EFFECT OF ULTRASONIC TREATMENT ON THE TRANSVERSE STRENGTH OF RELINED
DENTURE BASE WITH AUTOPOLYMERIZING ACRYLIC HARD RELINE MATERIAL

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ผลของการทำอัลตราโซนิกต่อความแข็งแรงเชื่อมขวางของวัสดุฐานฟันเทียมที่เสริมฐานด้วยวัสดุเสริมฐานอะคริลิกชนิดบ่มตัวเอง

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ฐิติพร อรุณวิจิตร : ผลของการทำอัลตราโซนิกต่อความแข็งแรงเชื่อมขวางของวัสดุฐานฟัน
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การศึกษานี้มีวัตถุประสงค์ เพื่อศึกษาผลของการทำอัลตราโซนิก และการปรับสภาพพื้นผิวต่อ
ความแข็งแรงเชื่อมขวางของฐานฟันเทียมอะคริลิกเรซินบ่มตัวด้วยความร้อน ที่ได้รับการเสริมฐานด้วย
อะคริลิกเรซินชนิดบ่มตัวเอง กลุ่มการศึกษาใช้อะคริลิกเรซินบ่มตัวด้วยความร้อนขนาด 64×10×2 มม
จำนวน 90 ชิ้น แบ่งสุ่มตามการปรับสภาพพื้นผิวก่อนการเสริมฐานเป็น ไม่ทาสารปรับสภาพพื้นผิว (N) ทา
MMA 180 วินาที (MM) และทาMFMA 15 วินาที (MF) จึงเสริมด้วยอะคริลิกเรซินบ่มตัวเองให้มีขนาด
โดยรวมเป็น 64×10×3.3 มม แล้วจึงแบ่งต่อตามกระบวนการหลังการบ่มด้วยอัลตราโซนิก 3 แบบดังนี้
ไม่ทำอัลตราโซนิก (X) ทำอัลตราโซนิกในน้ำ (W) และทำอัลตราโซนิกในเอทานอลความเข้มข้นร้อยละ 30
(E) จึงมีกลุ่มชิ้นงานทั้งหมดเป็นดังนี้ NX, NW, NE, MMX, MMW, MME, MFX, MFW, MFE (n=10)
ชิ้นงานทั้งหมดได้รับการทดสอบแรงดัดแบบ 3 จุด นำข้อมูลมาวิเคราะห์ความแปรปรวนสองทาง พบว่า
ปัจจัยเรื่องการทำอัลตราโซนิกและการปรับสภาพพื้นผิวมีปฏิสัมพันธ์กัน จึงวิเคราะห์ความแปรปรวนทาง
เดียวพบว่า ปัจจัยการทำอัลตราโซนิกทำให้ค่าความแข็งแรงของชิ้นงานเพิ่มขึ้น (p<0.05) การทำอัลตรา
โซนิกในเอทานอลให้ค่าความแข็งแรงมากกว่าในน้ำ (p<0.05) และมีค่าสูงที่สุด ปัจจัยเรื่องการปรับสภาพ
พื้นผิว เมื่อเปรียบเทียบในกระบวนการอัลตราโซนิกที่เหมือนกัน การใช้ MMA และ MF-MA เพิ่มค่าความ
แข็งแรงของชิ้นงานเมื่อเทียบกับกลุ่มที่ไม่ทา (p<0.05) กลุ่ม MM และ MF มีค่าความแข็งแรงของชิ้นงาน
ไม่ต่างกัน (p>0.05)

สาขาวิชา	ทันตกรรมประดิษฐ์	ลายมือชื่อนิสิต ....
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KEYWORD: Ultrasonic Relining auto-polymerizing acrylic resin surface treatment transverse strength

Tithiporn Arunwichit : EFFECT OF ULTRASONIC TREATMENT ON THE TRANSVERSE STRENGTH OF RELINED DENTURE BASE WITH AUTOPOLYMERIZING ACRYLIC HARD RELINE MATERIAL . Advisor: Assoc. Prof. CHAIRAT WIWATWARRAPAN

This study evaluated the effect of ultrasonic treatment and surface treatments on transverse strength of denture bases which relined with auto-polymerizing acrylic resin. The study groups, ninety heat-polymerized acrylic resins ($64 \times 10 \times 2$ mm) were divided by three surface treatments: no surface treatment (N), applied MMA for 180 seconds (MM), applied MF-MA for 15 seconds (MF). They were relined with auto-polymerized acrylic resin to create $64 \times 10 \times 3.3$ mm specimens. Then, they were divided further by ultrasonic which were no ultrasonic (X), ultrasonic in water (W), ultrasonic in 30% ethanol (E). As a result, all test groups were classified as NX, NW, NE, MMX, MMW, MME, MFX, MFW, MFE (n=10). All samples were performed three-point bending test. Two-way ANOVA presented that there was an interaction between ultrasonic and surface treatments. Then One-way ANOVA was analyzed. Ultrasonic could increase transverse strength of all relined specimens ($p < 0.05$). Ultrasonic in ethanol increased strength superior to water ($p < 0.05$) and presented the highest mean strength. In the same ultrasonic treatment, MMA and MF-MA increased the transverse strength of relined denture bases compared with no applying groups (N) ($p < 0.05$). There was no difference between all MM and MF groups ($p > 0.05$).

Field of Study:	Prosthodontics	Student's Signature ....
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CHAPTER I

INTRODUCTION

Background and rationale

Poly (methyl methacrylate) or PMMA is the most common material for removable denture fabrication. This polymer is also known as "acrylic resin". Acrylic resin was first introduced in dentistry in 1937[1] because of its characteristics such as mechanical properties, practical fabrication, cost effectiveness, light weight, chemical stability, etc. This material is very efficacious in several parts of dental appliances. These include denture bases, denture teeth, provisional crowns, maxillofacial prostheses, surgical stents, and orthodontic appliances. In addition, acrylic resin is the general material which is used for correcting and repairing dental appliances. Accordingly, PMMA is the material of choice for removable prostheses.[2, 3]

As a matter of fact, the underlining tissue support is inconstant. Edentulous ridges always resorb after tooth extraction, the resorption has an effect on the prostheses especially in the lower arch.[4] The alveolar bone resorption decreases stability of removable prostheses.[5] Poor prostheses probably cause injuries to the mucosal tissues and the abutment teeth which lead to mastication roughness.[6] Relining is one of the methods to maintain function and stability of the prostheses. Removable prostheses should be relined every 1-5 years.[7] The techniques for relining can be achieved either by direct or indirect action.[8] However, the direct method gives the benefits of short processing time. Because of low polymerization temperature, auto-polymerized acrylic resin is preferred because it can reduce distortion of the denture base.[9] The adjusted prostheses are returned to the patients within the same dental appointment. The lining materials are segregated into several classifications. Importantly, the hard liners which are generally used, are divided into MMA-based and non MMA-based. The MMA-based material provided better mechanical properties[10, 11] and reduced adhesive failure mode.[12] Moreover, chemical surface treatments are necessary substances for bonding either reline[13] or repair.[14, 15] There

are several chemical substances for treating the bonding interface of existing denture bases such as MMA (methyl methacrylate)[16, 17], acetone[18], chloroform[16] etc. Nowadays, the "CU Acrylic bond" is used to improve bond strengths of acrylic resin junctions. The CU Acrylic Bond (MF-MA) is another choice of chemical surface treatment because of low toxicity, rapid action, and no residual monomer.[19-22]

Auto-polymerized acrylic resin (cold-cure acrylic or self-cure resin) is available for chair-side operations but the residual monomer is the most unfavorable issue.[9] The residual monomer which left over after the polymerization, is an unavoidable element. The unreacted monomer influences on physical, mechanical properties and biological properties. For example dimensional stability, water absorption, transverse strength, surface hardness are adverse effects.[23, 24] There were reports of inflammation[25] and allergy[26, 27] caused by residual monomer. The presence of saliva can gradually reduce these effects but seeking the strategies to reduce the residual monomer is still necessary. The irritation mostly occurs within the first 24 hours.[28] The residual monomer was found highly within the first week after manufacture.[29, 30] Sometimes residual monomer could be found for 40 days[31] and up to a year or more.[32, 33] Therefore, the residual monomer issue need a proper management. There are two concepts to overcome the unfavorable effects of monomer.[34] First, by increasing polymerization rates, more monomers are consumed in the polymerization reaction resulting in a decrease of excess monomer. Thus, high temperature curing process was required.[24, 35] Second, the unreacted monomer could be solved by the elimination techniques. The residual monomer could be discharged in the appropriate conditions.[34] For intra-oral relining, the polymerization process might be encountered under the undesirable environments. Therefore, post-polymerization techniques should be advised for those reasons. Ultrasonic cleaners used to be advised for post-polymerization method instead of microwave radiation. Microwave post-polymerization suspiciously caused undesirable effects to prostheses and should be used with cautions.[36, 37] In comparison, ultrasonic baths could provide both source of energy and substances extraction. Gu et al,2002[38] confirmed that ultrasonic influenced in the bulk-polymerization of PMMA. Charasseangpaisarn and Wiwatwarrapan, 2015[39] suggested that ultrasonic treatment could minimize the

unreacted monomer more rapidly than other methods. In addition, ethanol immersion could subside the unreacted monomer more efficiently than water. According to Arriwiratchakun et al,2019[40], the immersion method could reduce the unreacted monomer moreover ultrasonic treatment in ethanol solutions could be more effective. Advantageously, the ultrasonic cleaning machines are generally available in dental clinics. As a result, dentists can simply use the machines they have. However, this method needs the verification that it might not cause undesirable effects to the relined prostheses.

The purpose of this study are as follows. First, to evaluate transverse strength of relined prostheses with auto-polymerizing acrylic resin after ultrasonic treatment. Second, to evaluate the transverse strength of relined prostheses after ultrasonic immersion in two different medias. Third, to evaluate the effect of chemical surface treatment before relining on the transverse strength of relined denture base which passed ultrasonic post-polymerization.

Research questions

1. Is there a difference between the transverse strength of relined denture base before and after the ultrasonic post-polymerization?
2. Does the ultrasonic post-polymerization in 30% ethanol solution affect on the transverse strength of relined denture base?
3. Does the different chemical surface treatments before relining affect on the transverse strength of the relined denture base which passed the same ultrasonic post-polymerization?

Research objectives

1. To evaluate the effect of ultrasonic on the transverse strength of relined denture base.
2. To observe the effect of 30% ethanol solution in ultrasonic treatment on the transverse strength of relined denture base.

3. To evaluate the effect of chemical surface treatments on the transverse strength of relined denture base which passed the same ultrasonic post-polymerization.

Research hypotheses

1. - H0: The ultrasonic post-polymerization treatment dose not significantly affect on the transverse strength of relined denture base at the 95% confidence level.
   - H1: The ultrasonic post-polymerization treatment significantly affects on the transverse strength of relined denture base at the 95% confidence level.
2. - H0: The 30% ethanol solution immersion in ultrasonic post-polymerization treatment dose not significantly affect on the transverse strength of relined denture base at the 95% confidence level.
   - H1: The 30% ethanol solution immersion in ultrasonic post-polymerization treatment significantly affects on the transverse strength of relined denture base at the 95% confidence level.
3. - H0: The chemical surface treatment does not significantly affect on the transverse strength of the same post-polymerization of relined denture base at the 95% confidence level.
   - H1: The chemical surface treatment significantly affects on the transverse strength of the same post-polymerization of relined denture base at the 95% confidence level.

Scope of the Research

1.This research is an in-vitro study

2. A type of heat-cure acrylic resin is Meliodent® (Kulzer, Germany) which is the most common material used in faculty of dentistry Chulalongkorn university.

3. A type of commercial hard reline materials used in this study are Unifast Trad®(GC America, USA).

4. A single investigator performed this study

Keywords

Ultrasonic, transverse strength, flexural strength, surface treatment, hard relining materials, hard liners, denture base, self-cure, auto-polymerized acrylic resin, denture base, PMMA, CU Acrylic Bond, Methyl formate, Methyl acetate, MF-MA

Expected benefits

1. To verify that the ultrasonic cleanser is the effective equipment for reducing unreacted monomer of relined prostheses without the adverse effects.

2. To find the rapid and user-friendly strategies reducing residual monomer of auto-polymerized acrylic resin.

3. To approve that the operators can work with the practical material; auto-polymerizing acrylic resin, for relining to obtain the better mechanical properties.

4. To ensure that CU Acrylic Bond can be used as a chemical surface treatment without mechanical properties interference.

Conceptual framework

Modifying factor
Chemical Surface Treatment

- Type of substance
- Concentration
- Time
- Surface Contamination

Relining

-Material type
-Thickness
-Curing method
-Temperature
-Curing time
-Curing cycle

Post Polymerization Treatment

Ultrasonic

Relined Acrylic Denture Base

Transverse Strength

- Oral Environment
- Temperature
- water Absorption
- Thickness
- Contamination
- Thermal Stress

CHAPTER II

REVIEW OF LITERATURES

Denture base polymer

Poly (methyl methacrylate)

"PMMA" Poly (methyl methacrylate) is a kind of polymer material which has been used in dental appliance fabrications. It was introduced in 1937 with cost effective and biocompatibility after complete polymerization (figure 1).[1]

Figure 1. History of denture base polymer

PMMA is a transparent and colorless material, so it profits for aesthetics. With its light weight, patients benefit from easy adaptation. In addition, it has favorable mechanical properties that it can serve the masticatory function. The material can serve the requirements of denture base materials. For example, the tensile strength is about 60 MPa. Knoop hardness is 18-20 KHN. The polymer also tolerates to chemical at below 125°C. Degree of conversion effects on the mechanical properties of the polymer. Polymerization of PMMA from MMA may get 21% average for volumetric shrinkage after the curing process.[2, 3]

Table 1. Ideal properties of denture base materials.[9]

Property	Requirements
Biological	1.Non-toxic, non-irritant, non-carcinogenic effects.
Chemical	1.Not dissolve in oral fluids and any other intake substances.
	2.Not absorb oral fluids and any other intake substances which might affect dimensional stability.
	3.Bond well with artificial teeth and liners.
Mechanical	1.High modulus of elasticity which can endure the masticatory force.
	2.High resilience to protect the underlying soft tissue while

	functioning. 3.High elastic limit and proportional limit to prevent permanent distortion. 4.Adequate mechanical strength to against fracture from repeated load. 5.Dimensional stable. 6.Adequate abrasive resistance. 7.Low specific gravity for the light weight.
Thermal	1.Adequate thermal conductor. 2.Co-efficient of thermal expansion (COTE) corresponds with the artificial teeth. 3.Softening temperature is higher than the boiling temperature of water.
Esthetic	1.Sufficient translucency to mimic the oral tissue. 2.Should be painted or pigmented.
Other	1.Maintain the desirable properties after fabrication. 2.Inexpensive. 3.Easy to manipulate. 4.Radiopaque. 5.Easy to repair. 6.Easy to clean. 7.Long shelf life.

The monomer of PMMA is methyl methacrylate (MMA) which chemical formula is $C_5H_8O_2$. At room temperature, it is clear liquid which can evaporate easily due to high vapor pressure. When the polymerization occurs (double bond turns into a single bond) to form poly (methyl methacrylate), the chain of polymer is longer to gain the appropriate properties then strengthened by the cross-link agents. The caution is that the residual monomer always presents.
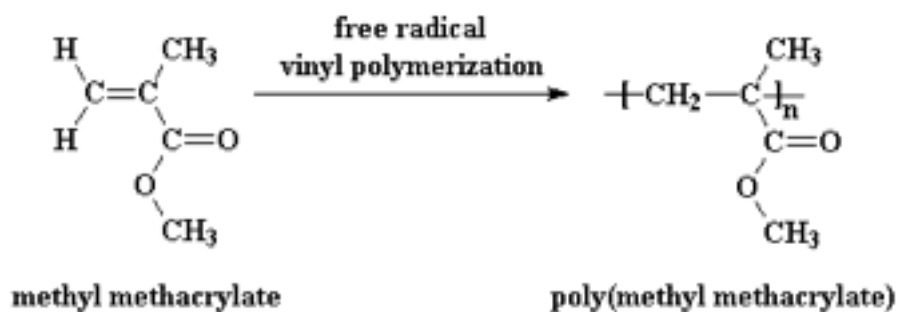
Figure 2. Polymerization of MMA

Table 2. The properties of MMA.[9]

Properties of MMA	
Molecular weight	100.12 g/mol
Melting point	-48ºC
Boiling point	100.8ºC
Density	0.945 g/cc at 20ºC
Polymerization shrinkage	21%
Heat of polymerization	12,900 cal/mole

Table 3. Classification of acrylic polymers.[41]

Classification of acrylics	
Type 1	Class 1: Heat processing polymers; powder and liquid
	Class 2: Heat processed; plastic cake
Type 2	Class 1: Auto-polymerized polymers; powder and liquid
	Class 2: Auto-polymerized polymers; pour type resins
Type 3	Thermoplastic blank or powder
Type 4	Light activated materials
Type 5	Microwave-cure materials

Acrylic resins are divided into 5 types as the following.[3, 9, 41]

Type 1, class 1 Heat-processed material is the general product which has been used for a long time. Low residual monomer and good mechanical properties are the benefits of heat-cure resin, but the processing takes long duration than others and volumetric shrinkage can occur. Transverse deformation of heat-cure resin is affected by thickness. (15-50 N at 2-5 mm. in thickness). The initiator is benzoyl peroxide (BPO) which can be detached at high temperature.

Table 4. Composition of heat-cure PMMA. (Type1) [9]

Powder	Liquid
1.Pre-polymerized poly (methyl methacrylate).	1.Methyl methacrylate.
2.Copolymers of of PMMA (5%): Ethyl or Butyl methacrylates	2.Co-monomers.
3.Initiator (0.2-1.5%): Benzoyl peroxide	3.Inhibitor: Hydroquinone (0.003%-0.1%).
4.Plasticizer: Dibutyl phthalate.	4.Plasticizers: Butyl or Octyl
5.Color pigments: Mercuric sulfide, cadmium sulfide, ferric oxide, carbon black.	methacrylate and Dibutyl phthalate.
6.Opacifiers: Zinc or titanium oxides	5.Cross-linking agent: Ethylene
7.Dyed synthetic fibers made from nylon or acrylic	glycol dimethacrylate.
8.Inorganic particles: Glass fibers, zirconium silicate, whiskers of alumina, SiC, boron nitride and carbon fibers.	
9.Heavy metal compounds: Barium, bismuth, etc.	

Table 5. Properties of heat-cure PMMA resin. (Type1) [9]

Solubility	In aromatic hydrocarbons, ketones, esters.	0.04 mg/cm^2
	In water.	0.02 mg/cm^2
Water sorption	0.69 mg/cm^2	
Modulus of elasticity	3.8x10$^{3.}$ MPa	
Proportional limit	26 MPa	
Compressive strength	76 MPa	
Tensile strength	48-62 MPa	
Percentage of elongation	1-2%	
Impact strength	0.98-1.27 J	
Surface hardness	18-20 KHN	
Density	1.16-1.18 g/cc	
Fatigue strength	1.5x10^6 cycles at 17.2 MPa	
Thermal conductivity	5.7x10^{-4} °C/Cm	
Coefficient of thermal expansion	81x10^{-6} /°C	
Heat distortion temperature	71-91 /°C	
Glass transition temperature	125 °C	
Depolymerization temperature	450°C	

Type 2, auto-polymerized resin (self-cure or cold-cure resin) is the material of choice for chair-side correction. Even though the material is convenient for users and low shrinkage. The more inferior properties and the higher residual monomer are the detriments. The process of material is initiated by a tertiary aromatic amine which allows free radicals at ordinary temperature. Color instability can be occurred from tertiary aromatic amine. The cold-cure material creates more inferior properties of acrylic than heat activating material.

Type 4, the light-activate resin has no methyl methacrylate but consists of urethane dimethacrylate. Finally, type 5, the microwave-process material has low residual monomer and improves mechanical strength. The microwave-cure resin is uncommon due to the high expenses.

Meliodent® (Kulzer, Germany) used in this study, is the heat polymerizing product which has been used in the faculty of dentistry Chulalongkorn University for long.

Table 6. Composition of auto-polymerized PMMA.[9]

Powder	Liquid
1.Poly (methyl methacrylate) or Copolymer beads.	1.Methyl methacrylate monomer.
2.Initiator: Peroxide (maximum –2.0%).	2.Cross linking agent: Ethyl glycol di methacrylate.
3.Pigments	3.Inhibitor: Hydroquinone.
4.Colored fibers (nylon/ acrylic)	4.Dibutyl phthalate.
	5. Activator: Tertiary amine (Di methyl para toludiene) – (maximum – 0.75%).

Bone Remodeling Process

Later tooth extraction, the alveolar bones continue to collapse. This incidence is always found among denture wearers. Particularly, the lower edentulous ridges collapse rapidly due to smaller area and less advantage ridge shape.[4] There are several factors that affect bone resorption such as bone remodeling after tooth removal, mechanical stress, systemic conditions, individual inflammatory mediators. Moreover, tooth is the factor that preserves alveolar bone density.

Hansson S and Halldin A, 2012[42] also confirmed that when the tooth was loaded, the alveolar bone gained tension stress and then initiated bone remodeling. After the tooth was removed there was no internal tension force, the alveolar ridges began to resorb especially in the removable prostheses supporting area.

When the edentulous ridges resorbed, the removable dentures perform inferior in functions especially in tissue-borne condition.[5, 6] The mucosal trauma, abutment teeth trauma and loose occlusal contact are the results of the edentulous ridges resorption. To maintain functions and stabilities of the prostheses, the removable dentures need to be relined. The patients need to follow up every 1-5 years [7, 8]
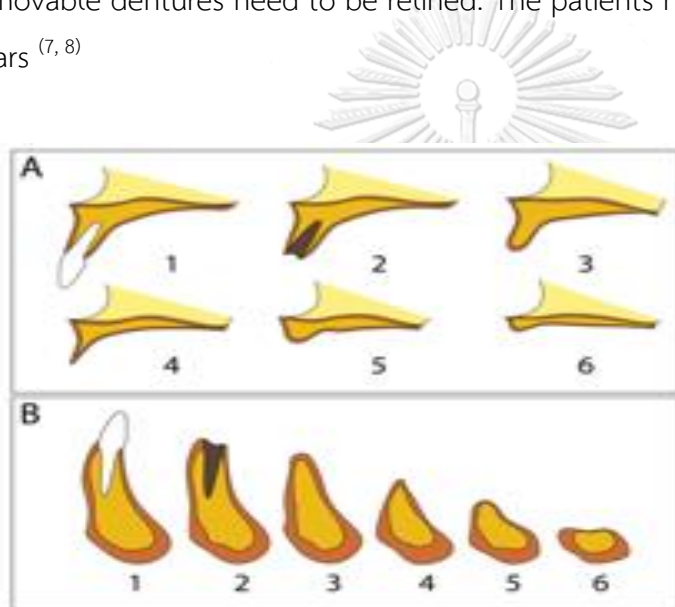
Figure 2: Classification system of six atrophy stages in the maxilla (A) and the mandible (B) according to Atwood (1963) and Cawood and Howell (1988). Atrophy stage 1: preextraction, stage 2: postextraction, stage 3: high well-rounded ridge, stage 4: knife-edge shaped ridge, stage 5: low well-rounded ridge, stage 6: depressed bone level.
85x76mm (600 x 600 DPI)

Figure 3. Bone remodeling process. [43]

Relining Method and Materials

Relining process has two techniques. First, direct (chair-side) relining technique: the auto-polymerized materials are allowed to set easily within patients' mouth. However, the polymerization process releases heat leading to mucosal burn. The materials also present poor color, unpleasant smell and taste. Furthermore, the

residual monomer is an undesirable compound. Second, indirect (laboratory) relining technique: the polymerization process takes place outside the mouth. The impression techniques are necessary to make the accurate oral tissue models. This method consumes for some time, but better results might be the outcomes.[8]

The materials for relining are divided into 4 classifications serving as hard-soft liners, curing process, temporary-permanent materials and resin-silicone based resins.[3] The hard liners are favorable materials for correcting the prostheses. Besides, monomethacrylates which is the material of choice for relining, can be classified by chemical compositions into two groups: methyl methacrylate-based (MMA-based) and non-methyl methacrylate based (non-MMA based). The most favorable of non-MMA based resin is poly (ethyl methacrylate) or PEMA. In addition, light activating resin can be used as a hard-lining material. The study showed that the MMA-based group superiorly satisfied the requirements of mechanical properties.[10] MMA-based resins gained flexural strength more than another group. Moreover, MMA-based group provides better shear bond strength than other group due to their similar components to denture bases.[44] It also benefited more esthetics, color stability, good adaptability, easy handling. Nevertheless, it caused more irritation to soft tissues. PEMA group was more biocompatibility. The material rarely generated heat during polymerization and low volumetric shrinkage. But it offered with inferior mechanical properties and low color stability. Composed of plasticizers, non MMA-based produced low mechanical properties than another such as flexural strength[11], hardness.[10]

Table 7. The auto-polymerized resin hard reline material is classified by compositions[9]

Type 1	Powder	
PMMA	Polymer beads	Poly(methyl methacrylate)
	Initiator	Benzoyl peroxide
	Liquid	
	Monomer	Methyl methacrylate
	Plasticizer	Di-*n*-butyphthalate
	Chemical activator	Tertiary amine
Type 2	Powder	
PEMA	Polymer beads	Poly(ethyl methacrylate)
	Initiator	Benzoyl peproxide
	Liquid	
	Monomer	Butyl methacrylate
	Cross-linking agent	Di-methacrylate
	Chemical activator	Tertiary amine

Chemical surface treatment

Chemical surface treatment can improve flexural strength of relined prostheses because of enhancing retention between 2 layers. The more monomer molecules penetrate, the more bond strength can be gained.
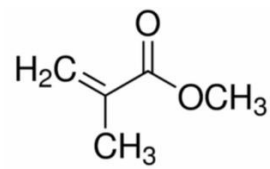
Chloroform[16] was used as a softening substance, but it is high toxicity. There are several alternative substances such as MMA[16, 17], acetone,[18] acetic acid[45] etc. A mixture of methyl formate-methyl acetate (MF-MA) can also be used as chemical surface treatment of denture base resins.[13, 19-22]
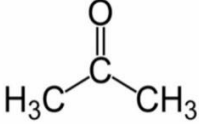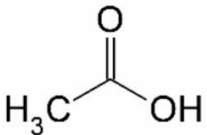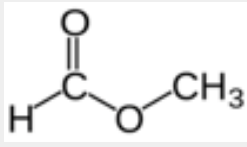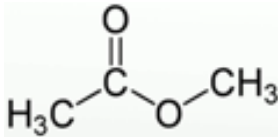
MMA can be used as a chemical surface treatment in relining dentures. As the result of softening theory (the materials can be dissolved by solutions if the solubility
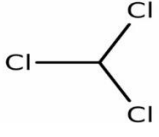
parameters and polarities of treated substances relate to each other). After solutions applied, surface of prepared dentures appeared more porosities. Vallittu et al, 1994[46] suggested that MMA solvent should be applied for 180 seconds. MMA is a part of PMMA then it can cause interface swelling of denture bases. The roughness surface of swelled dentures enhanced mechanical retention of relined materials. Thus, the resin monomer can easily penetrate. Moreover, MMA is the liquid portion of auto-polymerized acrylic resin, it is an available agent.

       Methyl formate-methyl acetate mixing solution is one of the surface treatment substances which less toxicity. It is called "CU Acrylic Bond". It is a colorless substance and evaporates easily due to low molecular weight and high vapor pressure. It is not a monomer because of no carbon double bond in its molecules. CU Acrylic Bond dose not interfere the bonding surface because it does not leave other remaining substances.[22] It's solubility parameter is 19.6-20.9 $MPa^{1/2}$. This parameter closes to PMMA (18.0 $MPa^{1/2}$).[47] Kungkapilas K and Santawisuk W, 2014[13] found that applying methyl formate-methyl acetate (MF-MA) 25:75 v/v for 15 seconds on acrylic lining interface trended to increase shear bond strength. Tanasamanchoke and Wiwatwarrapan, 2015[22] displayed that wetting MF-MA (25:75% volume) more than 30 seconds, acrylic resins presented too large porous appearance. Using methyl formate-methyl acetate (25:75%v) for 15 seconds treated on the acrylics surface, could increase tensile bond strength and shear bond strength of both acrylic denture bases[20, 22] and synthetic denture teeth.[21, 48]

Table 8. Substances for chemical surface treatment.[49]

Substance	Properties		Chemical Formula
Methyl methacry-late	Solubility parameter.	18 $MPa^{1/2}$	
	Molecular weight.	100.117 g/mol	
	Vapor pressure	29 mmHg (20°C)	
	Boiling point.	100.8°C	
	Density.	0.94 g/cm^3	

	Odor	unpleasant	
	Appearance.	Clear liquid	
Acetone	Solubility parameter.	19.9 MPa$^{1/2}$	
	Molecular weight.	58.08 g/mol	
	Vapor pressure	180 mmHg (20ºC)	
	Boiling point.	55.75ºC	
	Density.	0.79 g/cm^3	
	Odor	fruity	
	Appearance.	Clear liquid	
Acetic acid	Solubility parameter.	20.7 MPa$^{1/2}$	
	Molecular weight.	60.052 g/mol	
	Vapor pressure	11 mmHg (20ºC)	
	Boiling point.	117.9 ºC	
	Density.	1.05 g/cm3	
	Odor	vinegar-like	
	Appearance.	Clear liquid	
Methyl formate (Methyl methano-ate)	Solubility parameter.	20.9 MPa$^{1/2}$	
	Molecular weight.	60.052 g/mol	
	Vapor pressure	476 mmHg(20ºC)	
	Boiling point.	32ºC	
	Density.	0.98 g/cm3	
	Odor	pleasant	
	Appearance.	Clear liquid	
Methyl acetate (Methyl ethanoate)	Solubility parameter.	19.6 MPa$^{1/2}$	
	Molecular weight.	74.079 g/mol	
	Vapor pressure	173 mmHg(20ºC)	
	Boiling point.	56.8ºC	
	Density.	0.93 g/cm3	

	Odor	fruity	
	Appearance.	Clear liquid	
Chloroform	Solubility parameter.	19.0 MPa$^{1/2}$	
	Molecular weight.	119.36 g/mol	
	Vapor pressure	160 mmHg(20ºC)	
	Boiling point.	61.1ºC	
	Density.	1.48 g/cm3	
	Odor	pleasant	
	Appearance.	Clear liquid	

Residual monomer

Monomer which left after polymerization is called residual monomer or unreacted monomer. It is always true that the polymerization occurs incompletely once the process has passed. Residual monomer is always found in acrylic resin unavoidably, especially in auto-polymerized resin. It can be detected 0.2-0.5 % in heat-cure resin and 3-5% in cold-cure resin, respectively.[9]

Residual monomer can perform as a plasticizer which impacts on physical and mechanical properties for instance; flexural strength[50], microhardness[24], hardness[51], water absorption[23], tensile strength[23]. Promoting the polymerization helped reducing residual monomer and improved the mechanical properties. Unreacted monomer can diffuse out in liquid environments.[29] Residual monomer causes the adverse results not only in mechanical aspects but also biology aspects. Many studies reported that residual monomer caused tissue irritation and allergy.[25-27] Hashimoto et al, 2014[52] also reported that mucositis could be seen in a patient who took a self-cure acrylic provisional crown. In addition, self-cure acrylic can be used in orthopedic operations as bone cement. Their sessions still concern about unreacted MMA and biocompatibility. Incomplete polymerization can cause tissue irritation. Kostic.et al, 2018[53] and Mesquita et al, 2017[54] revealed that monomer initiated inflammation in subcutaneous tissue-layer. Even the toxic could be subsided, but the inflammation continued for period.

There are several parameters involved in severities of adverse effects, for example: type of acrylic, ratio, storage time, water immersion, polymerization cycle, polymerization method.[55]

Saravi et al, 2012[28] showed that the highest toxic level of residual monomer of cold cure resins was found in the first hour including heat-cure acrylics. Sometimes, left monomer had been found diffused out for over 17 years.[32] Lamb et al, 1982[29] reported that long-lived radical in self-cure resins could be found for 60 minutes after initial cured. Zissis et al, 2008[33] supported that residual monomer could be found released out high level in the first week then subsided after a year pass.

The other toxic substances which can be found leaching from acrylic denture base such as formaldehyde[56-58], methacrylic acid[59], biphenyl, phenyl benzoate.[60]

Formaldehyde, which is not composed of the initial substances, can be found from the leaching substances. It is a high toxic substance in spite of low dose. When the temperature is up to 37°C, formaldehyde may be formed by oxidation process of methacrylate group and degradation of methacrylate polymer. Ruyter IE, 1980[57] presented that formaldehyde released from denture base resins after storage.

According to the International Organization for Standardization, 2013[41] the limitation amount of residual monomer was announced. It should be less than 2.5% mass fraction for heat polymerizing acrylic and 4.5% for auto-polymerizing acrylic.

Methods to reduce residual monomer contents

Apart from proper powder/liquid ratio and curing process, the post-polymerization methods may influence the residual compounds.[31, 34] There are several methods for reducing residual monomer. Firstly, the chemical methods help changing residual monomer to other substances. Secondly, physical methods help for residual monomers extraction. In additions, the post-polymerization methods are chemical removal, radiation, devolatization, ion-exchange resin.[34]

Temperature is the most important factor that affects degree of conversion in acrylics both initial polymerization and post-polymerization. The amount of residual monomer is controlled by a thermodynamic consequence. Appropriate curing temperature and time, heat-cure acrylic rarely contained unreacted monomer due to diminishing unreacted monomer.[35, 61, 62] As a result, high temperature during curing process is an important factor for mechanical properties.[63] Harrison and Huggett, 1992[64] recommended that long curing cycle (70°C for 7 hours followed by 100°C for an hour) could reduce residual monomer. Without any treatments, auto-polymerizing acrylic contains more unreacted components. Curing in 30°C water for 30 minutes, auto-polymerized acrylic could significantly reduce residual monomer and increase tensile strength.[23]

The water immersion is a basic physical method for that purpose, but it takes long duration. Residual monomer could diffuse into liquid media.[29, 55] Sofou A et al, 2005[65] suggested that water immersion for 5 days in room temperature could reduce leaching substances. Lamb DJ et al, 1983[31] found that immersion in 37°C water for days could solve the remained monomer problems. The study showed that 55°C water immersion for 60 minutes improved biocompatibility of material.[66] Tsuchiya et al,1994[58] also suggested that immersion in 50°C water for 60 minutes helped reducing formaldehyde and methyl methacrylate concentrations.

Post-polymerization by ethanol immersion is a devolatization strategy. The residual monomer is carried away by the solvent. With the high temperature including the proper ethanol concentration, the more residual monomer can be eliminated. Ethanol concentrations, temperature, time and type of resins are the important factors in ethanol immersion. Previous studies showed that immersion in 20-50% volume ethanol's solution at 55°C for 10 minutes reduced residual monomer without disturbances of the mechanical properties.[67, 68] Ethanol solution reduced leaching substances effectively than distilled water despite heat-polymerized acrylic resin.[65]

Microwave treatment is a radiation post-polymerization method which can be done effortlessly and rapidly. Using microwave, internal heat will generate through all particles of the objects. The energy can increase degree of conversion thus the

mechanical properties of the auto-polymerizing acrylic can be improved. The microwave generates heat similar to the water bath method but dry condition. Microwave radiation could cause high rising temperature of acrylics. The mechanical properties of heat-polymerizing acrylic can also be impacted after microwave post-polymerization.[36, 37, 69] However, this technique should be use with specific conditions.[70] From microwave irradiation, the internal heat can be generated quickly. However, different resins could be treated by different radiations.[71, 72] Bural C et al,2011[73] revealed that even the microwave post-polymerization could improve degree of conversion of the incomplete-cured acrylics. But the highly cured acrylics might be depolymerized. Konchada J et al, 2013[74] suggested that using microwave 650 watt for 5 minutes in post-polymerization, self-cure acrylic would not cause the unfavorable results. Nevertheless, overheat issue is still concerned[75], this method needs more information.

Ultrasonic cleanser is another radical post-polymerization machine which is used for several purposes for example: scaling machine, endodontic treatment, cleaning etc. Ultrasonic shaking through the water generates bubbles and cavitation. Then the bubbles collapse and release more energy to induce the radicals. Energy from ultrasonic can provide both chemical and mechanical reaction.[76] As a result, ultrasonic bath is determined to solve residual monomer of self-cure acrylic. The residual monomer could be reduced by two mechanisms not only driving off the compounds but also increasing polymerization. According to the study of Gu et al, 2002[38] ultrasonic could control rate of polymerization. The appropriate ultrasonic irradiation could afford sufficient energy for bulk polymerization. Charasseangpaisarn T et al[39, 77] suggested that the ultrasonic treatment (100 W, frequency 40 kHz in 50ºC water for 5 minutes) on auto-polymerizing acrylic resin could reduce residual monomer below the determined level. Arriwiratchakun P and Wiwatwarapan C, 2019[40] also approved that ultrasonic treatment in 30% ethanol solution at 50ºC for 5 minutes solved leached unreacted monomer in self-cure acrylic. Thummawanich W and Wiwatwarapan C, 2018[78] confirmed that ultrasonic treatment in ethanol solution improved the mechanical properties of acrylics which used for orthodontic

base-plate material.

Flexural strength, transverse strength

Flexural strength or transverse strength is a represent for the material characteristics which comprise of compressive, tensile, and shear strength. The polymerization of MMA to PMMA is an important process which directly effects on mechanical properties of acrylics for instance molecular weight, characteristics, strengths, rigidity, melting temperature etc.[2, 79]

Transverse strength occurs as the masticatory force loading on the dentures. The bending force causes the stress then leading deformation. Excess stress, the tension-side areas are potentially initiated cracks. The repeating masticatory force does not cause permanent deformation of the dental appliances, but it may induce flexural fatigue. Three-point bending test is the imitating of this situation. The transverse strength depends on the material composition, degree of polymerization, and environments. The transverse strength is calculated by the formula as follow.[41]

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

F = maximum load (N)

l = the length between the two points support (mm)

b = the width of the specimen (mm)

h = the thickness or the height of the specimen (mm)

CHAPTER III

METHODOLOGY

Target population

Heat-polymerizing acrylic resin

Sample

Relined denture base 90 specimens

Table 9. The materials' names and manufacturers of samples used in this study.

Product name	Material	Lot. number	Manufacturer
Meliodent®	Powder: PMMA	K010035	Kulzer, Germany
	Liquid: MMA	k010124	
Unifast Trad®	Powder: PMMA	1907011	GC America, USA
	Liquid: MMA	1811221	
Methyl-methacrylate	Surface treatment agent (Liquid of Unifast Trad®)		GC America, USA
Methyl formate	Surface treatment agent		Merck Schuchardt OHG, Germany
Methyl acetate	Surface treatment agent		Merck KGaA, USA
Ethanol (LiChrosolv®)	Ultrasonic media		Merck Limited, Germany

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

Instruments

1. Heat-polymerized curing unit 'EWL 5518' (Kavo, Germany)
2. Hydraulic flask pressure 'EWG 5414' (Kavo, Germany)
3. Universal testing machine (Shimadzu, EZ-S 500N model, Japan)
4. Automatic grinding and polishing unit (Minitech 233, Metallography India, Maharashtra, India)
5. Digital vernier caliper (Mitutoyo, Japan)
6. Incubator 37°C (Contherm Scientific Ltd., New Zeland)
7. The ultrasonic cleaner (VGT 1990 QTD, Guangdong GT Ultrasonic Industrial Co., Shenzhen, China)
8. Hanau flasks
9. Sets of rectangular stainless-steel mold
10. Teflon sheet
11. Dental stone
12. Metallographic grinding paper P500, P1000 and P1200 (TOA, Thailand)

Sample preparations

Ninety specimens of heat-cured acrylic denture base (Meliodent[®] Kulzer, Germany) (64x10x2mm) were prepared in the denture flasks as recommended by manufacturer. Long cycle curing was performed at 70°C for 8 hours. Then the specimens were polished with 500,1000,1200-grit silicon carbide paper by using an automatic grinding and polishing unit (Minitech 233, Metallography India, Maharashtra, India) until precise. For the lining surface of heat-polymerized specimens, polishing was controlled meticulously (54 N, 450 rpm, 20 seconds). The dimension was controlled by a digital vernier caliper before water storage (37±2°C distilled water for 48±2 hours). Then, the specimens were randomly divided into 3 groups which classified by three surface treatments which were non-treatment: N, applied MMA for 180 seconds: MM, applied MF-MA (25:75% volume) for 15 seconds: MF. The auto-polymerizing acrylic resins were mixed as recommended by manufacturer and

relined on the heat-polymerizing specimens which had been placed in each split metal molds. Then the prepared molds were covered with a 5 kilograms metal block and were left polymerized for 8 minutes. After that, all relined specimens were divided to subgroups by post-polymerization (non post-polymerization: X, ultrasonic in water: W, ultrasonic in 30% ethanol: E) (n=10). Table no.10 showed the detail about specimens' preparation and post-polymerization as follow descriptions.

The specimens in group NX were relined without chemical surface treatment and without ultrasonic immersion. NW were relined without chemical surface treatment after relining they were immersed in water ultrasonic bath. NE were relined without chemical surface treatment after relining they were placed in 30% ethanol ultrasonic bath. MMX were applied MMA for 180 seconds without ultrasonic treatment, MMW were applied MMA for 180 seconds after relining they were immersed in water ultrasonic bath, MME were applied MMA for 180 seconds after relining they were immersed in 30% ethanol ultrasonic bath, MFX were applied MF-MA for 15 seconds without ultrasonic treatment, MFW were applied MF-MA for 15 seconds after relining they were immersed in water ultrasonic bath, MFE were applied MF-MA for 15 seconds after relining they were immersed in 30% ethanol ultrasonic bath.

After that all relined specimens were finished with 500-grit silicon carbide paper. The relined specimens were 3.3±0.02 mm in thick which each specimen was composed of heat-polymerizing resin 2 mm and auto-polymerizing resin 1.3 mm (Figure 5). Then, they were stored in 37±2°C water for 48±2 hours before testing. The flexural strength was measured by universal testing machine (Shimadzu, EZ-S 500N model, Japan) with the crosshead speed 5 mm/min. The distance between two supporting bars are 50 mm. There was a temperature control by testing under water at 37±2°C (figure 4).

Table 10. Description of experimental groups

Group	Surface Treatment		Relining	Ultrasonic Treatment	
	MMA 180 s	MF-MA 15 s		Water 50ºC, 5 min	30%v Ethanol 50ºC, 5 min
NX	-	-	+	-	-
NW	-	-	+	+	-
NE	-	-	+	-	+
MMX	+	-	+	-	-
MMW	+	-	+	+	-
MME	+	-	+	-	+
MFX	-	+	+	-	-
MFW	-	+	+	+	-
MFE	-	+	+	-	+

MMA: Methyl methacrylate, MF-MA: Methyl formate-methyl acetate, N: Non surface treatment, v: volume

X: No post-polymerization, W: Ultrasonic in water, E: Ultrasonic in 30%ethanol

Statistical analysis

Statistical analysis was performed using IBM SPSS version 23.0 (IBM Corp. 1989, 2014) as follows:

1. Descriptive analysis to determine mean and standard deviation of flexural strength

2. If the population was normally distribution and homogeneity of variance, two- way analysis of variance (two-way ANOVA) was applied to analyze the interaction effect between ultrasonic post-polymerization and the chemical treatment factors in each group of relined denture base at a 95% confidence level. If there is no statistically interaction, Post hoc (multiple comparison) test was used to interpret at a 95% confidence level. If there is a statistically interaction, Univariate analysis of variance test was used to interpret at a 95% confidence level.

3. If the population was normally distribution and homogeneity of variance, one- way analysis of variance (One-way ANOVA) was used to compare mean of flexural strength in each group at a 95% confidence level followed by Tukey's HSD Post-hoc test to compare between groups of non-reline denture base at a 95% confidence level. But if the population was not normally distributed, the Kruskal Wallis test was used at a 95% confidence level.
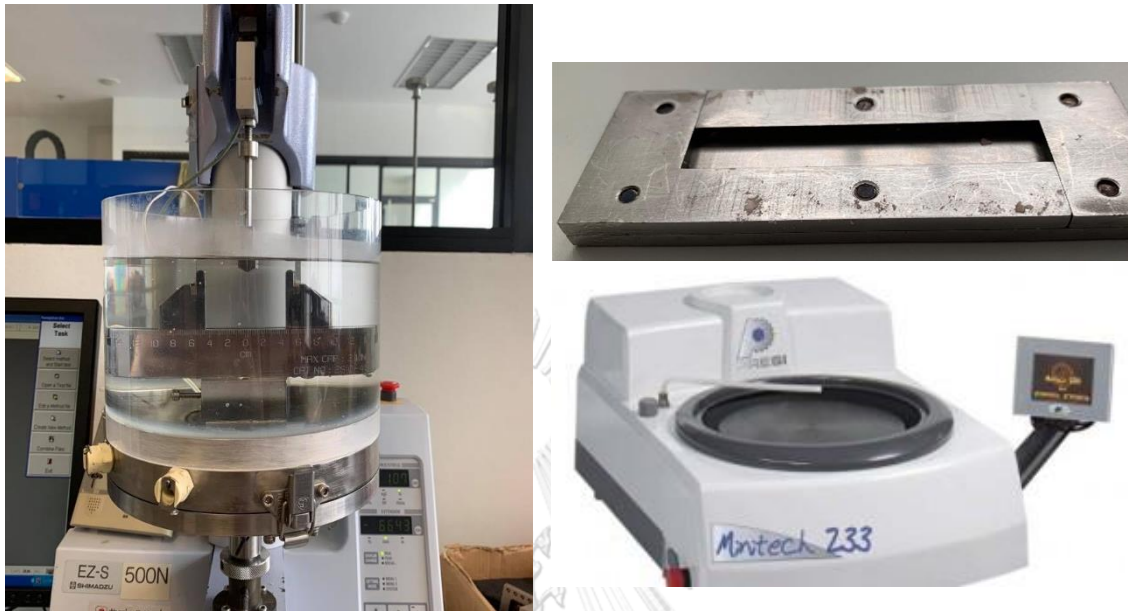
Figure 4. Instrument and machine

Left: the setting of three-point bending test, upper right: the split-metal mold, lower right: the polishing machine.
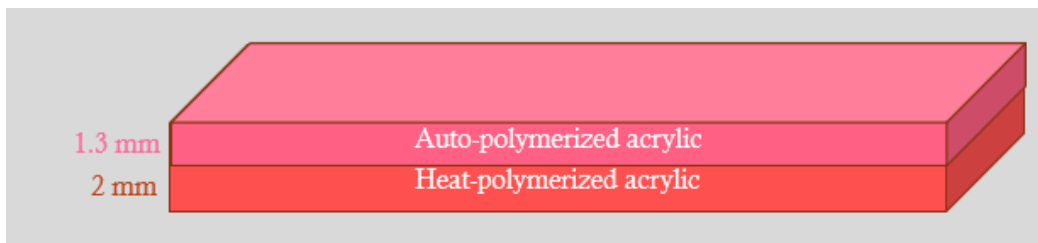
Figure 5. Size of each relined specimen.

CHAPTER IV

RESULT

Result

The data were analyzed normality using Kolmogorov-Smirnov test. All data

were normally distributed (p<0.05). The homogeneity of variance considered equal.

Two-way ANOVA analysis found an interaction effect of the two factors. Both

chemical surface treatments and post-polymerization factors significantly affected on

the transverse strength of relined denture bases at the 95% confidence level

(p<0.05) (Table 11). The interaction plot was shown in figure 6. Then, the data were

continuously analyzed by One-way ANOVA as presented in Table 12.

Table 11. Two-way ANOVA analysis of post-polymerizations and chemical surface
treatments effected on relined denture based.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	630.829[a]	8	78.854	48.427	.000	.827
Intercept	459703.616	1	459703.616	282319.941	.000	1.000
chem	141.962	2	70.981	43.592	.000	.518
postpoly	468.243	2	234.121	143.782	.000	.780
chem * postpoly	20.624	4	5.156	3.167	.018	.135
Error	131.893	81	1.628			
Total	460466.338	90				
Corrected Total	762.721	89				

a. R Squared = .827 (Adjusted R Squared = .810)

Figure 6. Profile plot presented an interaction effect between two factors.

The mean transverse strength and standard deviation of each groups (n=10) were revealed in the Table 12.

Within the same post-polymerization factors, the results revealed that the mean transverse strength of relined denture bases applied MMA and MF-MA as chemical surface treatments (MM and MF groups) were significantly higher than non-surface treatment groups (N groups) (p<0.05).

Table 12. The mean transverse strength and standard deviation of each relined groups (MPa)

		Surface treatment		
		Non-surface Treatment (N)	MMA (MM)	MF-MA (MF)
Post-polymerization	No US (X)	66.83 (1.05) [Aa]	69.75 (1.19) [Ab]	69.81 (1.22) [Ab]
	US water (W)	69.11 (1.35) [Ba]	72.86 (1.1) [Bb]	71.77 (1.16) [Bb]
	US 30% ethanol (E)	73.15 (1.42) [Ca]	74.36 (1.42) [Cab]	75.60 (1.51) [Cb]

US : ultrasonic, N : non surface treatment, MM : MMA 180 seconds, MF : MF-MA 15 seconds, X : without ultrasonic , W : water, E : 30% ethanol solution

***Same uppercase latter indicates no significant difference between the groups in each column (p>0.05).

***Same lowercase latter indicates no significant difference between the groups in each row (p>0.05).

In case of the same surface treatments, the transverse strength of relined denture base revealed the same significant ascending trends after ultrasonic post-polymerization (p<0.05) (Figure 6). The results of using MMA and MF-MA were not significantly different within the same post-polymerization groups (Table 12). The lowest transverse strength presented in the non-surface treatment group (NX group = 66.83±1.05 MPa). The groups of 30% ethanol ultrasonic post-polymerization (all E groups) presented the highest mean transverse strength among the same surface treatment groups. Thus, the highest transverse strength was found in MFE group (75.60±1.51 MPa).
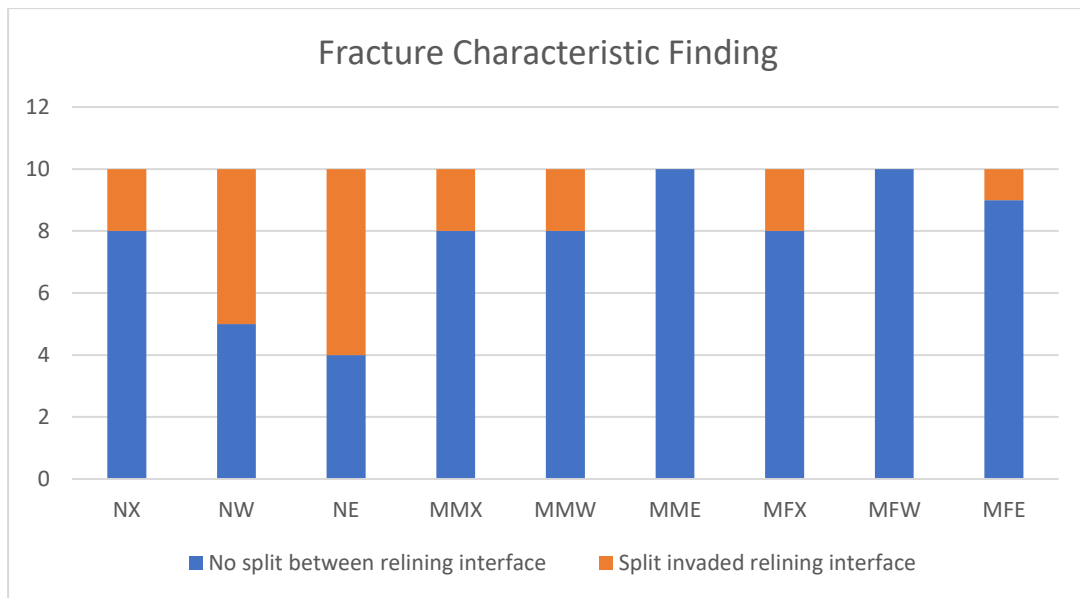
Figure 7. Specimens' fracture characteristic and chart presented number of each fracture characteristics (pcs). A: auto-polymerizing acrylic resin, H: heat-polymerizing acrylic resin.

Fracture characteristic along the fracture surface of all tested specimens were presented in figure 7. Fracture line invaded through relining-interface was slightly found with the groups of chemical surface treatment. Non-surface treatment groups presented the highest fracture line between relining-interface especially after ultrasonic treatment. The non-surface treatment group (NE group) showed the maximum interface fracture of specimens (60% inter-relining surface fracture). Only groups of MME and MFW presented no fracture between relining interfaces.

Each of chemical surface treatments effects the surface morphology of heat-polymerized acrylic resins in the different appearances (Figure 8). Applied MMA for 180 seconds generated the small-size porosities. The solution of methyl formate and methyl acetate could cause the large-size porosities within 15 seconds.

Heat-polymerizing and auto-polymerizing acrylic resins after 30% ethanol ultrasonic treatment rarely appealed any different appearance from the control and water ultrasonic treatment in figure 9 and 10.

Maginification 1500X	Maginification 6000X
Non-surface treat-ment (N)	
MMA 180 seconds (MM)	
MF-MA 15 seconds (MF)	
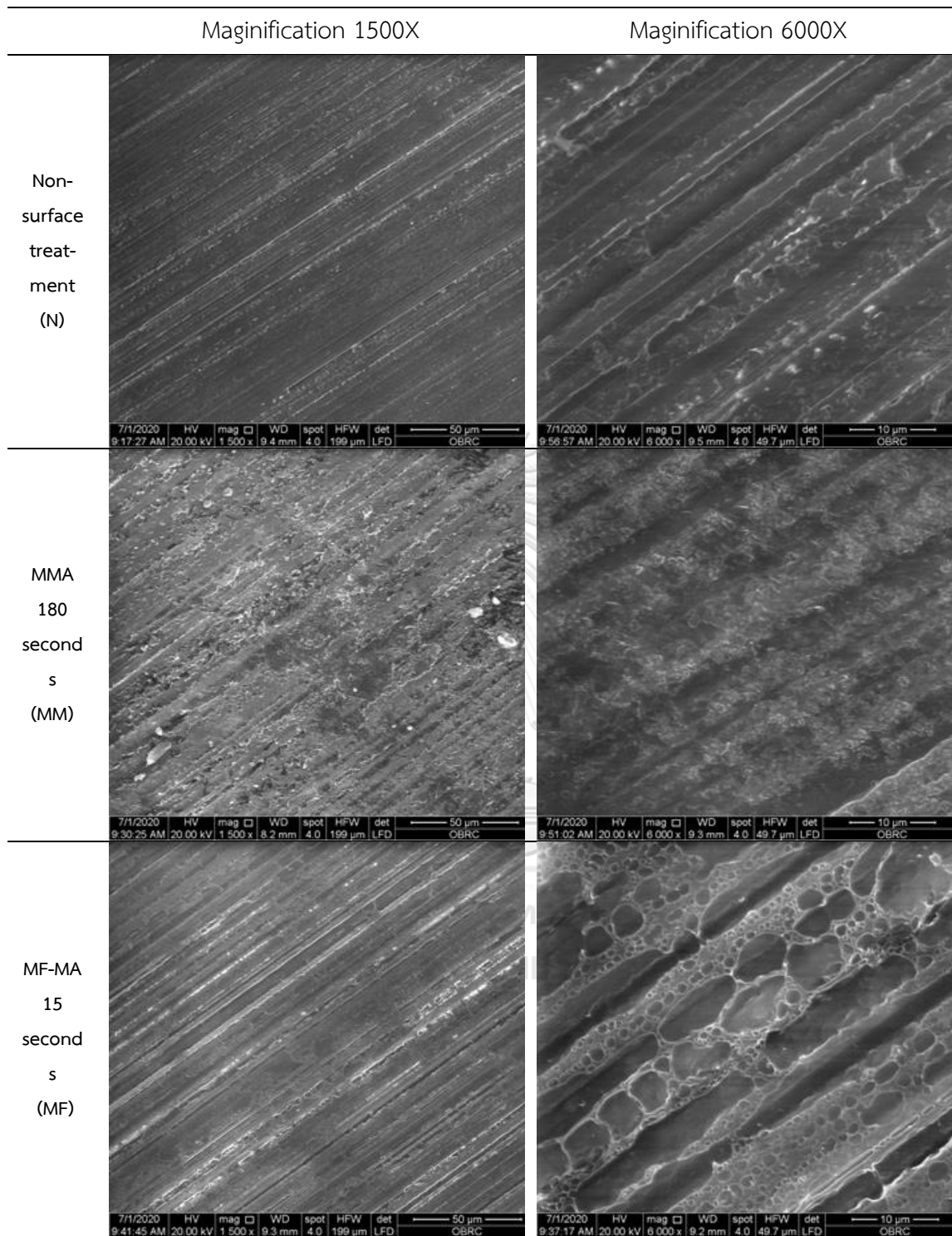
Figure 8. Scanning electron microscope of denture base with different surface treatments.

	Maginification 1500X	Maginification 6000X
Heat-polymeri-zing specimens (HX)		
Heat-polymeri-zing specimens after ultrasonic in water (HW)		
Heat-polymeri-zing specimens after ultrasonic in 30% ethanol (HE)		
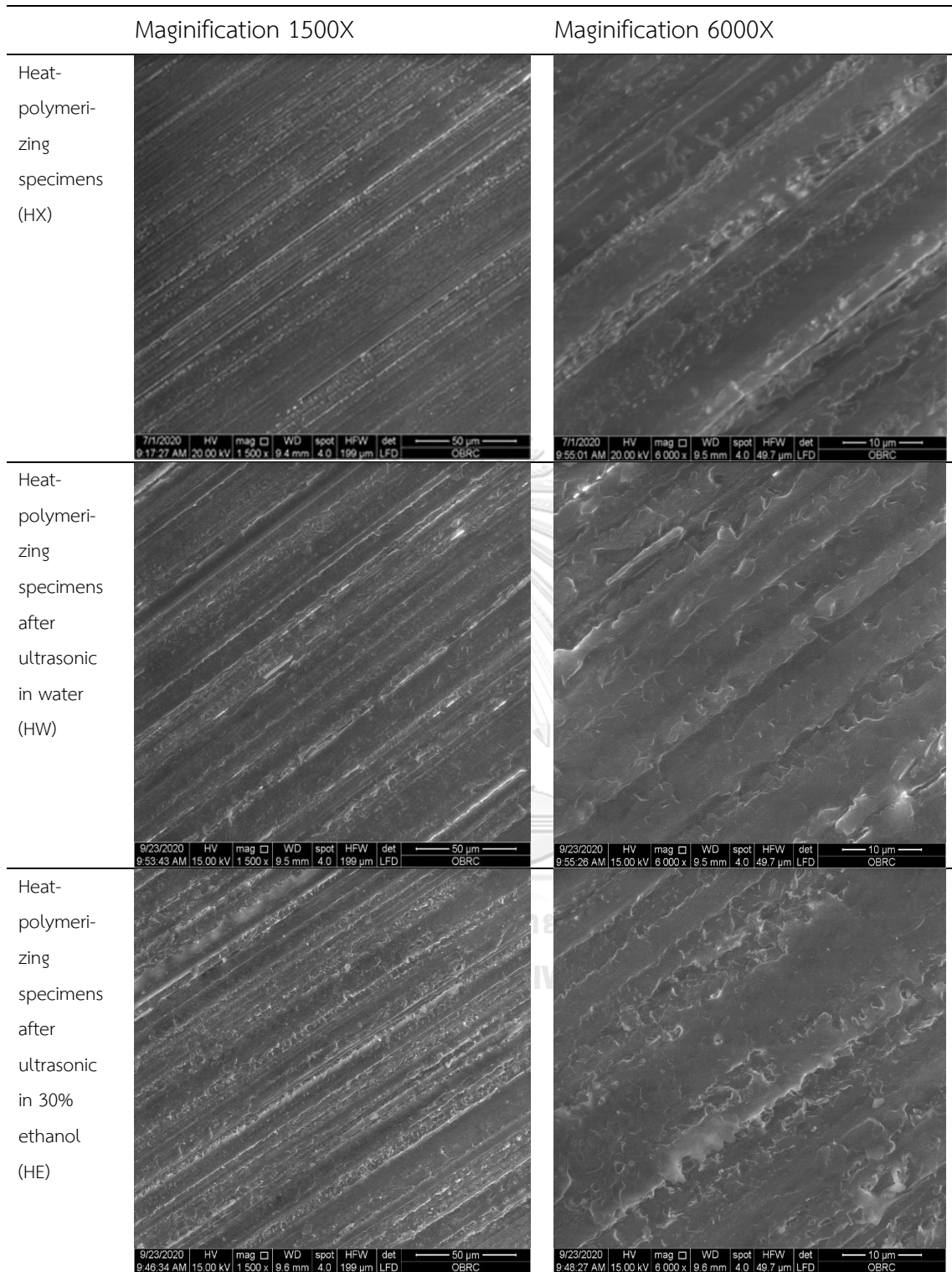
Figure 9. Scanning electron microscope of heat-polymerizing specimens with different ultrasonic treatments.

	Maginification 1500X	Maginification 6000X
Auto-polymerizing specimens (AX)		
Auto-polymerizing specimens after ultrasonic in water (AW)		
Auto-polymerizing specimens after ultrasonic in 30% ethanol (AE)		
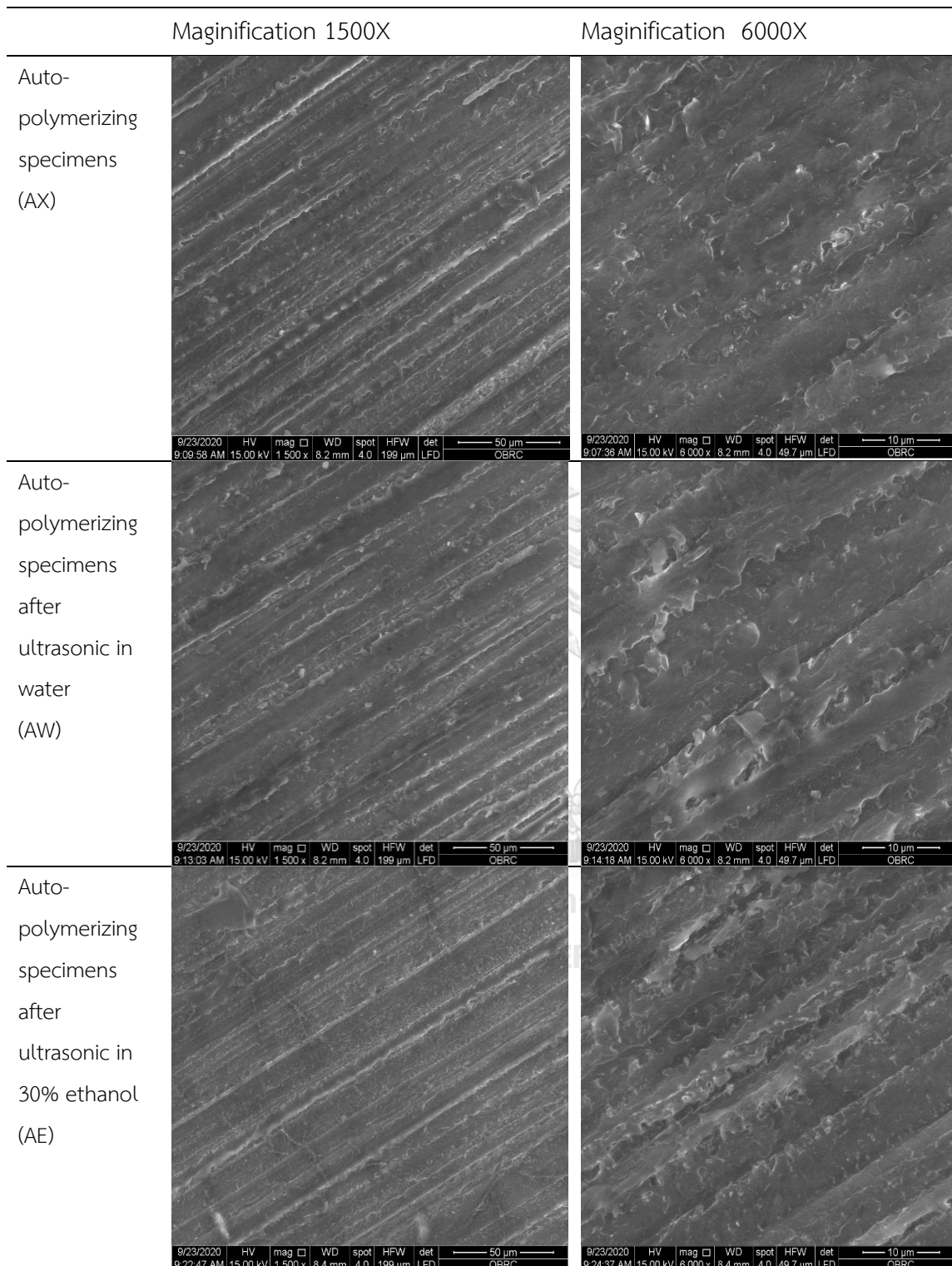
Figure 10. Scanning electron microscope of auto-polymerizing specimens with different ultrasonic treatments.

CHAPTER V

DISCUSSION AND CONCLUSION

Discussion

Based on the results, all relined groups presented higher mean transverse strength after water and 30% ethanol solution ultrasonic post-polymerization (Table 12). The result pointed out that ultrasonic post-polymerization effected transverse strength of relined denture-base. The first null hypothesis that the ultrasonic post-polymerization treatment had no effect on the transverse strength of relined denture base, was rejected.

The post-polymerization process might reduce the level of residual monomers. Residual monomers might cause the plasticizing effect which affected the mechanical properties of acrylic resins.[80] Studies presented effect of residual monomer to the mechanical properties.[23, 72] Thus, reduction of residual monomers improved the transverse strength. Studies reported that post-polymerizations could improve mechanical properties of acrylic resins.[50, 81, 82]

For water ultrasonic treatment groups (NW, MMW, MFW groups), showed higher mean transverse strength which might be the effects of warm water immersion combined with the energy from ultrasonic. First, residual monomers could leach out in liquid environments.[29] Tsuchiya H et al, 1994[58] suggested that preleaching the dentures in warm water could subside the amount of leaching residual monomers. Vallittu PK et al, 1995[83] confirmed that dentures soaking in 37°C water for 1-2 days could reduce residual monomers. Sometimes, high temperature immersion probably caused additional polymerization from residual monomers. Lamb DJ, 1982[29] revealed that active radical for auto-polymerizing acrylic resins could present for 60 minutes after polymerization passed. Furthermore, many studies supported and discussed about further polymerization concept after post-polymerization. Urban VM et al, 2009[84] found that post-polymerization in 55°C water for 10 minutes could increase degree of conversion. From the study of Bural C et al, 2011[73] auto-polymerized acrylic resins were performed post-polymerization by 60°C immersion for 30 minutes. They found that elution level of residual monomers decreased

(p<0.05) and increased degree of conversion. Seo SR et al, 2007[69] found that flexural strength of materials trended to raise after warm immersion. In contrast to Srihatajati J et al, 2017[85] the flexural strength could not significantly different after water immersion. This might be the result from difference time and temperature immersion.

Another factor influenced for these results was ultrasonic energy. Ultrasonic was suspected to be a relevant accelerator which provided better results within short post-polymerization time. Cavitation bubbles generated by ultrasonic might affect the specimens in two mechanisms when they exploded. The first, ultrasonic stimulated high velocity liquid-movement which helped in residual monomers diffusion out. The second, the more energy was released from the process which might accelerate further polymerization. Ultrasonic could generate the radical which initiated polymerizations.[76] Especially, the specimens in this study were immersion in ultrasonic bath immediately after relining process, some leftover active radical was possibly behaved for additional polymerization. Accordingly, Charasseangpaisarn T and Wiwatwarrapan C 2015, 2016[39, 77] found that ultrasonic treatment (100 watt, 40kHz, 50ºC water, 5 minutes) could reduce residual monomers of auto-polymerizing acrylic resin to the safety level significantly (p<0.05). In additions, Thummawanich W and wiwatwarrapan C, 2018[78] found that flexural strength of auto-polymerized orthodontic-acrylic resins increased significantly after water ultrasonic post-polymerization (p<0.05). As a result of the previous information, it could be hypothesized that water ultrasonic post-polymerization might help reduced residual monomers and raised transverse strength of acrylic resins.

The second hypothesis that 30% ethanol solution used in ultrasonic post-polymerization treatment dose not significantly effect on the transverse strength of relined denture base at the 95% confidence level was rejected.

All relined groups showed the highest mean flexural strength after 30% ethanol ultrasonic post-polymerization compared with water ultrasonic immersion significantly (p<0.05) (Table 12). These higher results might be the effect from ultrasonic heat-treatment and immersion in 30% ethanol solution together.

According to the effect of ultrasonic heat-treatment, the specimens might generate further polymerization which probably provided better transverse strength. Together with the present of ethanol solution, residual monomers might be extracted more easily than water. This effect could be explained by solubility parameter of the chemical and dissolving constant of the polymer. Because the solubility parameter (Hildebrand parameter) and Hydrogen-bonding capabilities of ethanol was closer to methyl methacrylate than water (26.0, 18.0 ,47.9 MPa$^{1/2}$ respectively).[47] Bettencourt et al, 2002[86] presented that ethanol concentration related to the amount of residual monomer elution. Even diluted concentration of ethanol, the elution effect could present remarkably when compared with water. Neves CB et al, 2013[68] found that high ethanol concentration could enlarge inner porous of acrylic resins. Their study supported that Lower ethanol concentration auto-polymerizing acrylic resins trended to provide better flexural strength. These might be explained by plasticizer effect of ethanol which influenced on glass transition temperature. Arriwiratchakun P and Wiwatwarrapan C, 2019[40] revealed that the both water and 30%ethanol ultrasonic immersion (40 kHz, 50°C, 5 minutes) could diminish the leaching unreacted monomer better than warm water immersion (50°C, 1 hour). However, higher ethanol concentration, more residual monomers released but the flexural strength could be diminished. As the result, dilute ethanol solution (<42%) could not cause significant adverse effects to materials such as hardness, roughness, flexural strength.[87] Importantly, the temperature at which the solvent behaved with the polymers had a significant effect in the degree of plasticizing. High temperature might help increasing materials' rigidity which counteracted plasticizing effect.[68] This implied that high temperature resulted in a lesser plasticizing effect. Moreover, the specimens were stored in distilled water for 48±2 hours before testing. This might reduce plasticizing effect of residual ethanol. Nevertheless, the study of Thummawanich W et al, 2018[78] presented that the flexural strength of auto-polymerized orthodontic acrylic resin performed ultrasonic post-polymerization in either water or 30% ethanol were not significant difference. The different materials and some methods might be involved.

For this study, the results of all 30% ethanol ultrasonic treatment groups (NE, MME, MFE) might be hypothesized that the equilibrium between ultrasonic heat-treatment and low ethanol concentration dictated the transverse strength of relined denture bases.

Pilot study revealed that heat-polymerization groups (H) presented the descending trend after ultrasonic post-polymerization. After 30% ethanol ultrasonic immersion, H groups (no reline) showed lower mean transverse strength than non-ultrasonic treatment group (HX= 79.27±1.61 MPa, HW= 78.68±0.77 MPa, HE= 77.53±1.26 MPa). It might be assumed that ultrasonic medium probably influenced. Heat apparently displayed the contrast from the relined dentures (NE, MME, MFE groups in table 12). From the information that ethanol could dissolve residual monomers better than water.[47] It could be conceived that ethanol might directly attack on the polymer's uncomplicated regions which were residual monomers. For heat-polymerized acrylic resins, polymerization process occurred more complete than auto-polymerized acrylic resins. Thus, amounts of residual monomers could be lower in heat-polymerized materials.[80] The target of ethanol might not primarily attack remaining monomers as in auto-polymerized resins. It meant that depolymerization could possibly occur when heat-polymerization acrylic resins had been in ethanol environment. Combined with ultrasonic, deterioration effect could possibly be magnified. Ultrasonic depolymerization also become more pronounced when the molecular weight of polymer increased. It was a limitation for the maximum molecular weight achievable.[76] Furthermore, the pilot study of auto-polymerization acrylic resins (A) showed that the transverse strength had raised after ultrasonic treatments (AX = 66.94±2.54 MPa, AW = 68.298±2.67 MPa, AE = 68.56±2.73 MPa). The pilot results might possibly support this conjecture. Basavarajappa S et al, 2017[88] confirmed that denture bases could be deteriorated by using ethanol solution. They found that the semi-interpenetrating layer between PMMA bead and polymer matrix was more prone to be dissolved than the core of the polymer bead and the cross-linked polymer matrix. They discussed that PMMA polymer beads were syndiotactic polymer which effectively prevented dissolving and crazing effects

compared to that of the semi-IPN structure. Sofou A et al, 2005[65] also confirmed that the heat-polymerization acrylic resins considerably leached monomer substances after ethanol immersion. Moreover, any dilute alcohol solution containing possibly attacked PMMA.[89] These information could support the present results of heat-polymerizing acrylics. However, the transverse strength of relined groups resulted from the combine effects of further polymerization and depolymerization. Especially, the effect of further polymerization could predominantly show in the presence of auto-polymerizing acrylic resins.

According to the above information, it possibly implied that ethanol solution used in ultrasonic process might be the factor which modified the transverse strength of acrylic resin materials.

In the aspect of chemical surface treatments, some significant difference (p<0.05) in mean transverse strength were revealed. The third hypothesis that chemical surface treatment dose not significantly effect on the transverse strength of relined denture base at the 95% confidence level was rejected.

The present results uncovered that mean transverse strength of relined denture bases which applied chemical surface treatments (MM, MF groups), increased altogether significantly (p<0.05) when compared with non-surface treatment groups (N groups) within the same post-polymerization actions (Table 12). It referred that chemical surface treatments possibly influenced the transverse strength of relined denture bases. In additions, mean transverse strength of relined denture bases using MMA (MM groups) were not significant difference compared with utilizing MF-MA (MF groups) in the same post-polymerization conditions.

According to Hout D et al, 2017[11] the bond strength of hard liner to denture bases correlated with the flexural strength of the whole pieces. Chemical surface treatments used in this study helped increase the bond strength by providing the micro-mechanical retention. Scanning electron microscope (SEM) discovered that there were some irregularities on the surface of heat-polymerized acrylic resins (Figure 8). In the spite of the fact that methyl methacrylate (MMA) and methyl formate-methyl acetate (MF-MA) seem swell denture base surface in different

appearances. However, they did not provide significant difference transverse strength of each other for this study.

As the theory of solubility, the solvent having the similar "solubility parameter" to the denture base could dissolve and swell the denture base surface generated surface irregularities. The solubility parameter of denture base, MMA, MF, MA were 18.3, 18.0 , 20.9 and 19.6 MPa$^{1/2}$ respectively.[47, 90] These irregular surfaces benefited for bonding. The monomer of hard liner acrylic resins could easily penetrate into those surface irregularities and polymerized forming the hybrid layer which increased the bond strength. Vallittu PK and Ruyter IE, 1997[91] also confirmed that acrylic resins could be swelled and allow interprenating network formation. From SEM images MF-MA generated large size porosities than MMA (Figure 8).

Kungkapilas K and Santawisuk W, 2014[13] found that MMA did not provide the significant difference shear bond strength when compared with MF-MA and control (p>0.05). With the presence of adhesive Tokuyama Rebase II, MF-MA group raised higer shear bond strength of relined dentutre bases than MMA group significantly (p<0.05).

The transverse strength of relined denture bases using MMA and MF-MA in this study did not differed from each other. Nevertheless, Osathananda R and Wiwatwarrapan C, 2014[20] found that appling MF-MA 25:75 could enhance shear bond strength of relined acrylic resin superior to appling MMA significantly. Tanasamanchoke C and Wiwatwarrapan C, 2015[22] figured out that wetting MF-MA (25:75% volume) for 15 seconds, relined specimems had higher tensile bond strength than non-wetting specimens significantly (p<0.05) (4.95±0.75 MPa, 7.38±0.40 MPa respectively). Moreover, Thunyakitpisal N et al, 2011[19] presented that transverse strength of repair denture bases raised by MF-MA (65.6±3.7 MPa) higher than MMA (58.5±2.8 MPa) significantly (p<0.05). It was that transverse strength of relined specimens might be influenced by combinding effect of heat and auto-polymerized acrylic resin. Thus, transverse strength in relining might not directly refer to the effect of bond strength between these two chemical surface treatments.

In additions, fracture characteristic in this study could support that chemical surface treatment benefited for relining. After ultrasonic post-polymerization, the specimens of no surface treatment groups presented higher relining-interface fracture while the samples treated with MMA or MF-MA groups showed the reduction of inter-relining surface fracture (Figure 7). Chemical surface treatments could increase flexural strength and decrease relining-interface fracture. With those benefits from proper bonding, chemical surface treatments were still recommended for denture relining.

Conclusion

Either ultrasonic with water or 30% ethanol could enhance the transverse strength of relined denture base. Therefore, ultrasonic post-polymerization profited for denture base relined with auto-polymerized acrylic resin.

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

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APPENDIX

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Table 13. Analysis of the data distribution.

Tests of Normality

	Group	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
Transverse Strength	1 HX	.172	10	.200[*]	.931	10	.457
	2 HW	.191	10	.200[*]	.905	10	.246
	3 HE	.220	10	.186	.848	10	.055
	4 NX	.171	10	.200[*]	.939	10	.541
	5 NW	.198	10	.200[*]	.904	10	.242
	6 NE	.191	10	.200[*]	.911	10	.290
	7 MMX	.169	10	.200[*]	.927	10	.418
	8 MMW	.218	10	.194	.941	10	.563
	9 MME	.249	10	.080	.848	10	.056
	10 MFX	.185	10	.200[*]	.905	10	.246
	11 MFW	.157	10	.200[*]	.958	10	.759
	12 MFE	.228	10	.149	.835	10	.038

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Table 14. Descriptive statistics of relined specimens' data.

Descriptive Statistics

Dependent Variable: Transverse strength

chem	postpoly	Mean	Std. Deviation	N
Non surface treatment	No US	66.8290	1.05268	10
	water	69.1060	1.35191	10
	ethanol	73.1450	1.41631	10
	Total	69.6933	2.93087	30
MMA	No US	69.7450	1.18500	10
	water	72.8560	1.10039	10
	ethanol	74.3620	1.41852	10
	Total	72.3210	2.29334	30
MF-MA	No US	69.8130	1.21779	10
	water	71.7650	1.15530	10

	ethanol	75.6000	1.50600	10
	Total	72.3927	2.74884	30
Total	No US	68.7957	1.80037	30
	water	71.2423	1.98068	30
	ethanol	74.3690	1.72917	30
	Total	71.4690	2.92744	90

Table 15. Levene's Test of Equality of Error

Variances[a]

Dependent Variable: Transverse strength

F	df1	df2	Sig.
.381	8	81	.928

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Design: Intercept + chem + postpoly + chem * postpoly

Table 11. Two-way ANOVA analysis of post-polymerizations and chemical surface treatments effected on relined denture based.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	630.829[a]	8	78.854	48.427	.000	.827
Intercept	459703.616	1	459703.616	282319.941	.000	1.000
chem	141.962	2	70.981	43.592	.000	.518
postpoly	468.243	2	234.121	143.782	.000	.780
chem * postpoly	20.624	4	5.156	3.167	.018	.135
Error	131.893	81	1.628			
Total	460466.338	90				
Corrected Total	762.721	89				

a. R Squared = .827 (Adjusted R Squared = .810)

Table 16. Estimated Marginal Means

1. chem

Dependent Variable: Transverse strength

chem	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
no treatment	69.693	.233	69.230	70.157
MMA	72.321	.233	71.857	72.785
MF-MA	72.393	.233	71.929	72.856

2. post-polymerization

Dependent Variable: Transverse strength

Post-polymerization	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
No US	68.796	.233	68.332	69.259
water	71.242	.233	70.779	71.706
ethanol	74.369	.233	73.905	74.833

Table 17. One-way ANOVA transverse strength by chemical surface treatments of relined specimens.

Test of Homogeneity of Variances

Transverse strength

Levene Statistic	df1	df2	Sig.
1.804	2	87	.171

ANOVA

Transverse strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	141.962	2	70.981	9.948	.000
Within Groups	620.760	87	7.135		
Total	762.721	89			

Multiple Comparisons

Dependent Variable: Transverse strength

	(I) chem	(J) chem	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	no treatment	MMA	-2.62767*	.68969	.001	-4.2722	-.9831
		MF-MA	-2.69933*	.68969	.001	-4.3439	-1.0548
	MMA	no treatment	2.62767*	.68969	.001	.9831	4.2722
		MF-MA	-.07167	.68969	.994	-1.7162	1.5729
	MF-MA	no treatment	2.69933*	.68969	.001	1.0548	4.3439
		MMA	.07167	.68969	.994	-1.5729	1.7162
Games-Howell	no treatment	MMA	-2.62767*	.67944	.001	-4.2644	-.9909
		MF-MA	-2.69933*	.73362	.001	-4.4641	-.9345
	MMA	no treatment	2.62767*	.67944	.001	.9909	4.2644
		MFMA	-.07167	.65359	.993	-1.6451	1.5018
	MF-MA	no treatment	2.69933*	.73362	.001	.9345	4.4641
		MMA	.07167	.65359	.993	-1.5018	1.6451

*. The mean difference is significant at the 0.05 level.

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Homogeneous Subsets

Transverse strength

	chem	N	Subset for alpha = 0.05	
			1	2
Tukey HSD[a]	no treatment	30	69.6933	
	MMA	30		72.3210
	MF-MA	30		72.3927
	Sig.		1.000	.994

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 30.000.

Table 18. One-way ANOVA transverse strength by post-polymerization of relined specimens.

Test of Homogeneity of Variances

Transverse strength

Levene Statistic	df1	df2	Sig.
.335	2	87	.716

ANOVA

Transverse strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	468.243	2	234.121	69.168	.000
Within Groups	294.479	87	3.385		
Total	762.721	89			

Robust Tests of Equality of Means

Transverse strength

	Statistic[a]	df1	df2	Sig.
Welch	74.474	2	57.823	.000

a. Asymptotically F distributed.

Multiple Comparisons

Dependent Variable: Transverse strength

	(I) postpoly	(J) postpoly	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	No US	water	-2.44667[*]	.47503	.000	-3.5794	-1.3140
		ethanol	-5.57333[*]	.47503	.000	-6.7060	-4.4406
	water	No US	2.44667[*]	.47503	.000	1.3140	3.5794
		ethanol	-3.12667[*]	.47503	.000	-4.2594	-1.9940
	ethanol	No US	5.57333[*]	.47503	.000	4.4406	6.7060
		water	3.12667[*]	.47503	.000	1.9940	4.2594
Games-Howell	No US	water	-2.44667[*]	.48869	.000	-3.6224	-1.2709
		ethanol	-5.57333[*]	.45575	.000	-6.6696	-4.4771
	water	No US	2.44667[*]	.48869	.000	1.2709	3.6224
		ethanol	-3.12667[*]	.48004	.000	-4.2819	-1.9715
	ethanol	No US	5.57333[*]	.45575	.000	4.4771	6.6696
		water	3.12667[*]	.48004	.000	1.9715	4.2819

*. The mean difference is significant at the 0.05 level.

Homogeneous Subsets

Transverse strength

	postpoly	N	Subset for alpha = 0.05		
			1	2	3
Tukey HSD[a]	No US	30	68.7957		
	water	30		71.2423	
	ethanol	30			74.3690
	Sig.		1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 30.000.

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