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Original Article

Increased Wetting Time of Methyl Formate-Methyl Acetate did not Increase Tensile Bond Strength of Relined Denture Base Resin

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Abstract

Objective The interface between denture base and hard reline resin can be improved with chemical surface treatment, methyl formate-methyl acetate (MF-MA) solution, by dissolving the relined surface.This study evaluated the effect of various MF-MA wetting times on the tensile bond strength between a non-methyl methacrylate (MMA) based reline material and denture base.

Materials and Methods One hundred plates of heat-cured denture base resin (Meliodent®) were prepared according to ISO 10139-2 and randomly divided into five groups: control and four experimental groups treated with CU Acrylic Bond, MF-MA at a ratio of 25:75 by volume, for 15, 30, 60, and 180 s, respectively. The groups were applied with a non MMA-based hard reline material (Kooliner®) between two plates of denture base resin. The tensile bond strength tests were performed using a universal testing machine. Denture base specimens were analyzed by scanning electron microscopy (SEM) to determine the surface morphology. The data were analyzed using one-way ANOVA and post hoc Tukey's analysis at $p<0.05$.

Results The tensile bond strength of the experimental groups were significantly higher than that of the control group ($p<0.05$). There were no significant differences between the experimental groups ($p>0.05$). The SEM images of the denture base groups indicated that the denture base specimens treated with MF-MA demonstrated a porous appearance compared to the control group.

Conclusion Surface treatment with MF-MA increased the bond strength between denture base resin and hard reline material. However, the length of time of treatment had no effect on bond strength.

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Key words: *chemical surface treatment, denture base, methyl formate-methyl acetate, reline material, tensile bond strength*

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Introduction

Denture base is made from poly (methyl methacrylate) resin, which is formed by the polymerization of methyl methacrylate monomer to form long polymer chains (Powers and Wataha, 2008). The fabrication of a well-fitting denture base requires certain mechanical and physical properties. However, even initially well-fitting dentures become less so over time. This decrease in denture fit occurs because alveolar bone resorption is a continuous process due to tooth loss, causing the denture base to be less stable on the residual ridges (Tallgren, 1972). Poor fitting dentures affect patients both physically and socially. Loose dentures may drop when a person speaks and can cause pain at the residual ridge as well as chewing problems, which leads to poor nutrition. Therefore, a dental prosthesis should be examined periodically and treated to increase their adaptation to the oral tissue. Relining a denture base with a lining material is a common procedure to reproduce the original fit of the denture and improve masticatory function (Pisani et al, 2013). There are two main types of denture lining materials, which are classified by their consistency into soft and hard liners (McCabe, 1990). Soft liners are designed to use for reducing masticatory force to the residual ridge. These liners consist of plasticizers, which serve as stress absorbers between the denture and underlying tissue (Anusavice, 2003). However, prolonged exposure of soft reliners to water produced significantly higher hardness values and lower bond strength values (Mese and Guzel, 2008). Hard relining materials contain methyl methacrylate (MMA) or other types of monomer (Tallgren, 1972). MMA can dissolve and penetrate into the denture base forming an adhesive bond (Vallittu et al, 1994). After this type lining material sets, residual monomer can still leach out for a month causing oral tissue inflammation by direct contact (Lamb, 1938, Fisher, 1954, Guinta and Zablotsky, 1976). Non-MMA based lining materials have a large amount of cross-linking agents added to theirliquid part, which promotes greater transverse bending strength (Arima, 1995). The interface betweena relining material and denture base resin depends on the ability of the monomer in the lining resin to diffuse and penetrate into the denture base, forming inter-penetrating polymer networks (IPN) (Takahashi and Chai, 2001). Adhesive failure of the reline material promotes microleakage, which enhances staining and bacteria accumulation (Takahashi and Chai, 2001, Chai et al, 1998). Thus, surface treatment has been suggested to improve poor bonding (Minami et al, 2004, Mutluay and Ruyter, 2005, Takahashi and Chai, 2001). A previous study reported that chemical surface treatment increased flexural strength between autopolymerized resin and denture base, while mechanical surface treatment did not (Pereira, 2012). Chemical agents dissolve the denture base surface and improve diffusion of the reline resin monomer into the denture base (Vallittu, 1994, Mutluay and Ruyter, 2005). Chloroform and methylene chloride have been used as softening agents, providing a better bond between acrylic artificial teeth or repair materials and denture base (Shen et al, 1984, Rupp et al, 1971). Methyl formate and methyl acetate have been demonstrated to effectively promote adhesion, similar to methylene chloride (Asmussen and Peutzfeldt, 2000). The use of MMA monomer and chloroform provides a high bond strength when compared with acetone and isobutyl methacrylate monomer (Leles et al, 2001). However, chloroform and methylene chloride are carcinogenic substances, which should not be used in humans (Groger and Grey, 1979). A mixture of methyl formate-methyl acetate (MF-MA) monomer has been investigated in recent years, because it provides a high bond strength similar to that of MMA (Thunyakitpisal et al, 2011). A study has shown that an MF-MA solution at a ratio of 25:75 by volume significantly increased the shear bond strength between denture base resin and reline resin (Osathananda and Wiwatwarrapan, 2014). However, the effect of various MF-MA wetting times

on the tensile bond strength between a non MMA-based lining material and denture base has not yet been investigated. Therefore, the objective of this study was to evaluate the effect of various MF-MA wetting times on the tensile bond strength between a non-methyl methacrylate based reline material and denture base.

Materials and Methods

The specifics of the heat-cured acrylic resin, hard lining material, methyl formate and methyl acetate used in our study are shown in Table 1. The following procedure was performed according to ISO 10139-2 (International Standards Organization, 2009). One hundred plates of heat-cured acrylic resin (Meliodent®) (25±3 mm square and 3±0.5 mm thick) were prepared as recommended from ISO standard. The plates were finished with 500-grit silicon carbide paper using an automatic grinding and polishing unit (NANO 2000, Pace Technologies, USA). The plates were then stored in a water bath at 37±1°C for 28±2 days. After removing the plates from the storage water, the denture base plates were randomly divided into five groups: control (group I) and four experimental groups (groups II-V). The specimens in groups II-V were treated on the surface to be relined with CU Acrylic Bond, methyl formate-methyl acetate (MF-MA) solution at a ratio of 25:75 (by volume) for 15, 30, 60, and 180 s, respectively. Two plates of heat-cured acrylic resin were used to form one test specimen, which was separated by a 10 mm inner diameter and 3 mm thick PTFE collar containing the non MMA-based hard reline resin (Kooliner®). For each group, ten specimens were constructed using a split metal mold (Fig. 1) at room temperature and stored in a water bath at 37±1°C for 23±1 hrs. Each of the 50 test specimens was attached to a Universal testing machine (8872, INSTRON, UK) in a vertical alignment. The tensile bond strength (B) was measured at a crosshead speed of 10 mm/min. The following equation was used to calculate the tensile bond strength: $B = F/A$. Where F is the maximum load (N) before debonding occurred and A is the adhesive area (mm^2).

The data were analyzed using SPSS software version 17.0 (SPSS Inc., Chicago, IL, USA). The results were statistically analyzed by one-way *ANOVA* and *post hoc* Tukey's analysis. Statistical significance was set at $p<0.05$.

Table 1 Type of each material and their manufacturer.

Product name	Material	Manufacturer	Composition	
			Powder	Liquid
Meliodent	Heat-activated acrylic resin	Tokuyama Dental Corp., Japan	PMMA	MMA
Kooliner	Self-cured hard reline	GC America, USA	PEMA	IBMA
CU Acrylic Bond	Surface treatment agent	Faculty of Dentistry, Chulalongkorn University, Thailand		MF:MA=25:75

PMMA, Poly(methyl methacrylate); MMA, Methyl methacrylate; PEMA, Poly(ethyl methacrylate); IBMA, Isobutyl methacrylate.

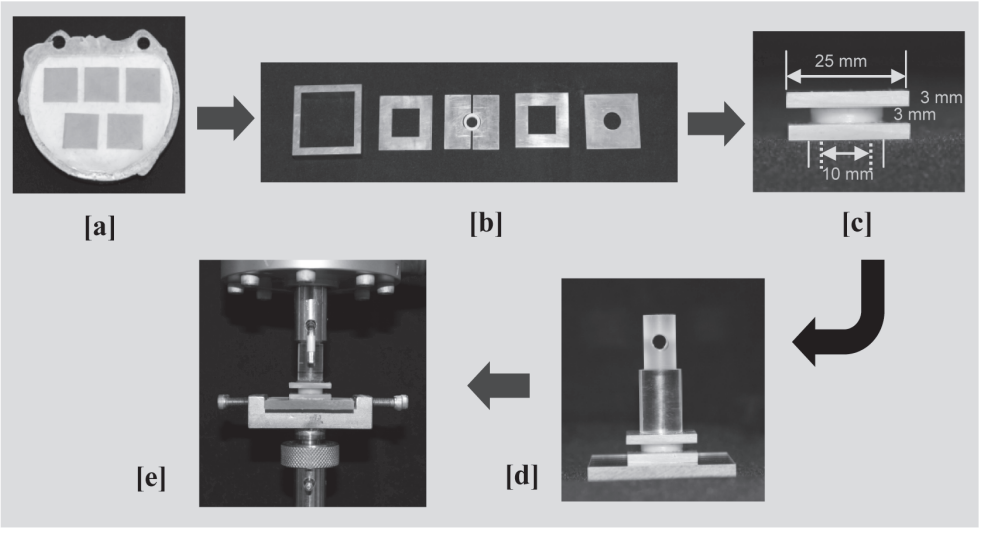

Fig. 1 Specimen preparation. [a] heat-cured denture base in a dental flask, [b] metal split mold, [c] test specimen, [d, e] test specimen in vertical alignment.

Table 2 Mean tensile bond strength with standard deviation of the different MF-MA wetting time groups.

Group	Wetting time with MF-MA (s)	Mean tensile bond strength ± SD (MPa)
I	0	4.94 ± 0.75^{a}
II	15	7.38 ± 0.40^{b}
III	30	7.82 ± 0.92^{b}
IV	60	7.50 ± 0.68^{b}
V	180	7.98 ± 0.54^{b}

The same superscript letter indicates no significant difference ($p>0.05$).

Results

The mean tensile bond strength and standard deviation of each group are presented in Table 2. The tensile bond strength of the experimental groups were significantly higher than that of the control group ($p<0.05$). The highest tensile bond strength was found in group V (180 s wetting time), however,there were no significant differences between the experimental groups ($p>0.05$).

The SEM images of denture base treated with MF-MA solution at a ratio of 25:75 for 15, 30, 60, and 180 seconds and no treatment are shown in Figure 2. The image of the non-treated denture base surface (Fig. 2a) shows parallel scratches in one direction because of the roughness of the silicon carbide paper and the specimen was polished in one direction. The denture base surfaces treated with MF-MA (Fig. 2b-e) demonstrated numerous porosities compared with the non-treated surface. The SEM appearance of the specimens treated with different wetting times showed different sizes and patterns of pores.

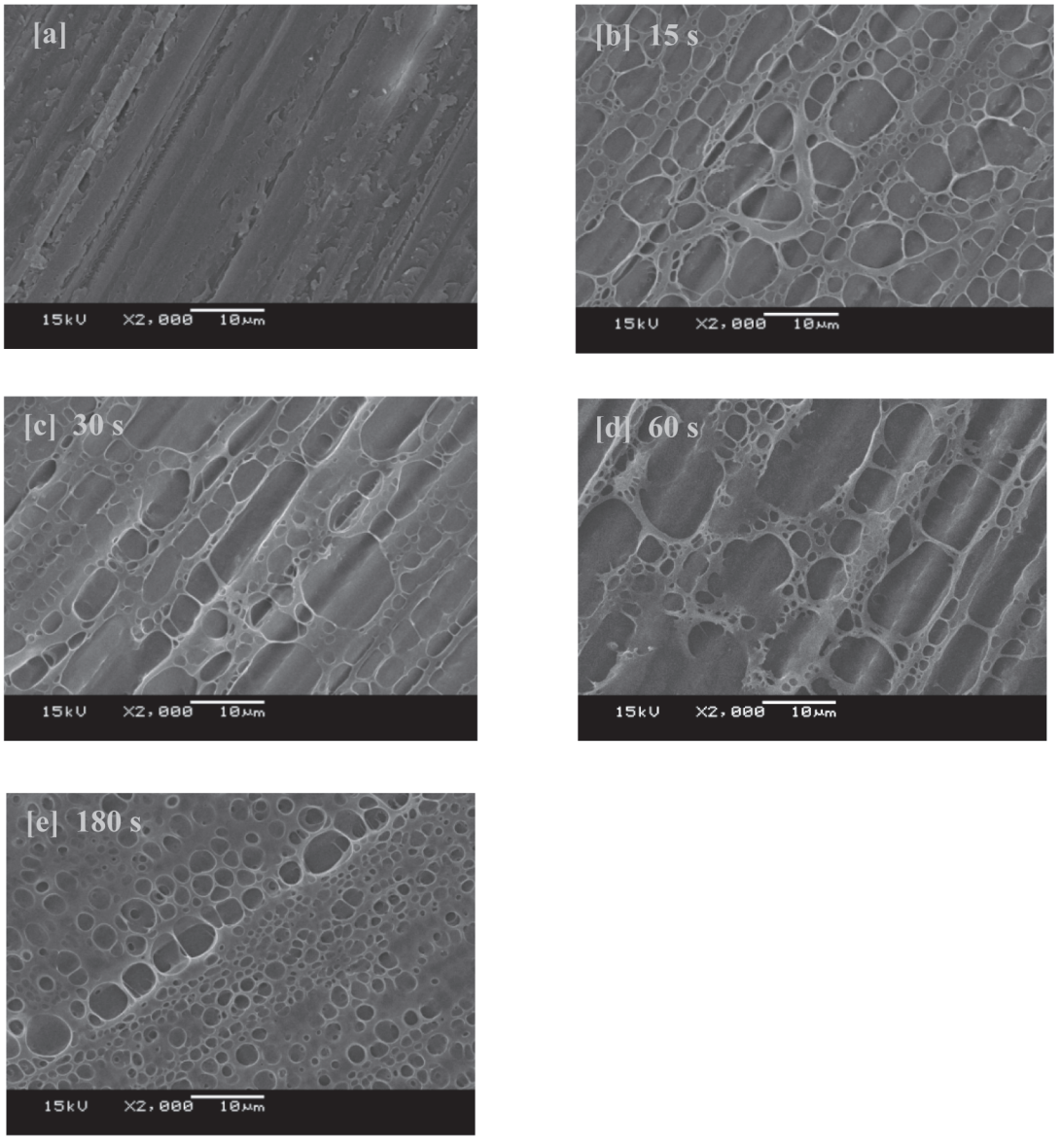

Fig. 2 SEM analysis of the surface of heat-cured denture base resin. [a] no treatment, [b] MF-MA solution 15 s, [c] 30 s, [d] 60 s, and [e] 180 s, respectively.

Discussion

In the present study, the tensile bond strength of relined denture surfaces was compared when treated with MF-MA using wetting times of 15, 30, 60, 180 s, and no treatment. These wetting times were selected based on a previous study that found that increased MMA wetting caused increased thickness of the swollen layer at the denture base surface (Vallittu and Ruyter, 1997). Vallittu et al concluded that a MMA wetting time of 180 s was recommended to strengthen repaired acrylic resin (Vallittu et al, 1994). For this reason, we used wetting times ranging from 15 s to 180 s to observe the trend of tensile bond strength. We found that prolonged MF-MA exposure time did not result in significantly greater tensile bond strength.

The results of the present study showed that the mean tensile bond strength of the MF-MA treated groups were higher than that of the untreated group,

which is in accordance with previous studies (Thunyakitpisal et al, 2011, Osathananda and Wiwatwarrapan, 2014). These finding can explained by the solubility parameters and polarities of PMMA and the solvent, which should be near each other for efficiency of dissolution (Asmussen and Peutzfeldt, 2000). The solubility parameters of PMMA, MF, and MA are 18.3, 20.9, and 19.6 $MPa^{1/2}$, respectively (Grulke, 1999). Moreover, MF and MA have methyl ester groups that enhance their ability to soften PMMA (Grulke, 1999). In addition, the low molecular weight of MF (60.05 Da) and MA (74.08 Da) promotes greater solubility to the denture base (Evchuk et al, 2005). The MF-MA solutions were observed to evaporate with no remaining on the bonding surface after their application. The molecular structure of methyl formate and methyl acetate shows that they do not have carbon-carbon double bonds (C=C), which could cause polymerization with the monomer in autopolymerized reline denture materials. Thus, there was neither residual monomer nor by-products to obstruct the bonding area. The bonding interface between denture base and reline resin was created by dissolution with MF-MA, causing porous layers on the denture base surface. These pores allowed the monomer of the reline material to penetrate, then polymerize tocreate a mechanical interlocking bond at the molecular level between the heat polymerized denture base and infiltrated autopolymerized reline material.

The molecular mechanism of MF-MA treatment corresponds with our SEM findings, which demonstrated that many porosities were formed by wetting with MF-MA. Although different patterns of porosities were found in each experimental group, the mean tensile bond strengths were not significantly different to each other. This suggested that the size and pattern of the porosities were not related to the tensile bond strength.

Conclusion

Surface treatment with MF-MA solutions generated higher bond strength between denture base resin and hard reline materials, compared with no treatment. MF-MA wetting time as short as 15 s is practically appropriate to be used prior to lining a denture base with a reline material to improve bond strength.

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