

ความแข็งแรงพันธะดีระหว่างอะคริลิกเรซินชนิดบ่มด้วยตัวเอง กับซีฟีนเทียมอะคริลิกประเภทต่างๆ  
ที่ปรับสภาพผิวด้วยสารละลายเมทิลฟอร์มเมตและเมทิลอะซิเตต



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CHULALONGKORN UNIVERSITY

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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

TENSILE BOND STRENGTH BETWEEN SELF-CURED ACRYLIC RESIN  
AND VARIOUS ACRYLIC DENTURE TEETH TREATED WITH METHYL FORMATE-  
METHYL ACETATE SOLUTION

Miss Ticha Thongrakard



A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Master of Science Program in Prosthodontics

Department of Prosthodontics

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ทิตา ทอระภาค : ความแข็งแรงพันธะตั้งระหว่างอะคริลิกเรซินชนิดบ่มด้วยตัวเอง กับซีฟันเทียมอะคริลิกประเภทต่างๆที่ปรับสภาพผิวด้วยสารละลายเมทิลฟอร์มเมตและเมทิลอะซิเตต (TENSILE BOND STRENGTH BETWEEN SELF-CURED ACRYLIC RESIN AND VARIOUS ACRYLIC DENTURE TEETH TREATED WITH METHYL FORMATE-METHYL ACETATE SOLUTION) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ชัยรัตน์ วิวัฒน์วรพันธ์ , หน้า.

งานวิจัยนี้ศึกษาผลของการทาสารเคมีบนผิวซีฟันเทียมอะคริลิกเพื่อปรับปรุงพื้นผิว ก่อนนำไปเชื่อมด้วย อะคริลิกเรซินชนิดบ่มด้วยตัวเอง โดยนำฟันตัดหน้าซี่กลางบน 3 ยี่ห้อ (Yamahachi New Ace: YA, Cosmo HXL: CH, Trubyte Bioform IPN: TB) มาขัดกระดาษทรายตำแหน่งผิวด้านประชิดสันเหงือก โดยมีการแบ่งซีฟันแต่ละผลิตภัณฑ์เป็น 2 ส่วน ส่วนแรกแบ่งเป็นทั้งหมด 7 กลุ่ม (n=10) ดังนี้ : กลุ่มไม่ทาสาร, กลุ่มทาสารละลายเมทิลฟอร์มเมต และเมทิลอะซิเตต (MF-MA) ที่อัตราส่วน 25:75 โดยปริมาตร (CU Acrylic Bond) เป็นเวลา 15, 30, 60, 120, 180 วินาที และกลุ่มทาสารเมทิลเมทาคริเลต (MMA) 180 วินาที ส่วนที่สองคือกลุ่มที่นำไปแช่เครื่องสลับน้ำร้อนน้ำเย็น แบ่งเป็น 3 กลุ่ม (n=10) คือ กลุ่มไม่ทาสาร, กลุ่มทา MMA 180 วินาที และกลุ่มทา MF-MA ตามเวลาแนะนำจากผลการทดลองในส่วนแรก จากนั้นนำชิ้นงานมายึดด้วยอะคริลิกเรซินชนิดบ่มด้วยตัวเอง (Unifast Trad) แล้วจึงนำมาทดสอบแรงดึงด้วยเครื่องทดสอบแรงดึงแรงอัด นำค่าเฉลี่ยความแข็งแรงพันธะตั้งของแต่ละกลุ่มมาวิเคราะห์ข้อมูลทางสถิติด้วยการวิเคราะห์ความแปรปรวนแบบทางเดียว ผลการศึกษาคือ กลุ่มที่ทาสารปรับสภาพผิวมีความแข็งแรงตั้งสูงกว่ากลุ่มที่ไม่ทาสารอย่างมีนัยสำคัญทางสถิติในซีฟันยี่ห้อเดียวกัน ( $p < 0.05$ ) ยกเว้นกลุ่มทา MF-MA 15 วินาทีของยี่ห้อ TB ( $p > 0.05$ ) กลุ่มทา MMA 180 วินาที ไม่มีความแตกต่างกันทางสถิติกับกลุ่มทา MF-MA 15 และ 30 วินาที ( $p > 0.05$ ) และพบว่า กลุ่มซีฟันทุกผลิตภัณฑ์ที่ไม่มีการทาสารและผ่านการแช่เครื่องสลับน้ำร้อนน้ำเย็น มีค่ากำลังความแข็งแรงตั้งไม่แตกต่างจากกลุ่มที่ไม่ผ่านการแช่อย่างมีนัยสำคัญทางสถิติ ( $p < 0.05$ ) สรุป MF-MA จึงสามารถเป็นสารเคมีทางเลือกในการปรับปรุงผิวซีฟันเทียมอะคริลิกก่อนการเชื่อมด้วยอะคริลิกเรซินชนิดบ่มด้วยตัวเอง โดยทาเป็นเวลา 15 หรือ 30 วินาทีแล้วแต่ชนิดของซีฟันเทียม ในการแช่เครื่องสลับน้ำร้อนน้ำเย็น กลุ่มซีฟันที่ทาสารทุกกลุ่ม มีค่าความแข็งแรงพันธะตั้งลดลง แต่ยังคงมีค่าไม่แตกต่าง หรือสูงกว่ากลุ่มที่ไม่ทาสาร

ภาควิชา ทันตกรรมประดิษฐ์

ลายมือชื่อนิสิต .....

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KEYWORDS: TENSILE BOND STRENGTH / DENTURE TEETH / DENTURE BASE / SELF-CURED ACRYLIC / METHYL FORMATE / METHYL ACETATE / THERMOCYCLING

TICHA THONGRAKARD: TENSILE BOND STRENGTH BETWEEN SELF-CURED ACRYLIC RESIN AND VARIOUS ACRYLIC DENTURE TEETH TREATED WITH METHYL FORMATE-METHYL ACETATE SOLUTION. ADVISOR: ASSOC. PROF. CHAIRAT WIWATWARRAPAN, pp.

This study evaluated the use of methyl formate-methyl acetate (MF-MA) solution on the tensile bond strength between acrylic denture teeth and self-cured acrylic resin. Maxillary central incisor acrylic denture teeth (Yamahachi New Ace: YA, Cosmo HXL: CH, Trubyte Bioform IPN: TB) were ground on their ridge lap surfaces. This study was divided into two parts. First part, the teeth of each brand were divided into seven groups (n=10) (no treatment, MF-MA for 15, 30, 60, 120, 180 s, and MMA for 180 s. Second part, the teeth of each brand were divided into three groups (n=10) with thermocycling (no treatment, MMA for 180 s, and MF-MA for the optimum time determined in the first part). After their respective treatments, self-cured acrylic resin (Unifast Trad) was applied. The results were analyzed using one-way analysis of variance (ANOVA). The results of the first part indicated that the surface treatment groups had significantly higher tensile bond strengths compared with no treatment group ( $p < 0.05$ ) within the same brand, except for TB MF-MA 15 s group ( $p > 0.05$ ). There were no significant differences in tensile bond strength between MMA 180 s, MF-MA 15 and 30 s groups ( $p > 0.05$ ). In second part, there were no significant differences between the thermocycling and non-thermocycling control groups of all brands ( $p > 0.05$ ). However, significant differences were present between some treatment groups ( $p < 0.05$ ). The results indicated that application of MF-MA for 15 s or 30 s can be an alternative chemical surface treatment for rebonding acrylic denture teeth with self-cured acrylic resin. Although thermocycling reduced bond strength of treatment groups, it did not in the control groups.

Department: Prosthodontics Student's Signature .....

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

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# CHAPTER I

## INTRODUCTION

### Background and rationale

The most common type of acrylic denture failure is debonding or fracture of the denture teeth, accounting for approximately 33% of failures.(1) This failure usually occurs in the anterior region of the denture. Dentists and patients both prefer chair-side repair using self-cured acrylic resin, because it is a straight-forward procedure requiring less chair time than repair in the laboratory. A likely explanation for teeth debonding is contamination of the tooth surface with substances such as wax during laboratory processing. Another cause of debonding is a chemical difference between the tooth and the denture base due to different processing methods.(2) A weak repaired interface can lead to recurrent debonding. Therefore, the surface treatment method (mechanical or chemical) used on the denture teeth is important for successful denture repair. The placement of a diatoric recess (mechanical) or the use of a bonding agent (chemical) on denture teeth resulted in higher bond strengths compared with no treatment.(3, 4) Methyl methacrylate (MMA) is the most frequently used chemical surface treatment agent. MMA is effective in promoting adhesion because of its chemical similarities to denture base resin. However, surface treatment with MMA requires 3 minutes to effectively prime the surface and diminish adhesive failures,(5) resulting in excessive chair time. Other solutions have been used as chemical surface treatments, e.g., chloroform, methylene chloride (or dichloromethane), and 4-methacryloxyethyl trimellitate anhydride (4-META). Although chloroform and methylene chloride have been used as surface treatment agents, these are now known to be carcinogenic.(6) This study found that methyl formate (MF) and methyl acetate (MA) solutions were non-toxic and their use as surface treatment agents resulted in similar bond strengths to poly(methyl methacrylate) (PMMA) to those obtained when methylene chloride was used. Another study found that MF, MA, and an MF-MA mixture significantly enhanced the flexural strength of heat-cured acrylic denture base resin that had

been repaired with self-cured acrylic resin. The scanning electron micrographs in their study demonstrated that the application of these solutions to the surface of heat-cured acrylic resin resulted in a 3D honeycomb appearance, whereas specimens treated with MMA developed shallow pits.(7) In addition, other studies have been conducted investigating the surface treatment of acrylic denture base and relined resin with MF-MA, finding that that MF-MA significantly enhanced the bond strength between these materials.(8, 9)

A study of the application of MF-MA on acrylic denture teeth revealed that a 15 s application before packing with heat-cured acrylic denture base resulted in a significantly higher micro-tensile bond strength between the teeth and the denture base, compared with the non-surface treated group. Moreover, there was no significant difference between the MF-MA and MMA groups, using a 15 s and a 180 s, respectively, on micro-tensile bond strength.(10) Therefore, MF-MA may be an acceptable alternative for MMA because it is less toxic.

Acrylic denture tooth structure can also influence its bond strength to a denture.(3, 11) Conventional denture teeth have low wear resistance,(12) and cross-linking the polymer matrix of the denture teeth results in increased wear resistance.(13) Because of their complex structure, cross-linked acrylic denture teeth have less polymer penetration compared with conventional denture teeth, resulting in a lower bond strength. In contrast, another study found that there was no significant difference in bond strength between conventional and cross-linked denture teeth bonded to self-cured acrylic resin.(14) Thus, crosslinking is not a major factor in reducing the strength of the adhesion between denture teeth and denture base.

There are various methods for determining the bond strength between denture teeth and denture base, such as American Dental Association Specification number 15 (ADA 15),(15) International Organization for Standardization for synthetic resin teeth (ISO 3336),(16) or the finite element stress analysis technique.(17) However, these methods can be criticized concerning their accuracy in determining bond strength. Moreover, the lack of uniformity in the tooth-denture base testing

methods does not allow bond strength to be investigated in a standardized manner or the results of studies using different methods to be directly compared(2)

During denture use, the temperature and fluid in the oral cavity can affect the denture teeth-denture base interface. These factors deteriorate the bond strength by leaching monomer and absorbing water, resulting in occasional debonding.(18) Thermocycling also affects certain acrylic tooth/denture base resin combinations, with the effects varying based on the materials used.(19)

Past studies of the use of MF-MA solution have indicated that it is a non-toxic surface treatment agent that improved the bond strength of acrylic resin materials. However, there have been no studies of the bond strength when using MF-MA for the repair of denture teeth with self-cured acrylic resin.



## Objective

To evaluate the chemical surface-treatment effect of various MF-MA solution wetting times on bond strength between three types of acrylic denture teeth repaired with acrylic resin before and after thermocycling.

## Hypothesis

- $H_0$ : There is no significant difference in tensile bond strength between MF-MA and MMA treatment at the 95% confidence level.

$H_1$ : There is a significant difference in tensile bond strength between MF-MA and MMA treatment at the 95% confidence level.
- $H_0$ : There is no significant difference in tensile bond strength between the MF-MA application-time groups at the 95% confidence level.

$H_1$ : There is a significant difference in tensile bond strength between the MF-MA application-time groups at the 95% confidence level.
- $H_0$ : There is no significant difference in tensile bond strength between acrylic-teeth types in the same surface treatment groups at the 95% confidence level.

$H_1$ : There is a significant difference in tensile bond strength between acrylic-teeth types in the same surface treatment groups at the 95% confidence level.
- $H_0$ : There is no significant difference in tensile bond strength between the thermocycling and non-thermocycling groups at the 95% confidence level.

$H_1$ : There is a significant difference in tensile bond strength between the thermocycling and non-thermocycling groups at the 95% confidence level.



### **Study limitations**

1. This research was conducted in a laboratory setting.
2. Conventional, semi-interpenetrating polymer network, and interpenetrating polymer network type acrylic denture teeth were used.
3. Unifast TRAD was the self-cured acrylic used.
4. All procedures were performed by a single investigator using the same instruments.

### **Benefits of this study**

MF-MA is less toxic compared with other surface treatment agents. Therefore it might be an alternative chemical surface treatment solution for repairing denture base with all types of acrylic denture teeth.

### **Key words**

acrylic denture teeth, methyl acetate, methyl formate, self-cured acrylic, methyl acetate, tensile bond strength, thermocycling

## CHAPTER II

### LITERATURE REVIEW

#### Denture base

A denture base is defined as that part of a denture that rests on the foundation tissue and to which teeth are attached.(20) Denture base can be classified according to its chemical composition and curing method based on ISO 20795-1: 2008. Denture base classification is shown in Table 1.

Table 1 Denture base classification.

Type	Material	Curing method
1	Heat-curing polymers	Cured at temperature more than 65°C
2	Self-curing polymers	Cured at temperature less than 65°C
3	Thermoplastic materials	Polymers formed when heated
4	Light-curing materials	Cured with Ultraviolet ray or visible light
5	Materials for microwave polymerization	Cured with microwave

The most commonly employed material in fabricating denture bases is acrylic resin (PMMA). Although the use of PMMA is popular and adequate in satisfying esthetic demands, it is far from ideal in fulfilling the mechanical requirements of a denture. This is reflected in the common problem of denture fracture.(1)

When dentures are fractured or their teeth debond, heat-cured or self-cured denture resin should be used in their repair. Heat-cured resin requires a laboratory process, while self-cured resin can be used for chair-side repairs. Thus, repairs using self-cured resin take less time and patients get their prosthesis back in one visit.

## Self-cured acrylic resin



<http://www.gcasia.info/proddet.asp?prodid=118>

Figure 1 Self-cured acrylic resin products.

Self-cured acrylic resins (chemically curing acrylic resin, cold-curing acrylic resin, and auto-polymerized acrylic resin), are shown in Figure 1, and the composition of self-cured acrylic resins are shown in Table 2. The composition of self-cured acrylic resin is the same as that of heat-cured acrylic resin; however, self-cured acrylic resin also contains a chemical polymerization activator (Figure 2). In heat-cured acrylic resin, the polymerization process is activated solely by heat.

### Recommended indications

- Repair dentures.
- Construction of temporary crowns.
- Impression trays for individual teeth.
- Orthodontic plates, splints, and other general purposes.

### Direction for use

The recommended powder/liquid ratio is 1 g of powder to 0.5 mL of liquid. The two components are combined and mixed for 10-15 s. The mixture reaches the dough-stage 20-30 s after mixing. All manipulation should be finished before 2 minutes after mixing because the mixture begins to set at that time.

Table 2 Self-cured acrylic resin compositions..

powder		liquid	
content	function	content	function
-Poly(methyl methacrylate)	-Dissolved by monomer to form dough	-Methyl methacrylate monomer	-Dissolves/plasticizes polymer
-Benzoyl peroxide	-Initiator	-Dimethyl-para-toluidine	-Activator
-Compounds of mercuric sulphide, cadmium sulphide	-Dyes	-Dibutyl phthalate	-Plasticizer
-Zinc or titanium oxide	-Opacities	-Glycol dimethacrylate	-Cross-linking agent
-Dibutyl phthalate	-Plasticizer	-Hydroquinone	-Inhibitor
-Dyed organic fillers, inorganic particles like glass fibers or bead	-Esthetics		

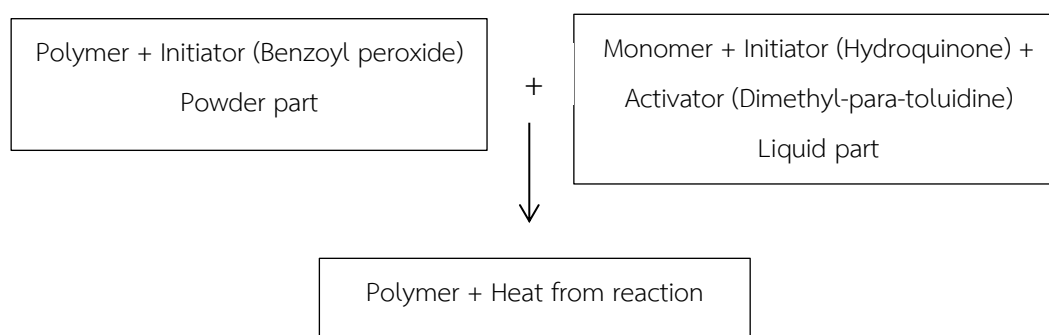


Figure 2 Polymerization reaction mechanism

### Storage

Store in a cool place away from direct sunlight. (Shelf life: 3 years from date of manufacture)

### Disadvantages

- Self-cured resin may cause sensitivity or tissue irritation in some people. The maximum amount of monomer released by self-cured denture base resin in the first hour in whole saliva was 29.5  $\mu\text{g/mL}$ , which, while not a toxic or primary irritant dose, may sensitize patients or elicit an allergic reaction. The denture should be immersed for 24 hours in water before being delivered to a patient.(21)
- Self-cured resin has lower strength compared with heat-cured resin.(22)

### Characteristics

- Heat is not necessary for polymerization.
- Porosity and distortion is greater than heat-cured acrylic resin.
- High residual monomer (0.2 weight% in heat-cured polymers and 4 weight% in self-cured polymers),(23) act as plasticizers that decrease the flexural strength of dentures.
- Poor color stability.
- Easy to deflask.

## Denture teeth

Denture teeth can be classified into 2 types according to the materials used.

### 1. Porcelain denture teeth

These artificial teeth were popular before the development of acrylic denture teeth. However these teeth present several problems. A common problem is enamel wear on the opposing natural teeth due to the hardness of porcelain, especially if it is not polished. In addition, porcelain teeth make a clicking sound when chewing. Moreover, they do not chemically adhere to the denture base. Porcelain teeth rely on mechanical mounting holes or grooves. One method to achieve chemical adhesion is by treating porcelain teeth with a silane coupling agent.(24)

### 2. Acrylic denture teeth

Acrylic resin polymer teeth are composed of PMMA beads and color pigments in a cross-linked polymer matrix. Acrylic teeth are currently popular due to their micromechanical bonding to denture base and ease in adjustment compared with porcelain teeth. Acrylic teeth are classified into 2 types.

#### *2.1 Conventional acrylic resin teeth*

These teeth are composed of one resin type, methyl methacrylate (linear PMMA). Their fracture resistance is higher than that of porcelain teeth, however, their wear resistance is lower.(12)

Examples of this tooth type are Major Dent (Major Prodotti Dentari, Italy), Basic (Heraus Kulzer, Germany), and Yamahachi FX (Yamahachi Dental Mfg.Co., Aichi Pref., Japan).

#### *2.2 Improved acrylic resin teeth*

These teeth have an improved structure that increases wear resistance compared with the conventional tooth type.(13, 25)

## 2.2.1 Cross-linked acrylic resin teeth

There are 2 types of cross-linked denture teeth

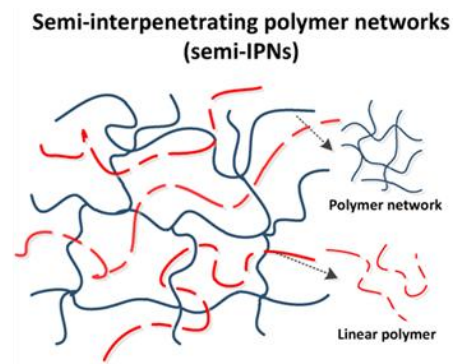
2.2.1.1 Semi-interpenetrating polymer network denture teeth (Semi-IPN): These teeth have a non-covalent Semi-IPN that has only one of the polymer systems cross-linked (Figure 3). An example of this type is Cosmo HXL (Dentsply International, Inc., York, USA).

2.2.1.2 Interpenetrating polymer network denture teeth (IPN): These teeth have a non-covalent full IPN that has two separate independently cross-linked polymers (Figure 4). Examples of this type of tooth are Trubyte Bioform IPN, Trubyte Portrait IPN, and Trubyte Bioform IPN (Dentsply International, Inc., York, USA).

### IUPAC definition(26)

A semi-interpenetrating polymer network (Semi-IPN) is a polymer comprising one or more networks and one or more linear or branched polymer(s) characterized by the penetration on a molecular scale of at least one of the networks by at least some of the linear or branched macromolecules (Figure 3).

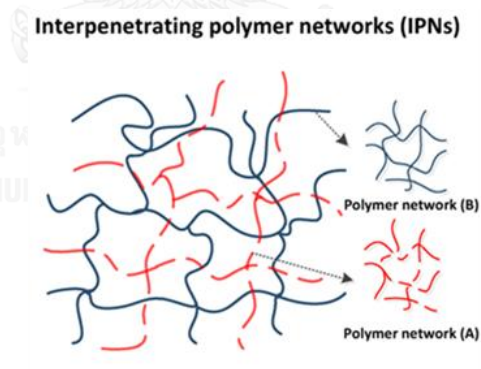
Semi-IPNs are distinguished from interpenetrating polymer networks because the constituent linear or branched polymers can, in principle, be separated from the constituent polymer network(s) without breaking chemical bonds, i.e. they are polymer blends.



<http://www.mdpi.com/2073-4360/4/2/913/htm>

Figure 3 Schematic representation of a Semi-IPN.

An interpenetrating polymer network (IPN) is a polymer comprising two or more networks that are at least partially interlaced on a molecular scale, but not covalently bonded to each other and cannot be separated unless chemical bonds are broken. A mixture of two or more pre-formed polymer networks is not an IPN (Figure 4).



<http://www.mdpi.com/2073-4360/4/2/913/htm>

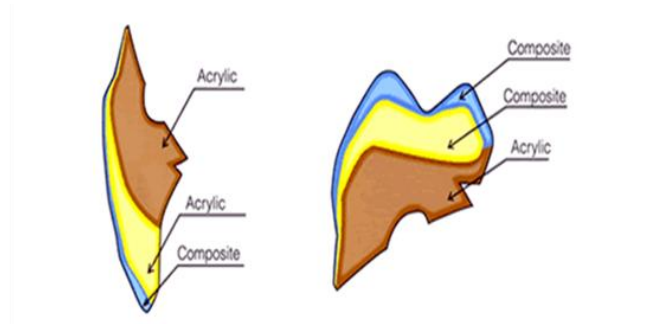
Figure 4 Schematic representation of an IPN.

### 2.2.2 Composite resin teeth

Composite resin teeth have a wear resistance that is higher than that of the conventional tooth type, however, there is no significant difference in wear resistance compared with porcelain



teeth.(27) Composite resin teeth also have a very esthetic appearance because the labial and occlusal surfaces are coated with composite resin. These teeth are known as Multilithic teeth (Figure 5).



[http://www.yamahachi-dental.co.jp/en/products/01artificial\\_resin\\_teeth/01composite\\_resin\\_teeth/01what\\_is\\_px/](http://www.yamahachi-dental.co.jp/en/products/01artificial_resin_teeth/01composite_resin_teeth/01what_is_px/)

Figure 5 Structural layers of composite resin teeth.

Examples of this type are Ivoclar Vivodent PE (Ivoclar Vivadent AG., Schaan, Liechtenstein), SR Orthosit PE (Ivoclar Vivadent, Naturns, Italy), Veracia (Shofu INC., Kyoto, Japan), Endura (Shofu INC., Kyoto, Japan), Yamahachi PX (Yamahachi Dental Mfg. Co., Aichi Pref., Japan), and SR Phonares II (Ivoclar Vivadent, Naturns, Italy).

#### Adhesion between the denture teeth and acrylic denture base

Adhesion between acrylic denture teeth and a denture base is due to chemical bonding. Bonding is caused by monomer from the uncured denture base contacting and penetrate into the polymer molecules in the denture teeth.(28) This bonding mechanism is called the swelling phenomenon (Figure 6).(29) The swelling phenomenon occurs via secondary-IPN formation. The requirements for secondary-IPN formation are swelling of the substrate and interlacing of the polymer chains of the repair resin and the damaged resin. MMA fulfills the requirements for being a good PMMA solvent.(30) This is due to the deeper penetration of the monomer of denture base polymers into the denture teeth, generating thicker secondary-IPN layer formation, resulting in higher bond strength.

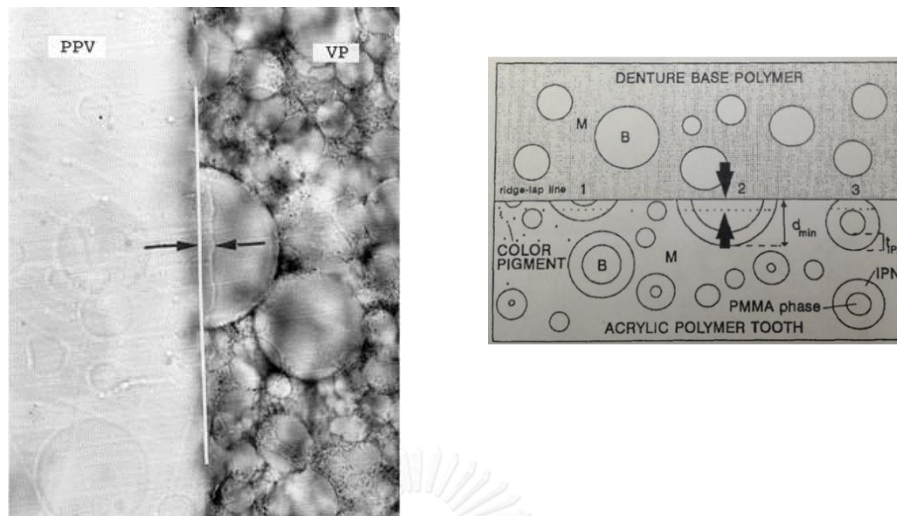


Figure 6 Bonding mechanism between denture teeth and acrylic denture base, known as the swelling phenomenon.(29)

The amount of monomer penetration depends on time, polymerization temperature, type of solution, polymer structure, cleanliness of the bonding surface, and polymer glass transition temperature.(31) A study found that surfaces to be repaired should be wet with MMA monomer for 3 minutes to diminish adhesive failure.(5) Increased polymerization temperature has also been suggested.(29) The recommended temperature should be above 50°C, because at this temperature the denture base monomer diffuses more effectively into the acrylic resin polymer teeth, increasing bond strength.(32)

### **Causes of denture teeth-denture base debonding**

Debonding of acrylic denture teeth from a denture base remains a major problem in prosthodontics. Debonding usually occurs at the anterior region of the denture, which may be attributed to less ridge lap surface area available for bonding and the direction of the stresses encountered during function.(1) Debonding may be the result of incompatible surface conditions at the tooth and base interface. The factors that contribute to debonding are: (i) contamination of the two surfaces such

as with wax, petroleum jelly, or sodium alginate solution(33) and (ii) difference in the structure of the two components because of their different processing methods.(3) There are many studies of modifying the ridge lap area to improve bond strength using mechanical, chemical, or a combination of these methods.(4, 25)

### **Testing the bond strength between acrylic denture teeth and denture base**

There are international standards concerning testing the adhesion between denture teeth and denture base. These standards vary in how the specimens are prepared, the testing methods, and the requirements for testing, e.g. The American National Standards/American Dental Association Specification number 15 (ANSI/ADA 15), The Australian Standard (AS1626), International Standard Organization for synthetic resin teeth (ISO 3336), and The Japanese Standard on Acrylic Resin Teeth (JIST 6506). There was a study also introduced a new method of testing.(34) This method attempted to overcome the weak point for the standard methods. However, there is no studies indicated which method is better.

The acceptable bond strength values of each standardized method differ. For example: for ADA 15, 31 MPa is acceptable, AS 1626 accepts minimum bond strength at 32 MPa, and JIST 6506 states that 110 N for upper teeth and 60 N for the lower teeth is sufficient. The bond strength value depends on the specimen fabrication method.

Self-cured acrylic denture base polymers showed lower bond strengths compared with heat-cured denture base.(22) No standard testing method uses self-cured acrylic resin in specimen preparation. Therefore, there is no bond strength standard for denture teeth repaired with self-cured acrylic resin.

## Agents for surface treatment

### 1. Chloroform

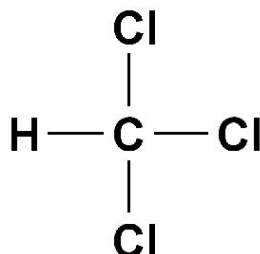


Figure 7 Structural formula of Chloroform.

Chloroform (Trichloromethane, Formyl trichloride, Methane trichloride, Methyl trichloride, Methenyl trichloride) is an organic compound with formula  $\text{CHCl}_3$  (Figure 7). Chloroform is a colorless, heavy volatile liquid with a sweet taste and odor, and was formerly used as an inhalation anesthetic.

Chloroform is also used in pesticide formulations, as a solvent for fats, oils, rubber, alkaloids, waxes, gutta-percha, and resins, as a cleansing agent, grain fumigant, in fire extinguishers, and in the rubber industry. However, based on evidence from animal studies, Chloroform is likely carcinogenic in humans.(35)

### 2. Methylene chloride

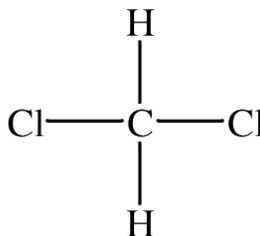


Figure 8 Structural formula of Methylene chloride.

Methylene chloride (Dichloromethane, Methylene dichloride) is an organic compound with the formula  $\text{CH}_2\text{Cl}_2$  (Figure 8). This colorless, volatile liquid with a

moderately sweet aroma is widely used as a solvent. Although Methylene chloride is not miscible with water, it is miscible with many organic solvents

Methylene chloride is used in various industrial processes, including paint stripping, pharmaceutical manufacturing, paint remover manufacturing, and metal cleaning and degreasing. The most common means of exposure to methylene chloride are by inhalation and skin exposure. OSHA considers methylene chloride to be a potential occupational carcinogen.

Methylene chloride is recommended to be applied on the denture teeth ridge-lap area prior to denture base processing. The use of Methylene chloride improved the bond strength of denture teeth.(36)

3. 4-methacryloxyethyl trimellitate anhrderide (4-META)

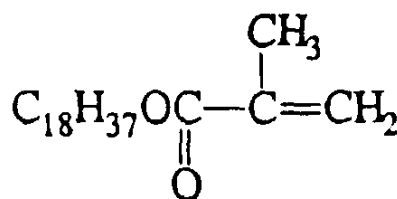


Figure 9 Structural formula of 4-methacryloxyethyl trimellitate anhrderide.

4-META (Figure 9) was developed by Nakabayashi in 1978 and patented in Japan in January 1979 and patented in the United States in April 1979.

4-META has been reported to be a suitable monomer promoting adhesion between PMMA rods and tooth substrates and metals. 4-META is an MMA derivative and polymerizes with MMA to form a co-polymer. 4-META is a reactive monomer that is used in promoting adhesion, especially in dental applications. A study showed that the application of 4-META adhesive bonding agents to denture teeth improved the bonding between highly cross-linked acrylic teeth and denture base (25) These authors reported that 4-META monomer promoted the infiltration of MMA monomer into cross-linked polymer surfaces.

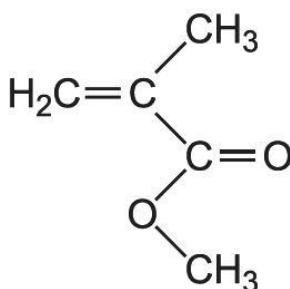
4. Methyl methacrylate (MMA)

Figure 10 Structural formula of Methyl methacrylate.

MMA is an organic compound with the formula  $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOCH}_3$  (Figure 10). This colorless liquid, the methyl ester of methacrylic acid, is a monomer produced on a large scale for the production of PMMA.

The acute toxicity of MMA is low. Irritation of the skin, eyes, and nasal cavities has been observed in rodents and rabbits exposed to relatively high concentrations of MMA. MMA is a mild skin irritant in humans and has the potential to induce skin sensitization in susceptible individuals. However, MMA may sensitize patients or elicit an allergic reaction.(21)

MMA is effective in promoting adhesion and is widely used as a chemical surface-treatment agent.(37) However, a clinical disadvantage of MMA is that it has a long wetting-time. MMA requires 3 minutes to reduce adhesive failure.(5)

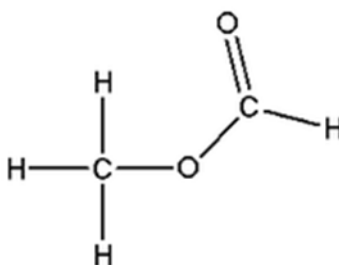
5. Methyl formate

Figure 11 Structural formula of Methyl formate.

MF (Figure 11), the methyl ester of formic acid, is a colorless and highly flammable liquid with an ether-like odor, and is soluble in water and miscible with most organic solvents. Its chemical formula is  $C_2H_4O_2$ . MF is readily hydrolyzed by water. Commercial MF is produced from methanol with carbon monoxide in the presence of a strong base.

The most common use of MF is in the preparation of formamide and dimethyl formamide. MF is used as the raw material for formic acid that is used as a chemical intermediate and solvent, in processing textiles, leathers, electroplating, in coagulating latex rubber, and as a disinfectant. MF has high volatility, high vapor pressure, low viscosity, and low surface tension. Accordingly, MF is commonly used as a component in solvent systems to achieve quick drying coating finishes and in spray applications. MF is also used as a blowing agent for expanded synthetic rubbers. MF is used as an intermediate in manufacturing various organic chemicals including pharmaceuticals, optical brighteners, and hydrocyanic acid.(38)

#### 6. Methyl acetate

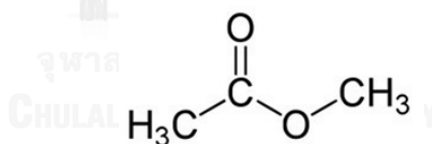


Figure 12 Structural formula of Methyl acetate.

MA or methyl ethanoate is the methyl ester of acetic acid with the chemical formula  $C_3H_6O_2$  (Figure 12). MA is a flammable liquid at room temperature with a fruity smell and has a high volatility. MA is produced by an esterification reaction of acetic acid and the corresponding alcohol in the presence of strong acids such as sulfuric acid. This solvent is typically used as a liquid that acts as a dissolving agent. MA is used as a solvent in nail polish, perfume, and glue.(39)

## Thermocycling

The international standard for testing dental adhesives stipulates the use of an ageing procedure in which the test specimens are placed first in cold water and then in hot water (one cycle) for a high number of cycles. Subsequent testing invariably demonstrates degradation in adhesive strength. It is important that this loss of bonding strength is limited.

Thermocycling requires moderately sophisticated equipment to ensure constant temperatures in the water baths and properly timed transfer of the specimens. The Nordic Institute of Dental Materials (NIOM) designed the first thermocycling apparatus 20 years ago, and has since supplied numerous laboratories with the equipment, with each new model incorporating new developments in temperature and mechanical control.

Although international standards specify immersion times and temperatures for established tests, new materials and test methods create new situations for which thermocycling is a useful method of experimentally ageing a material. Thermocycling is based on the diffusion of heat, and in porous materials also of moisture, in and out of the test specimen.(40) In denture teeth studies, thermocycling theoretically allows repeated expansion and contraction of the tooth and denture base resin components and hydrates the specimens stimulating the oral condition.(41)



## CHAPTER III

### METHODOLOGY

#### Target population

Removable denture that has debonding of the acrylic denture teeth

#### Sample

Specimens were prepared using 3 types of acrylic denture teeth and a self-cured acrylic repair resin.

#### Materials and Instruments

- I. Self-cured acrylic resin (Unifast TRAD, Accord Corporation, Ltd, Thailand)
- II. Denture teeth 3 brands (Yamahachi New Ace, Trubyte Bioform IPN, and Cosmo HXL) ;see Table 3
- III. Methyl methacrylate solution (the liquid part of Unifast TRAD, Accord Corporation, Ltd, Thailand)
- IV. Methyl formate methyl acetate solution (CU Acrylic Bond) (Faculty of Dentistry, Chulalongkorn University, BKK, Thailand)
- V. Water pipe
- VI. Autopolymerized (poly)methyl methacrylate (resin clear casting)
- VII. Silicon carbide paper no. 500, 1200
- VIII. Polyethylene sheet
- IX. Acrylic resin ring
- X. Acrylic resin rod
- XI. Cyanoacrylate glue
- XII. Deionized water
- XIII. Paintbrush
- XIV. Timer
- XV. Weight 1 kg
- XVI. Polishing machine (Nano2000, PACE Technologies, AZ, USA)

- XVII. Pressure cooker (In Motion Technology Co., Ltd., BKK, Thailand)
- XVIII. Universal testing machine (Shimadzu, EZ-S, Bara Scientific Co., Ltd., Thailand)
- XIX. Incubator 37 °C (Contherm Scientific Ltd., New Zealand)
- XX. Thermo Cycling unit (King Mongkut's Institute of Technology Ladkrabang, BKK, Thailand)
- XXI. Stereo microscope (Olympus Spacemed Inc.,TYO, Japan)



Table 3 Materials used in this study.

Material	Brand Name	Composition	Abbreviation	Batch No.	Manufacturer
Denture teeth	Yamahachi New Ace	Poly(methyl-methacrylate)	YA	HK2017	Yamahachi Dental Mfg., Co., Aichi Pref., Japan
	Cosmo HXL	10% Highly cross-linked poly(methyl-methacrylate)	CH	20140801C	Dentsply Dental Co., Ltd., Tianjin, China
	Trubyte Bioform IPN	Highly cross-linked poly(methyl-methacrylate)	TB	17404	Dentsply International Inc., York, PA, USA
Self-cured acrylic resin	Unifast Trad (powder)	Poly(methyl-methacrylate)	-	1503092	GC Dental product corp., Aichi., Japan
	Unifast Trad (liquid)	Methyl-methacrylate	-	1404012	GC Dental product corp., Aichi., Japan
	Unifast Trad (liquid)	Methyl-methacrylate	-	-	GC Dental product corp., Aichi., Japan
Chemical agents	Unifast Trad (liquid)	Methyl-methacrylate	MMA	-	GC Dental product corp., Aichi., Japan
	CU Acrylic Bond	Methyl formate, Methyl acetate	MF-MA	-	Faculty of Dentistry, Chulalongkorn university, Bangkok., Thailand

## Sample preparation

### Part I Non-thermocycling

Three brands of denture teeth were used (Table 3). Seventy maxillary central incisor acrylic denture teeth of each brand were embedded incisal surface down in auto-polymerized PMMA that was packed in a 20-mm diameter polyethylene pipe (Figure 13). Each tooth's ridge-lap surface was polished with 500- and 1200-grit silicon carbide paper in a polishing machine (Nano2000, PACE Technologies, St. Tucson, AZ). The specimens were then put in an ultrasonic cleaner for 2 minutes to remove surface debris (Figure 14).



Figure 13 Denture teeth embedded in auto-polymerized PMMA.



Figure 14 The ridge-lap surfaces of the denture teeth were polished and cleaned.

The specimens of each brand were divided into 7 groups (n=10). The specimens were readied (Figure 15) and chemical solutions were applied to the groups as follows: no treatment; MF-MA solution at a ratio of 25:75 (by volume) (CU Acrylic Bond, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand) for 15, 30, 60, 120, 180 s, and MMA 180 s (Table 4).



Figure 15 Preparation for chemical-solution application.

A polyethylene sheet with a 3-mm diameter hole was placed over the treated surface and a 10-mm diameter acrylic resin ring was placed centrally over the hole in the polyethylene sheet. Self-cured acrylic resin (Unifast Trad, GC Dental Products Corp, Aichi, Japan) was loaded into the ring to slight excess and compressed with a 1-kg weight. The specimen was placed in a pressure cooker at 2 MPa at 60°C. This process is shown in Figure 16. After the acrylic resin had set, an acrylic resin rod was attached on top of the specimen with cyanoacrylate glue (Super Glue, Alteco Chemical PTE Ltd, Japan) to connect to the tensile testing machine (Figure 17).

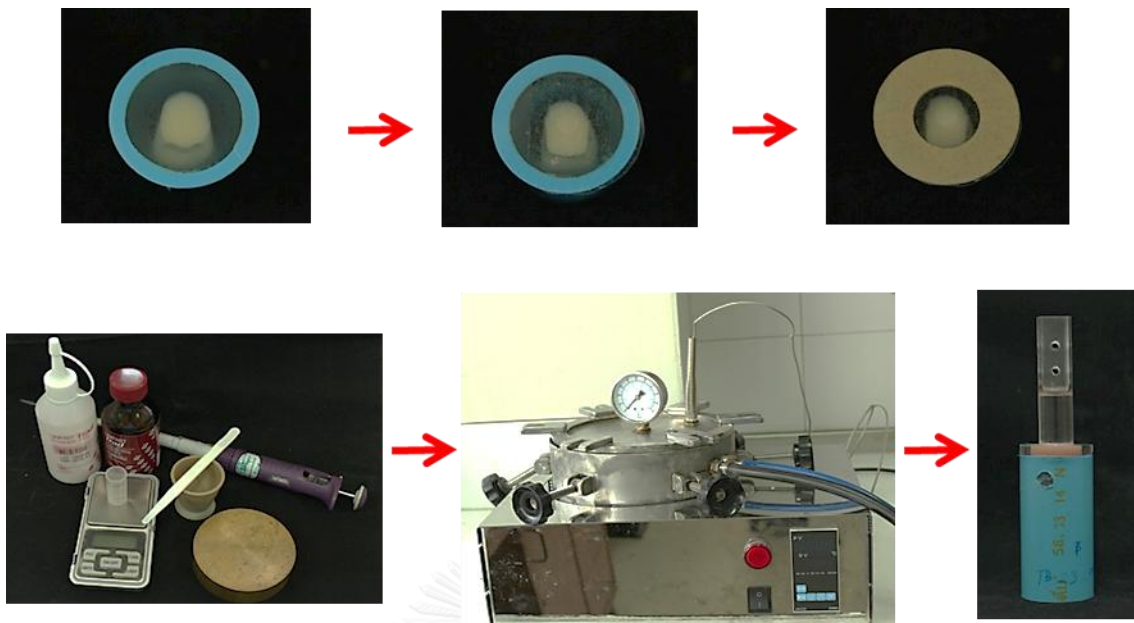


Figure 16 Specimen preparation after surface treatment for tensile bond strength testing.

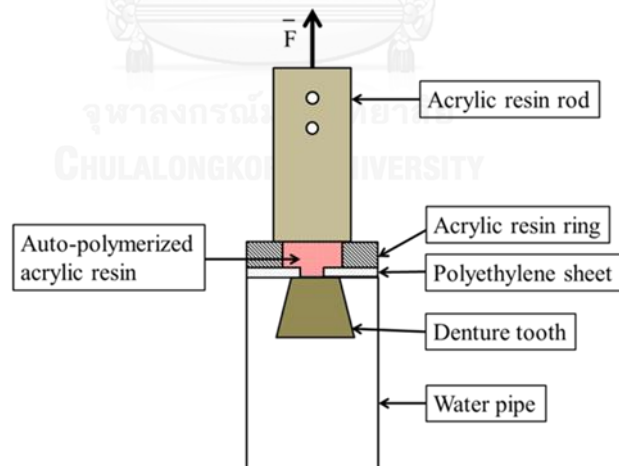


Figure 17 Schematic drawing of the specimens.

The polymerized specimens were stored in deionized water at  $37^{\circ}\text{C}$  for  $48 \pm 2$  h and tested to failure using a universal testing machine (Shimadzu, EZ-S, Bara

Scientific Co., Ltd., Thailand) with a 500 N load cell at a crosshead speed of 10 mm/min. The tensile bond strength (MPa) was calculated by dividing the failure force by the bond surface area. The following equation was used to calculate the tensile bond strength:

$$B = F/A$$

Where B is the bond strength in MPa, F is the maximum load (N) before debonding occurred, and A is the adhesive area (mm<sup>2</sup>).



Table 4 Details of the groups in part I. (n=10)

Denture teeth type	Group (Code name)	Chemical Agent		Application time (s)
		MF-MA	MMA	
Conventional (YA)	YA C	-	-	-
	YA MMA180s	-	+	180
	YA MF-MA15s	+	-	15
	YA MF-MA30s	+	-	30
	YA MF-MA60s	+	-	60
	YA MF-MA120s	+	-	120
	YA MF-MA180s	+	-	180
Semi-IPN (CH)	CH C	-	-	-
	CH MMA180s	-	+	180
	CH MF-MA15s	+	-	15
	CH MF-MA30s	+	-	30
	CH MF-MA60s	+	-	60
	CH MF-MA120s	+	-	120
	CH MF-MA180s	+	-	180
IPN (TB)	TB C	-	-	-
	TB MMA180s	-	+	180
	TB MF-MA15s	+	-	15
	TB MF-MA30s	+	-	30
	TB MF-MA60s	+	-	60
	TB MF-MA120s	+	-	120
	TB MF-MA180s	+	-	180

IPN, Interpenetrating polymer network



The debonded surface was inspected using a stereomicroscope at 30X magnification to determine the amount of self-cured acrylic resin remaining on the denture tooth surface. The mode of failure was categorized as adhesive (no self-cured acrylic material on the denture tooth), cohesive (self-cured acrylic material on more than 50% of the denture tooth), or mixed (self-cured acrylic material on less than 50% of the denture tooth). The surfaces were examined by one investigator.

#### Part II Thermocycling

The shortest MF-MA application time from the Part I result that generated higher bond strength than the control for each type of denture teeth was used in the tooth groups as seen in Table 5. The MMA application time was the same as part I (180 s).

Specimen preparation was performed as in part I. Before tensile testing, the specimens were thermocycled in water at 4°C and 60°C for 5000 cycles with an immersion time 30 s and transfer time of 10 s.

Table 5 Details of groups in part II. (n=10)

Denture teeth type	Group (Code name)	Thermocycling 5,000 cycles
Conventional (YA)	YA C-	-
	YA C+	+
	YA MMA180s-	-
	YA MMA180s+	+
	YA MF-MA- (time from part I)	-
	YA MF-MA+ (time from part I)	+
Semi-IPN (CH)	CH C-	-
	CH C+	+
	CH MMA180s-	-
	CH MMA180s+	+
	CH MF-MA- (time from part I)	-
	CH MF-MA+ (time from part I)	+
IPN (TB)	TB C-	-
	TB C+	+
	TB MMA180s-	-
	TB MMA180s+	+
	TB MF-MA- (time from part I)	-
	TB MF-MA+ (time from part I)	+

IPN, Interpenetrating polymer network

The data were statistically analyzed using SPSS for Windows 22 (IBM Corporation, New York, United States).

## CHAPTER IV

### RESULTS

The data were analyzed using the Kolmogorov-Smirnov test to determine data distribution. The results showed the data were normally distributed in all groups ( $p>0.05$ ) (Tables 8-9).

The part I data were then analyzed using Univariate two-way ANOVA, however, the variances between groups were not equal and there was an interaction effect between tooth brand and surface treatments ( $p<0.05$ ) (Table 11). Therefore, these data did not fit the two-way ANOVA's criteria, thus, they were analyzed using one-way ANOVA and the Dunnet T3 test ( $\alpha= 0.05$ ) (Tables 12-13).

The mean tensile bond strength (MPa) in each group in part I is shown in Table 6. The results demonstrated that the tensile bond strengths of the control groups (YAC, CHC, and TBC) were not significantly different from each other ( $p>0.05$ ). For each brand, the surface treatment groups had significantly higher tensile bond strengths compared with their negative control groups ( $p<0.05$ ), except for the TB MF-MA 15 s group ( $p>0.05$ ). The tensile bond strengths of the MMA 180 s group of each brand were not significantly different from their MF-MA 15 s, 30 s, 60 s, 120 s, and 180 s groups ( $p>0.05$ ), with the exception of the YA MF-MA 180 s and the TB MF-MA 60 s groups that had significantly higher tensile bond strengths than the MMA 180 s groups ( $p<0.05$ ).

Table 6 The mean and standard deviations of tensile bond strength (MPa) of the groups in part I.

	Yamahachi New Ace (YA)	Cosmo HXL (CH)	Trubyte Bioform IPN (TB)
Control	13.0 ± 2.09 <sup>A,a</sup>	11.4 ± 1.24 <sup>D,a</sup>	11.5 ± 2.02 <sup>G,a</sup>
MMA 180 s	19.5 ± 2.35 <sup>B,i</sup>	18.7 ± 2.33 <sup>EF,i</sup>	16.2 ± 2.0 <sup>HJ,i</sup>
MF-MA 15 s	18.6 ± 1.94 <sup>B,b</sup>	16.3 ± 2.65 <sup>E,bc</sup>	14.9 ± 1.44 <sup>GH,c</sup>
MF-MA 30 s	20.1 ± 2.39 <sup>B,d</sup>	19.2 ± 4.07 <sup>EF,d</sup>	18.0 ± 2.0 <sup>HI,d</sup>
MF-MA 60 s	21.4 ± 2.58 <sup>B,e</sup>	21.1 ± 1.96 <sup>F,e</sup>	20.1 ± 1.81 <sup>I,e</sup>
MF-MA 120 s	23.3 ± 3.99 <sup>B,f</sup>	19.0 ± 2.00 <sup>EF,f</sup>	18.3 ± 1.9 <sup>IJ,f</sup>
MF-MA 180 s	24.9 ± 1.29 <sup>C,g</sup>	18.2 ± 3.28 <sup>EF,h</sup>	18.6 ± 1.62 <sup>IJ,h</sup>

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

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

The data of the thermocycling part were tested for homogeneity of variance using the Levene test. The results showed that the variances between groups were not significantly different (Table 14). However, the data also showed an interaction effect between tooth brand and surface treatments ( $p<0.05$ ) (Table 15). The data were separately analyzed by one-way ANOVA and Tukey's HSD test ( $\alpha= 0.05$ ) to compare the mean bond strength in each group (Table 16-17).

The results of part II are shown in Table 7. There were no significant differences between the tensile bond strength of the YA, CH, and TB control groups before and after thermocycling ( $p>0.05$ ). However, significant differences in tensile bond strength were found between the YA MF-MA and MMA groups, CH MMA groups, and TB MF-MA groups ( $p<0.05$ ).

Percentage of failure mode for all groups was shown in Figure. 18-19 and their stereo microscope pictures of each group were shown in Figure 20-22.

Scanning electron microscope (SEM) analysis of the MF-MA and MMA treated surfaces (Figure 23-25) revealed different morphological patterns. The surface appearances varied by chemical-agent types and application time.



Table 7 The mean and standard deviations of tensile bond strength (MPa) of the groups in part II.

Group (Code name)	Bond strength $\pm$ SD (MPa)
YA C-	13.0 $\pm$ 2.09 <sup>bcd</sup>
YA C+	10.2 $\pm$ 2.02 <sup>ab</sup>
YA MMA180s-	19.5 $\pm$ 2.35 <sup>f</sup>
YA MMA180s+	15.0 $\pm$ 3.54 <sup>cde</sup>
YA MF-MA15s-	18.6 $\pm$ 1.94 <sup>f</sup>
YA MF-MA15s+	12.1 $\pm$ 2.04 <sup>bc</sup>
CH C-	11.4 $\pm$ 1.24 <sup>abc</sup>
CH C+	8.5 $\pm$ 2.18 <sup>a</sup>
CH MMA180s-	18.7 $\pm$ 2.33 <sup>f</sup>
CH MMA180s+	11.7 $\pm$ 1.51 <sup>abc</sup>
CH MF-MA15s-	16.3 $\pm$ 2.65 <sup>def</sup>
CH MF-MA15s+	14.0 $\pm$ 3.60 <sup>cd</sup>
TB C-	11.5 $\pm$ 2.02 <sup>abc</sup>
TB C+	12.5 $\pm$ 2.07 <sup>bc</sup>
TB MMA180s-	16.2 $\pm$ 2.0 <sup>def</sup>
TB MMA180s+	12.8 $\pm$ 1.72 <sup>bcd</sup>
TB MF-MA30s-	18.0 $\pm$ 2.0 <sup>ef</sup>
TB MF-MA30s+	11.7 $\pm$ 1.69 <sup>abc</sup>

-, Non thermocycling; +, Thermocycling

Same superscript letter indicates no significant difference between the groups ( $p > 0.05$ ).

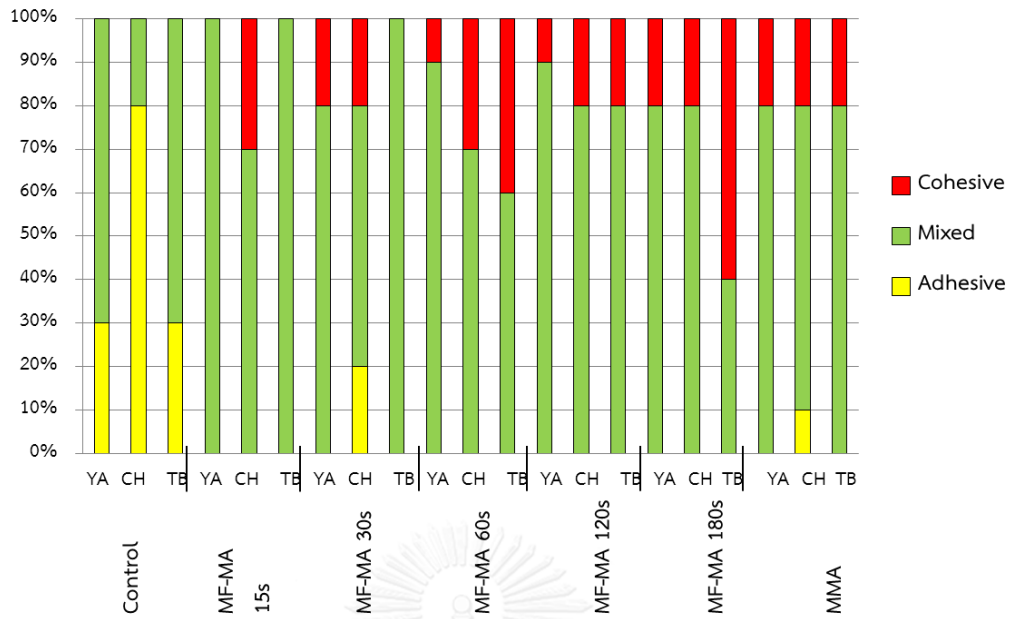


Figure 18 Percentage of failure modes of the part I groups.

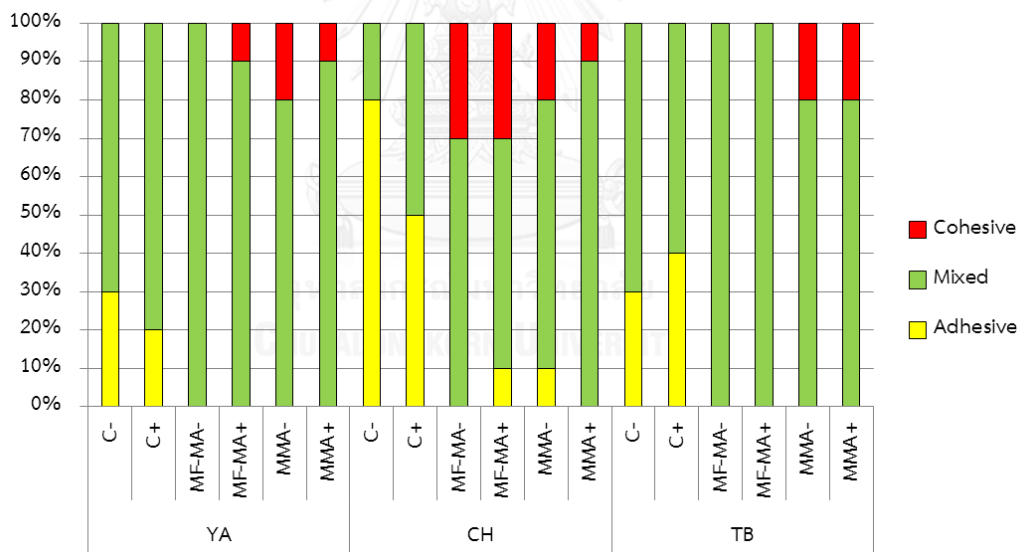


Figure 19 Percentage of failure modes of the part II groups compared with part I groups.

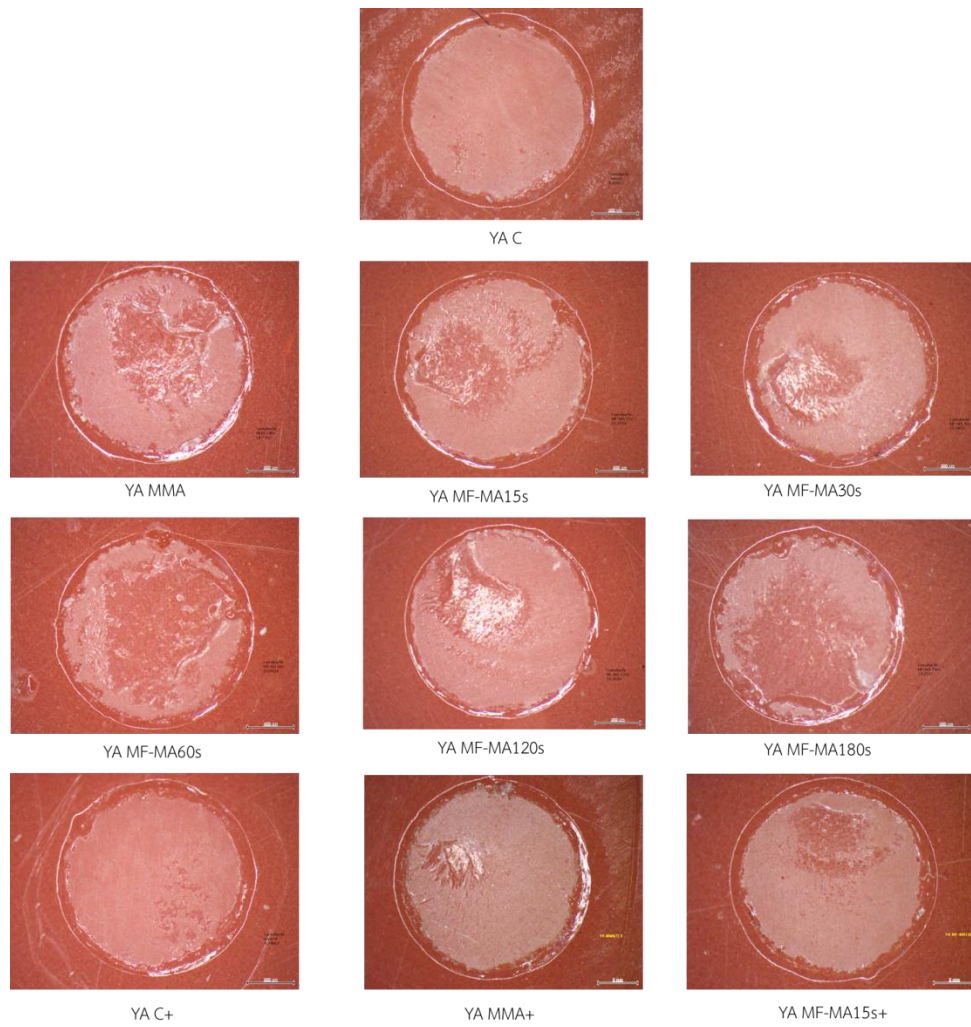


Figure 20 Failure patterns of the Yamahachi New Ace specimens



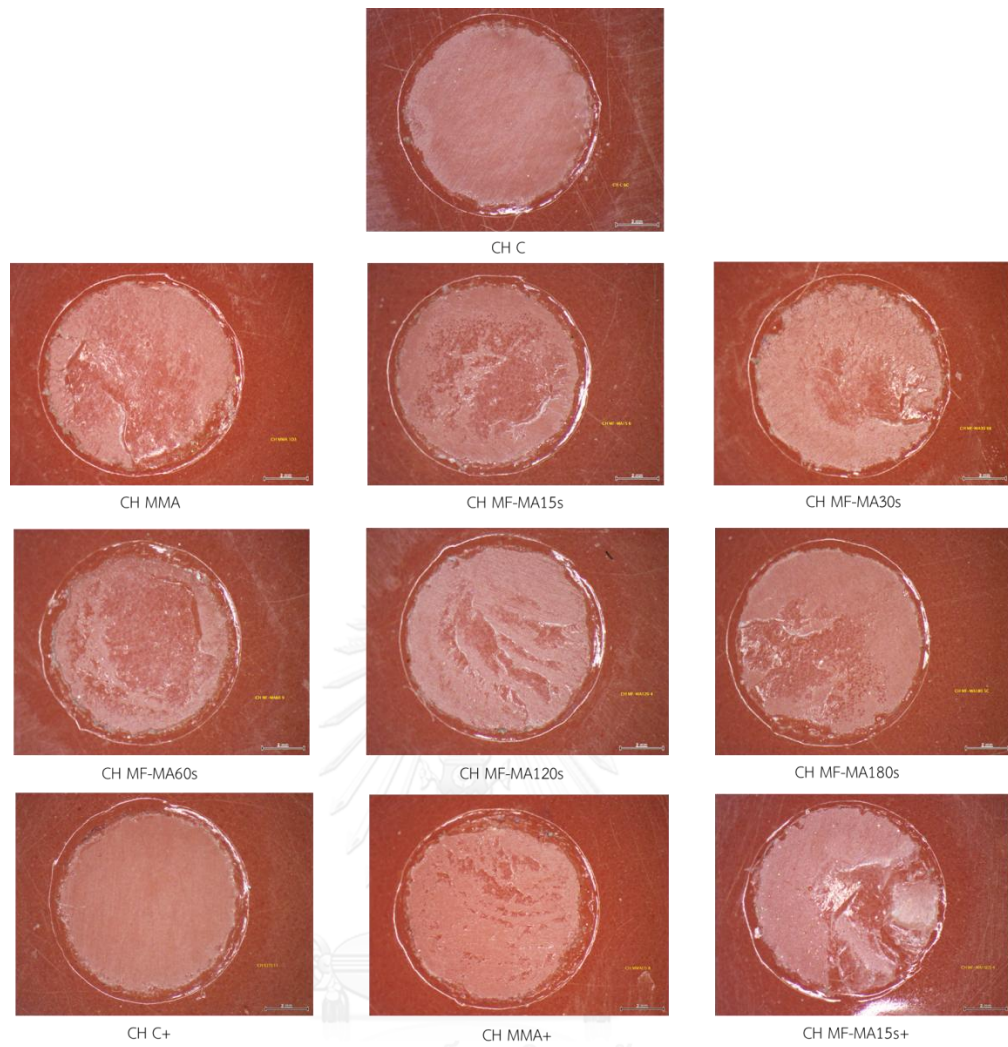


Figure 21 Failure patterns of the Cosmo HXL specimens

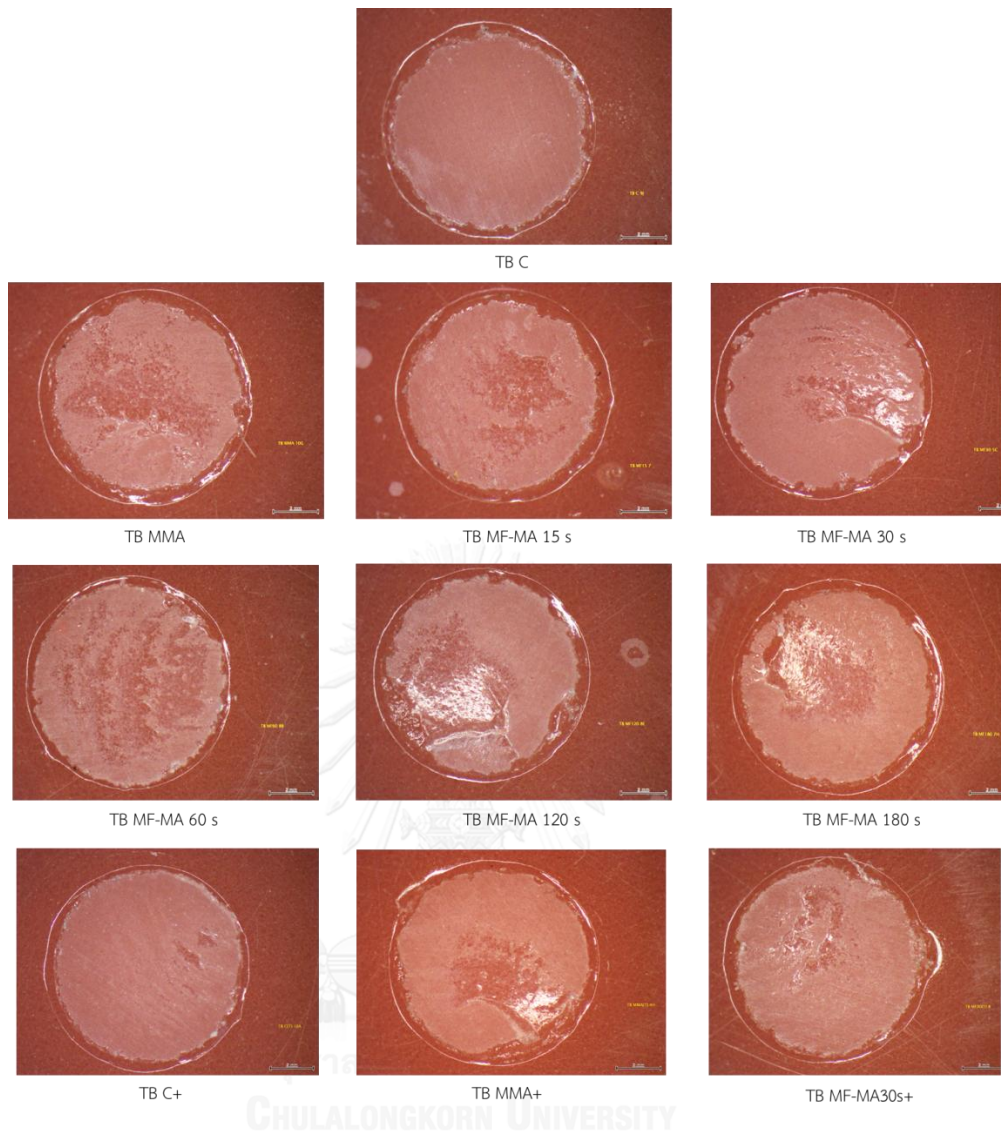


Figure 22 Failure patterns of the Trubyte Bioform IPN specimens

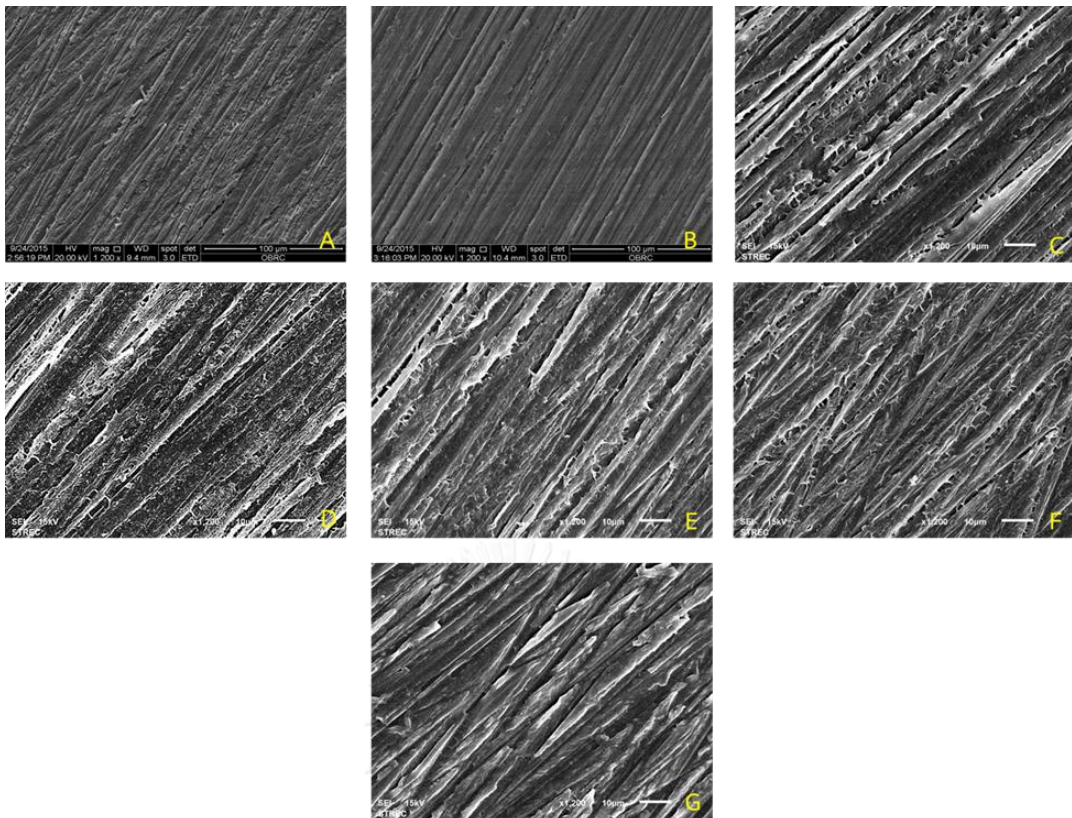


Figure 23 SEM analysis of the surface characteristics of the Yamahachi New Ace acrylic denture teeth. (A) no treatment, (B-G) MF-MA solution 15, 30, 60, 120, 180 s, and MMA 180 s, respectively



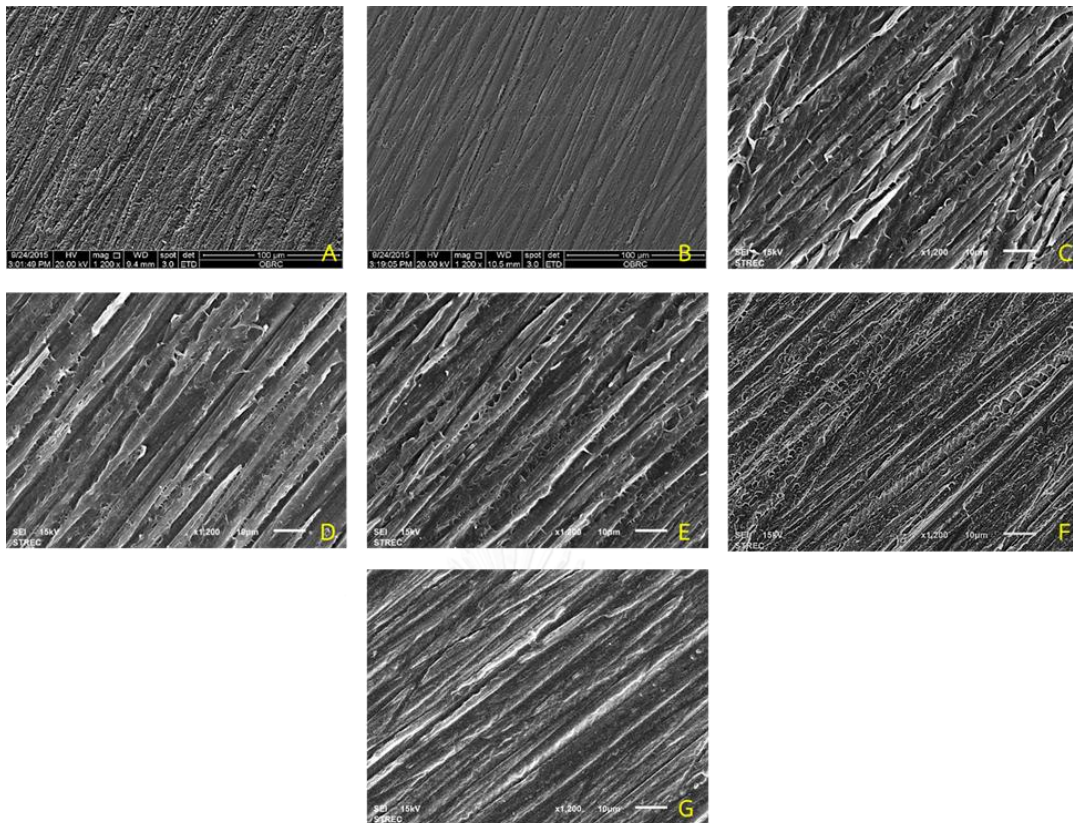


Figure 24 SEM analysis of the surface characteristics of the Cosmo HXL acrylic denture teeth. (A) no treatment, (B-G) MF-MA solution 15, 30, 60, 120, 180 s, and MMA 180 s, respectively.

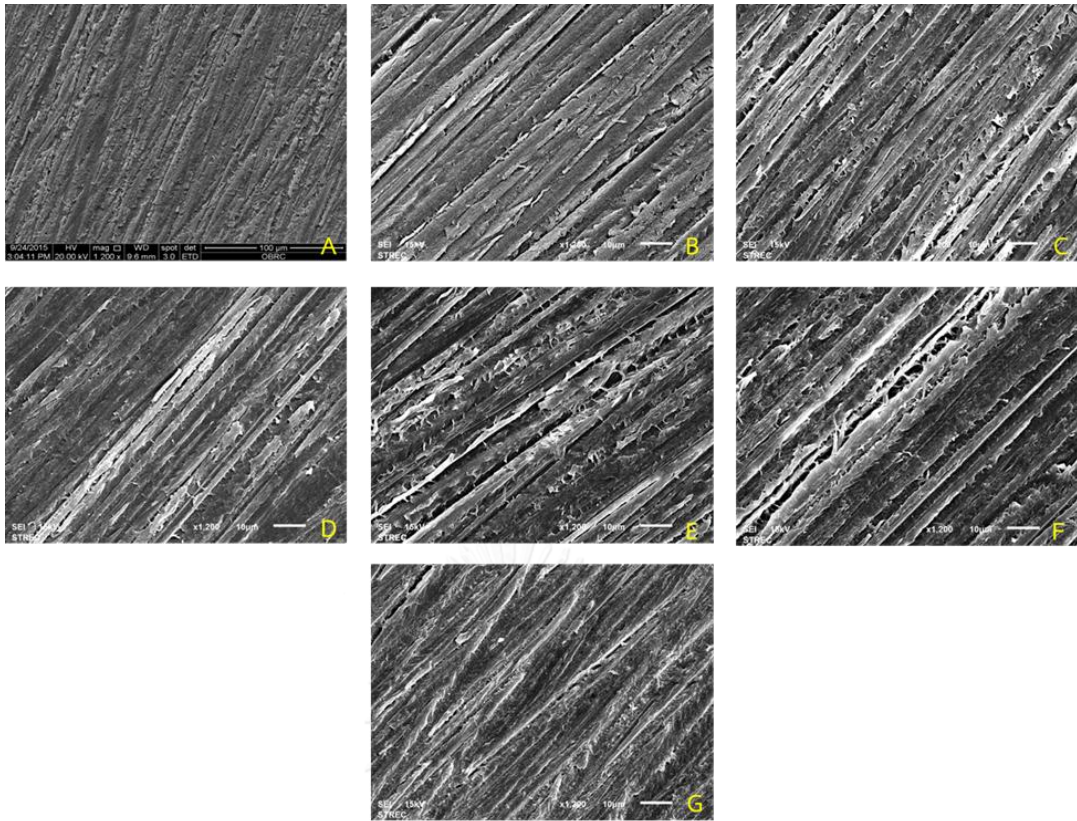


Figure 25 SEM analysis of the surface characteristic of the Trubyte Bioform IPN acrylic denture teeth. (A) no treatment, (B-G) MF-MA solution 15, 30, 60, 120, 180 s, and MMA 180 s, respectively.

## CHAPTER V

### DISCUSSION AND CONCLUSION

#### Discussion

The bond strength of acrylic denture teeth to self-cured acrylic resin can be improved by chemical surface treatment, as confirmed by the failure-mode results in this study. Adhesive failures decreased and mixed and cohesive failures increased in the surface-treatment groups compared with the control groups indicating that the bond strength of the treated groups had increased to greater than the material strength of the no-treated groups. This increased strength begins when the solvents in the surface treatment agent contact the denture tooth, dissolving its surface and cause swelling of the surface layer. Subsequently, the monomer of the self-cured acrylic resin material diffuses and penetrates into the IPN matrix of the denture teeth during polymerization, known as the swelling phenomenon.(29) The swelling phenomenon is affected by application time, polymerization temperature, type of solvent, denture teeth structure, and the glass transitional temperature of the denture teeth.(31)

When used on acrylic denture teeth as a surface treatment, an MF-MA solution acts by dissolving and swelling their surfaces, and then evaporating. In addition, there are no carbon-carbon double bonds (C=C) in MF or MA molecules to polymerize with the monomer in the self-cured acrylic material. Thus, MF-MA would not obstruct the interlocking of the self-cured resin polymer chains and the denture teeth, increasing the tensile bond strength.

Polymer solubility and swelling occurs when the polymer and solvent solubility parameters and polarities are close to each other. The solubility parameter of PMMA (acrylic denture teeth) is  $18.3 \text{ MPa}^{1/2}$ , whereas those of MMA, MF, and MA are 18.0, 20.9, and  $19.6 \text{ MPa}^{1/2}$ , respectively.(42) In addition, MMA, MF and MA molecules have the same methyl ester group, which increases their ability to soften PMMA.(6) MF, MA and the MF-MA mixture could increase the bond strength of repaired acrylic denture base and acrylic denture base relined with rebasing

material.(7, 9) Thus, it was hypothesized that an MF-MA mixture would result in a higher tensile bond strength between acrylic denture teeth and self-cured acrylic resin compared with using MMA liquid.

Although the results indicated that surface treatment with MF-MA and MMA improved the bond strength compared with no treatment, MF-MA is superior to MMA for treating the three brands of denture teeth prior to chair-side repair, due to its reduced application time and lack of tissue irritation. This study results indicated that the bond strengths using MMA for 180 s for all brands were not significantly different from the MF-MA groups that used less application time ( $p>0.05$ ). The shortest MF-MA application times that had a higher bond strength than that of the control were 15 s for YA and CH and 30 s for TB, thus, these are the optimum times for chair-side use for conventional, highly cross-linked, and IPN acrylic denture teeth.

Based on denture tooth type, the results showed that the bond strength between an IPN tooth (Trubyte Bioform IPN) and self-cured resin was lower than that of conventional (Yamahachi New Ace) and highly cross-linked (Cosmo HXL) teeth. The reason may be that Trubyte Bioform IPN has a true IPN structure. The IPN consists of two or more polymer networks (double cross-linked polymer), resulting in a more complex structure than that of Yamahachi New Ace and Cosmo HXL. Therefore, there is less space between the polymer chains (crosslink density) in IPN acrylic denture teeth compared with the conventional and highly cross-linked type. Thus, monomer from the self-cured acrylic resin and chemical agents (MF-MA or MMA) are difficult to diffuse into the IPN acrylic denture teeth and form swelled layer, resulting in decreased bonding. According to this reason, an IPN tooth required more application time than the other teeth types.

Cosmo HXL is a highly cross-linked acrylic denture tooth which has cross-linked structure more than Yamahachi New Ace, but Cosmo HXL and Yamahachi New Ace required the same MF-MA application time (15 s). The reason for this may be that the cross-linked structure of Cosmo HXL teeth, an IPN consisting of only 10% highly cross-linked PMMA resin, is not too complex to result in a different bond strength. Confirming with glass transition temperature, the crosslink density increases,

glass transition temperature shifts to higher temperatures.(43) Differential Scanning Calorimetry analysis showed that glass transition temperature of Yamahachi New Ace and Cosmo HXL were 114.1 °C and 112.8 °C, in order. (Figure 26-27) It indicates that Yamahachi New Ace has similar crosslink density to Cosmo HXL. In addition, glass transition temperature of Trubyte Bioform IPN was 123.3 °C. (Figure 28) It indicates that Trubyte Bioform IPN has higher crosslink density than the other teeth.

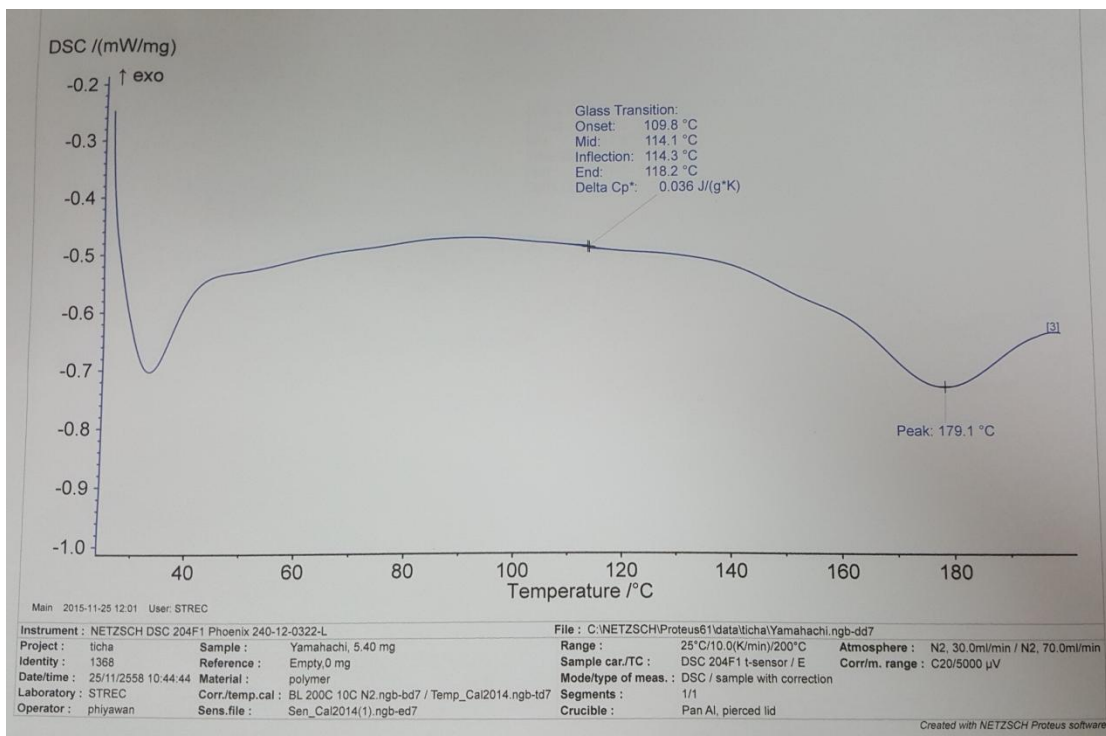


Figure 26 Differential Scanning Calorimetry (DSC) analysis of Yamahachi New Ace



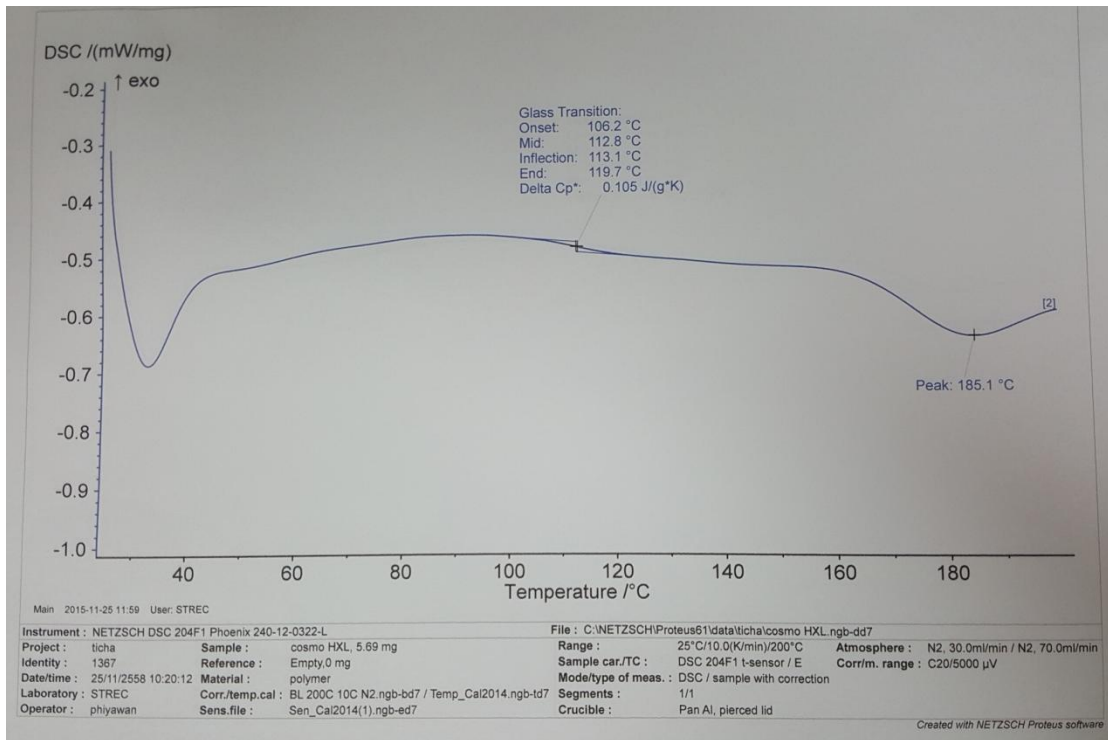


Figure 27 Differential Scanning Calorimetry (DSC) analysis of Cosmo HXL

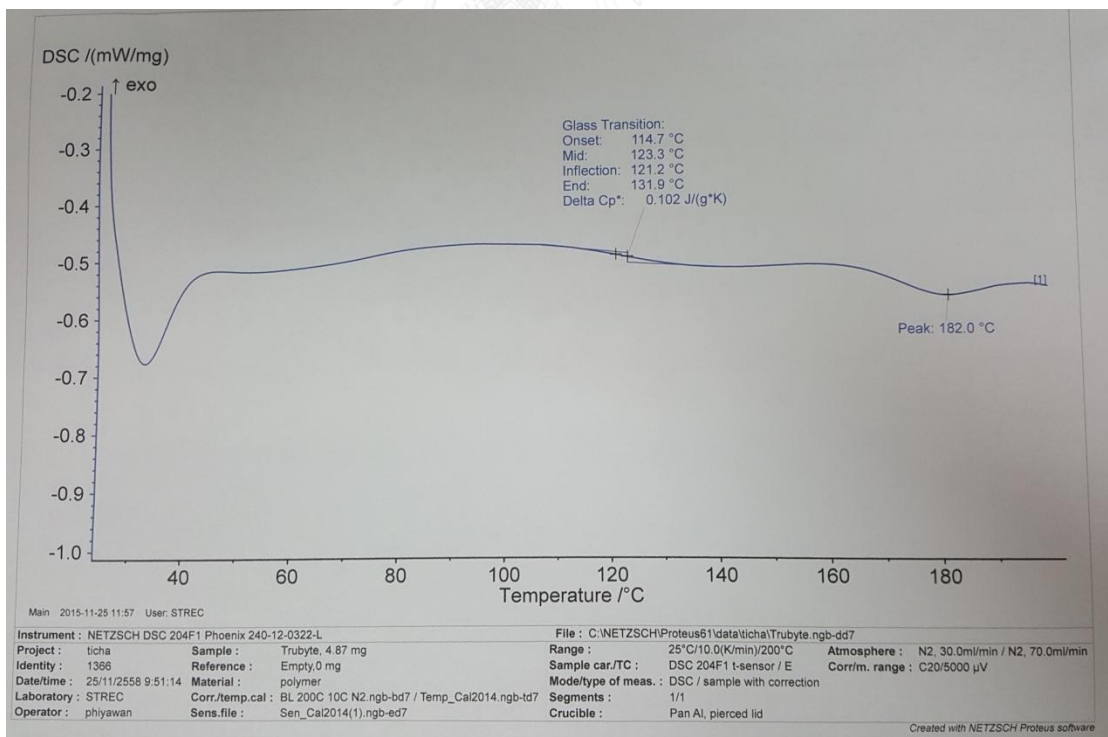


Figure 28 Differential Scanning Calorimetry (DSC) analysis of Trubyte Bioform IPN

Most of surface-treatment groups, the bond strengths between denture teeth and self-cured acrylic resin were significantly decreased by thermocycling. The reason for this might be the mechanical stress induced by the different cycling temperatures, allowing for crack propagation through the bond interface.(44) These results agreed with previous bonding studies involving thermocycling.(19) The bond strengths of the surface treatment with thermocycling groups were decreased compared with their non-thermocycling groups. However, the thermocycled control group values were similar to their control groups with no thermocycling. This could be explained that the bond strengths of non-thermocycling control groups were weak and easy to deteriorate by thermocycling. Therefore, tensile bond strength values between before and after thermocycling were not significant different ( $p>.05$ ).

The scanning electron microscope (SEM) examination of the MF-MA and MMA treated groups revealed differences in their morphological patterns. MF-MA treatment resulted in pits and holes with a honey-comb appearance, however most of MMA treated surface patterns were swelled and blurred rather than pits or holes. Thus, the MF-MA treatment resulted in a surface structure that would allow for better mechanical bonding compared with MMA treatment. However, the SEM results only evaluated the specimens' surfaces. A previous study reported that deeper penetration of monomer into the acrylic denture teeth might improve the bond strength.(45) Therefore, further studies should focus on analyzing MF-MA penetration in dissolving the acrylic denture teeth surface in a vertical section because this will generate additional data to further explain the results of this study.

## Conclusion

We found that CU Acrylic Bond (MF-MA solution) and MMA increased the bond strength of conventional, highly cross-linked, and IPN acrylic denture teeth to self-cured acrylic resin compared with no treatment. The application of MF-MA for 15 s can be an alternative chemical surface treatment for repairing a denture base and rebonding conventional and highly cross-linked acrylic denture teeth with self-cured acrylic resin. However, the IPN type teeth required a 30 s MF-MA treatment. Although some treatment groups were affected by thermocycling, the control groups were not.





Table 8 Kolmogorov-Smirnov test analysis of the data distribution in part I.

	order	Kolmogorov-Smirnov <sup>a</sup>		
		Statistic	df	Sig.
Bond strength	YA control	.250	10	.076
	YA MF-MA15s	.194	10	.200*
	YA MF-MA30s	.183	10	.200*
	YA MF-MA60s	.138	10	.200*
	YA MF-MA120s	.186	10	.200*
	YA MF-MA180s	.194	10	.200*
	YA MMA	.176	10	.200*
	CH control	.221	10	.180
	CH MF-MA15s	.141	10	.200*
	CH MF-MA30s	.169	10	.200*
	CH MF-MA60s	.205	10	.200*
	CH MF-MA120s	.158	10	.200*
	CH MF-MA180s	.226	10	.160
	CH MMA	.236	10	.122
	TB control	.215	10	.200*
	TB MF-MA15s	.222	10	.179
	TB MF-MA30s	.169	10	.200*
	TB MF-MA60s	.175	10	.200*
	TB MF-MA120s	.170	10	.200*
	TB MF-MA180s	.192	10	.200*
TB MMA	.202	10	.200*	

Table 9 Kolmogorov-Smirnov test analysis of the data distribution in part II.

		Kolmogorov-Smirnov <sup>a</sup>		
	Code	Statistic	df	Sig.
Bond strength	YA C-	.250	10	.076
	YA C+	.243	10	.095
	YA MF-MA-	.194	10	.200*
	YA MF-MA+	.163	10	.200*
	YA MMA-	.176	10	.200*
	YA MMA+	.162	10	.200*
	CH C-	.221	10	.180
	CH C+	.105	10	.200*
	CH MF-MA-	.141	10	.200*
	CH MF-MA+	.141	10	.200*
	CH MMA-	.236	10	.122
	CH MMA+	.209	10	.200*
	TB C-	.215	10	.200*
	TB C+	.207	10	.200*
	TB MF-MA-	.169	10	.200*
	TB MF-MA+	.206	10	.200*
	TB MMA-	.202	10	.200*
	TB MMA+	.103	10	.200*

Table 10 The Levene statistical analysis of the bond strength in part I.

Levene Statistic	df1	df2	Sig.
3.006	20	189	.000

Table 11 Univariate tests of between-subjects effects in part I.

Dependent Variable: Bond strength

Source	Type III Sum of Squares			F	Sig.
	Squares	df	Mean Square		
Corrected Model	2371.214 <sup>a</sup>	20	118.561	21.378	.000
Intercept	69617.535	1	69617.535	12552.903	.000
brand	407.683	2	203.841	36.755	.000
group	1767.543	6	294.590	53.118	.000
brand * group	195.989	12	16.332	2.945	.001
Error	1048.181	189	5.546		
Total	73036.930	210			
Corrected Total	3419.395	209			

Table 12 One-way ANOVA analysis of the bond strength in part I.

ANOVA					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	2371.214	20	118.561	21.378	.000
Within Groups	1048.181	189	5.546		
Total	3419.395	209			





Table 13 Dunnett T3 analysis of the bond strength in part I.

		Tensile bond strength							
		Subset for alpha = 0.05							
Code	N	1	2	3	4	5	6	7	8
Dunnett	CH C	10	11.42						
T3	TB C	10	11.54	11.54					
	YA C	10	12.95	12.95	12.95				
	TB MFMA15s	10		14.89	14.89	14.89			
	TB MMA180s	10			16.23	16.23	16.23		
	CH MMFMA15s	10			16.29	16.29	16.29	16.29	
	TB MF-MA30s	10				18.01	18.01	18.01	18.01
	CH MF-MA180s	10			18.22	18.22	18.22	18.22	18.22
	TB MF-MA120s	10					18.29	18.29	18.29
	TB MF-MA180s	10					18.57	18.57	18.57
	YA MF-MA15s	10					18.61	18.61	18.61
	CH MMA180s	10				18.72	18.72	18.72	18.72
	CH MFMA120s	10					19.00	19.00	19.00
	CH MF-MA30s	10			19.24	19.24	19.24	19.24	19.24
	YA MMA180s	10					19.46	19.46	19.46
	TB MF-MA60s	10						20.09	20.09
	YA MF-MA30s	10					20.13	20.13	20.13
	CH MF-MA60s	10							21.11
	YA MF-MA60s	10							21.42
	YA MF-MA120s	10							23.25
	YA MF-MA180s	10							24.93

Means for groups in homogeneous subsets are displayed.

Table 14 The Levene statistical analysis of the bond strength in part II.

Levene Statistic	df1	df2	Sig.
1.277	17	162	.213

Table 15 Univariate tests of between-subjects effects in part II.

Dependent Variable: Bond strength

Source	Type III Sum of			F	Sig.
	Squares	df	Mean Square		
Corrected Model	1774.927 <sup>a</sup>	17	104.407	20.738	.000
Intercept	35172.325	1	35172.325	6986.117	.000
group	845.522	8	105.690	20.993	.000
thermocycling	673.985	1	673.985	133.871	.000
group * thermocycling	255.420	8	31.927	6.342	.000
Error	815.606	162	5.035		
Total	37762.858	180			
Corrected Total	2590.533	179			

a. R Squared = .685 (Adjusted R Squared = .652)

Table 16 One-way ANOVA analysis of the bond strength in part II.

ANOVA					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1774.927	17	104.407	20.738	.000
Within Groups	815.606	162	5.035		
Total	2590.533	179			



Table 17 Tukey's HSD analysis of the bond strength in part II.

Tensile bond strength

Code	N	Subset for alpha = 0.05					
		1	2	3	4	5	6
Tukey HSD CH C+	10	8.4591					
YA C+	10	10.1725	10.1725				
CH C-	10	11.4209	11.4209	11.4209			
TB C-	10	11.5416	11.5416	11.5416			
TB MF-MA+	10	11.6876	11.6876	11.6876			
CH MMA+	10	11.7488	11.7488	11.7488			
YA MF-MA+	10		12.0620	12.0620			
TB C+	10		12.4923	12.4923			
TB MMA+	10		12.8103	12.8103	12.8103		
YA C-	10		12.9513	12.9513	12.9513		
CH MF-MA+	10			13.9865	13.9865		
YA MMA+	10			14.9731	14.9731	14.9731	
TB MMA-	10				16.2295	16.2295	16.2295
CH MF-MA-	10				16.2869	16.2869	16.2869
TB MF-MA-	10					18.0082	18.0082
YA MF-MA-	10						18.6116
CH MMA-	10						18.7177
YA MMA-	10						19.4552
Sig.		.107	.343	.051	.064	.201	.127

Means for groups in homogeneous subsets are displayed.

Table 18 The tensile bond strength values between self-cured acrylic resin and acrylic-denture teeth in each specimen. (MPa) Part I

Group	No	Tensile bond value	Failure mode	Mean	SD
YA C	1	9.249	adhesive	12.951	2.087
	2	9.424	adhesive		
	3	12.146	adhesive		
	4	13.215	mixed		
	5	13.401	mixed		
	6	13.465	mixed		
	7	14.211	mixed		
	8	14.748	mixed		
	9	14.816	mixed		
	10	14.840	mixed		
YA MMA180s	1	15.756	mixed	19.455	2.346
	2	17.569	mixed		
	3	18.192	mixed		
	4	18.289	mixed		
	5	18.720	cohesive		
	6	19.551	mixed		
	7	19.598	mixed		
	8	20.914	cohesive		
	9	22.045	mixed		
	10	23.919	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
YA MF-MA15s	1	16.163	mixed	18.612	1.942
	2	17.008	mixed		
	3	17.082	mixed		
	4	17.482	mixed		
	5	17.776	mixed		
	6	18.147	mixed		
	7	19.005	mixed		
	8	20.315	mixed		
	9	21.182	mixed		
	10	21.956	mixed		
YA MF-MA30s	1	16.252	mixed	20.127	2.386
	2	17.696	mixed		
	3	18.819	mixed		
	4	18.884	mixed		
	5	18.993	mixed		
	6	20.538	mixed		
	7	21.185	mixed		
	8	22.568	mixed		
	9	23.148	cohesive		
	10	23.191	cohesive		

Group	No	Tensile bond value	Failure mode	Mean	SD
YA MF-MA60s	1	17.479	mixed	21.423	2.582
	2	18.621	mixed		
	3	18.876	mixed		
	4	20.889	mixed		
	5	20.902	mixed		
	6	21.830	mixed		
	7	22.687	mixed		
	8	23.537	mixed		
	9	23.760	mixed		
	10	25.647	cohesive		
YA MF-MA120s	1	18.123	mixed	23.251	3.993
	2	18.527	mixed		
	3	19.268	mixed		
	4	20.931	mixed		
	5	21.833	mixed		
	6	24.280	mixed		
	7	26.416	mixed		
	8	27.124	mixed		
	9	27.605	mixed		
	10	28.406	cohesive		

Group	No	Tensile bond value	Failure mode	Mean	SD
YA MF-MA180s	1	23.345	mixed	24.927	1.290
	2	23.675	mixed		
	3	23.905	mixed		
	4	24.252	mixed		
	5	24.271	mixed		
	6	24.704	mixed		
	7	25.571	cohesive		
	8	25.723	mixed		
	9	26.851	mixed		
	10	26.972	cohesive		
CH C	1	9.171	adhesive	11.421	1.243
	2	9.563	adhesive		
	3	10.990	adhesive		
	4	11.488	adhesive		
	5	11.492	adhesive		
	6	11.671	adhesive		
	7	11.885	adhesive		
	8	12.260	adhesive		
	9	12.557	mixed		
	10	13.132	mixed		



Group	No	Tensile bond value	Failure mode	Mean	SD
CH MMA180s	1	15.113	adhesive	18.718	2.334
	2	17.228	mixed		
	3	17.298	mixed		
	4	18.132	mixed		
	5	18.146	mixed		
	6	18.526	mixed		
	7	19.370	mixed		
	8	19.555	cohesive		
	9	19.707	mixed		
	10	24.103	cohesive		
CH MF-MA15s	1	13.189	mixed	16.287	2.649
	2	13.482	mixed		
	3	14.117	mixed		
	4	14.575	mixed		
	5	15.714	mixed		
	6	16.053	mixed		
	7	16.814	mixed		
	8	18.415	cohesive		
	9	19.419	cohesive		
	10	21.090	cohesive		

Group	No	Tensile bond value	Failure mode	Mean	SD
CH MF-MA30s	1	14.048	adhesive	19.241	4.072
	2	14.324	mixed		
	3	15.527	adhesive		
	4	16.643	mixed		
	5	18.197	mixed		
	6	21.026	cohesive		
	7	21.373	mixed		
	8	21.515	mixed		
	9	24.846	mixed		
	10	24.910	mixed		
CH MF-MA60s	1	18.934	mixed	21.106	1.962
	2	19.053	mixed		
	3	19.124	mixed		
	4	19.420	mixed		
	5	21.012	mixed		
	6	21.192	mixed		
	7	21.940	cohesive		
	8	22.466	mixed		
	9	23.758	cohesive		
	10	24.163	cohesive		

Group	No	Tensile bond value	Failure mode	Mean	SD
CH MF-MA120s	1	16.517	mixed	18.996	2.002
	2	16.663	mixed		
	3	16.847	mixed		
	4	17.893	mixed		
	5	18.353	mixed		
	6	19.810	mixed		
	7	20.095	mixed		
	8	20.704	cohesive		
	9	21.016	mixed		
	10	22.059	cohesive		
CH MF-MA180s	1	15.445	mixed	18.221	3.276
	2	15.696	mixed		
	3	15.735	mixed		
	4	15.953	mixed		
	5	16.771	cohesive		
	6	17.454	mixed		
	7	18.009	cohesive		
	8	19.727	mixed		
	9	21.988	mixed		
	10	25.431	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
TB C	1	7.783	adhesive	11.542	2.017
	2	8.623	mixed		
	3	11.109	adhesive		
	4	11.215	adhesive		
	5	11.775	mixed		
	6	11.810	mixed		
	7	12.247	mixed		
	8	13.367	mixed		
	9	13.691	mixed		
	10	13.797	mixed		
TB MMA180s	1	14.112	mixed	16.229	2.007
	2	14.244	mixed		
	3	14.763	mixed		
	4	14.812	mixed		
	5	15.898	mixed		
	6	16.043	cohesive		
	7	16.217	mixed		
	8	16.973	mixed		
	9	19.481	cohesive		
	10	19.753	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
TB MF-MA15s	1	13.259	mixed	14.892	1.444
	2	13.281	mixed		
	3	13.419	mixed		
	4	13.561	mixed		
	5	14.888	mixed		
	6	15.445	mixed		
	7	15.594	mixed		
	8	15.636	mixed		
	9	16.893	mixed		
	10	16.945	mixed		
TB MF-MA30s	1	15.470	mixed	18.008	2.002
	2	15.654	mixed		
	3	15.767	mixed		
	4	17.536	mixed		
	5	18.165	mixed		
	6	18.264	mixed		
	7	18.463	mixed		
	8	18.792	mixed		
	9	20.669	mixed		
	10	21.302	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
TB MF-MA60s	1	17.422	mixed	20.090	1.806
	2	18.508	mixed		
	3	18.660	mixed		
	4	18.915	mixed		
	5	19.463	mixed		
	6	20.326	mixed		
	7	20.499	cohesive		
	8	22.166	cohesive		
	9	22.331	cohesive		
	10	22.615	cohesive		
TB MF-MA120s	1	15.700	mixed	18.291	1.902
	2	16.322	mixed		
	3	16.814	mixed		
	4	17.089	cohesive		
	5	17.849	mixed		
	6	17.956	mixed		
	7	19.367	mixed		
	8	19.885	mixed		
	9	20.871	cohesive		
	10	21.062	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
TB MF-MA180s	1	16.503	cohesive	18.568	1.617
	2	16.608	mixed		
	3	17.126	mixed		
	4	17.252	cohesive		
	5	18.158	mixed		
	6	19.209	mixed		
	7	19.790	cohesive		
	8	20.239	cohesive		
	9	20.263	cohesive		
	10	20.535	cohesive		

Table 19 The tensile bond strength values between self-cured acrylic resin and acrylic-denture teeth in each specimen after thermocycling. (MPa) Part II

Group	No	Tensile bond value	Failure mode	Mean	SD
YA C+	1	8.097	adhesive	10.173	2.016
	2	8.455	mixed		
	3	8.584	mixed		
	4	8.623	adhesive		
	5	8.854	mixed		
	6	9.839	mixed		
	7	10.639	mixed		
	8	12.608	mixed		
	9	12.655	mixed		
	10	13.373	mixed		
YA MF-MA15s+	1	8.309	mixed	12.062	2.040
	2	9.179	mixed		
	3	11.299	mixed		
	4	11.874	mixed		
	5	12.272	mixed		
	6	12.524	mixed		
	7	12.835	cohesive		
	8	13.291	mixed		
	9	14.462	mixed		
	10	14.577	mixed		



Group	No	Tensile bond value	Failure mode	Mean	SD
YA MMA180s+	1	10.470	mixed	14.973	3.539
	2	11.516	mixed		
	3	12.843	mixed		
	4	13.045	mixed		
	5	13.580	mixed		
	6	14.422	mixed		
	7	15.884	cohesive		
	8	16.634	mixed		
	9	19.807	mixed		
	10	21.532	mixed		
CH C+	1	5.174	mixed	8.459	2.182
	2	6.400	adhesive		
	3	6.599	mixed		
	4	7.355	adhesive		
	5	7.905	adhesive		
	6	8.937	adhesive		
	7	8.984	mixed		
	8	10.246	adhesive		
	9	10.906	mixed		
	10	12.086	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
CH MF-MA15s+	1	7.645	adhesive	13.986	3.604
	2	10.626	mixed		
	3	11.809	mixed		
	4	12.485	cohesive		
	5	13.882	mixed		
	6	14.718	mixed		
	7	15.212	cohesive		
	8	15.466	mixed		
	9	17.659	cohesive		
	10	20.363	mixed		
CH MMA180s+	1	9.960	mixed	11.749	1.513
	2	10.451	mixed		
	3	10.586	mixed		
	4	11.033	mixed		
	5	11.181	mixed		
	6	11.328	mixed		
	7	12.308	mixed		
	8	12.428	mixed		
	9	13.252	mixed		
	10	14.962	cohesive		

Group	No	Tensile bond value	Failure mode	Mean	SD
TB C+	1	8.297	mixed	12.492	2.067
	2	9.687	adhesive		
	3	12.007	adhesive		
	4	12.242	adhesive		
	5	12.980	mixed		
	6	12.999	adhesive		
	7	13.776	mixed		
	8	13.777	mixed		
	9	14.257	mixed		
	10	14.900	mixed		
TB MF-MA30s+	1	8.882	mixed	11.688	1.690
	2	9.105	mixed		
	3	10.548	mixed		
	4	11.711	mixed		
	5	11.954	mixed		
	6	12.145	mixed		
	7	12.191	mixed		
	8	13.273	mixed		
	9	13.437	mixed		
	10	13.629	mixed		

Group	No	Tensile bond value	Failure mode	Mean	SD
TB MMA+	1	9.365	mixed	12.810	1.718
	2	11.306	mixed		
	3	11.912	mixed		
	4	12.290	mixed		
	5	12.824	mixed		
	6	13.178	mixed		
	7	13.281	mixed		
	8	13.948	mixed		
	9	14.772	cohesive		
	10	15.227	cohesive		

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