ระยะเวลาในการปรับสภาพผิวด้วยสารละลายเมทิลฟอร์เมต-เมทิลอะซิเตต ที่มีผลต่อความแข็งแรงพันธะดึงระหว่างฐานฟันเทียมอะคริลิกเรซิน และเรซินเสริมฐานแบบแข็งชนิดส่วนประกอบพื้นฐานไม่เป็นเมทิลเมทาคริเลต

นางสาวชลิตา ธนะสมานโชค

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
สาขาวิชาทันตกรรมประดิษฐ์ ภาควิชาทันตกรรมประดิษฐ์
คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2558

WETTING TIME OF METHYL FORMATE-METHYL ACETATE SOLUTION AFFECTS THE TENSILE BOND STRENGTH BETWEEN DENTURE BASE RESIN AND NON MMA-BASED HARD RELINE RESINS

Miss Chalita Tanasamanchoke

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Prosthodontics
Department of Prosthodontics
Faculty of Dentistry
Chulalongkorn University
Academic Year 2015

Thesis Title	WETTING TIME OF METHYL FORMATE-METHYL ACETATE SOLUTION AFFECTS THE TENSILE BOND STRENGTH BETWEEN DENTURE BASE RESIN AND NON MMA-BASED HARD RELINE RESINS
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ชลิตา ธนะสมานโชค : ระยะเวลาในการปรับสภาพผิวด้วยสารละลายเมทิลฟอร์เมต-เมทิลอะซิเตตที่มีผลต่อความแข็งแรงพันธะดึงระหว่างฐานฟันเทียมอะคริลิกเรซินและเรซินเสริมฐานแบบแข็งชนิดส่วนประกอบพื้นฐานไม่เป็นเมทิลเมทาคริเลต (WETTING TIME OF METHYL FORMATE-METHYL ACETATE SOLUTION AFFECTS THE TENSILE BOND STRENGTH BETWEEN DENTURE BASE RESIN AND NON MMA-BASED HARD RELINE RESINS) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ชัยรัตน์ วิวัฒน์วรพันธ์, หน้า.

งานวิจัยนี้ เป็นการศึกษาผลของระยะเวลาในการปรับสภาพผิวด้วยสารละลายเมทิลฟอร์เมต-เมทิลอะซิเตต ที่มีผลต่อความแข็งแรงพันธะดึงระหว่างฐานฟันเทียมอะคริลิกเรซิน และเรซินเสริมฐานแบบแข็ง ชนิดส่วนประกอบพื้นฐานไม่เป็นเมทิลเมทาคริเลต โดยเตรียมชิ้นงานอะคริลิกชนิดบ่มด้วยความร้อน (Meliodent®) แบ่งออกเป็น 3 กลุ่มตามวัสดุเสริมฐานชนิดแข็ง (Kooliner®, Tokuyama® Rebase II และ Ufi Gel Hard®) แต่ละสารมีรูปแบบการปรับสภาพพื้นผิว ดังนี้ กลุ่มควบคุม (ไม่ทาสาร) ทาด้วยสารยึดติด (จากบริษัท) ทาด้วยสารละลายเมทิลฟอร์เมต-เมทิลอะซิเตตเป็นเวลา 15, 30 60 และ 180 วินาที และทาด้วยสารเมทิลเมทาคริเลต 180 วินาที นำชิ้นงานไปทดสอบด้วยเครื่องทดสอบแรงดึงแรงอัดระบบไฮดรอลิก วิเคราะห์ข้อมูลทางสถิติด้วยการวิเคราะห์ความแปรปรวนทางเดียวและ Tukey HSD ผลการทดลอง พบว่า ค่าเฉลี่ยความแข็งแรงพันธะดึงของกลุ่มที่ได้รับการปรับสภาพผิวด้วยสารต่างๆ มากกว่ากลุ่มควบคุมอย่างมีนัยสำคัญทางสถิติ ที่ระดับความเชื่อมั่น 95% สำหรับ Kooliner ค่าเฉลี่ยความแข็งแรงพันธะดึงของกลุ่มที่ได้รับการปรับสภาพผิวด้วยสารต่างๆ ไม่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติ ที่ระดับความเชื่อมั่น 95% สำหรับ Tokuyama® Rebase II และ Ufi Gel Hard® การปรับสภาพผิวด้วยสารละลายเมทิลฟอร์เมต-เมทิลอะซิเตตเป็นระยะเวลา 180 วินาที ให้ค่าเฉลี่ยความแข็งแรงพันธะดึงสูงที่สุด อย่างมีนัยสำคัญทางสถิติ ที่ระดับความเชื่อมั่น 95% ดังนั้น การปรับสภาพผิวด้วยสารละลายเมทิลฟอร์เมต-เมทิลอะซิเตต สามารถเพิ่มค่าความแข็งแรงพันธะดึงให้กับการยึดติดระหว่างฐานฟันเทียมอะคริลิกเรซิน และเรซินเสริมฐานแบบแข็ง ชนิดส่วนประกอบพื้นฐานไม่เป็นเมทิลเมทาคริเลตได้ ส่วนระยะเวลาที่เหมาะสมในการทาสารนี้กับ Kooliner® คือ 15 วินาที สำหรับ Tokuyama® Rebase II และ Ufi Gel Hard® คือ 180 วินาที

ภาควิชา	ทันตกรรมประดิษฐ์	ลายมือชื่อนิสิต	....
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ปีการศึกษา	2558		

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KEYWORDS: TENSILE BOND STRENGTH, METHYL FORMATE, METHYL ACETATE, CHEMICAL SURFACE TREATMENT, DENTURE BASE, RELINE MATERIAL

CHALITA TANASAMANCHOKE: WETTING TIME OF METHYL FORMATE-METHYL ACETATE SOLUTION AFFECTS THE TENSILE BOND STRENGTH BETWEEN DENTURE BASE RESIN AND NON MMA-BASED HARD RELINE RESINS. ADVISOR: ASSOC. PROF. CHAIRAT WIWATWARRAPAN, pp.

This study investigated the effect of 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 of hard reline resins (Kooliner®, Tokuyama® Rebase II and Ufi Gel Hard®). Each group of reline material consists of 6 - 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. The data were analyzed using one-way ANOVA and *post hoc* Tukey's analysis at $p<0.05$. The mean TBS of the treated groups were significantly higher compared with those of the control group ($p<0.05$). In the Kooliner® group, there were no significant differences in TBS between the MF-MA and MMA treatment groups ($p>0.05$). In the Tokuyama® Rebase II group, application with MF-MA solutions for 180 s produced the significantly 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-based 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.

Department: Prosthodontics Student's Signature ....

Field of Study: Prosthodontics Advisor's Signature ....

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CHAPTER I

INTRODUCTION

Background and rational

A person's oral health affects their general health. Poor oral health can cause considerable pain and suffering in the mouth, and also by change a person's diet, speech and quality of life. Proper oral hygiene (e.g. brushing, flossing, and professional care) can maintain the teeth and gums in a healthy condition throughout one's life. However, oral problems still occur in people of all ages that result in tooth loss. The 7th Thailand National Oral Health Survey (2012) found that tooth loss was the main oral health problem in elderly people.[1] This report indicated that 88.3% of people age 60-74 were partially edentulous and 7.2% were fully edentulous. In addition, the report indicated that up to 32.2% of people age 80-89 were fully edentulous. Thus, the Ministry of Public Health initiated a dental eldercare policy that included the fabrication of dentures to be performed by dentists working at public hospitals in all districts of Thailand. Once a patient has been wearing their dentures for many years, they usually complain that the prosthesis no longer fits well. An ill-fitting denture occurs because tooth loss results in continuous alveolar bone resorption, causing the denture base to be less stable on the residual ridges.[2] A poorly fitting denture affects a patient both physically and socially. Poor fitting dentures may drop when a person speaks, and they can also cause residual ridge pain and chewing problems leading to poor nutrition. Therefore, a denture should be examined periodically and a proper fit to underlying tissues should be re-established using reline material to increase its tissue adaptation.

Research questions

1. Does surface treatment with methyl formate-methyl acetate affect the tensile bond strength between non methyl methacrylate (MMA) based lining materials and denture base resin?

2. What is the most appropriate MF-MA wetting time for enhancing the tensile bond strength between non MMA-based lining materials and denture base resin?

Research objective

To evaluate the effect of various MF-MA wetting times on the tensile bond strength between three non MMA-based reline materials and denture base resin.

Hypothesis

$H_0$1 : There is no significant difference in tensile bond strength between MF-MA-treated and non-surface treated denture base with non MMA-based hard reline resin.

H_a1 : There is significant difference in tensile bond strength between MF-MA-treated and non-surface treated denture base relined with non MMA-based hard reline resin.

$H_0$2 : There is no significant difference in tensile bond strength between denture base relined with non MMA-based hard reline resin when the duration of MF-MA application varies.

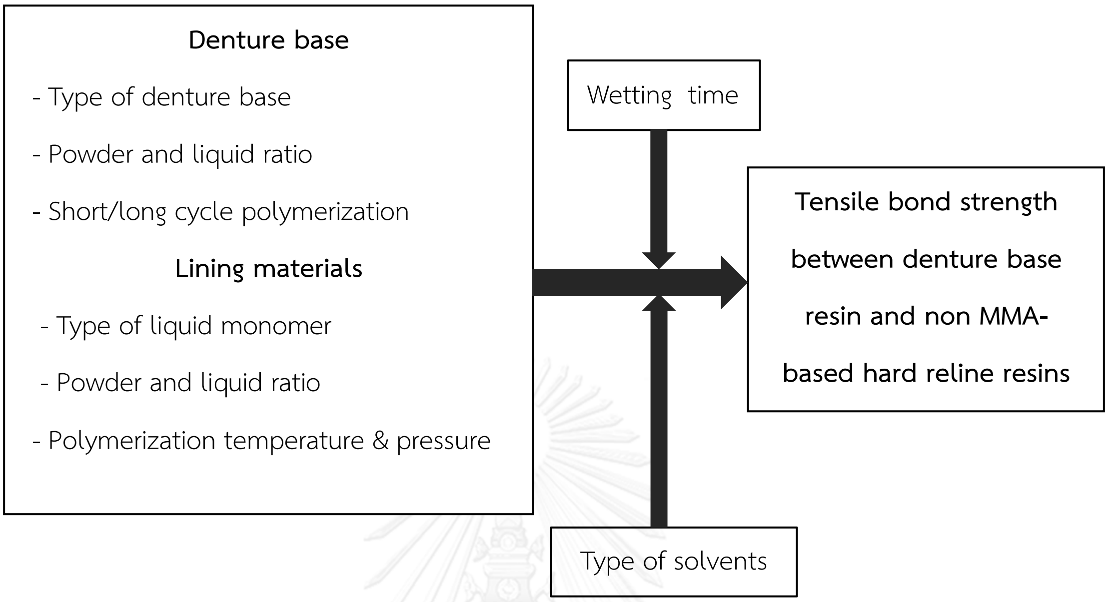
H_a2 : There is a significant difference in tensile bond strength between denture base relined with non MMA-based hard reline resin when the duration of MF-MA application varies.

For all hypotheses, the significance level α is 0.05

Limitation

1. This was an in vitro study, following by previous protocol, which might not adequately simulated oral conditions.
2. The materials used in this study were Meliodent® (Heat-cured acrylic resin), Kooliner®, Tokuyama® Rebase II Fast and Ufi Gel Hard® (hard lining materials) that are commonly used in prosthodontic treatment.
3. A single investigator performed all experiments and evaluations.

Conceptual framework



CHAPTER II

LITERATURE REVIEW

Denture base polymers

Before 1940, vulcanite was used to fabricate denture base polymers. Because valcanite contains highly cross-linked natural rubber, it is hard to add color to and is prone to accumulate bacteria. Currently, acrylic resin is used worldwide rather than vulcanite when fabricating a denture base.[3] Denture bases are made from poly(methyl methacrylate) resins that are constructed by polymerization of monomers to form polymer chain.[4] In 1963, Bowen introduced adding cross-linking monomers (e.g. dimethacrylate monomer) to the monomer liquid of denture base resins.[5] This method resulted in the formation of a multiphase in denture base polymers, the so called 'Interpenetrating Polymer Networks (IPNs)'. IPNs are a combination of two or more polymers in network form that are synthesized juxtaposed to each other.[6] IPNs are composed of finely divided 5-10 nm phases. However, IPNs are not joined by chemical bonds at the molecular scale. Several advantages of the IPNs-like structures in dental materials are improved toughness and increased mechanical interlocking at the nanometer level.

Requirements of a denture base polymer

An ideal denture base polymer should include the following properties.[3]

1. Physical properties
    - Match the appearance of the oral soft tissue
    - Higher glass transition temperature (Tg) than normal mouth temperature and hot food
    - Good dimensional stability
    - Low specific gravity
    - Thermal conductivity
    - Radiopacity
2. Mechanical properties

- High modulus of elasticity and elastic limit
- Sufficient flexural strength and impact strength to resist fracture
- Adequate fatigue life and high fatigue limit
- Sufficient abrasive resistance

3. Chemical properties
    - Chemically inert
    - Low water and saliva sorption
4. Biological properties
    - Non-toxic to technician and patient
    - Impermeable to oral fluid
5. Miscellaneous properties
    - Inexpensive
    - Long shelf life
    - Easy to manipulate

Classification of denture base polymers

Denture base polymers classified by ISO 20795.[7] (Table 1)

Table 1. Classification of denture base polymers.

Type	Class	Description
1	1	Heat-processing polymers, powder and liquid
1	2	Heat-processing (plastic cake)
2	1	Autopolymerized polymers, powder and liquid
2	1	Autopolymerized polymers (powder and liquid pour type resin)
3	-	Thermoplastic blank or powder
4	-	Light-activated materials
5	-	Microwave-cured materials

Heat activated denture base resins

Denture base fabrication typically uses a heat-activated process, where heat (e.g. from microwave or water bath) increases polymerization of these materials).[8] The stages of the PMMA polymerization reaction are shown in Figure 1.

Initiation

This step requires an activator (e.g. heat or light) to produce free radicals, which initiate polymer chain formations.

Propagation

The polymer free radicals react with available double-bond-containing monomers to lengthen the chains.

Termination

As a result of propagation, large amount of polymer chains are created, resulting in branched and cross-linked networks. In addition, tetramethylene dimethacrylate, which is a cross-linking agent, has two double bonds per molecule and it can cross-link to two polymer chains, forming a netlike structure (Figure 2).

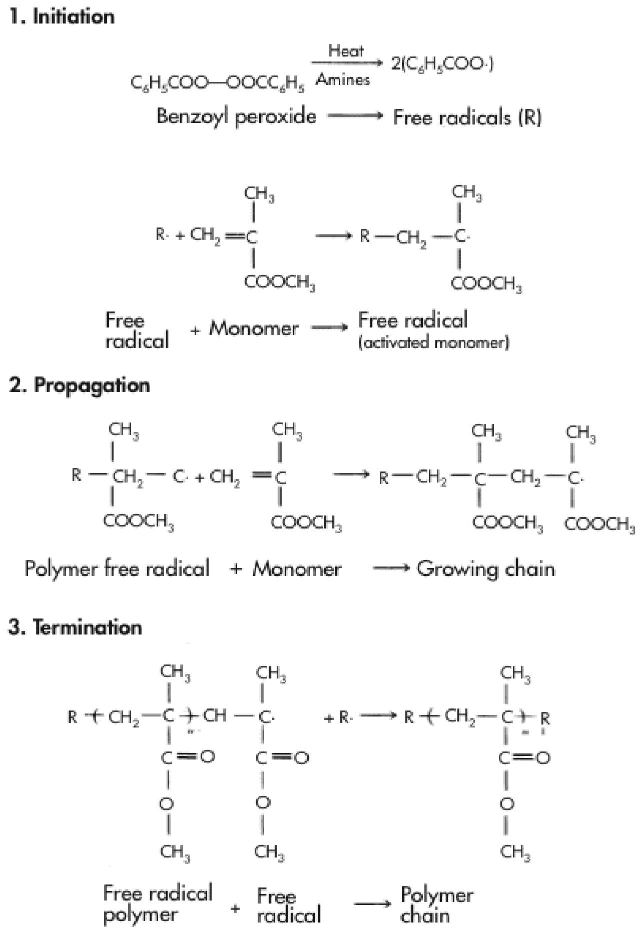


Figure 1. The three steps of poly(methyl methacrylate) polymerization.

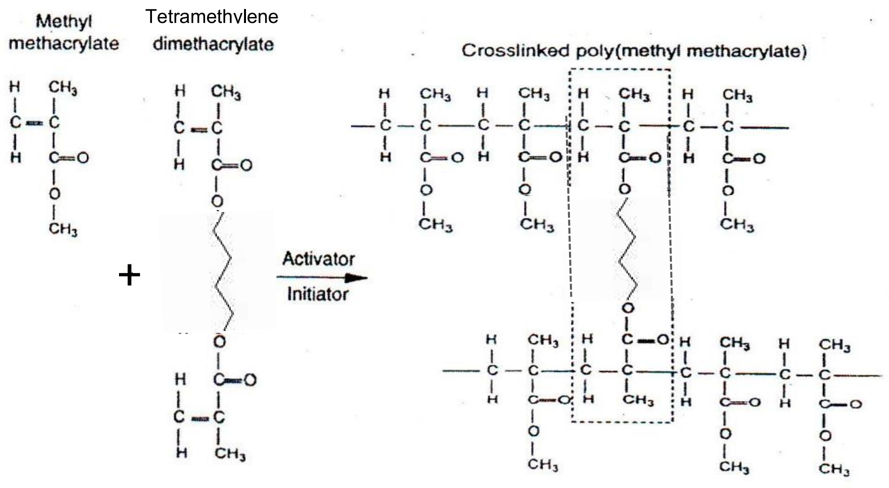


Figure 2. The cross-linking formation of tetramethylene dimethacrylate into a polymer chain.

The polymerization reaction ultimately results in polymer chains and unreacted monomers, known as residual monomers. The amount of polymerized monomer is measured by the ‘degree of conversion’. Polymers with a high degree of conversion have a low level of residual monomers.[4]

Self-cured acrylic resin

The main component of self-cured acrylic resin liquid is similar to that of heat-cured acrylic resin except that tertiary aromatic amine is added to the self-cured acrylic resin liquid. The polymerization process begins with monomer dissolving the polymer beads and the beads become swollen. The tertiary aromatic amine reacts with benzoyl peroxide at room temperature. Subsequently, the monomers link to form a polymer chain, resulting in the higher viscosity. Several factors, such as size, molecular weight and plasticizer, affect with the penetration of the resin into the repaired denture or plastic tooth. A low viscosity consistency is required for high penetration. However, this type of resin also has a large amount of residual monomer, which decreases the mechanical properties of the denture base.[3, 4, 8]

Acrylic resin material composition

Most acrylic resins are composed of powder and liquid parts.[3]

Powder

A major component of the acrylic resin powder is poly(methyl methacrylate) (PMMA), a glass-like polymer, that is molded into 100 μm diameter beads.

Polymer: PMMA beads

Initiator: Peroxide such as benzoyl peroxide (approximately 0.5%)

Pigments: cadmium salts, iron or organic dyes

Liquid

A major component of the acrylic resin liquid is methyl methacrylate (MMA) monomer. This monomer is clear, colorless, and has a pungent odor and low viscosity with a boiling point of 100.3°C.

Monomer: MMA

Cross-linking agent: ethylene glycol dimethacrylate (approximately 0.5%)

Inhibitor: Hydroquinone (trace)

Activator: *N*,*N*-dimethyl-*p*-toluidine (approximately 1%) – only in self-curing materials

Denture lining materials

Relining a denture base with lining materials is a common procedure to restore the initial oral tissue fit of the denture and improve masticatory function.[9] This technique can be performed in a patient's mouth (with self-cured resins) or in a laboratory (with heat-cured resins). The lining materials are recommended to be applied when they are still viscous and the relined denture should then be inserted into the mouth using a 'closed mouth technique' where the patient's is asked to gently close their mouth until the opposing teeth are in contact.[3] Direct relining with a self-cured acrylic resins is faster than the laboratory procedure, and can reproduce the morphologic features of the oral tissue directly on the denture base.[10] The laboratory relining method involves an extra patient visit, laboratory fee and causes the patient to be without their denture for a period of time.[11]

Lining materials are differentiated by their consistency into two types, hard and soft liners.

Hard liners

Hard liners usually used to improve the fit of unstable dentures. The two types of hard liners are distinguished by containing MMA in the liquid part or containing other monomer types.[4]

Type I (MMA-based)

MMA can dissolve and penetrate into the denture base forming an adhesion between the relining material and the denture base. After the lining materials set, residual monomer leaches out for up to a month causing inflammation by direct contact of monomer with the oral tissue.[12-14] Thus, protecting the oral tissue with petroleum jelly before a relining procedure is recommended. The composition of the MMA-based hard liner material is shown in Table 2.

Table 2. Composition of the MMA-based hard liner material.

Powder	Liquid
Powder beads: PMMA Initiator: benzoyl peroxide	Monomer: MMA Plasticizer: di-*n*-butylphthalate Chemical activator: tertiary amine

Type II (Non MMA-based)

Non MMA-based lining materials have a large amount of cross-linking agents added to their liquid part, which promotes greater transverse bending strength.[15] However, the high molecular weight cross-linking agents have less monomer penetration, resulting in a weak bond.[16, 17] This non-MMA material is safer than MMA-type because when polymerized, it has less residual monomer. The composition of the non MMA-based hard liner material is shown in Table 3.

Table 3. Composition of the non MMA-based hard liner material.

Powder	Liquid
Powder beads: poly(ethyl methacrylate) Initiator: benzoyl peroxide	Monomer: butyl methacrylate or isobutyl methacrylate or some other higher methacrylate monomer Cross-linking agent: di-methacrylate Chemical activator: tertiary amine

Soft liners

Soft liners are used in patients with bony undercuts on their edentulous ridge or as a treatment denture after oral surgery. These liners are also fabricated as obturators for cleft palate patients. Soft liners are classified as short and long-term materials. Short-term materials have plasticizers that leach out of the material after a few days and the denture then becomes harder. Therefore, short-term soft liners should be replaced every 3 days. Long-term materials consist of methacrylate-based or silicone-based polymers. Methacrylate-based materials increase their hardness by slowly releasing plasticizers. Silicone-based materials have an advantage of not hardnening, because they lack plasticizers.[4]

Bond strength evaluation between these two types of lining materials indicated that soft liners had significantly lower bond strength compared with hard liners materials.[18] The bond strength between a relining material and denture base resin affects the mechanical strength of the relined denture base.[19] The interface between a reline material and denture base resin depends on the ability of the monomers in the relining resin to diffuse and penetrate into the denture base, forming IPN.[6, 20] Failure of adhesion between a reline material and denture base resin promotes microleakage, enhancing staining and bacteria accumulation.[19, 20] Thus, surface treatment has been suggested to improve poor bonding by relining material (e.g. mechanical and chemical surface treatment).[16, 21, 22]

Surface treatment

Surface treatment has been suggested to improve poor bonding between a reline material and denture base resin. This treatment can be classified into mechanical and chemical treatment.

Mechanical treatment

Mechanical surface treatment (e.g. polishing with silicon carbide paper or air abrasion) increases the bond strength of relined denture by removing contaminants and preparing a rough surface for proper mechanical retention.[22] Preparing the denture base surface with a bur also increases surface roughness that can result in a higher bond strength, depending on the bur type used.[23] However, roughening the surface with a bur can creates microvoids and overhanging grooves covered with debris that reduce adhesion.[24] The use of plasma treatment to modify the surface of a denture base has been studied. It was found that plasma treatment increased shear bond strength. This is because plasma treatment improves the wettability of PMMA denture base by reducing the water contact angle.[25] However, preparation of a denture base surface with sandblasting and carbon dioxide laser are not effective in improving the adhesion between resilient liners and denture bases.[26] In contrast, the application of a bonding agent on artificial teeth increased the bond strength to the denture base, whereas the use of an Er:YAG laser was not effective.[27]

Chemical treatment

Pereira et al. found that chemical surface treatment increased the flexural strength between the denture base and reline material, while mechanical surface treatment had no effect on flexural strength.[28] Application of a chemical agent dissolves the denture base surface and improves the diffusion of reline resin monomer into the denture base.[21, 29]

- Chloroform and methylene chloride

For the last 40 years, chloroform and methylene chloride have been used as softening agents, providing a better bond between acrylic artificial teeth or repair materials and denture bases.[24, 30, 31] However, chloroform and methylene chloride are now known to be carcinogenic, and should not be used in patients.[32, 33]

- Methyl methacrylate

The use of MMA monomers or chloroform results in a higher bond strength compared with the use of acetone or isobutyl methacrylate monomer.[34] A prolonged wetting time of MMA provides a strong bond between the repaired surface and self-cured acrylic resin.[29] Vallittu et al. reported that 180 s of MMA exposure reduced adhesive failure.[17]

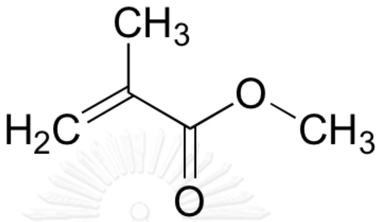
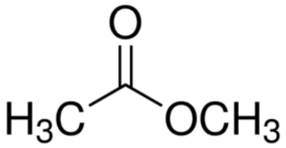


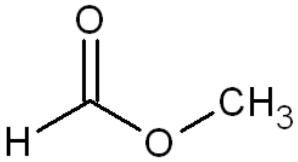
Figure 3. Methyl methacrylate chemical structure.

- Methyl formate and methyl acetate

Methyl formate and methyl acetate have been demonstrated to effectively promote adhesion, similar in effect to methylene chloride in dissolving PMMA.[35]



Methyl formate



Methyl acetate

Figure 4. Methyl formate and methyl acetate chemical structure.

- Methyl formate-methyl acetate (MF-MA) solutions

The use of a mixture of MF-MA monomer has been investigated in recent years, finding that this mixture generated a high bond strength similar to that of MMA treatment.[36] Considering the ratio of MF-MA solutions, Ratchanee et al. demonstrated that an MF-MA ratio of 25:75 significantly increased the bond strength between denture base resin and reline resins than those of other ratios.[37] Scanning electron microscopy (SEM) evaluation found that denture base surfaces treated with MF-MA at a ratio of 25:75 had the largest pore sizes when compared with other ratios.[37] Furthermore, larger pore sizes in the denture base surface generated the greatest bond strength with the reline resins.[36, 37] However, only MMA-based lining materials were evaluated in these two studies. The effect of various MF-MA wetting times on the tensile bond strength between non MMA-based lining materials and denture base has not yet been investigated.

CHAPTER III

METHODOLOGY

Materials

The materials used in this study and their details are shown in Table 4.

- Heat-cured acrylic resin: Meliodent®
- Reline materials (non MMA-based): Kooliner®, Tokuyama® Rebase II fast and Ufi Gel Hard®
- Methyl formate-methyl acetate solution (ratio of 25:75 by volume)

Table 4. Trade name, manufacturer and chemical composition of 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-acetate 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

Instruments

2.1 Curing unit, EWL 5518 (Kavo, Germany)

2.2 General incubator, Contherm 160M (Contherm Scientific Ltd., New Zealand)

2.3 Hydraulic flask pressure, 5414 EWG (Kavo, Germany)

2.4 Compressed air-driven deflasking unit (Renfert, Germany)

2.5 Polishing machine, NANO2000 (Pace Technologies, USA)

2.6 Digital Vernier caliper (Mitutoyo Corporation, Japan)

2.7 Universal Testing Machine, 8872 (INSTRON, UK)

2.8 Stereo Microscope SZ61 (OLYMPUS, China)

2.9 metal split mold

Sample preparation

The method of this study mainly followed ISO10139-2:2009(E).[38] Four hundred heat-cured acrylic resin plates were prepared (25±3 mm^2 and 3±0.5 mm thick) as recommended by the manufacturer. The plates were finished with silicon carbide paper (P500) using an automatic grinding and polishing unit. A digital vernier caliper was used to verify samples' dimensions after polishing. The plates were stored in a water bath at 37±1°C for 28±2 days. The surface of each heat-cured acrylic plate was using stereo microscope before receiving surface treatment. Next, the samples were randomly divided into three groups of self-cured acrylic resin [Group I: Kooliner® (n=60), Group II: Tokuyama® Rebase II fast (n=70), Group III: Ufi Gel Hard® (n=70)]. Each group consists of six to seven subgroups (n=10), differentiated by surface treatment (Figure 5).

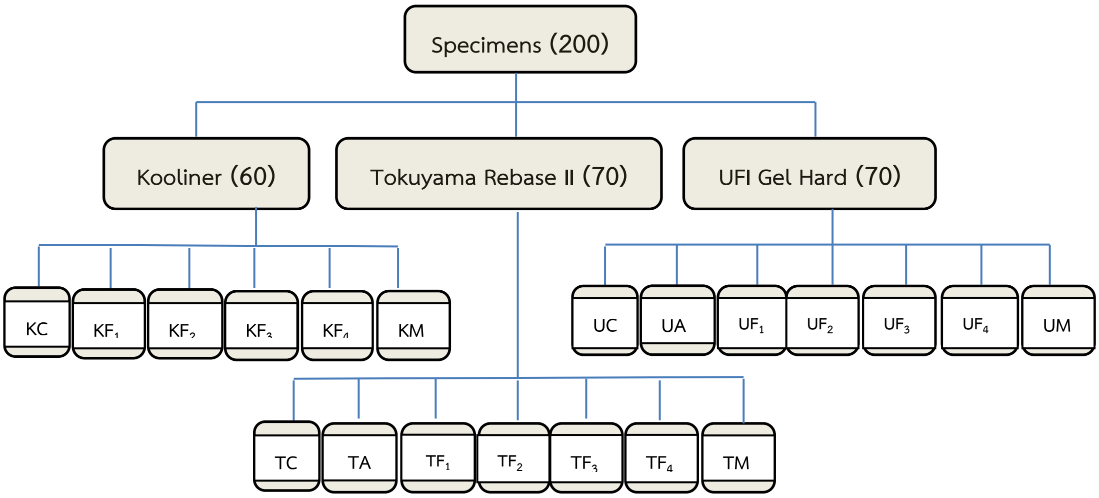


Figure 5. The distribution of the specimens from each material .

Key

“K”: Kooliner®, “T”: Tokuyama® Rebase II fast, “U”: Ufi Gel Hard®.

“C”: negative control groups (3 groups). These groups were not treated with any solution on the bonding surface, only lined with the three lining materials.

“M”: positive control groups (3 groups). These groups were 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 was applied before the relining material was loaded. Kooliner® does not use 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 relining material.

The specimens were constructed in a metal split mold (Figure 6[B]) at room temperature. A bond area was controlled by Teflon collar in 10 mm diameter and 3 mm height. Two plates of heat-cured acrylic resin that were separated by hard lining material were used to form one test specimen that was pressed with a 4 kg metal pendulum, simulating complete denture bite force.[39] After the hard lining material was set, the test specimens 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 (Figure 7). The tensile bond strength was measured by a Universal testing machine with crosshead speed 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 is the tensile bond strength in MPa, F is the maximum load (N) before debonding occurred and A is the adhesive area (mm^2).

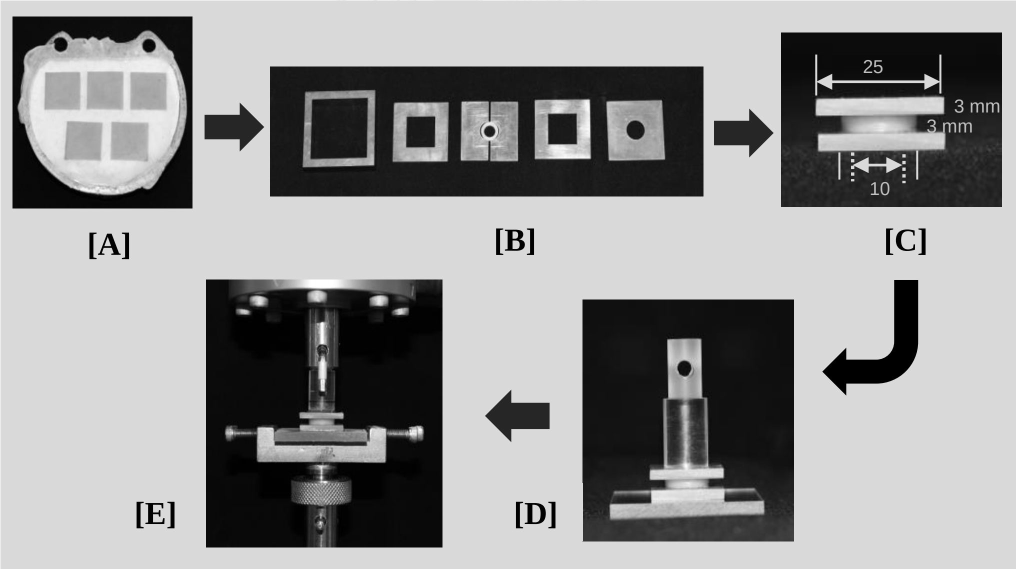
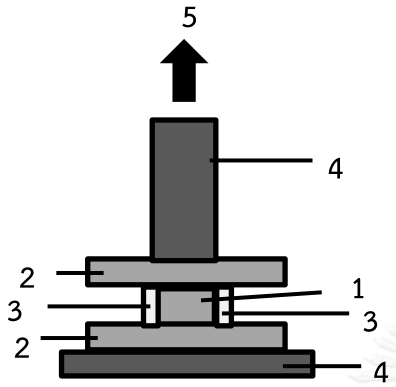


Figure 6. Specimen preparation. [A] heat-cured denture base in a dental flask, [B] split metal mold, [C] test specimen, and [D,E] test specimen in a vertical alignment.



Key

1. Test material (lining material)
2. Acrylic denture base plate
3. Collar
4. Polymethyl methacrylate rods (optional)
5. Direction of the tensile stress

Figure 7. Test specimen components.

Failure analysis

The mode of failure of the debonded surface was determined (cohesive, mixed or adhesive failure) using a stereomicroscope at 10x magnification. Cohesive failure was defined as when there was more than 50% of reline material on the denture base surface. Adhesive failure was defined as when there was no traces of reline material on the denture base surface. Mixed failure was defined as there was less than 50% of reline material on the denture base surface.[40]

Statistical analysis

The data were analysed using SPSS for Windows 22.0 (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. If the data showed a normal distribution ($p>0.05$) and homogeneous variances ($p>0.05$), Two-way ANOVA would be used. If the data did not meet these criteria, Non parametric statistic, Kruskal Wallis test was used.

CHAPTER IV

RESULTS

Results

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.[41] Thus, the results were statistically analyzed by One-Way ANOVA and Tukey HSD test.

The mean tensile bond strength and standard deviation of each group and the percentage of each failure type are presented in Table 5 and Figure 8 and Table 6, respectively. 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 (AT) and MMA 180 s (MT) groups ($p>0.05$). However, the mean tensile bond strength of the AT group was significantly lower than that of the MT group ($p<0.05$). In the Ufi Gel Hard groups, there were no significant differences in tensile bond strength between the groups applied with MF-MA for 15, 30 and 60 s and the Adhesive (AU group) ($p>0.05$). However, the mean tensile bond strength of the MF-MA 15 s, 30 s, 60 s and Adhesive groups were significantly lower than those of MF-MA 180 s and MMA 180 s groups ($p<0.05$). The mean tensile bond strength of the MF-MA 180 s group was not significantly different compared with the MMA 180 s group (MU) group ($p>0.05$).

Table 5. The mean tensile bond strength of each reline material differentiated by surface treatment. The same superscript letter indicates no significant difference between groups (p>0.05).

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}

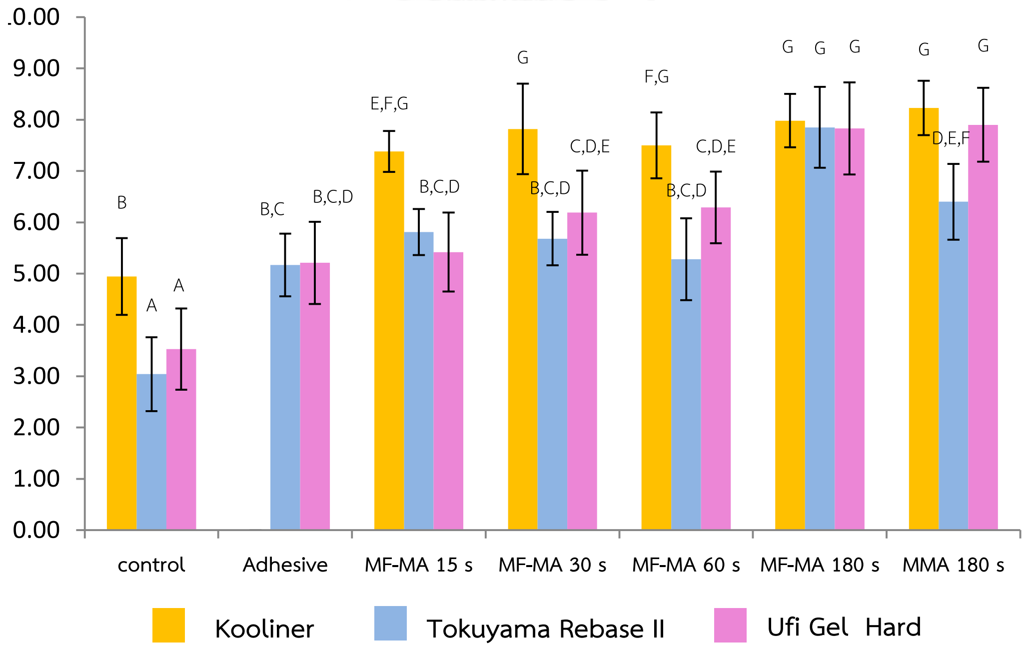


Figure 8. Bar graph of the mean tensile bond strength of all groups. Groups with the same letter were not significantly different (p>0.05).

Failure type analysis demonstrated that all 3 relining material control groups had 100% adhesive failure. Most of failure, and in some cases all, in the adhesive, MF-MA and MMA groups were mixed failures. The Tokuyama Rebase II and 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 is shown in Table 7. The failure patterns stereo microscopy images are shown in Figure 9, 10, and 11.

Table 6. The percentage of failure pattern of the three relining 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	-

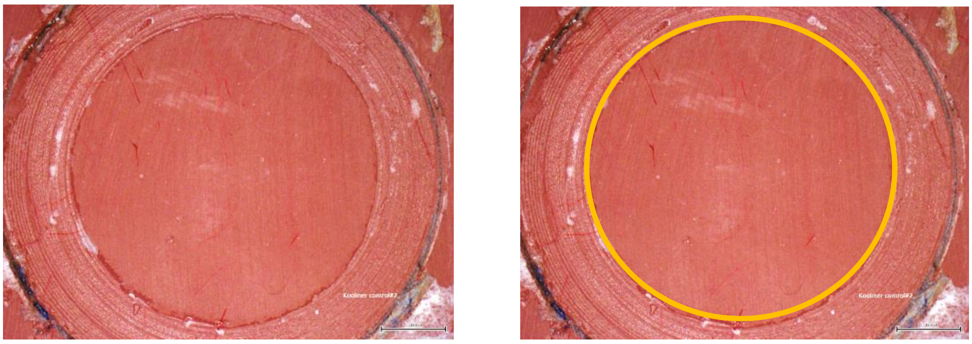


Figure 9. Adhesive failure at denture base surface.

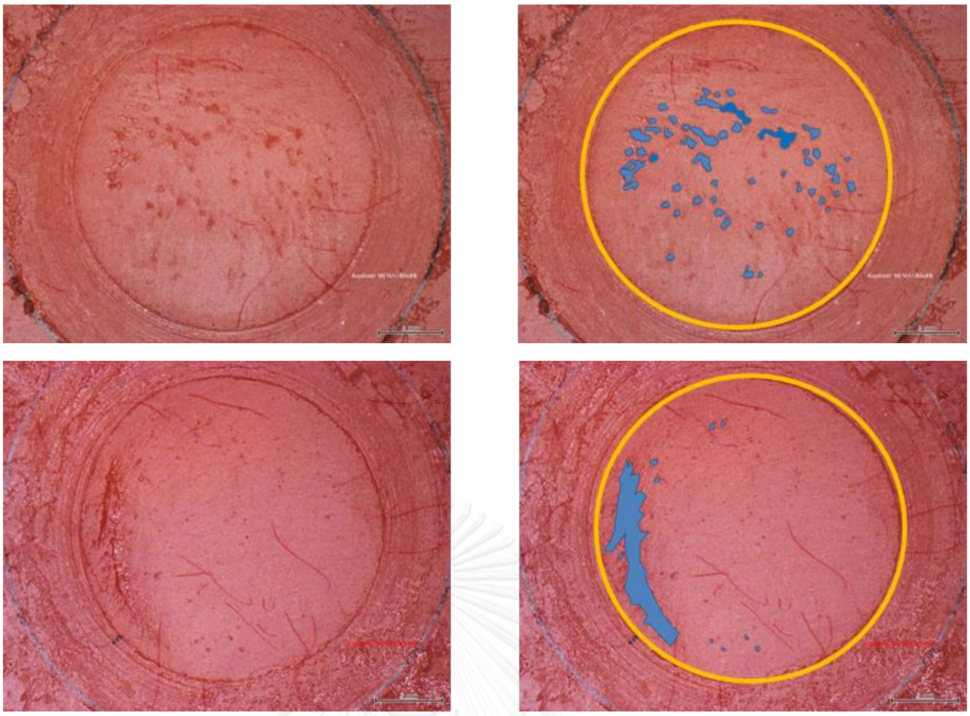


Figure 10. Mixed failure showing the reline material attached to some of the denture base surface.

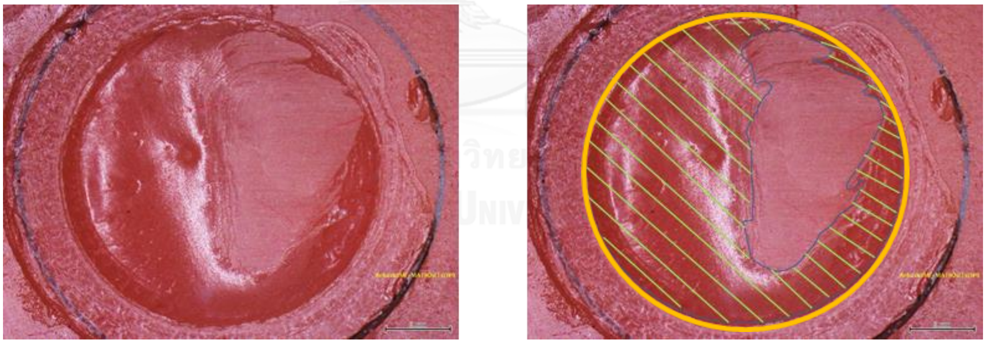


Figure 11. Cohesive failure showing most of the reline material attached to the denture base surface.

SEM examination was used to observed the morphological changes on the denture base surface after surface treatment (Figure 12). The untreated denture base surface, the control group, exhibited scratch lines in a single direction with some acrylic debris from polishing (Figure 12[A]). The surface 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

(Figure 12[B,C]). Denture 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 (Figure 12[D]). The denture resin treated with MF-MA for 180 s demonstrated a honeycomb appearance with 3-dimensional pores from the superficial into the deep layer (Figure 12[E]). The denture base treated with MMA for 180 s had irregular scratch lines similar to the denture resin applied with Tokuyama Rebase II adhesive (Figure 12[F,G]). The Ufi Gel Hard adhesive created a smoother denture base surface (Figure 12[H]).

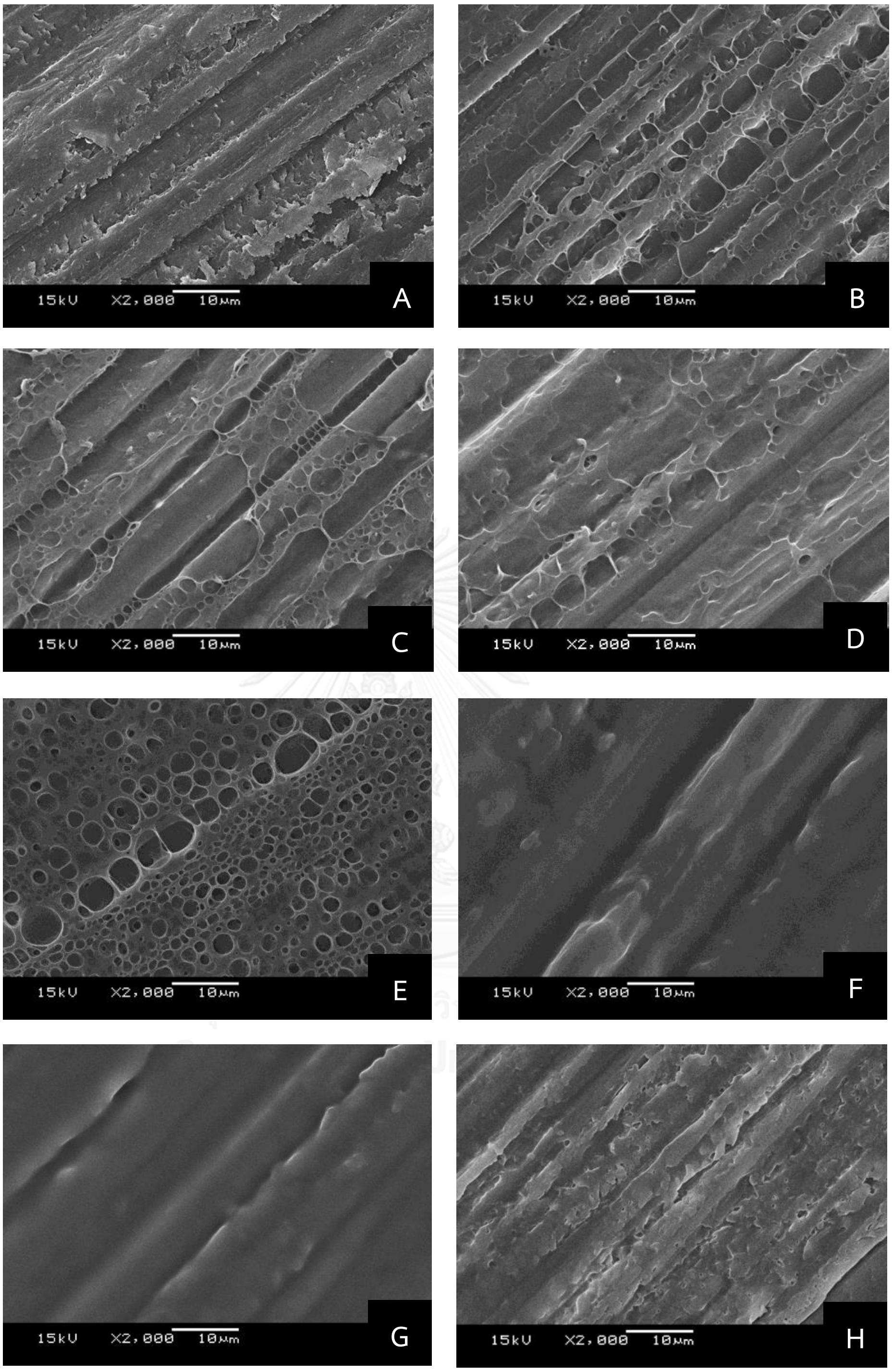


Figure 12. SEM analysis of the morphological changes of heat-cured denture base surface treated with different surface treatment. [A] no treatment, [B] MF-MA solutions 15 s, [C] MF-MA solutions 30 s, [D] MF-MA solutions 60 s, [E] MF-MA solutions 180 s, [F] MMA 180 s, [G] Tokuyama Rebase II adhesive, [H] Ufi Gel Hard adhesive, respectively.

CHAPTER V
DISSCUSSION AND CONCLUSION

Discussion

This study was designed to determine how various MF-MA wetting times affected the tensile bond strength between 3 non MMA-based relining materials and denture base resin. The tensile bond strength of specimens treated with MF-MA for 15, 30, 60, 180 s and no treatment was compared. These wetting times were selected based on a previous study that found that increased MMA wetting time caused increased thickness of the swollen layer at the denture base surface.[17] Vallittu et al. concluded that an MMA wetting time of 180 s was sufficient to provide a strong bond.[29] 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, surface treatment and reline materials. Surface treatment refers to two factors, type of solvent and wetting time.

Four solvents were used for denture base surface treatment (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.[35] The solubility parameter of PMMA is 18.3 MPa$^{1/2}$, while those of MF, MA, MMA, ethyl acetate and acetone are 20.9, 19.6, 18.0, 18.2 and 19.7 MPa$^{1/2}$, respectively.[42] The solubility parameter of 2-HEMA (26.93 MPa$^{1/2}$) 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 being 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.[35]

The molecular weight of solvent has an effect on the softening efficacy which lower molecular weight promotes the faster kinetics of diffusion.[35] Acetone (58.08 Da) has a molecular weight close to MF (60.05 Da). The other four solvents have the higher molecular weight than acetone and MF which are MA (74.08 Da), ethyl acetate (88.11 Da), MMA (100.12 Da) and 2-HEMA (130.14 Da). Boiling point affects to the bonding process that lower boiling point of solvent causes an easier evaporation and takes less chair-time. Methyl formate (31.8°C) has the least 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.

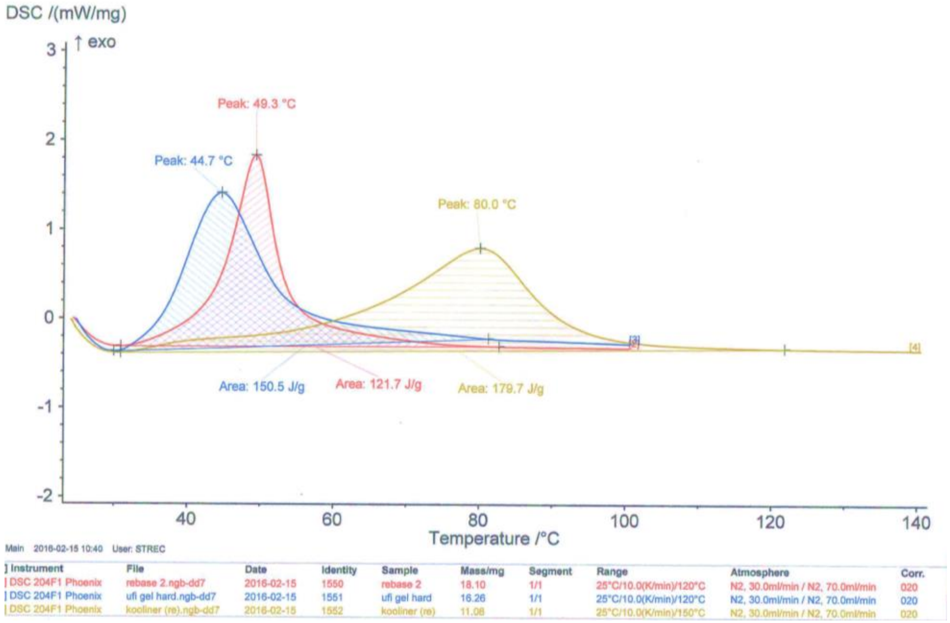
The morphological surface changes of the variously treated denture base resins were observed using scanning electron microscopy. The control surface was demonstrated scratch lines from the silicon carbide paper. The SEM images of pores resulting from MF-MA application revealed the ability of MF-MA to dissolve the denture base surface. Increased MF-MA wetting times increased the number of porosities with different sizes and patterns. The appearance of the MMA treated denture base had blurred and irregular scratch lines similar to that observed after the application of Tokuyama Rebase II adhesive. However, the Ufi Gel Hard adhesive treatment resulted in a smoothened denture base surface.

As above, MF and MA have a low boiling point, 31.8°C and 56.9°C, respectively, compared to the other solvents[43, 44] that allows for the solution to evaporate with none remaining on the bonding surface after their application. The bond mechanism between 2 materials has two processes, dissolution and penetration.[6] First, the solvent dissolves and swells to the denture base surface. This process depends on the solubility parameter, polarity and the concentration of the solvent in the polymer.[45] Due to the similar solubility parameter and polarity of MF-MA compared to PMMA are the one of the reason provided a good bond at relined interface. The second process is that the MF-MA solution generates a swollen gel-like pattern and then penetrates into the denture base surface. This process is related to the size of the solvent molecules.[45] MF and MA have smaller molecules compared with MMA and the other two adhesives. The molecular structures of MF and MA also do not contain carbon–carbon double bonds (C=C) that might polymerize with the monomer of autopolymerized relining materials. Thus, using MF-MA solution can create a proper bond area without any residual material that can block bonding. The large amount of pores at the interface of the MF-MA treated relined denture base surfaces allowed 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 was formed between the denture base and relining material.

Methyl methacrylate is a solvent commonly used for surface treatment. This solvent has similar solubility parameter and polarity compared to PMMA. However, a higher molecular weight and boiling point of MMA might be provided lower solubility to the denture base material compared to MF-MA. Ethyl acetate and acetone has similar solubility parameter compared to PMMA but they have different functional groups in their chemical structure. 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 considerably a higher molecular weight and boiling point compared to the other solvents and also has dissimilar solubility parameter and polarity. Thus, it would explain that 2-HEMA is not the good effective promotor to dissolve PMMA.

Based on the results, the treated denture base surface groups (i.e. adhesive, MF-MA solutions, and MMA) had significantly higher tensile bond strength compared with the non-treated group of the 3 relining materials. According to the reasons above, the first null hypothesis was rejected. These findings are in accordance with previous studies that have demonstrated that chemical surface treatment increased the bond strength between denture base resin and relining materials.[21, 31, 34, 37]

The mean tensile bond strength of the Kooliner groups was significantly higher compared with those of the Tokuyama Rebase II and Ufi Gel Hard groups. The molecular weight of the liquid part of relining materials plays a role in its viscosity.[13] 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 Kooliner, or the 1,6 HDMA (254.32 Da) in Ufi Gel Hard. According to diffusion theory, a material with high viscosity will move slower compared with a low viscosity material.[46] 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 relining materials from after mixing until 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 (Figure 13). The heat generated during polymerization stimulates the rate of diffusion of monomer molecule into the denture base material, enhancing tensile bond strength.[13] These two reasons, molecular weight of monomer and exothermic energy, were explained to the higher tensile bond strength of Kooliner comparing to other materials.



Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
DSC 204F1 Phoenix	rebase 2.ngb-dd7	2016-02-15	1550	rebase 2	18.10	1/1	25°C/10.0(K/min)/120°C	N2, 30.0ml/min / N2, 70.0ml/min	020
DSC 204F1 Phoenix	ufi gel hard.ngb-dd7	2016-02-15	1551	ufi gel hard	16.26	1/1	25°C/10.0(K/min)/120°C	N2, 30.0ml/min / N2, 70.0ml/min	020
DSC 204F1 Phoenix	kooliner (re).ngb-dd7	2016-02-15	1552	kooliner (re)	11.08	1/1	25°C/10.0(K/min)/150°C	N2, 30.0ml/min / N2, 70.0ml/min	020

Figure 13 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)

Consequently, 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 strength of the Tokuyama Rebase II and Ufi Gel Hard were significantly different in 180 s-MF-MA wetting time compared to that of 15, 30 and 60 s-MF-MA groups. The mean tensile bond strength of the Tokuyama Rebase II and Ufi Gel Hard of 15, 30 and 60 s-MF-MA groups were not significantly different from each other. It might be that the three-dimension appearance from 180 s-MF-MA SEM image (Figure 12[E]) was allowed the monomer of Tokuyama Rebase II and Ufi Gel Hard to penetrate and form the better bond compared with 15, 30 and 60 s-MF-MA wetting time. Based on these data, the second null hypothesis was rejected.

In order to the failure patterns of specimens, the amount of tensile bond strength positively related to the type of failure observed. From the correlation analysis, the higher tensile bond strength tends to be cohesive failure more than mixed and adhesive type (Table 11). However, this analysis was not represented the relations between the mean tensile bond strength and failure pattern in Kooliner groups. The Kooliner groups had a higher mean tensile bond strength compared with the two other materials, however, these groups 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 Kooliner specimens.[22, 47-51] 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 was found in MF-MA 180 s and MMA 180 s groups. The non-harmonized mixing and the powder-liquid ratio of Tokuyama Rebase II and Ufi Gel Hard might affect the failure results of these two materials that could create voids blending in the piece of relining materials. Once the test specimens were applied on the tensile force, it would be broke at the weakest area, sometimes at the void in the reline material.

Per the National Fire Protection Association 704 code: Chemical identifiers for hazardous materials[52], the methyl methacrylate[53] and methyl formate[43] are level 2 materials that cause temporary incapacitation or residual injury. Methyl acetate is level 1 material[44], where exposure to this material would cause irritation with only minor residual injury. Therefore, MF and MA are equal to or less hazardous compared with methyl methacrylate. Furthermore, the American Conference of Governmental Industrial Hygienists recommends that the air borne exposure limits of MMA, MF and MA for an 8- hour work shift are 50, 100, and 200 ppm, respectively.[54-56] Consequently, MF-MA solution is the solvent of choice that provides many advantages for surface treatment and is safer compared with MMA.

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 relining materials and, also reduces adhesive failure at the relined interface.

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APPENDIX

Table 7. Kolmogorov-Smirnov test analysis of the data distribution.

Number of groups	Group	N	Asymp. Sig.(2-tailed)
1	Koo con	10	.200
2	Koo MF-MA 15s	10	.139
3	Koo MF-MA 30s	10	.200
4	Koo MF-MA 60s	10	.200
5	Koo MF-MA 180s	10	.200
6	Koo MMA 180s	10	.200
7	Re con	10	.127
8	Re Ad	10	.200
9	Re MF-MA 15s	10	.200
10	Re MF-MA 30s	10	.200
11	Re MF-MA 60s	10	.158
12	Re MF-MA 180s	10	.200
13	Re MMA 180s	10	.075
14	Ufi con	10	.200
15	Ufi Ad	10	.200
16	Ufi MF-MA 15s	10	.062
17	Ufi MF-MA 30s	10	.200
18	Ufi MF-MA 60s	10	.200
19	Ufi MF-MA 180s	10	.200
20	Ufi MMA 180s	10	.200

Table 8. The Levene statistical analysis of the groups.

Levene Statistic	df1	df2	Sig.
1.163	19	180	.294

Table 9. One-way ANOVA analysis of the groups.

ANOVA

Bond strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	437.736	19	23.039	41.897	.000
Within Groups	98.981	180	.550		
Total	536.717	199			

Table 10. Tukey's HSD analysis of the bond strength of the groups.

Group 1way	N	Subset for alpha = 0.05						
		1	2	3	4	5	6	7
Re con	10	3.0410						
Ufi con	10	3.5320						
Koo control	10		4.9430					
Re Ad	10		5.1680	5.1680				
Ufi Ad	10		5.2130	5.2130	5.2130			
Re MF-MA60s	10		5.2780	5.2780	5.2780			
Ufi MF-MA15s	10		5.4170	5.4170	5.4170			
Re MF-MA30s	10		5.6800	5.6800	5.6800			
Re MF-MA15s	10		5.8080	5.8080	5.8080			
Ufi MF-MA30s	10			6.1870	6.1870	6.1870		
Ufi MF-MA60s	10			6.2850	6.2850	6.2850		
Re MMA180s	10				6.4020	6.4020	6.4020	
Koo MF-MA15s	10					7.3790	7.3790	7.3790
Koo MF-MA60s	10						7.4960	7.4960
Koo MF-MA30s	10							7.8170
Ufi MF-MA180s	10							7.8290

Re MF-MA180s	10							7.8480
Ufi MMA180s	10							7.8990
Koo MF-MA180s	10							7.9760
Koo MMA180s	10							8.2320
Sig.		.995	.503	.098	.052	.050	.118	.531

Means for groups in homogeneous subsets are displayed.

Table 11. The regression analysis between the tensile bond strength and mode of failure.

Bond strength	Co. efficient	Standard error	t	P>ltl	[95% conf.interval]	
Cohesive	4.34	0.51	8.52	0.00	3.33	5.34
Mixed	2.24	0.21	10.56	0.00	1.83	2.66

Adhesive failure is a reference group.

Table 12. The tensile bond strength of the surface treatments in the Kooliner group.

No	control	adhesive	MF-MA 15s	MF-MA 30s	MF-MA 60s	MF-MA 180s	MMA 180s
1	6.65	-	8.01	9.20	8.68	8.75	9.27
2	5.39	-	7.93	9.19	8.46	8.70	8.88
3	5.12	-	7.61	8.24	7.96	8.28	8.54
4	5.10	-	7.59	8.22	7.61	8.14	8.28
5	5.07	-	7.35	7.91	7.40	8.04	8.27
6	5.06	-	7.34	7.66	7.29	7.99	8.23
7	4.42	-	7.00	7.39	7.02	7.75	7.95
8	4.25	-	7.00	6.98	6.96	7.73	7.71
9	4.21	-	6.99	6.87	6.86	7.38	7.60
10	4.16	-	6.97	6.51	6.72	7.00	7.59
Mean	4.94	-	7.38	7.82	7.50	7.98	8.23
SD	0.75	-	0.40	0.88	0.64	0.52	0.53

Table 13. The tensile bond strength of the surface treatments in the Tokuyama Rebase II group.

No	control	adhesive	MF-MA 15s	MF-MA 30s	MF-MA 60s	MF-MA 180s	MMA 180s
1	4.13	6.02	6.36	6.51	7.00	8.94	8.34
2	3.80	5.78	6.33	6.09	6.19	8.59	7.02
3	3.79	5.74	6.23	6.08	5.74	8.58	6.52
4	3.78	5.70	6.07	6.08	5.42	8.53	6.40
5	2.85	5.44	6.03	5.76	5.41	8.28	6.10
6	2.85	5.06	5.71	5.70	4.77	7.58	6.05
7	2.55	4.62	5.70	5.48	4.77	7.44	6.05
8	2.22	4.48	5.43	5.42	4.55	6.98	6.04
9	2.22	4.43	5.14	4.85	4.47	6.89	5.75
10	2.22	4.41	5.08	4.83	4.46	6.67	5.75
Mean	3.04	5.17	5.81	5.68	5.28	7.85	6.40
SD	0.72	0.61	0.45	0.52	0.80	0.79	0.74

Table 14. The tensile bond strength of the surface treatments in the Ufi Gel Hard group.

No	control	adhesive	MF-MA 15s	MF-MA 30s	MF-MA 60s	MF-MA 180s	MMA 180s
1	4.77	6.68	7.19	7.09	7.33	9.27	9.22
2	4.44	6.37	6.41	7.08	7.02	9.10	8.59
3	4.20	5.74	5.73	7.03	7.02	8.60	8.59
4	4.13	5.42	5.49	6.74	6.75	8.27	8.28
5	3.52	5.11	5.09	6.36	6.37	7.96	7.98
6	3.50	5.06	4.94	6.07	6.05	7.33	7.65
7	2.85	4.77	4.91	6.05	6.04	7.27	7.32
8	2.84	4.44	4.83	5.79	5.75	7.01	7.30
9	2.78	4.42	4.80	5.13	5.43	6.78	7.05
10	2.29	4.12	4.78	4.53	5.09	6.70	7.01
Mean	3.53	5.21	5.42	6.19	6.29	7.83	7.90
SD	0.79	0.80	0.77	0.82	0.70	0.90	0.72

VITA

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