rebase II and Ufi Gel Hard retards the diffusion reaction in the polymerization process. Differential scanning calorimetry was used to calculate the exothermic energy of the 3 reline materials after mixing until the complete setting. The released energy of Kooliner, Tokuyama Rebase II and Ufi Gel Hard were 179.7, 121.7 and 150.5 J/g, respectively (Fig. 7). The heat generated during polymerization stimulates the rate of diffusion of the monomer molecules into the denture base material, enhancing the tensile bond strength. These two reasons, molecular weight of monomer and exothermic energy, account for the higher tensile bond strength of the Kooliner compared to other materials. The third null hypothesis was rejected.

For the failure patterns of specimens, the amount of tensile bond strength is positively related to the type of failure observed. From the correlation analysis, the higher tensile bond strength tends to be cohesive failure more than the mixed or the adhesive failure. However, this analysis cannot be applied to the relation between the mean tensile bond strength and the failure pattern in Kooliner group. The Kooliner group had a higher mean tensile bond strength compared with the two other materials, however, this group only exhibited mixed and adhesive failures. Previous studies have found in the same way with the failure result of this study that adhesive failure was generally occurred in the Kooliner specimens.[29-34] The Tokuyama Rebase II groups showed mixed and adhesive failures for all treatments except for the MF-MA 180 s group that showed cohesive failure. The Ufi Gel Hard groups showed all three failure types with cohesive failure in the MF-MA 180 s and the MMA 180 s groups. The non-harmonized mixing and the

powder-liquid ratio of the Tokuyama Rebase II and the Ufi Gel Hard might affect the failure results of these two materials by possibly creating voids in the reline materials. Once the test specimens were applied on the tensile force, it would be broken at the weakest area, sometimes at the void in the reline material.

Further researches using the flexural strength test, similar to the oral cavity condition, and thermocycling condition are required to confirm the effect of MMA and MF-MA solution on the bond strength between hard reline materials and a heated-polymerized acrylic denture base.
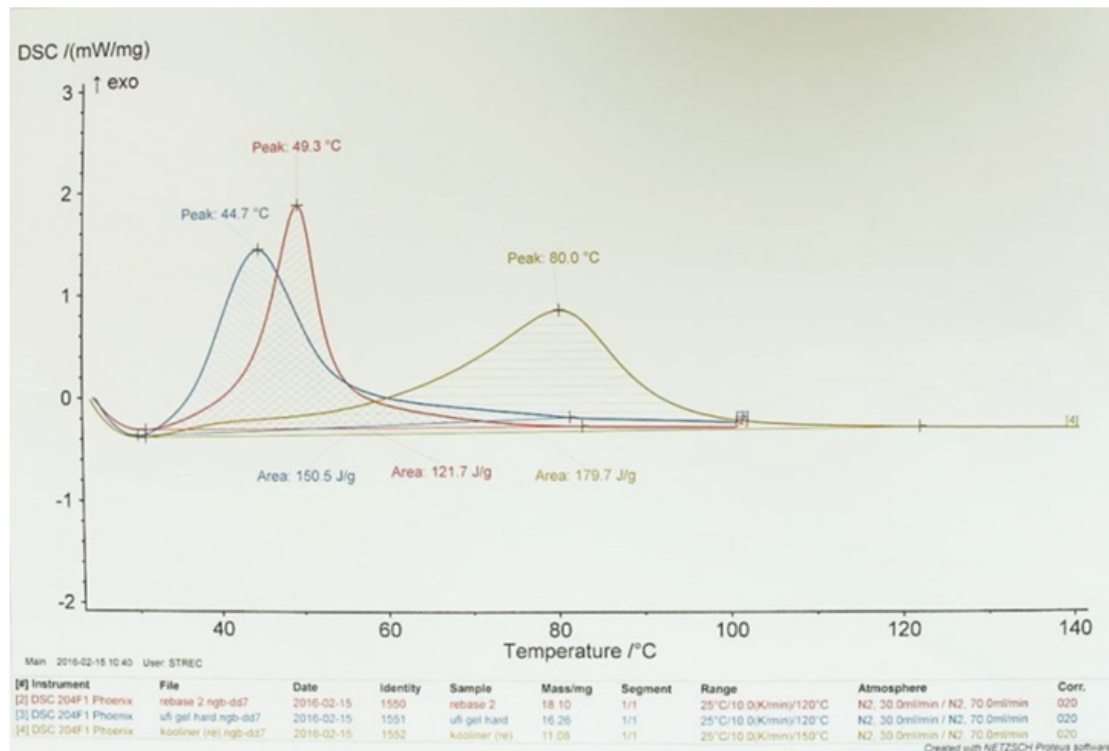

Figure 7 *Differential scanning calorimetry (DSC) analysis of each reline material (Kooliner in yellow line, Tokuyama Rebase II in red line and Ufi Gel Hard in blue line)*

Conclusion

Surface treatment with MF-MA solutions significantly increases the tensile bond strength between denture base resin and non-MMA based hard reline resins. This study suggests that a 15 s-MF-MA wetting time is adequate for creating a strong bond when using Kooliner as a reline material. MF-MA at a 180 s wetting time significantly enhances the tensile bond strength of the Tokuyama Rebase II fast and Ufi Gel Hard reline materials, and also reduces adhesive failure at the relined interface.

Clinical suggestions arising from this research

MF-MA solution is a solvent of choice in the surface treatment prior to relining denture base surface with a hard reline material.

Acknowledgments

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