

THE EFFECT OF DIFFERENT SURFACE TREATMENTS ON TENSILE BOND STRENGTH OF
ACRYLIC DENTURE TEETH AND LIGHT-CURED COMPOSITE RESIN



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

Department of Prosthodontics

FACULTY OF DENTISTRY

Chulalongkorn University

Academic Year 2019

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สาขาวิชาทันตกรรมประดิษฐ์ ภาควิชาทันตกรรมประดิษฐ์
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Thesis Title THE EFFECT OF DIFFERENT SURFACE TREATMENTS ON
TENSILE BOND STRENGTH OF ACRYLIC DENTURE TEETH
AND LIGHT-CURED COMPOSITE RESIN

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โชติบุช วงษ์พรไพโรจน์ : ผลของการทาสารปรับสภาพพื้นผิวต่อความแข็งแรงพันธะดึงระหว่างซีฟันเทียมอะคริลิกและคอมโพสิตเรซินชนิดแข็งตัวด้วยแสง. (THE EFFECT OF DIFFERENT SURFACE TREATMENTS ON TENSILE BOND STRENGTH OF ACRYLIC DENTURE TEETH AND LIGHT-CURED COMPOSITE RESIN) อ.ที่ปรึกษาหลัก : รศ.ชัยรัตน์ วิวัฒน์วรพันธ์

งานวิจัยนี้มีวัตถุประสงค์เพื่อศึกษาผลของการทาสารปรับสภาพพื้นผิวชนิดต่าง ๆ ที่มีต่อความแข็งแรงพันธะดึงระหว่างซีฟันเทียมอะคริลิกและเรซินคอมโพสิตชนิดแข็งตัวด้วยแสง โดยการขัดผิวด้านริมฝีปากของฟันัดบนซีฟันชนิดดั้งเดิม(Yamahachi New Ace) และชนิดร่างแหพอลิเมอร์แบบสอดไขว้ (Trubyte Bioform IPN) ฟันเทียมแต่ละชนิดถูกแบ่งออกเป็น 6 กลุ่ม (n=10) ตามการทาสารปรับสภาพพื้นผิวได้แก่ กลุ่มไม่ทาสาร (กลุ่มควบคุม), กลุ่มทาสารเมทิลเมทาคริเลต (MMA) 180 วินาที, กลุ่มทาสารละลายเมทิลฟอร์เมตและเมทิลอะซิเตต (MF-MA) ที่อัตราส่วนความเข้มข้น 25:75 โดยปริมาณเป็นเวลา 15 วินาที, กลุ่มทาสารยึติดคอมโพสิตเรซิน, กลุ่มทาสาร MMA (180วินาที)ตามด้วยการทาสารยึติดคอมโพสิตเรซิน และกลุ่มทาสาร MF-MA (15วินาที) ตามด้วยการทาสารยึติดคอมโพสิตเรซิน จากนั้นนำขึ้นทดสอบมาบรูณะด้วยคอมโพสิตเรซิน แล้วจึงนำมาทดสอบแรงดึงด้วยเครื่องทดสอบเอนกประสงค์ที่ระดับความเร็ว 0.5 มิลลิเมตรต่อนาที วิเคราะห์ข้อมูลทางสถิติโดยใช้การวิเคราะห์ความแปรปรวนสามทาง (ชนิดของซีฟันเทียม, การทำตัวทำละลายเคมี และการทาสารยึติด) และทดสอบความแตกต่างระหว่างค่าความแข็งแรงพันธะดึงโดยเฉลี่ยของกลุ่มต่าง ๆ โดยใช้การทดสอบ Dunnett T3 ที่ระดับความเชื่อมั่นร้อยละ 95 ผลการทดสอบพบว่าชนิดของซีฟันเทียม, การทำตัวทำละลายเคมี และการทาสารยึติดส่งผลต่อความแข็งแรงพันธะดึงระหว่างซีฟันเทียมอะคริลิกและคอมโพสิตเรซินอย่างมีนัยสำคัญทางสถิติที่ระดับความเชื่อมั่นร้อยละ 95 ค่าความแข็งแรงพันธะของกลุ่มที่ไม่สารปรับสภาพพื้นผิว, กลุ่มที่ทา MMA และกลุ่มที่ทา MF-MA ไม่แตกต่างกันอย่างมีนัยสำคัญทางสถิติ สำหรับฟัน Yamahachi New Ace ความแข็งแรงพันธะดึงของกลุ่มที่ทา MF-MA ร่วมกับการทาสารยึติดมีค่าสูงกว่ากลุ่มอื่นและ กลุ่มฟัน Trubyte IPN ที่ทา MF-MA ร่วมกับการยึติดอย่างมีนัยสำคัญทางสถิติ สำหรับฟัน Trubyte IPN กลุ่มที่ทา MMA ร่วมกับการยึติดให้ค่าความแข็งแรงพันธะดึงสูงกว่ากลุ่มอื่นอย่างมีนัยสำคัญทางสถิติ ที่ระดับความเชื่อมั่นร้อยละ 95 การศึกษานี้แนะนำการทาสารละลายเมทิลฟอร์เมต และเมทิลอะซิเตตเป็นเวลา 15 วินาทีตามด้วยการทาสารยึติดคอมโพสิตเรซิน ก่อนการซ่อมซีฟันเทียม ซึ่งสามารถเพิ่มความแข็งแรงพันธะดึงระหว่างซีฟันเทียมอะคริลิกชนิดดั้งเดิมและคอมโพสิตเรซิน

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KEYWORD: Acrylic denture teeth, Composite resin, Methyl acetate, Methyl formate, Tensile bond strength

Chotinut Wongpornpirot : THE EFFECT OF DIFFERENT SURFACE TREATMENTS ON TENSILE BOND STRENGTH OF ACRYLIC DENTURE TEETH AND LIGHT-CURED COMPOSITE RESIN. Advisor: Assoc. Prof. CHAIRAT WIWATWARRAPAN

This study evaluated the effect of different surface treatments on the tensile bond strength between acrylic denture teeth and composite resin. Maxillary lateral incisors of conventional acrylic denture teeth (Yamahachi New Ace) and highly cross-linked acrylic denture teeth (Trubyte Bioform IPN teeth) were ground on the labial surfaces. Each type of denture teeth were divided into six groups (n=10) according to surface treatment procedures which are no surface treatment (control), methyl methacrylate treatment (MMA) for 180 seconds, methyl formate-methyl acetate mixture (MF-MA) solution at a ratio of 25:75 (v/v) treatment for 15 seconds, composite bonding agents, MMA (180 seconds) with the application of bonding agent, and MF-MA (15 seconds) with a bonding agent. After surface treatments, light-cured composite resin was packed onto the treated surface. The tensile strength was measured using a Universal testing machine at a crosshead speed of 0.5mm/min. The data were analyzed using three-way ANOVA and post hoc Dunnett T3 test at a 95% confidence level. The denture teeth type, chemical solvents, and the use of a composite bonding agent significantly affected the tensile bond strength between acrylic denture teeth and composite resin ($p < 0.05$). The bond strengths of the control group, MMA treated, and MF-MA treated group were not significantly different ($p > 0.05$). For Yamahachi teeth, the bond strength of the MF-MA with bonding agent group was significantly higher than the other group ($p < 0.05$) and the MF-MA-bonding agent treated Trubyte IPN teeth. For Trubyte IPN teeth, the MMA-bonding agent treated group showed significantly higher tensile bond strength than the other group ($p < 0.05$). This study suggests the application of MF-MA solution for 15 s followed by a composite bonding agent before repair procedure can increase the tensile bond strength between conventional acrylic denture teeth and resin composite.

Field of Study: Prosthodontics

Student's Signature

Academic Year: 2019

Advisor's Signature

ACKNOWLEDGEMENTS

I would like to express my deep gratitude to Associate Professor Chairat Wiwatwarrapan, my advisor, for his kind support and useful advices of this research work. My grateful thanks are also extended to the staffs in the Oral Biology Research Center and the Dental Material Science Research Center for their assistance. Finally, I would like to thank my parents for their support and encouragement throughout my study.

Chotinut Wongpornpirot



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CHAPTER I

INTRODUCTION

Background and rationale

Currently, the older population tends to increase continuously around the world due to the better medical technology which provides health care and prolongs the lifespan. According to WHO, the global population is increasing 1.7% each year, while the elderly that age over 65 years is increasing at a rate of 2.5% [1]. In 2018, people older than 60 years constitute 13% of the global total population, which amounts to 990 million. Similarly, Thailand population in 2018 is 66 million which 18% of the total population is the elderly that amounts to 12 million [2]. Losing tooth support is the major dental complication in the elderly that consequently affects their mastication efficiency, nutrition and eventually their physical health. Removable partial denture and complete denture are commonly used to replace the missing teeth in the elderly.

Acrylic resin teeth are often preferred due to their structure that similar to denture base material structure, so they can chemically bond to the denture base [3]. Moreover, acrylic denture teeth are easy to adjust, do not wear the natural opposing teeth, do not cause a clicking sound when chewing, and they can provide a satisfying esthetic result. However, acrylic denture teeth debonding, fracture and wear can occur over time and require the repair procedure.

Light-cured composite resins are the most common direct restorative materials. Composite resins can be used both anterior and posterior teeth due to the improvement in filler particles and resin matrix that resulted in not only good mechanical properties but also satisfying esthetic results. Composite resin can also be used for prosthodontic purposes in various applications including the replacement of lost or broken denture teeth, the correction of acrylic resin denture teeth wear, and the modification labial surfaces of denture teeth to harmonize esthetic of the prosthetic teeth with the adjacent natural teeth [4-6].

It has been reported that fracture and denture teeth debonding damages were accounted for 26% [7] to 33% [8] of all denture repairs. In order to repair the broken

acrylic denture teeth, the in-office procedure may be preferred because it is less time consuming, less cost, aesthetically pleasing, and it is not required that the patient be without prosthesis. In an attempt to repair the acrylic denture teeth, the chemical bond may not occur since acrylic denture teeth are highly polymerized. Consequently, the surface treatments of acrylic denture teeth are required to provide the micromechanical retention and ensure the longevity of the repaired denture.

Chemical solvents can cause the swelling phenomenon on the polymethyl methacrylate (PMMA) which result in micro-irregularity of the surface. The diffusion of the solvent into the polymer is dependent on time, temperature, type of solvent, the polymeric structure, and glass transition temperature (T_g) of the polymer [9]. Many chemical solvents such as chloroform, methylene chloride, 4-methacryloxyethyl trimellitate anhydride (4-META), and methyl methacrylate (MMA) have been used to treat acrylic denture teeth surface, however chloroform and methylene chloride were identified as carcinogenic potential agents. MMA is the most common chemical solvent for treating the surface of acrylic denture teeth. It has been proved that MMA can enhance the bond strength of repaired acrylic denture base, relined acrylic denture base, and the bond strength between acrylic denture teeth and acrylic resin. Moreover, it had been reported that surface treatment with MMA enhanced the bond strength between acrylic denture teeth and resin composite [4], especially when combined with the use of a bonding agent [4, 6, 10-12]. Asmussen and Peutzfeldt (2000) found that low molecular weight methyl esters such as methyl formate (MF) and methyl acetate (MA) provided the softening effect on the acrylic denture base as effectively as methylene chloride [13].

The best wetting time to achieve the effective primed surface and diminish the adhesive failure for repair heat-polymerized acrylic resin using MMA as surface treatment is 180 seconds [9]. Whereas, a study showed that treating surface with MF, MA, or MF-MA mixture solution for 15 seconds significantly enhanced the flexural strength of repaired heat-polymerized denture base material, which comparable to the use of 180-second MMA [14]. Also, there was a study revealed that 15-second MF-MA treatment increased the bond strength between the acrylic denture base and relined material [15]. Another study found that increased wetting time of MF-MA (15, 30,

60, and 180 seconds) did not increase the tensile bond strength of relined denture base resin [16]. Moreover, the tensile bond strength between auto-polymerized acrylic resin and acrylic denture teeth treated with MF-MA solution for 15 seconds was significantly higher than the no-treatment surface but was not significantly different compared with the MF-MA 30 seconds, 60 seconds, 120 seconds, 180 seconds, and the 180-second MMA treated surface [17]. The 25:75 %v/v concentration was the best concentration of MF-MA mixture solution to promote the bond strength of the acrylic denture base [14, 18-20].

The MF, MA, and MF-MA mixture significantly increased the bond strength of repaired acrylic denture base, acrylic denture base relined with rebasing material, and the bond strength between acrylic denture teeth and auto-polymerized acrylic resin. Thus, it is hypothesized that the mixture of MF-MA could improve the tensile bond strength between acrylic denture teeth and resin composite.

Past studies indicated that using the non-toxic MF-MA solutions as a surface treatment agent can enhanced the bond strength of acrylic resin material with the advantage of less time-consuming. However, there is no study of the effect of using MF-MA solution to the bond strength between acrylic denture teeth and resin composite. Moreover, past studies about the effect of different surface treatment on the bond strength between acrylic denture teeth and resin composite were focus only on the shear bond strength. The purposes of this study are to investigate and compare the effect of the surface treatment with MMA, MF-MA, and composite bonding agent on the tensile bond strength between acrylic denture teeth and resin composite.

Objective

1. To evaluate and compare the effect of different surface treatments on tensile bond strength of acrylic denture teeth and composite resin
2. To evaluate and compare the effect of different denture teeth type on tensile bond strength of acrylic denture teeth and composite resin

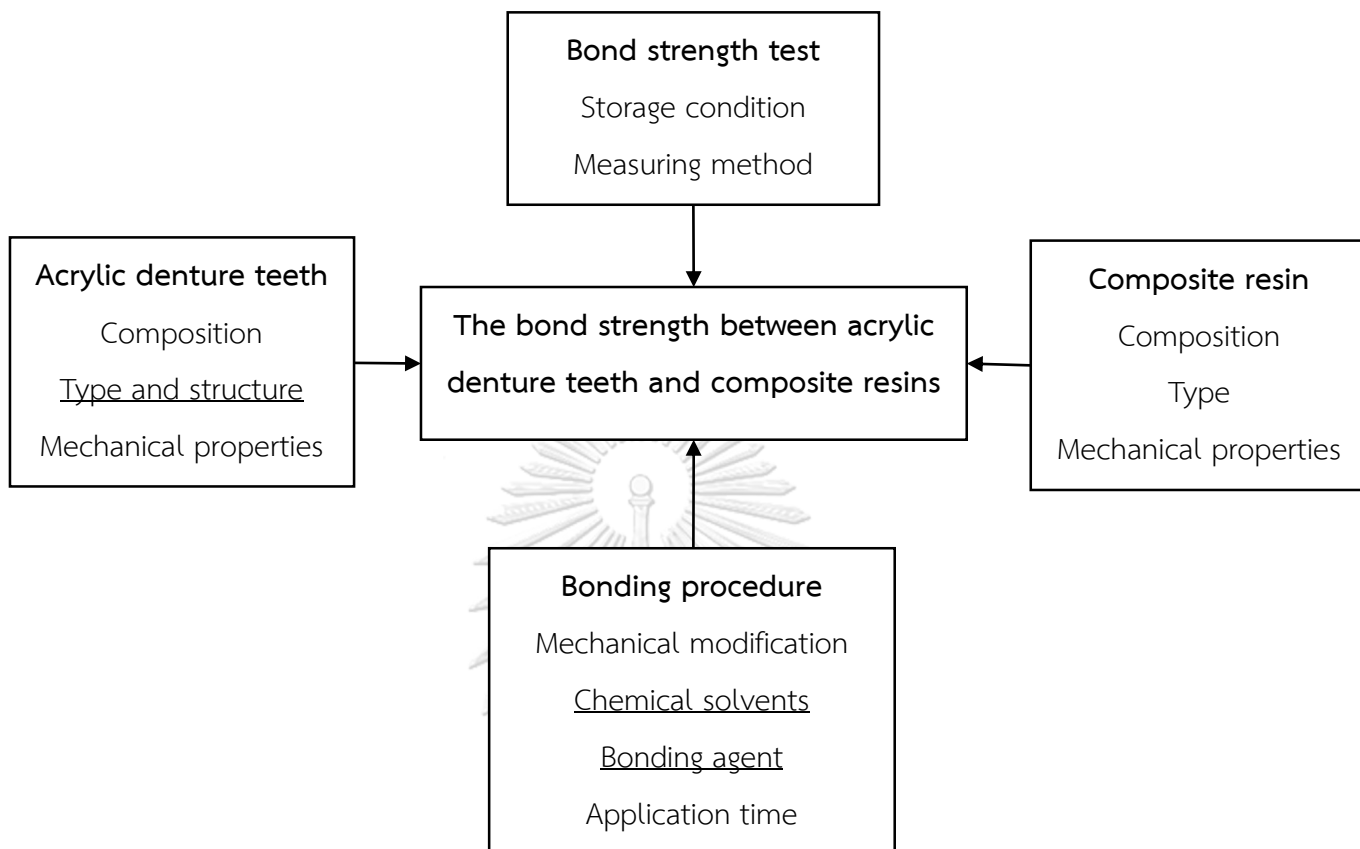
Research question

1. Do the different surface treatment methods effect on the tensile bond strength of acrylic denture teeth and composite resin?
2. Do the type of denture teeth effect on the tensile bond strength of acrylic denture teeth and composite resin?

Hypotheses

1. H_0 : there is no significant difference in tensile bond strength among different surface treatment methods at the 95% confidence level.
 H_1 : there is a significant difference in tensile bond strength among different surface treatment methods at the 95% confidence level.
2. H_0 : there is no significant difference in tensile bond strength among denture teeth type in the same surface treatment group at the 95% confidence level.
 H_1 : there is a significant difference in tensile bond strength among denture teeth type in the same surface treatment group at the 95% confidence level.

Conceptual framework



Keywords

Acrylic denture teeth, composite resin, methyl acetate, methyl formate, tensile bond strength

Research design

Laboratory and experimental research

Expected benefit

The alternative surface treatment protocol to enhance the effective bonding of denture teeth and composite resin.

CHAPTER II

REVIEW OF RELATED LITERATURE

Denture teeth

Denture teeth can be divided into 3 types according to the material used

1. Porcelain teeth

The composition of porcelain denture teeth is mainly feldspar, 15% quartz and 4% Kaolin.[21] The outstanding properties of porcelain teeth consist of highly resistant to abrasion, long-lasting cusp height, resistant to dental plaque deposition, and resistant to discoloration. [22] However, because of their hardness, porcelain teeth are difficult to polish after grinding and may produce excessive wear on enamel. In addition, porcelain teeth produce a clicking sound when contacting with opposing teeth. Porcelain teeth do not form a chemical bond to the polymer denture base, so they can attach to denture base by using only mechanical retention such as holes or pins.

In an attempt to enhance the bond strength, Paffenbarger et al. found that using a silane coupling agent can increase the bonding of porcelain teeth to self-cured and heat-cured denture bases.[23] Marchack et al. showed that high energy abrasion, hydrofluoric acid etching and the use of a general purpose bonding agent resulted in improved bond strength of heat-cured denture base resin bonded to porcelain teeth. Since high energy abrasion and hydrofluoric acid etching change the surface morphology of porcelain teeth and increase the surface roughness, the mechanical bonding between porcelain teeth and PMMA base is improved. Additionally, a multipurpose bonding agent monomer can penetrate tiny irregularities and copolymerize with resins to form a micromechanical bond to porcelain teeth. However, they found that silane coupling agent did not improve bond strength because heated polymerization of denture base in a water bath and water storage of samples may allow water ingress that could cause hydrolysis.[24] Another study in 2013 by El-Sheikh and Powers concluded that surface treatment of porcelain teeth with hydrofluoric acid etching, sandblasting and air abrasion can increase the bond

strength to denture base especially when using auto-polymerized resin. And the application of ceramic primer and bonding agent will also improve the bond strength of porcelain teeth.[25]

2.Acrylic resin teeth

Although ceramic denture teeth were first used in the removable partial and complete denture, ceramic denture teeth are less popular today. Acrylic resin teeth or polymer teeth are often preferred over ceramic teeth because they are easier to adjust, do not wear the natural opposing teeth and do not cause a clicking sound when chewing. Moreover, they are fabricated from polymethyl methacrylate, the same materials as denture base resin, so they can chemically bond to the denture base. The major difference in the composition of polymer teeth is that many pigments are used to create a variety of tooth shades. Acrylic resin teeth can provide satisfying esthetic since they are made in layers of different colors, translucencies, and thicknesses.[3]

Acrylic resin teeth are composed of PMMA beads and color pigments in a cross-linked polymer matrix. However, there also are differences in the polymer network itself. The degree of cross-linking in gingival, or body portion is much lesser than other portions to ensure the bonding ability to denture base material during the packing step of processing. On the other hand, the occlusal or incisal portions of acrylic resin teeth exhibit higher cross-linked polymer to provide resistance to crazing when the teeth are exposed to solvents such as alcohol. The increased cross-linking of polymer teeth together with the addition of inorganic filler also provide better wear resistance and a more solid feel when the patient chews. [3, 26]

Acrylic resin teeth can be classified into 2 types, based on their composition and method of polymerization

2.1 Conventional acrylic resin teeth

These teeth are homogenous in their composition and compose of a polymer matrix that is basically only one type of resin i.e. methyl methacrylate. Their impact resistance is higher than porcelain teeth resulted in less likely to harm the supporting tissue.[27] However, conventional acrylic resin teeth are

less wear resistance when compared to porcelain teeth or composite resin teeth.[28]

Examples of conventional acrylic resin teeth are Major dent (Major Prodotti Dentari, Italy), Yamahachi FX (Yamahachi Dental Mfg.Co., Aichi Pref., Japan), Basic (Heraus Kulzer, Germany), and Trubyte Bioform (Dentsply International, Inc., York, Pa)

2.2 Modified acrylic resin teeth

These teeth have an improved structure by adding cross-linked polymer to provide better wear resistance compared with the conventional type.

2.2.1 Semi-interpenetrating polymer network denture teeth (Semi-IPN)

According to IUPAC definition[29], semi-interpenetrating polymer network (SIPN) 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.

Semi-interpenetrating polymer networks 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; they are polymer blends.

An example of semi-IPN teeth is Cosmo HXL (Dentsply International, Inc., York, USA)

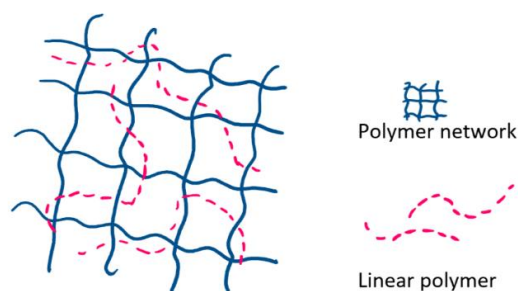


Figure 1 Schematic representation of a semi-IPN polymer

2.2.2 Interpenetrating polymer network denture teeth (IPN)

According to IUPAC definition, Interpenetrating polymer network is a polymer comprising two or more networks which 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. [29] However, a mixture of two or more pre-formed polymer networks is not an IPN.

Examples of IPN teeth are Trubyte Bioform IPN (Dentsply), Trilux , Vivodent (Ivoclar-Vivadent, Liechtenstein)

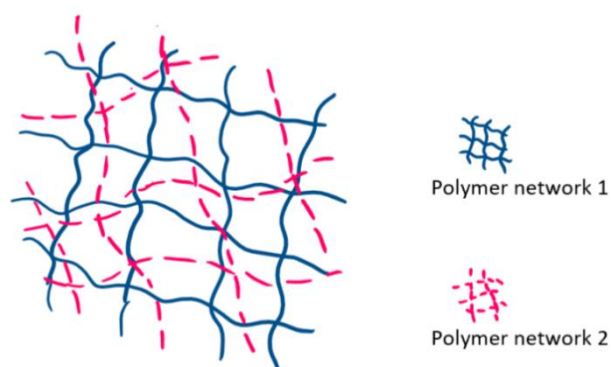
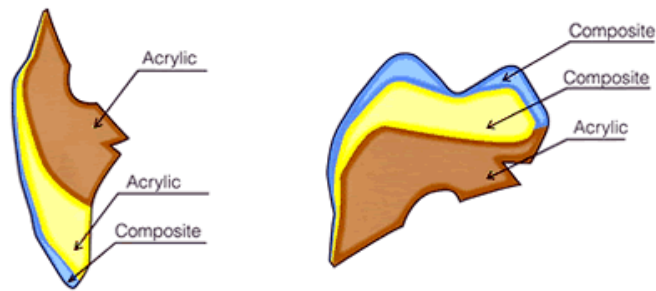


Figure 2 Schematic representation of an IPN polymer

3. Composite resin teeth

Composite resin denture teeth usually include composite resin-based and polymethyl methacrylate components which composite resins are found primarily on the labial, incisal or occlusal surface in order to provide esthetic and improved wear resistance. Since, the chemical bonding between composite resin and heat-polymerized denture base materials is lower than that which occurs between acrylic denture teeth and denture base materials, to overcome this problem, the manufacturers have incorporated polymethyl methacrylate at the ridge-lap and lingual surface of composite resin teeth.



http://www.yamahachi-dental.co.jp/en/products/01artificial_resin_teeth/01composite_resin_teeth/01what_is_px/

Figure 3 Structural layers of composite resin tooth

Examples of composite resin teeth are Yamahachi PX (Yamahachi Dental, Aichi Pref., Japan), Ivoclar PE (Ivoclar Vivadent AG., Schaan, Liechtenstein), SR Orthosit PE (Ivoclar Vivadent, Naturns, Italy), and Condyliform II NFC (Candulor, Wangen, Switzerland)

The longevity of removable denture

According to the investigation by Purcell et al. about the longevity of complete denture, the three most common complications encountered in this investigation were: replacement of acrylic resin posterior teeth due to wear, a requirement for maxillary complete denture laboratory heat-processed hard reline, and fractures of acrylic resin anterior teeth.[30]

As a result, wear resistance is one of the important properties of denture teeth to maintain the esthetic and function of removable dentures, thus sustain their longevity. Additionally, in order to maintain the denture esthetic, color stability of denture teeth material is one of the most important factors.

Wear resistance of denture teeth

Wear is a process that the materials have progressive loss of substance resulting from mechanical interaction between two contacting surfaces, which are in relative motion. When choosing proper denture teeth materials to use in removable partial

dentures or removable complete denture, wear resistance should be taken into consideration.

Wear resistance is important to maintain the denture's esthetic and stable occlusal relationship over time. Excessive wear of denture teeth might result in the reduction of the occlusal surface, loss of posterior teeth support, loss of vertical dimension of occlusion, loss of masticatory efficiency, alteration in the functional path of masticatory movement, the fatigue of masticatory muscles, faulty tooth relationship, and loss of aesthetics.[31]

Tooth wear in oral environment is a complex process that involves many factors, such as the abrasive nature of food, parafunctional habits, neuromuscular force, antagonistic material, enamel thickness and hardness, and chewing pattern.[28] Wear depends on three broad components of a system. First is the material structure which includes the type of materials and the geometry of their interaction. Secondly, the interaction conditions which are forces, stress applied to the system and duration of the interaction. The element is the environment and surface condition such as the ambient temperature, surface chemistry, topography, and environment. Wear can be classified into 4 types by its mechanism:

1) *Adhesive wear* which occurs when two surfaces are pressed together by a load and create local welding at the contact point. These welds are subjected to shear forces as the surface slide on another surface and can be broken. When this breakage occurs, some material is transferred from one surface to the other and often remains attached or may even transfer back to the original surface. The particles that have been created can contribute to abrasive wear. This type of wear is less likely to occur in the oral cavity because saliva acts as a lubricant and reduced friction.

2) *Abrasive wear* is the event that material is scraped off a surface either by hard protuberances on the other surface, which is called two-body abrasion or by hard particles at the surface that is called three-body abrasion.

3) *Fatigue wear* is caused by the repeated stressing and unstressing of materials which lead to the formation of microcracks at or below the surface. These microcracks can expand and join leading to the particle detachment from the material's surface.

4) *Corrosive wear* can be occurred by a chemical reaction between the surface and the environment.

Coffey et al. (1985)[32] has been studied in vitro wear characteristics of natural and artificial teeth under an artificial oral environment. This study used conventional acrylic teeth and IPN acrylic teeth, Trubyte Bioform (Dentsply International, Inc.), opposed by acrylic resin, IPN, and natural teeth. The result found that IPN teeth opposing IPN teeth demonstrated lower volume loss than acrylic resin teeth opposing acrylic resin teeth and IPN denture teeth were more resistant to wear than acrylic resin teeth when opposing each other. They also found that natural teeth did not exhibit measurable wear either when opposing IPN or acrylic teeth.

Whitman et al. (1987) studied the in vitro wear rates of three types of commercial denture teeth materials including conventional acrylic denture teeth (Trubyte/Bioform), acrylic interpenetrating-polymer network denture teeth (Trubyte/Bioform IPN), and microfilled composite denture teeth (Isosit) using the pin-and-disk wear apparatus. The report showed that interpenetrating-polymer network (IPN) and microfilled composite denture teeth are more wear resistant than conventional acrylic resin. Moreover, ethanol-soaked conventional acrylic resin specimens showed significantly greater wear than water-soaked acrylic resin and ethanol-soaked specimens of IPN and microfilled composite. Although IPN and microfilled composite showed a softening effect by ethanol, it did not significantly increase wear rates. The result of this study suggests that IPN and microfilled composite denture teeth are more chemically stable than conventional acrylic resin teeth.[33]

In 1998, Hirano et al. compared the in vitro wear of four different resin denture teeth against human enamel using mechanical wear testing device for two 5,000 cycles

wear periods under 13.4N load. The study showed the amount wear after 10,000 cycles of acrylic resin denture teeth (Kenson : 0.010 ± 0.003 mm and Classic : 0.008 ± 0.004 mm) was much higher than composite teeth (DB plus and MLI: 0.004 ± 0.002 mm).[34]

Suzuki (2004) investigated the Knoop hardness and in vitro wear of nano-composite denture teeth (Veracia), microfilled composites teeth (SR-Orthosit, Endura, Duradent, Surpass), cross-linked acrylic teeth (SR-Postaris, Genios-P, Creaparl, Vitapan Physiodens, Premium 8, Integral) and conventional acrylic teeth (Biotone). The study demonstrated Knoop hardness values of denture teeth materials as follows: microfilled composite had Knoop hardness ranged from 28.2 to 29.8, for nano-composite was 22.7, for cross-linked acrylic was 18.9 to 21.6, and 18.6 for conventional acrylic teeth. The results of wear depth recorded in this study showed that microfilled composites were more resistance than nano-composites, cross-linked acrylic, and conventional acrylic, respectively. The worn surface areas for denture teeth tested in this study exhibited similar tendencies and are presented in Figure 4. The wear value of the microfilled composite, the nano-composites, and the cross-linked acrylic was not statically different. However, the conventional acrylic's wear value was statistically higher from all tested denture teeth ($p\leq 0.0001$).[35]

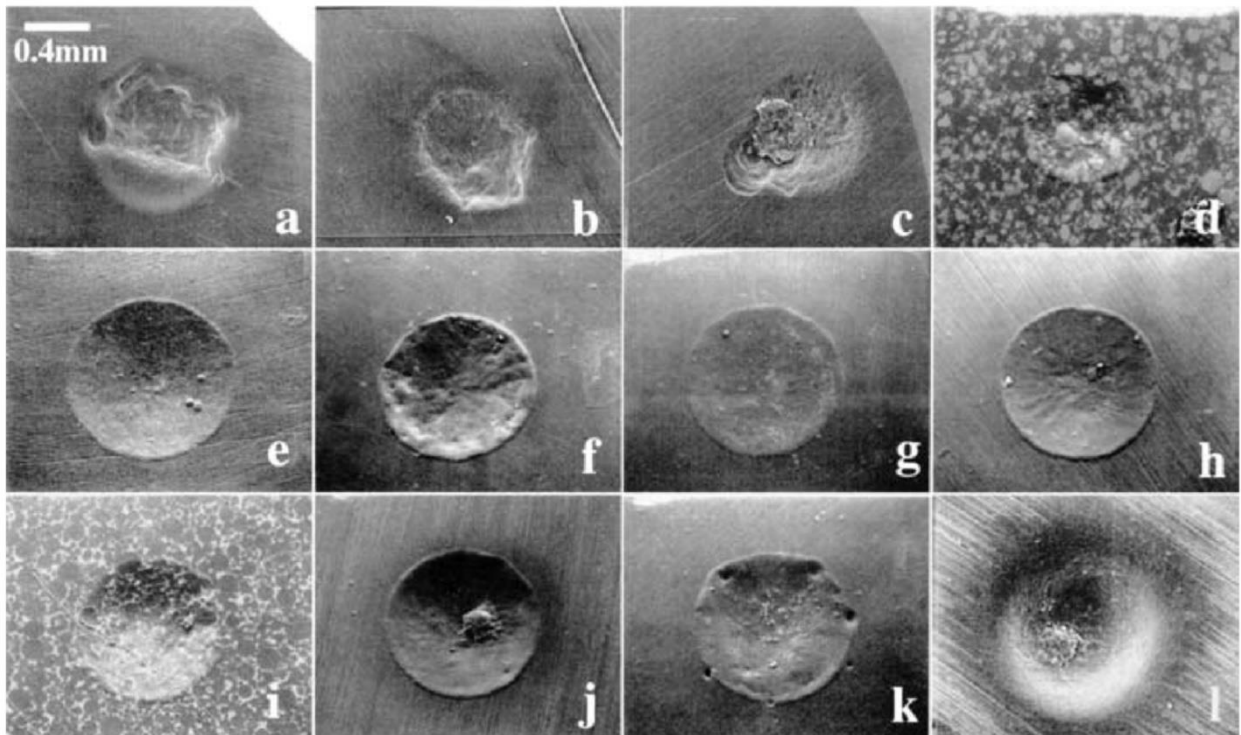


Figure 4 Suzuki (2004), SEM photographs of worn surfaces (magnification 50 \times).
 (a)–(d): Micro-filled composite teeth. [(a) SR-Orthosit. (b) Endura. (c) Duradent. (d) Surpass]. Note that the worn surfaces showed a brittle pattern. (e) Nano-composite tooth (Veracia). Note that the worn surface is very smooth. (f)–(k): Cross-linked teeth [(f) SR Postaris. (g) Genios-P. (h) Creapearl. (i) Vitapan Physiodens. (j) Premium 8. (k) Integral]. Note that the worn surfaces are smooth. Various sized spherical particles are seen in Physiodens specimen. (l) Acrylic control (Biotone) Note that the worn area is smooth but quite extended.

Study about the in vitro two-body wear resistance of three types of polymethyl methacrylate denture teeth, including 1 conventional PMMA denture tooth (Trubyte biotone) and 2 highly cross-linked IPN PMMA denture teeth (Trilux, Vivodent), by Reis et al. in 2008 found that there were no statistically significant differences ($p > 0.05$) in wear among the 3 denture teeth when opposed by glazed ceramic antagonist. On the other hand, the wear of conventional acrylic tooth that evaluated against airborne particle abraded ceramic was statistically significantly higher than highly cross-linked IPN teeth.[36]

Several studies found that improved acrylic resin denture teeth were more resistant to wear than conventional acrylic teeth. However, there are some study showed that the type of acrylic resin teeth did not affect the wear resistant. For example, the study by Ghazel et al.(2008) that evaluated the two-body wear resistance of the ceramic tooth (Bonartic CT[®]), the composite resin tooth (Condyloform II NFC[®]), and the acrylic resin teeth, including conventional acrylic teeth (Orthognath[®]) and cross-linked acrylic teeth (Bonartic TCR[®], Polystar Selection[®], SR Orthotyp DCL[®], and Vitapan Cuspiform[®]), when opposing to steatite ceramic balls in a dual-axis chewing stimulator. After 1,200,000 chewing cycles, the mean vertical substance loss and volume loss were measured using profilometry and an optical microscope. The results exhibited that the vertical substance loss of a ceramic tooth (36 μ m) was less than enamel (56 μ m), although the difference was not statistically significant. Among denture teeth types, all acrylic resin teeth showed statistically more wear than enamel, ceramic teeth, and composite resin teeth. However, there was no statistical difference between the acrylic resin teeth ($p>0.05$).[37] As well as study in 2011 by Suwannaroop et al. about the in vitro two-body wear resistance of artificial denture teeth which included 3 conventional acrylic resin teeth (Cosmo HXL, Major Dent, and Yamahachi FX), 1 high cross-linked acrylic resin teeth (Trubyte Bioform IPN), 2 composite resin teeth (SR Orthosit PE, and Yamahachi PX), and 1 porcelain teeth (ACE Teeth). They found that wear resistance of high cross-linked acrylic teeth was the lowest. This study also investigated the relation between wear resistance and some mechanical properties including hardness and elastic modulus, however, they could not find a definite relation.[31]

Dentures fracture

The causes of denture damage are accidental dropping, poor retention stability, poor occlusion, acrylic denture base defects, fatigue, and faulty teeth arrangement [38, 39]. The most common type of denture damages were the fracture and debonding of

the acrylic denture teeth which were accounted for 26% [7] to 33% [40] of all denture damages that need to be repaired. Although the chemical bonding between acrylic denture teeth and denture base materials has proven extremely effective, debonding of acrylic denture teeth can occur if the ridge-lap surfaces are contaminated by residual wax or separating media or there is a difference in structure of the two components due to their different processing method [3, 41]. Modification of the ridge-lap surface using mechanical, chemical, or a combination of these methods can improve the bond strength between acrylic denture tooth and denture base material.

Chemical surface treatment of heat-polymerized acrylic resin

Valittu et al. (1997) stated that when a solvent comes in contact with the polymer, the solvent will diffuse into the polymer and causing swelling of the polymer surface [42]. The surface of dissolve PMMA can chemically bond to the auto-polymerized acrylic resin base due to the formation of new polymer chains between the heat-polymerized acrylic resin pieces. The diffusion of the solvents is dependent on time exposure, temperature, type of solvent, the polymeric structure, and glass transition temperature (T_g) of the polymer [9].

Temperature

The diffusion of monomers of the denture base polymers can be promoted by increasing the polymerization temperature that consequently increase the bond strength between the acrylic denture teeth and the denture base polymer. When the auto-polymerized polymer was cured at 30°C, the denture teeth surface swelled to a depth of 3µm. Thickness of the swell layer was increased to 21µm when cured polymer at 70°C [42].

The polymeric structure

Although crosslinked networks make polymers harden and improve the wear resistant, highly crosslinked denture teeth (such as IPN teeth) show poor adhesive

bonding to the denture base material [27, 43, 44]. This might be due to the inhibition of the MMA monomer penetration by the crosslinked polymer surface.

Type of solvent

Factors that need to be considered in selection chemical surface treatment to increase the bond strength are solubility parameter and polarity

Solubility parameters can be used for selecting compatible solvents for coating resin, predicting the swelling of cured elastomers by solvents, estimating solvent vapor pressure in polymer solution for devolatilization and reaction systems, and predicting phase equilibria[45]. Base on the general softening theory, a liquid will act as plasticizer of a polymer solid when the polarities of the two are similar and the solubility parameters are close to each other[13]. The solubility parameters of solvents that had been used for acrylic resin surface treatment are presented in Table 1.

Table 1 the solubility parameters of acrylic resin polymer and chemical solvents that had been used for surface treatment

Name	Solubility parameter (MPa ^{1/2})
Poly (methyl methacrylate)	18.3
Acetone	20.3
Chloroform	19.0
Methyl acetate	19.6
Methylene chloride	19.8
Methyl formate	20.9
Methyl methacrylate	18.0

Acetone

Acetone (CH_3COCH_3) is a clear, colorless liquid with a molecular weight of 58.08. It is used as a solvent for fats, oils, waxes, resins, plastics, varnishes, for making other chemicals, and in nail polish remover [46, 47]. The structural formula of acetone is presented in Figure 5.

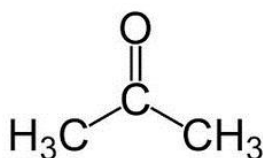


Figure 5 Structural formula of acetone

Sarac et al. (2005) studied the effect of chemical surface treatments on the shear bond strength of denture repair. They reported that the application of acetone for 30s prior denture repair showed significantly improvement of the shear bond strength [48]. Similar result was found in the study of the influence of chemical surface treatments in the repair bond strength of a heat-polymerized acrylic resin [49]. However, acetone can cause skin irritation.

Chloroform

Chloroform (CHCl_3) is a colorless, volatile, organic liquid solvent derivative of trichloromethane with a pleasant, sweet odor. The structural formula of chloroform is presented in Figure 6. It is widely used in biochemistry and molecular biology. Formerly, it was used as an inhaled anesthetic surgery, the primary use of chloroform is presently in industry as a solvent and in the production of the refrigerant [50, 51].

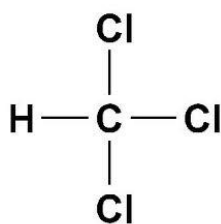


Figure 6 Structural formula of chloroform

Studies indicated that using chloroform as chemical surface treatment improved the bond strength of the repaired denture [52] and enhanced the bond strength between acrylic denture teeth and denture base [53]. Chloroform is identified as a carcinogen and should be handle with extreme caution. Acute toxicity of chloroform results in impaired liver function, cardiac arrhythmia, nausea and central nervous system dysfunction.

Methylene chloride

Methylene chloride (CH₂Cl₂) or dichloromethane is a colorless, volatile liquid with a sweet odor. It is used as a paint and varnish remover, solvent for plastics, degreasing agent, propellant, and blowing agent. The structural formula of methylene chloride is presented in Figure 7.

Methylene chloride has been marketed as adhesion promotor in rebasing acrylic denture base with an acrylic relining material [13]. In addition, using methylene chloride prior denture base repair can improve the shear bond strength [48]. Methylene chloride should be handled with extreme caution because it has carcinogenic and mutagenic potentials.

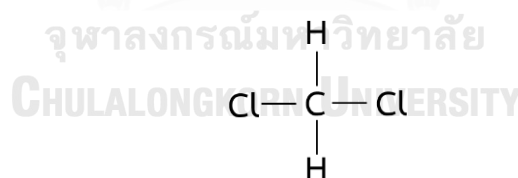


Figure 7 Structural formula of methylene chloride

Methyl methacrylate

Methyl methacrylate (MMA) is a colorless liquid with a sharp and fruity odor. It is used to make resin, plastics, and acrylic dentures. According to the New Jersey department of health and senior services, the odor threshold of MMA is 0.049 ppm [54]. The centers of disease control and prevention (CDC) reported that MMA has 100

ppm 8-hour workday exposure limits (NIOSH recommended exposure limit and OSHA permissible exposure limit) [55]. The structural formula of MMA is presented in Figure 8.

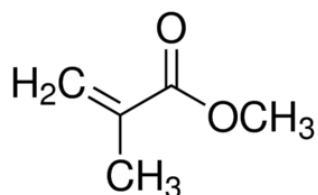


Figure 8 Structural formula of methyl methacrylate

Valittu et al. (1994) studied the effect of proper wetting time of MMA to the transverse strength of the repaired heat-polymerized acrylic resin. They suggested that the proper MMA wetting time of the repair surface significantly affected to the strength of repaired acrylic resin. A 180-second wetting time is the most effective wetting time which diminished the number of adhesive failures in repaired acrylic resins[9]. It has been demonstrated that the application of MMA effectively improved the bond strength of repaired acrylic resin, the bond strength between acrylic denture teeth and acrylic denture base [17, 56, 57], and the bond strength between reline material and acrylic denture base [58].

Methyl acetate and methyl formate

Methyl acetate (MA) ($\text{CH}_3\text{COOCH}_3$) is a colorless liquid with fruity odor. It is used as a solvent in lacquers and paint removers, and to make pharmaceuticals. According to the New Jersey department of health and senior services, the odor threshold of methyl acetate is 180 ppm [59]. And according to the national institute for occupational safety and health (NIOSH), the 8-hour workday exposure limits of methyl acetate is 200ppm [55].

Methyl formate (MF) (HCO_2CH_3) is a colorless liquid with a pleasant odor. It is used as an insecticide, a solvent, and to manufacture various organic chemicals. The odor threshold of methyl formate is 2,000 ppm [60]. The centers of disease control and prevention (CDC) suggested that methyl formate has 100 ppm 8-hour workday

exposure limits (NIOSH recommended exposure limit and OSHA permissible exposure limit) [55].

Asmussen and Peutzfeldt (2000) suggested the low molecular weight methyl esters including methyl formate (MF) and methyl acetate (MA) were as effectively softening the polymethyl methacrylate denture base as methylene chloride but less toxic. The effect of using MA, MF, and the mixture of both solvents as chemical surface treatment has been investigated. It has been found that MA, MF, and the mixture of MF-MA at various concentration significantly enhanced the flexural strength of repaired acrylic denture base and the tensile bond strength between acrylic denture teeth and denture base. The application of 25:75 %v/v concentration of MF-MA mixture solution showed highest strength, however there was no significantly different compared with other concentration [14, 61]. Moreover, it has been demonstrated that using MF-MA mixture solution as chemical surface treatment for 15 seconds improved the bond strength of relined denture base resin [15, 16, 18], the tensile bond strength between acrylic denture teeth and denture base resin [17], and the tensile bond strength between denture base resin and soft lining material [20].

The structural formula of methyl acetate and methyl formate are presented in Figure 9 and 10, respectively.

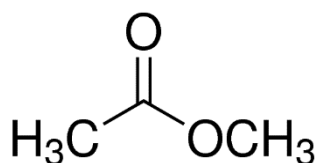


Figure 9 Structural formula of methyl acetate

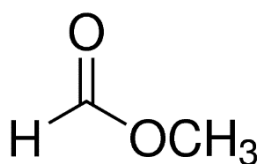


Figure 10 Structural formula of methyl formate

Composite resin

Composite resins for dental use were introduced in the 1960s, they were developed to combine the esthetic and ease of use for restoration in anterior teeth. Composite resins are majority composed of a soft polymerizable dimethacrylate resin base and hard inorganic filler particles.

The composition of composite resins

Dental composite resins can be distinguished by differences in the formulation. However, these materials are similar in composition as they are all composed of a polymeric matrix, reinforced inorganic filler particle, coupling agent, initiator of polymerization and other ingredients including the oxide pigments to provide a range of shade.

1) Polymer matrix

The organic polymer matrix in currently available composites is commonly an aromatic or urethane diacrylate oligomer such as bisphenol A glycidyl methacrylate (Bis-GMA). Oligomers have reactive double bonds at each end of the molecule that can be able to undergo addition polymerization in the presence of free radicals. Since the oligomer molecule is highly viscous, the addition of low-molecular-weight diluent monomers, such as triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA) is required in order to make composite resin more clinically workable consistency even when the filler is incorporated.

2) Coupling agent

The silane coupling agent is important to bond the filler particles to the polymer matrix of composite resin. The silane molecule has reactive groups at both ends and is coated on the filler particle surface before mixing with the oligomer. While polymerization reaction occurs, double bonds on the silane molecule will react with the polymeric matrix. When filler and polymeric matrix are bonded properly, the

stresses generated during function can be distributed from the soft polymeric matrix to hard fillers. The bonding between filler particles and the polymeric matrix is the most important to reinforce the strength of composite resin. Additionally, bonding will enhance the retention of filler particle at the composite surface during abrasive action, therefore the wear resistance of composite is improved. Effective bonding between filler particles and polymeric matrix achieving by silane coupling agent also play an important role to reduce the water resorption of materials and resulted in better dimensional and color stability of composite resin over time.

3) Initiators

Most composite resins currently are light activated, either as the sole polymerization initiator or in a dual cure formation containing a chemically cured component. In chemically activated system, the addition polymerization can be initiated by an organic peroxide initiator (or catalyst) reacts with a tertiary amine accelerator, producing free radicals that attack the double bonds of monomer molecules.

Light-activated polymerization can be initiated by the generation of free radicals with a photoinitiator when irradiated with blue light. The most common photoinitiator is camphoquinone, accelerated by an aromatic tertiary amine. Other photoinitiators that are less yellow than camphoquinone can be formulated to provide more color stability, such as PPD (1-phenyl-1,2-propanedione), Lucirin TPO (monoacylphosphine oxide), and Irgacure 819 (bisacyl-phosphine oxide).[62]

4) Filler composition

Filler particles are added to composites in order to enhance mechanical properties. Filler particles are inorganic composition, typically made from radiopaque glass. In addition to quartz, fine-sized particles may be composed of barium or lithium aluminum silicate glasses (borosilicate glass), or barium, strontium, or zinc glasses.[21] Filler not only directly determine the mechanical properties of composite resin

materials but also allow reducing the monomer content and consequently the polymerization shrinkage, optimizing wear, translucency, opalescence, radiopacity, intrinsic surface roughness, so the polishability of the material is also improved and enhance its esthetics and handling properties.[63]

Type of composite resins

Composite resins can be used for many applications in dentistry and can be distinguished by differences formulation tailored to their particular requirements as restoratives materials, pits and fissure sealants, cement, provisional materials, etc. Restorative composite resins materials are distinguished by their consistency which depending upon the formulation. The flowable composites, which are typically less viscous due to the reducing of the filler particles or the adding of other modifying agents such as surfactants, are designed to be dispensed from very fine bore syringes into small spaces for enhanced adaptation. The packable composites are the thicker consistency restorations which are produced by modification of the filler size distributions or the addition of other types of particles, such as fibers, however, the overall filler level is usually not increased. [62]

Packable composites are designed to provide significant resistance to a condenser or other instrument in order to avoid slumping and to enhance the formation of tight interproximal contacts. The materials were developed in an attempt to limit wear and fracture of restoration, to reduce polymerization shrinkage and the technique sensitivity. there was a study have found that the mechanical properties of packable resin composites (ALERT, Pyramid-Dentin, Pyramid-Enamel, Solitaire, SureFil) are similar to typical hybrid resin composites (Z100) except Solitaire which performed worse.[64] Another purported advantage of high-viscosity packable composites was better handling characteristic and adequate proximal contact however some studies reported that there was no difference in the proximal contact tightness created with either packable or hybrid composite.[65] Additionally, some authors concluded that

there was no clinically advantages of using the packable composites when compared to hybrid composites.[63] Furthermore, a study about the packable composites reports that the mechanical properties and wear behavior of the composite resins are highly influenced by the filler system and not by the material category.[66]

Most of the composite resin classification criteria are influenced by the filler system. These criteria are primarily based on the amount of inorganic filler fraction in volume percent or the mean particle size.

1) Conventional dental composites or macrofilled composite

Conventional composites had average particle sizes exceeded 1 μm and typically had fillers close to or exceeding the diameter of a human hair which approximately 50 μm . Because of the large size filler particles, conventional macrofilled composites were very strong but difficult to polish and impossible to retain surface smoothness.

2) Microfilled resin composites

Microfilled composites were produced to enhance polishability. Actually, these materials were truly nanocomposites because the average size of the spherical colloidal silica reinforcing particles was approximately 40nm (0.01-0.12 μm). Filler loading in these composites is low and limited to about 30% to 55% by volume or 35% to 60% by weight. However, filler content may be increased, and properties improved by incorporating pre-polymerizing resin fillers (PRPF) 10 to 20 μm in diameter. In order to enhance the polishability and esthetics of microfilled composites, the strength of these composites must be compromised due to their relatively low filler content.

3) Microhybrid composites

Further refinements in the particle size through enhanced milling and grinding techniques resulted in composites with sub-micron particles. Therefore, microhybrid composites are composed of a combination of microfillers and ultrafine glass particles.

Their particles size was reduced to 0.04 – 1.0 μ m. Microhybrid composites are generally considered to be universal composites which can offer both esthetic and enhanced strength for use in both anterior and posterior applications.

4) Nanofilled composites

Nanofilled composites contain only nanoscale particles. Most manufacturers have modified the formulations of microhybrid composites with smaller sized particles prepared using nanotechnology and possibly include pre-polymerized resin fillers and have named this group nanohybrid.

Properties of dental composites

Direct restorative composite materials have been developed and improved since their first introduced in the 1960s. Therefore, present methacrylate-based direct composites are excellent accepted. The most significant modify in commercial composites in the last decades have been made through the improvements in the filler system, because fillers directly determine the mechanical properties and the esthetic of restoration.

Ilie and Hickel (2009) investigated the mechanical behavior of 72 dental composites including hybrid, nanohybrid, microfilled, packable composites, flowable composites, compomers, flowable compomers, and ormocer-based composites. The last one is the inorganic-organic hybrid restorative materials which ormocer stands for originally modified ceramics. According to this report, the hybrid, nanohybrid, packable and ormocer-based composite do not have significant differences in mechanical properties—including flexural strength, flexural modulus, diametric tensile strength, and compressive strength—among each other material groups and also reaching the highest flexural strength. The microfilled composites achieved the lowest mean mechanical properties. Although the flowable composites and compomers showed similar mechanical properties to microfilled composites, the diametric tensile strength

of the last one was lower. They also found that filler volume was the strongest influence on the mechanical properties, followed by filler weight.[63]

In general, dental composites have flexural strength, fracture toughness, and tensile strength superior to glass ionomer and similar to those of porcelain and amalgam. Dental composites nowadays have adequate mechanical properties for use in all areas of the mouth except for high-stress situations, such as bruxism or parafunctional habits. Due to their lower elastic modulus when compared to amalgam, composite resins may allow enhanced deformation and dimensional change under high stress and lead to defect formation.[62]

The wear resistance of composite resins is considered to be a lesser problem for current materials because of the refinement of reinforcing fillers size which consequently reduced the magnitude of abrasive wear. However, when placed in large restoration or for those patients with bruxism and clenching behavior, the wear of composites may still be a concern. Several studies found that nanofilled composites (Filtek Z350 XT) have high compressive strength, hardness, and greater wear resistance when compared to microfilled composite, resin-modified glass ionomer cement [67] and microhybrid composites.[68, 69] However, Yesil et al. (2008) evaluated the 3-body abrasive wear resistance of nanofilled composite resin (Filtek Supreme), nanohybrid composite (Premise), microfilled composite (Heliomolar RO), and microhybrid composite (Point 4). They found that the abrasive wear of conventional microfilled material was not significantly different from the wear data of the nanofilled and microhybrid materials, but it was less than the wear of nanohybrid.[70]

According to a systemic review of wear resistance of dental tissues and materials in clinical studies by Wulfman et al. in 2018, composite materials appeared to be more resistant to wear than PMMA resin denture teeth. Composite resins were shown to have mean vertical height loss varied between 20 and 40 μm after one year whereas resin-based denture tooth materials, which used very similar wear

measurement protocols, were reported values varied between 140 and 225 μm after one year.[71] When compared the wear behavior between enamel cusp and esthetic restorative materials which include zirconia, porcelain, and composite resin, Jang et al. (2019) found that zirconia (Lava) with polished surface, microhybrid composite with PRPF (Gradia Direct), and nanofilled composite (Filtek Z350) exhibited less wear of antagonist enamel cusp. However, the nanofilled composite showed high wear on its own surface. The greatest wear of enamel was found when opposed to porcelain followed by microhybrid composite (Z250).[72]

The abrasive wear of resin composites is influenced by many factors including the fillers, the resin matrix, and the bonding between fillers and resin matrix. First, the shape, size, orientation, distribution, and the amount of filler are important. Second, the type of resin matrix and the polymerization initiators of resin composite which influence the hardness of the resin composite surface. Third, bonding between fillers and resin matrix affects the likelihood of plucking of filler particles. Because if the bonding between filler and resin matrix is not adequate, when the loading is applied, the filler will detach from the resin matrix and may lead to 3-body abrasive wear. The abrasive wear is reduced when bonding between the filler and the resin matrix is improved as well as when the size of or spacing between filler particles is reduced and when the degree of conversion of the resin matrix is increased.[73]

Although the mechanical properties of composite resin can be improved by higher filler volume, the flexural strength and flexural modulus tend to be enhanced until a filler volume of 60%. It seems like introducing a higher filler volume than 60% will probably also introduce a higher amount of defects.[63] Another study also reported a similar statement about fracture toughness. Kim et al. (2002) studied the effect of filler loading on the mechanical properties of composites and concluded that the improvement of composite resin fracture toughness was affected by an increasing of filler loading up to a threshold level of approximately 55% filler volume.[74]

Bonding of composite resin to acrylic denture teeth

The contamination of separating media on the ridge lap area of the denture teeth will lead to the debonding of denture teeth from denture base resin. In addition, the denture teeth may have been worn or fractured over time. In order to repair removable dentures, laboratory process requires the transportation of denture to a remote laboratory and causing the patient to be without the prosthesis for several days. Due to the various shade and great wear resistance of composite resin, therefore, composite materials may be suitable not only for the replacement of the lost or broken denture teeth but also for the restoration of the worn cusp. Repairing the denture teeth with composite resins can be performed as a chair-side procedure which will provide the esthetic satisfying result and eliminate the need for costly and time-consuming processing. Moreover, composite resins can be used to build up or modify the facial surface of denture teeth to harmonize the esthetics of denture with characteristics of adjacent natural teeth or with the facial characteristics of the patient.

When repaired composite resins restoration, the connection between old and new material has been described to occur by three possible mechanisms. First, the micromechanical retention created by the penetration of the new monomer into the irregularities of the treated old composite surface. The second mechanism is through the chemical bonding of the new monomers to the resin matrix. The last one is through the chemical bonding with the exposed filler particles. [75]

Wendler et al. (2016) had been discussed about the effect of different surface treatment on repaired bond strength of composite resin. They found that the presence of unreacted C=C double bonds in the treated surface layer plays an important role in creating the covalent chemical bonds to the monomer of the newly applied composites. Additionally, micromechanical retention on the old composite surface had been reported as one of the key mechanisms to achieve reliable repair bond strength. Mechanical surface modification such as sandblasting and grinding the surface with dental bur can provide irregularities of the surface and serves as macro- and micro-

retentive features. However, there was no consistent correlation between the surface roughness profile and the bond strength achieved.[76, 77] Silane coupling agents slightly enhanced the bond strength when applied on the silica-coated surface compared to the solely silanized surface. The silane coupling agent can form covalent bonds with the exposed fillers in the old composite resin surface and co-polymerizes with methacrylate groups of the repair material. Furthermore, silane promotes the diffusion of the bonding agent into the micro-retentions in the substrate by improving the wettability of the surface.

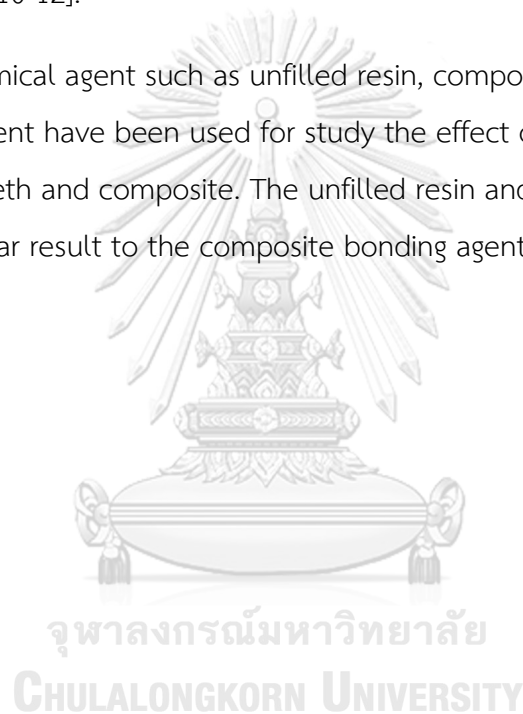
To provide the successful results and longevity of the repaired denture, the most important factor is a reliable bonding between composite resins and acrylic denture teeth. Since the acrylic denture teeth rarely have unreacted C=C double bonds on the surface layer, therefore the chemical bond between acrylic denture teeth and composite resins may not occur. Surface preparations of the acrylic denture teeth such as the application of phosphoric acid, a bonding system, MMA monomer, and other solvents will play an important role to provide the micromechanical retention and ensure a long service life of repaired denture.

Effect of chemical surface treatment on the bond strength between acrylic denture teeth and composite resin

Many studies proved that surface treatment acrylic denture teeth with MMA monomer improved the bond strength to the composite resin [11, 78]. Since the polymerization process of MMA and Bis-GMA follow a similar pattern of activation and cross-linking, due to the similarity of the reactive methacrylate groups, some chemical bonding between composite and acrylic resin may occur. However, it can be possible only if crosslinks are available on the acrylic resin teeth for bonding to the composite [78]. Since MMA can swell the acrylic denture tooth surface, the predominant bond could be a mechanical bond that is provided by the infiltration of the composite into the surface microirregularities.

The poor wettability properties of the high-viscosity composite material inhibited the penetration of composite resin into the acrylic denture teeth surface. With the composite bonding agent, a better surface wetting may be achieved by the infiltration of the resin into microscopic surfaces. Several studies demonstrated the important of using adhesive agent as surface treatment for bonding composite resin to acrylic denture teeth [5, 11, 12, 79]. The highest bond strength between acrylic denture teeth and composite resin obtained by the combination use of MMA and composite bonding agent [6, 10-12].

Other chemical agent such as unfilled resin, composite color modifier, and silane coupling agent have been used for study the effect of the bond strength of acrylic denture teeth and composite. The unfilled resin and composite color modifier provided the similar result to the composite bonding agent [78].



CHAPTER III

RESEARCH METHODOLOGY

Materials and instruments

1. Maxillary lateral incisor denture teeth 2 brands (Yamahachi New Ace and Trubyte Bioform IPN)
2. Resin composite (Filtek Z350XT, 3M ESPE, St. Paul, MN, USA)
3. Methyl methacrylate solution (the liquid part of Unifast Trad, GC Dental Product Corp, Aichi, Japan)
4. Methyl formate (Merck Schuchardt OHG, Germany)
5. Methyl acetate (Merck KGaA, USA)
6. Composite bonding agent (Adper Single Bond 2 Adhesive, 3M ESPE, St. Paul, MN, USA)
7. Polyethylene pipes with 20mm diameter
8. Potting resin
9. Silicon carbide paper no. 280, 600
10. Polypropylene film
11. Acrylic resin ring
12. Acrylic resin rod
13. Cyanoacrylate glue (Super Glue, Alteco Chemical PTE Ltd., Osaka, Japan)
14. Distilled water
15. Microbrush
16. Timer
17. Polishing machine (Nano2000, PACE Technologies, Si. Tucson, AZ, USA)
18. Ultrasonic cleaner (GT-1730QTS GT Sonic, GuangDong GT Ultrasonic Co.,Ltd, Shenzhen City, China)

19. LED light curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA, light output: 1200 mW/cm²)

20. Universal testing machine (Shimadzu, EZ-S 500N mode, Kyoto, Japan)

21. Incubator 37°C (Contherm Scientific Ltd., New Zealand)

22. Stereo microscope (Olympus SZH10, Olympus Optical, Tokyo, Japan)

Table 2 Materials used in this study

Material	Product name	Composition	Manufacturer
Denture teeth	Yamahachi New Ace	Poly(methyl methacrylate)	Yamahachi Dental Mfg.Co.,Aichi Pref., Japan
	Trubyte Bioform IPN	Highly cross-linked poly(methyl methacrylate)	Dentsply International, Inc., York, PA, USA
Resin composite	Filtek Z350 XT	Bis-GMA, Bis-EMA, UDMA, PEGDMA, TEGDMA, zirconia and silica particles(5-20nm nonagglomerated)	3M ESPE, St. Paul, MN, USA
Chemical agents	Unifast Trad (liquid)	Methyl methacrylate	GC Dental product corp., Aichi., Japan
	Methyl formate	Methyl formate	Merck Schuchardt OHG, Germany
	Methyl acetate	Methyl acetate	Merck KGaA, USA
	Adper Single bond 2	Ethyl alcohol, Bis-GMA, silane treated silica, HEMA, hydroxy-1,3-dimethacryloxypropane, UDMA	3M ESPE, St. Paul, MN, USA

Bis-GMA: bisphenol A diglycidyl dimethacrylate, Bis-EMA: Bisphenol A polyethylene glycol diether dimethacrylate, UDMA: Diurethane dimethacrylate, PEGDMA: Polyethylene Glycol Dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, HEMA: 2-Hydroxyethyl methacrylate

Sample preparation

Sixty maxillary lateral incisor acrylic denture teeth of each brand were embedded with labial surface down inside polyethylene pipes with auto-polymerized potting resin (Figure 11(a-b)). Then the labial surfaces of all specimens were polished to a depth of proximately 1-1.5mm with 280-grit and 600-grit silicon carbide paper in a polishing machine (Nano2000, PACE Technologies, Si. Tucson, AZ, USA) (Figure11(c)). The specimens were ultrasonically cleaned (GT-1730QTS GT Sonic, GuangDong GT Ultrasonic Co.,Ltd, Shenzhen City, China) with distilled water for 10 min and were left to air dry.

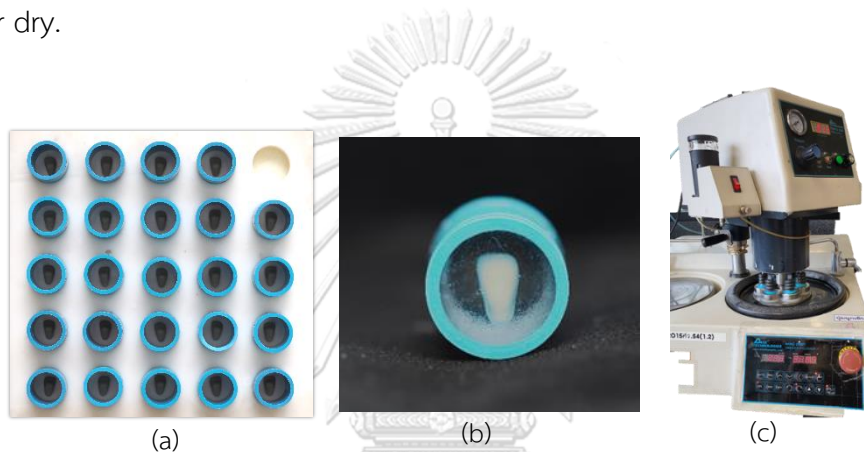


Figure 11 Specimen preparation.

(a) Acrylic denture teeth were embedded in potting resin. (b) The specimen with labial surface down. (c) The labial surfaces of acrylic denture teeth were polished.

The specimens of each brand were divided into 6 groups (n=10) and chemical surface treatment were applied as following:

Group I (YA C) Control specimens of Yamahachi New Ace teeth (Yamahachi Dental Mfg.Co.,Aichi Pref., Japan). No surface treatment was performed and a clear 50- μ m thickness polypropylene film specimens with a 3-mm diameter hole was placed over the surface.

Group II (YA MMA) MMA monomer (the liquid part of Unifast trad, GC Dental product corp., Aichi., Japan). MMA were applied by brush every 3 seconds for 180 seconds. Then the excess liquid was blown away and a clear polypropylene film with a 3-mm diameter hole was placed over the treated surface.

Group III (YA MF-MA) MF-MA mixture solution at a ratio of 25:75 by volume. MF-MA solution was applied by brush every 3 seconds for 15 seconds. Then the excess liquid was blown away and a clear polypropylene film with a 3-mm diameter hole was placed over the treated surface.

Group IV (YA B) Adper Single Bond 2 composite bonding agent (3M ESPE, St. Paul, MN, USA). After the specimen dried, the surface of the specimen was covered with a clear polypropylene film with a 3-mm diameter hole. Two layers of composite bonding agent were applied for 15 seconds, air thinned with oil-free air for 5 seconds. Then polymerized with LED light curing unit for 20 seconds.

Group V (YA MMA+B) MMA monomer with a composite bonding agent. MMA was applied for 180 seconds (by brush every 3 seconds). After the excess liquid was blown away, a clear polypropylene film with a 3-mm diameter hole was placed over the treated surface. Two layers of composite bonding agent were applied for 15 seconds, air thinned for 5 seconds and then light-cured for 20 seconds.

Group VI (YA MF-MA+B) MF-MA solution with a composite bonding agent. MF-MA mixture solution was applied for 15 seconds (by brush every 3 seconds). After the excess liquid was blown away, a clear polypropylene film with a 3-mm diameter hole was placed over the treated surface. Two layers of composite bonding agent will applied for 15 seconds, air thinned for 5 seconds and then light-cured for 20 seconds.

Group VII (TB C) the control specimens of Trubyte Bioform IPN denture teeth. No surface treatment was performed. All surface specimens were covered with a clear 50- μm thickness polypropylene film specimens with a 3-mm diameter hole.

Group VIII (TB MMA) Trubyte Bioform IPN denture teeth were prepared the same method as group II

Group IX (TB MF-MA) Trubyte Bioform IPN denture teeth were prepared the same method as group III

Group X (TB B) Trubyte Bioform IPN denture teeth were prepared the same method as group IV

Group XI (TB MMA+B) Trubyte Bioform IPN denture teeth were prepared the same method as group V

Group XII (TB MF-MA+B) Trubyte Bioform IPN denture teeth were prepared the same method as group VI

After surface treatment, all specimens were centrally placed with a 5-mm diameter acrylic ring over the hole of polypropylene film. Resin composite (Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA) was packed in the ring hole with 2mm in height and light-cured for 40 seconds. The specimens were then kept in distilled water at 37°C for 24 hours before testing (Figure 12).

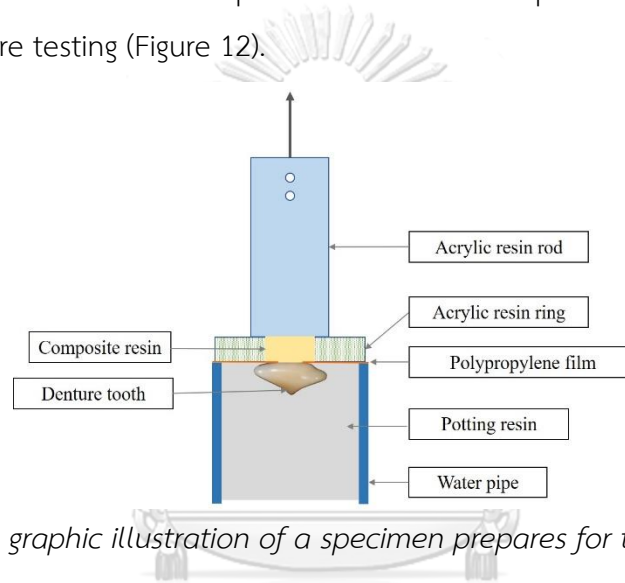


Figure 12 The graphic illustration of a specimen prepares for tensile strength testing.

Tensile bond strength test

After the storage period, each specimen was left to air dry at room temperature and then the acrylic resin rod was attached to top part of the specimen using cyanoacrylate glue (Super Glue, Alteco Chemical PTE Ltd., Osaka, Japan). Specimens were fixed in metal mounting jig and connected to the tensile testing machine by securing the acrylic resin rods in a vertical position to the testing apparatus (Figure 13). The tensile strengths were measured by a universal testing machine (Shimadzu, EZ-S 500N mode, Kyoto, Japan) with a 500N load cell at a crosshead speed of 0.5mm/min. The tensile bond strength value (MPa) was calculated by dividing the failure force by the bond surface area.

The following equation will be used to calculate the tensile bond strength:

$$B = \frac{F}{A}$$

Where B is the bond strength value in MPa, F is the maximum load (N) before debonding occurred, and A is the adhesive area in mm².



Figure 13 Specimen placed in the testing machine for the tensile bond strength test.

Failure mode determination

To determine the mode of fracture failure, all fracture surfaces were visually examined using a stereomicroscope (Olympus SZH10, Olympus Optical, Tokyo, Japan) at 35X magnification. Failure modes were classified as follow: adhesive failure between the denture teeth and composite resin, cohesive failure within denture teeth, cohesive failure within bonding layer, cohesive failure within composite resin, and mixed type of failure. The mixed failure mode can be subclassified as mixed primarily adhesive (the majority of fracture is at the interface), mixed primarily cohesive in the denture teeth (the majority of fracture occur in denture teeth), mixed primarily cohesive in

bonding layer (most of the fracture area is covered by bonding agent), and mixed primarily cohesive in composite resin (the majority of fracture occur in composite resin).

Statistical analysis

The data were statistically analyzed using SPSS software version 22.0 (IBM corporation, New York, NY, USA). The normal distribution of data was examined using the Kolmogorov-Smirnov test. The mean and standard deviation (SD) for the tensile bond strength of each group were calculated and statistically analyzed using three-way analysis of variance (ANOVA) and the post hoc Dunnett T3 test at the 95% confidence level to detect significant difference of tensile bonding strength according to the chemical solvent factor, composite bonding factor, and denture teeth brand factor.



CHAPTER IV

RESULT

Result

The mean tensile bond strength and standard deviation of each group (n=10) were presented in Table 3. The data were tested for normality using the Kolmogorov-Smirnov test and found that all data were normally distributed in all groups ($p>0.05$) (Table 5 in appendix).

Table 3 The mean tensile bond strength and standard deviation (MPa) of each group.

Surface treatment	Denture teeth brand	
	Yamahachi New Ace (YA)	Trubyte Bioform IPN (TB)
Control	0.45 ± 0.07 ^{A, a}	0.10 ± 0.03 ^{A, b}
MMA	0.42 ± 0.08 ^{A, a}	0.09 ± 0.02 ^{A, b}
MF-MA	0.47 ± 0.12 ^{A, a}	0.08 ± 0.01 ^{A, b}
Composite bonding (B)	7.36 ± 1.76 ^{B, a}	7.39 ± 0.54 ^{B, a}
MMA + composite bonding (MMA+B)	11.71 ± 2.36 ^{C, a}	10.00 ± 1.20 ^{C, a}
MF-MA + composite bonding (MF-MA+B)	16.28 ± 2.36 ^{D, a}	7.39 ± 0.78 ^{B, b}

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

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

Even though the homogeneity of variance is violated (Table 6 in appendix), the results of the three-way ANOVA of all groups were showed that the chemical solvent treatment, the use of a composite bonding agent, and the denture teeth type significantly affected on the tensile bond strength between acrylic denture teeth and composite resin at the 95% confidence level ($p<0.05$). There are interaction effects between the three factors including denture teeth type-the use of composite bonding, denture teeth type-chemical solvents, the use of composite bonding-chemical

solvents, and the denture teeth type-chemical solvents-the use of a composite bonding ($p < 0.05$) (Table 4).

Table 4 Three way-ANOVA analysis of denture teeth type, chemical solvents, and the use of a composite bonding agents.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	3484.034 ^a	11	316.730	228.762	.000	.959
Intercept	3175.597	1	3175.597	2293.608	.000	.955
Tooth_type	112.772	1	112.772	81.451	.000	.430
Bonding	2852.753	1	2852.753	2060.430	.000	.950
Solvent	109.499	2	54.750	39.544	.000	.423
Tooth_type * Bonding	75.129	1	75.129	54.263	.000	.334
Tooth_type * Solvent	113.326	2	56.663	40.926	.000	.431
Bonding * Solvent	109.882	2	54.941	39.682	.000	.424
Tooth_type * Bonding * Solvent	110.672	2	55.336	39.967	.000	.425
Error	149.531	108	1.385			
Total	6809.161	120				
Corrected Total	3633.564	119				

a. R Squared = .959 (Adjusted R Squared = .955)

The results demonstrated that the mean tensile bond strengths of the negative control group, MMA group, and MF-MA group were not significantly different from each other ($p > 0.05$) for both denture teeth types. The mean tensile bond strengths of the tested groups, that composite bonding agent was used as a part of surface treatment method (group IV, V, VI, X, XI, XII), were significantly higher than that of the groups that did not involve the use of composite bonding agent (group I, II, III) (Table 8 in appendix).

For Yamahachi New Ace teeth, the mean tensile bond strengths of the samples treated with MMA and bonding agent (group V) along with the samples treated with MF-MA and bonding agent (group VI) were significantly higher than that of those treated

only with a bonding agent alone (group IV) ($p < 0.001$) and the MF-MA with bonding agent group (group VI) had the significantly highest mean tensile bond strength ($p < 0.001$) (Table 8 in appendix).

For Trubyte IPN teeth, the samples that treated with a composite bonding agent alone (Group X) showed the similar mean tensile bond strengths to the sample treated with both MF-MA and a bonding agent (Group XII) ($p > 0.05$). Whereas, the tensile bond strength of the samples treated with MMA and bonding agent (Group XI) showed the significantly highest result ($p < 0.001$) (Table 8 in appendix).

The percentage of mode of failure for all groups is presented in Figure 14. The stereomicroscope images of failure surface for all groups are presented in Figure 15-16. There was no cohesive failure in this study. All specimens in negative control group, MMA treated group, and MF-MA treated group of both denture teeth types were 100% adhesive failure. The failures of specimens in Yamahachi teeth treated with bonding agent treated were predominantly mixed failure (60%) due to the majority of fracture was at the interface and some part of fracture surface was covered by bonding agent layer. The pure adhesive failure was not found in Yamahachi teeth and Trubyte IPN teeth that were treated with the combination of MMA with a bonding agent. For samples that were treated with the combination of MF-MA with a bonding agent, Yamahachi teeth showed 100% mixed failure, whereas Trubyte IPN teeth showed 10% of adhesive failure.

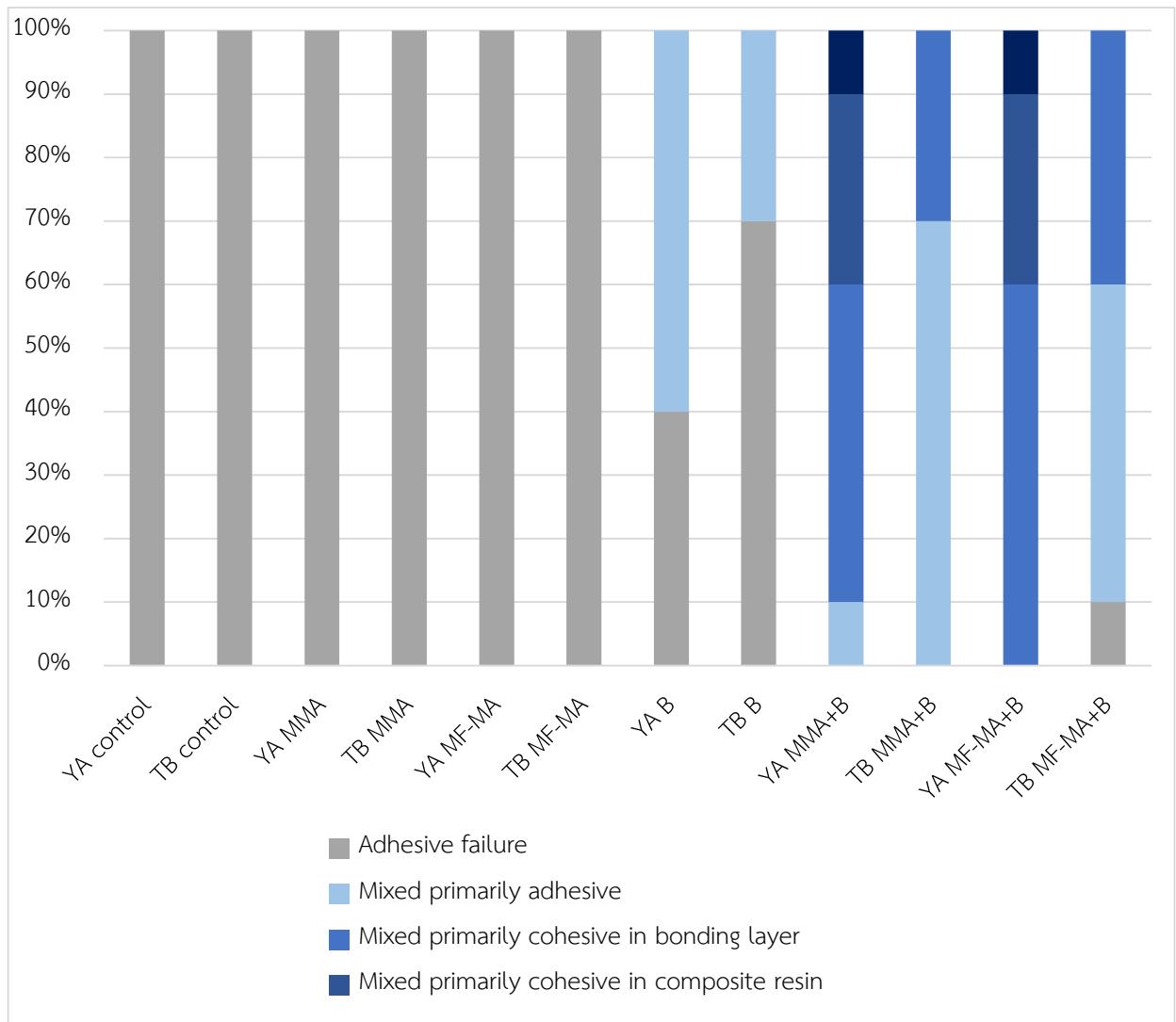


Figure 14 Failure mode in percentage (%)

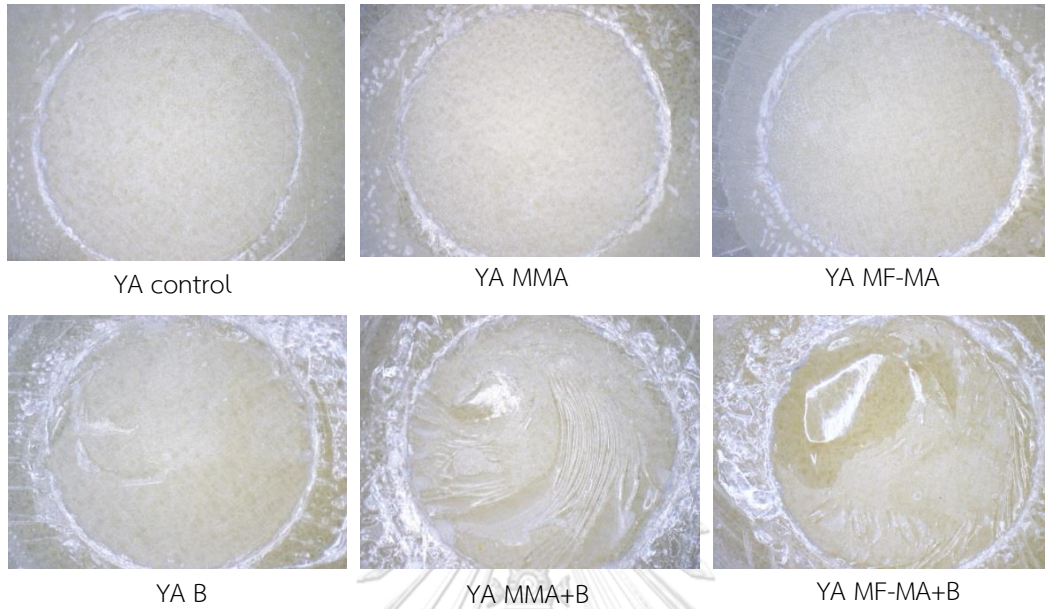


Figure 15 Failure pattern of Yamahachi New Ace specimen.

Note that the majority failure surface of YA MMA+B is covered with composite resin and the partial of denture teeth surface of YA MF-MA+B is missing.

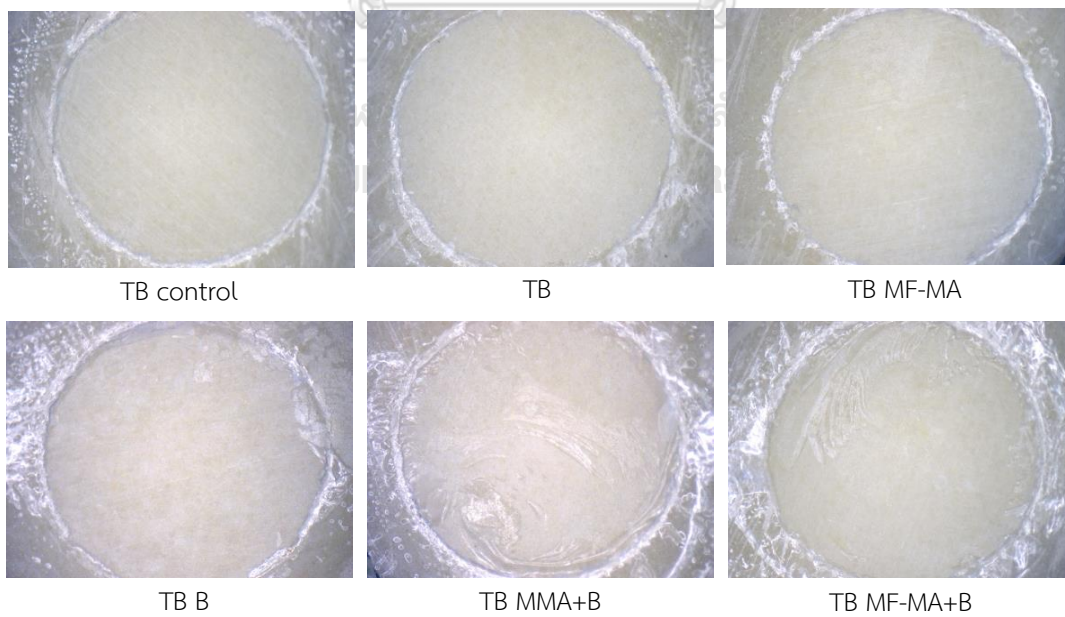


Figure 16 Failure pattern of Trubyte IPN specimen.

Scanning electron microscope (SEM) photographs of the sample treated surfaces were shown in Figure 17-18. The SEM analysis of the untreated (control) denture teeth showed a homogenous surface with irregularities from grinding (Figure 17-20A). Surface treatment of Yamahachi New Ace teeth with MMA monomer for 180s resulted in smoother and blended surface with shallow pits (Figure 17-18B) and when treated Yamahachi teeth with MF-MA for 15s resulted in small pits and holes of various diameter and depth with a honey-comb appearance (Figure 18C). Trubyte IPN teeth also showed blended and smooth surface with shallow pits when treated with MMA monomer for 180s (Figure 19-20B), while the application of 15-second MF-MA showed similar surface morphology to the untreated surface (Figure 20C, 20A). The use of a composite bonding agent created the smooth homogenous surface whether the surface treated were using a composite bonding agent alone or the combination with the use of MMA or MF-MA (Figure 17D-F, 19D-F).

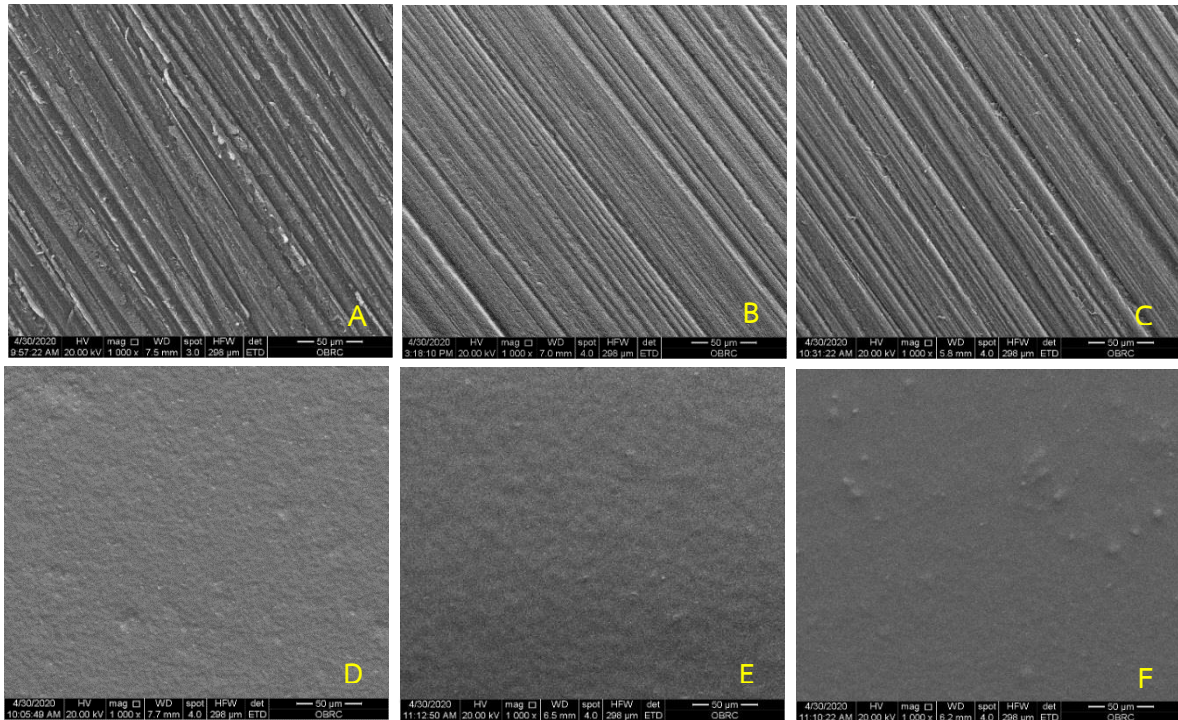


Figure 17 SEM analysis of the surface characteristics of Yamahachi New Ace acrylic denture teeth (magnification 1000X).

(A) no treatment, (B) MMA 180s, (C) MF-MA solution 15s, (D) bonding agent, (E) MMA 180s+bonding agent, (F)MF-MA 15s +bonding agent.

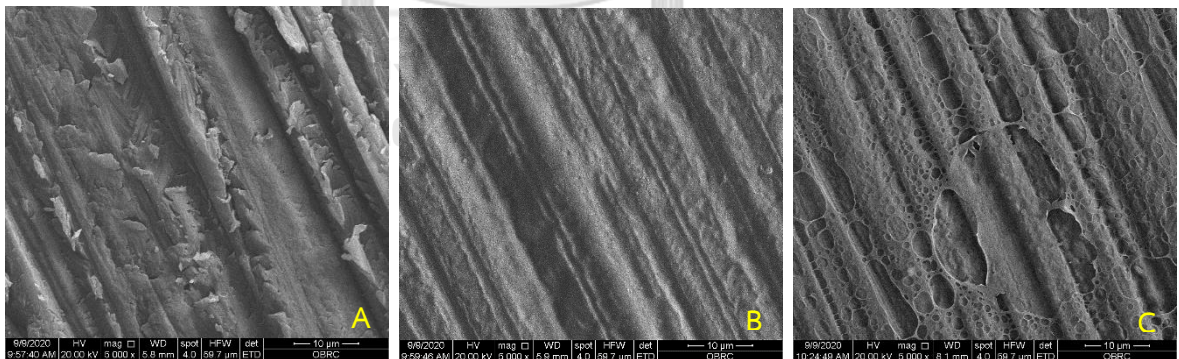


Figure 18 SEM analysis of the surface characteristics of Yamahachi New Ace acrylic denture teeth (magnification 5000X).

(A) no treatment, (B) MMA 180s, (C) MF-MA solution 15s

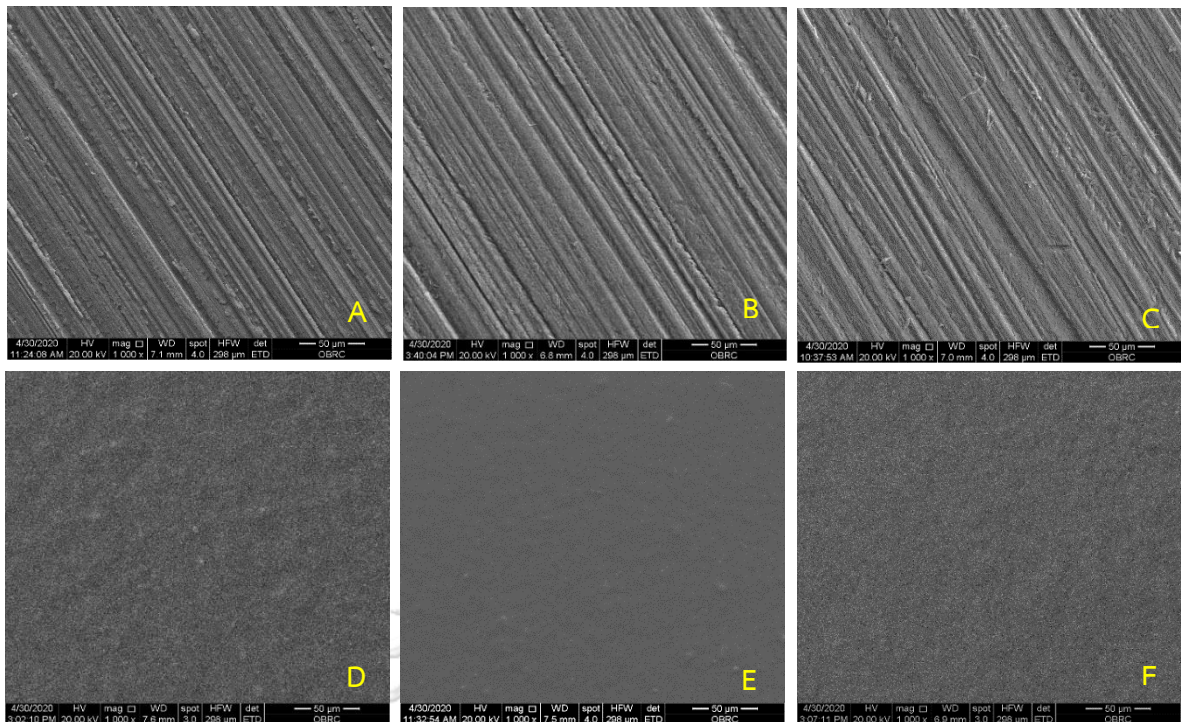


Figure 19 SEM analysis of the surface characteristics of Trubyte Bioform IPN acrylic denture teeth (magnification 1000X).

(A) no treatment, (B) MMA 180s, (C) MF-MA solution 15s, (D) bonding agent, (E) MMA 180s+bonding agent, (F) MF-MA 15s +bonding agent

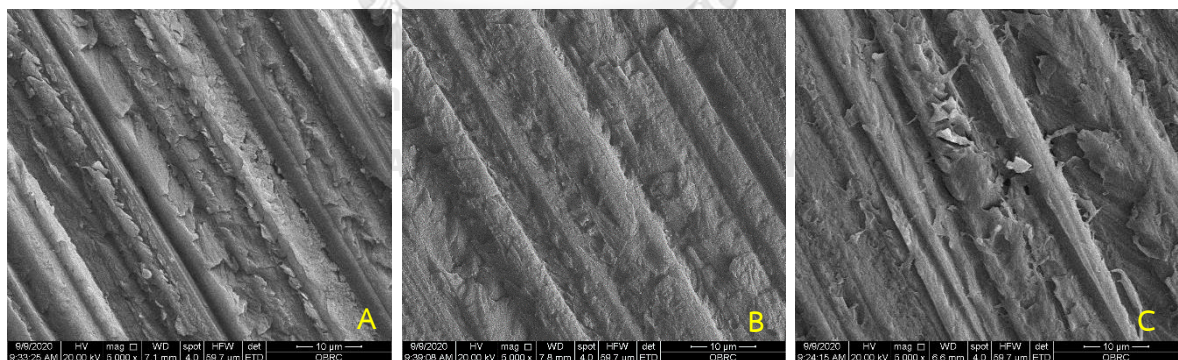


Figure 20 SEM analysis of the surface characteristics of Trubyte Bioform IPN acrylic denture teeth (magnification 5000X).

(A) no treatment, (B) MMA 180s, (C) MF-MA solution 15s

CHAPTER V

DISCUSSION AND CONCLUSION

Discussion

Based on the obtained data, the null hypotheses were rejected. Different surface treatments and different denture teeth types affected the tensile bond strength between acrylic denture teeth and composite resin. The bonding strength between two materials is dependent on their ability to create a physical, chemical, or mechanical bond at the interface [80]. A reliable strong bond is the most important factor for clinical success and longevity of the repairing of acrylic denture teeth with composite resin.

Using light-cured composite resin, as the in-office procedure for repair broken or fracture acrylic denture teeth has many advantages including less time consuming, less cost, aesthetically satisfying, and sufficiently strong. Acrylic resin and composite resin have similar reactive methacrylate molecules [81]. Therefore, the polymerization process of MMA and Bis-GMA in the composite resin matrix will follow a similar pattern of activation and cross-linking. Papazoglou and Vasilas (1999) suggested that some chemical bonding between acrylic resin and composite resin may occur if the acrylic resin teeth surfaces provide cross-links for bonding to composite. When acrylic resin denture teeth were bonded to resin composite, there is probably a combination of chemical and mechanical bonding [4]. However, the degree of conversion of acrylic denture teeth is relatively high because of the manufacturing process, that using heat curing or cross-linking methods, therefore the chemical bond may rarely occur.

In order to improve the mechanical retention of the acrylic denture teeth, surface treatment with chemical solvents such as acetone, chloroform, ethyl acetate, 4-META, methylene chloride, and MMA monomer can be used. However, methylene chloride and chloroform were identified as noxious compounds and carcinogenic potential [50, 82]. MMA monomer is the most frequently used chemical solvent to treat the acrylic denture teeth. MMA can act as an effective adhesion promoter due

to the similarity of chemical structure to the denture base material. Chemical solvents can enhance the bond strength of acrylic denture teeth through the swelling phenomenon which is the dissolution and swelling of the surface layer when the solvents come in contact and diffuse into the polymer [42]. Consequently, the morphology of denture teeth surfaces is altered, and the monomer of the denture base materials or auto-polymerized acrylic resin can penetrate the micro irregularities which result in micromechanical retention. However, the surface treatment with MMA requires 180 seconds of wetting time to effectively prime the surface and ultimately reducing adhesive failures [9].

To replace the use of methylene chloride and chloroform, Asmussen and Peutzfeldt (2000) found that the low molecular weight methyl esters including MF and MA were as effectively softening the polymethyl methacrylate denture base as methylene chloride but less toxic [13]. Polymer dissolving and swelling occur when the solubility parameters and polarities of the polymer and solvent are close to each other. The solubility parameter of acrylic denture teeth material (PMMA) is $18.3 \text{ MPa}^{1/2}$, whereas MMA, MF, and MA have solubility parameters approximately 18.0, 20.9, and $19.6 \text{ MPa}^{1/2}$, respectively [45]. Many studies had proved that using MF-MA mixture solution as chemical surface treatment for 15 seconds improved the bond strength of relined denture base resin [15, 16, 18], the tensile bond strength between acrylic denture teeth and denture base resin [17, 61], and the tensile bond strength between denture base resin and soft lining material [20].

In this study, the tensile bond strengths of MMA-treated (YA MMA, TB MMA) and MF-MA-treated group (YA MF-MA, TB MF-MA) were not significantly different the control group (no treatment) ($p < 0.05$). Furthermore, the modes of failure of control, MMA treated, and MF-MA treated groups were 100% completely adhesive failure, these results indicated that using MMA or MF-MA alone to swell the acrylic denture teeth surface did not provide sufficient micromechanical retention when repaired with resin composites. These findings were corresponding to the previous studies [6, 10, 11]. The results in these groups could be influenced by the poor wettability property of the

high viscosity resin composite, so the material cannot penetrate into the microstructure occurring from the swelling phenomenon.

The composite bonding agent is essential to enhance the bond strength of resin composite restoration in operative dentistry. One of the important properties of bonding agents is its low viscosity due to the presence of solvents and diluted monomers. Using a bonding agent to treat acrylic denture teeth before repair with resin composite could improve the surface wettability and promote the penetration and infiltration of the bonding agent itself which enable the adhesion to composite resin. According to the result, the groups that treated with Adper Single Bond 2 composite bonding agent (YA B, TB B) showed significantly higher tensile bond strength than the non-using bonding agent groups (YA control, YA MMA, YA MF-MA, TB control, TB MMA, TB MF-MA) and the failure modes of Yamahachi and Trubyte IPN groups that treated with a composite bonding agent were found as the 60% and 40% of mixed primarily adhesive. These results were concurrent with the previous studies that the bonding agent significantly improved the shear bond strength between acrylic denture teeth and composite resin [5, 79, 83, 84]. Adper Single Bond 2 contains ethyl alcohol that can swell and dissolve the denture teeth surface then evaporate which resulted in the surface microstructure allowing other components containing carbon-carbon double bonds (C=C) molecules penetrate deeper and polymerized to the resin composite monomer. Therefore, using Adper Single bond 2 not only improve the surface wettability but may also promote the micromechanical retention. Thus, a composite bonding agent should be used as part of surface treatment when repair acrylic denture teeth with resin composite.

Several studies found that using the composite bonding agent with prior treatment of MMA for 180 seconds increased the shear bond strength between acrylic denture teeth and resin composite [4, 6, 10-12]. In this study, Yamahachi teeth that using the combination of chemical solvent (MMA, MF-MA) and bonding agent (YA MMA+B, YA MF-MA+B), as well as Trubyte IPN teeth that were treated with MMA and

a bonding agent (TB MMA+B), had significantly higher tensile bond strengths than those using the bonding agent alone (YA B, TB B) ($p < 0.05$). Whereas, using both MF-MA solution and a composite bonding agent to treat Yamahachi teeth resulted in the highest tensile bond strength. Mode of failure of these groups was also better the mode of failure of the group that using a composite bonding agent alone since 100% mixed failure were observed for both 2-step surface treatment of Yamahachi teeth (YA MMA+B, YA MF-MA+B) and Trubyte IPN teeth with MMA and a bonding agent (TB MMA+B). Whereas Trubyte IPN teeth treated with MF-MA and a bonding agent (TB MF-MA+B) were found with 90% mixed failure. These results could be explained by the swelling phenomenon and the improvement of surface wettability. MMA and MF-MA dissolved and swelled acrylic denture teeth surface leading to micro irregularity, so when bonding agent was applied, resin composite can be infiltrated into the microstructure and then polymerized to form the micromechanical retention. Since the solubility parameters of MMA, MF, and MA are closer to the solubility parameter of PMMA than the solubility parameter of ethyl alcohol ($26.0 \text{ MPa}^{1/2}$) [45], therefore MMA and MF-MA can dissolve the denture teeth surface better than Adper single bond 2.

The scanning electron microscope (SEM) examination of the MMA and MF-MA treated Yamahachi denture surface revealed differences in their morphological patterns. Treating Yamahachi teeth with MMA monomer resulted in swelled and blurred surface with shallow pits whereas MF-MA treatment showed 3D pores of various diameter and depth with a honey-comb appearance, hence the surface treatment with MF-MA would allow better micromechanical retention. The results of tensile bond strength and SEM examination showed the superiority of using MF-MA solution and composite bonding agent as a surface treatment method for bonding conventional acrylic denture teeth to composite resin over the use of MMA and a composite bonding agent. The benefits of using MF-MA solution as part of the surface treatment method are not only less application time but also less tissue irritation and

less toxic [54, 55, 59, 60]. For Trubyte IPN teeth, surface treatment with MMA for 180s also showed swelled and blurred surface with shallow pits. However, 15-second treatment of MF-MA showed similar surface morphology when compared to the control teeth. This results of SEM examination and tensile bond strength test suggested that MF-MA treatment may require longer wetting time than 15s.

Shen et al. (1984) indicated that the chemical softening effect of the polymer was depended on the degree of cross-linking [52]. Highly cross-linked denture teeth (such as IPN teeth), which contain two or more interlaced polymer networks, show poor adhesive bonding to the denture base material [27, 43, 44]. According to the master thesis of Thongrakard (2015), the tensile bond strength of cross-linked denture teeth increased when increasing the wetting time. They also stated that IPN teeth required a 30-second MF-MA treatment [85]. In this study, Trubyte IPN teeth of the control group (TB control), the MMA treated group (TB MMA), the MF-MA treated group (TB MF-MA), and the 2-step application of MF-MA and composite bonding agent group (TB MF-MA+B) showed significantly lower tensile bond strength when compared to Yamahachi teeth with the same surface treatment method ($p < 0.05$). Because of the high complexity and density of cross-linking in the interpenetrating polymer networks, the spaces between the polymer chain networks in the cross-linked acrylic denture teeth are smaller and lesser than the conventional type [17, 86]. Thus, monomers from the bonding agent and composite resin can penetrate more into the conventional acrylic denture teeth matrix which resulting in higher bond strength. Moreover, the high degree of cross-linking in IPN denture teeth can lead to less availability of unlinked polymer chains for the development of an interwoven polymer between denture teeth and the bonding agent and/or composite resin [44, 87].

When using a composite bonding agent to treat the acrylic denture teeth surfaces (YA B, TB B), the tensile bond strength was increased and was no significant difference among denture teeth types ($p > 0.05$). The bonding agent consists of a solvent and diluted monomer which can dissolve the polymer matrix and provide the carbon-carbon double bonds (C=C) molecules to polymerized with the composite resin, therefore the polymer structure of acrylic denture teeth does not affect the

infiltration of a bonding agent and, consequently, does not affect the bond strength of acrylic denture teeth and composite resin.

In addition, treating denture teeth surface with a 2-step application of MMA and a composite bonding agent showed no statistically significant difference of tensile bond strength between conventional and IPN denture teeth ($p>0.05$). This result could be due to the characteristic of MMA which can act as both chemical solvent to swell the teeth surface and monomer for polymerization with the monomers of a bonding agent and result in increased bond strength. On the other hand, MF-MA has a low boiling point and does not contain carbon-carbon double bonds (C=C) in the molecules, therefore it will dissolve the acrylic denture teeth surface and then evaporate leaving only the microstructure. If the monomer of a bonding agent cannot penetrate into the deepest cavity, the bond strength will be decreased. As well as, the difficulty of chemical solvents diffusion into the complex cross-linked polymer surface of interpenetrating network denture teeth which result in decreased bond strength. Therefore, IPN teeth may require more application time of MF-MA solution than the other denture teeth types.

The present study is an in vitro study that may be limited in predicting the success of a technique in clinical use. Several factors in the oral cavity may influence the bond degradation such as saliva which contains numerous molecules with a high affinity for adsorption to the denture surface and may interfere in the bonded area. Further study should investigate on the effect of complex denture-oral environment conditions, the effect of long periods of water storage, the effect of thermocycling, and the effect of longer wetting time of MF-MA surface treatment such as 30s and 60s on IPN acrylic teeth. Although this experimental method does not imitate the intraoral condition, it does provide an effective means comparing the influence of different surface treatments on the tensile bond strength between acrylic denture teeth and composite resin.

Conclusion

Within the limitation of the present study, the following conclusions can be drawn:

1. Using MMA or MF-MA mixture solution alone cannot improve the tensile bond strength between the acrylic denture teeth and composite resin when compared to the negative control group.
2. The application of the bonding agent significantly influences on the tensile bond strength between the acrylic denture teeth and resin composite.
3. The use of MMA monomer for 180s followed by the composite bonding agent significantly increases the tensile bond strength between acrylic denture teeth and composite resin.
4. IPN cross-linked denture teeth possess lower tensile bond strength to composite resin than conventional acrylic denture teeth except when treating the surface with a composite bonding agent alone or with the 2-step application of MMA and a composite bond agent.
5. The application of the bonding agent with prior treatment of MF-MA mixture solution (25:75 %v/v) for 15 seconds can be an alternative surface treatment for repairing and bonding the conventional acrylic denture teeth with composite resin.

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APPENDIX

จุฬาลงกรณ์มหาวิทยาลัย
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Table 5 Analysis of the data distribution.

		Tests of Normality					
		Kolmogorov-Smirnov ^a			Shapiro-Wilk		
	Group	Statistic	df	Sig.	Statistic	df	Sig.
Tensile bond strength	Yamahachi control	.214	10	.200*	.907	10	.261
	Yamahachi MMA	.153	10	.200*	.953	10	.705
	Yamahachi MF-MA	.234	10	.127	.882	10	.136
	Yamahachi Bonding agent	.166	10	.200*	.886	10	.151
	Yamahachi MMA + Bonding	.230	10	.142	.866	10	.089
	Yamahachi MF-MA + Bonding	.209	10	.200*	.938	10	.529
	Trubyte control	.185	10	.200*	.958	10	.760
	Trubyte MMA	.200	10	.200*	.961	10	.795
	Trubyte MF-MA	.228	10	.152	.867	10	.092
	Trubyte Bonding agent	.130	10	.200*	.947	10	.633
	Trubyte MMA + Bonding	.164	10	.200*	.894	10	.190
	Trubyte MF-MA + Bonding	.179	10	.200*	.926	10	.412

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

a. Lilliefors Significance Correction

Table 6 The Levene's Test of Equality of Error.

Dependent Variable: Tensile bond strength

F	df1	df2	Sig.
9.326	11	108	.000

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

a. Design: Intercept + Tooth_type + Bonding + Solvent + Tooth_type

* Bonding + Tooth_type * Solvent + Bonding * Solvent + Tooth_type

* Bonding * Solvent

Table 7 Descriptive Statistics of all data.

Descriptives

Tensile bond strength

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Min	Max
					Lower Bound	Upper Bound		
					Yamahachi control	10		
Yamahachi MMA	10	.42100	.078944	.024964	.36453	.47747	.310	.540
Yamahachi MF-MA	10	.47200	.122184	.038638	.38459	.55941	.330	.680
Yamahachi Bonding	10	7.35800	1.764425	.557960	6.09581	8.62019	5.610	10.440
Yamahachi MMA + Bonding	10	11.70600	2.356519	.745197	10.02025	13.39175	9.180	16.070
Yamahachi MF-MA + Bonding	10	16.27800	2.361580	.746797	14.58863	17.96737	12.270	19.590
Trubyte control	10	.10000	.034319	.010853	.07545	.12455	.050	.170
Trubyte MMA	10	.09000	.017638	.005578	.07738	.10262	.060	.120
Trubyte MF-MA	10	.08100	.014491	.004583	.07063	.09137	.060	.100
Trubyte Bonding	10	7.39300	.537836	.170079	7.00826	7.77774	6.670	8.540
Trubyte MMA + Bonding	10	10.00000	1.200213	.379541	9.14142	10.85858	8.230	11.390
Trubyte MF-MA + Bonding	10	7.38500	.783670	.247818	6.82440	7.94560	6.330	8.500
Total	120	5.14425	5.525772	.504432	4.14542	6.14308	.050	19.590

Table 8 One-way ANOVA analysis and Post Hoc Tests of all groups

Test of Homogeneity of Variances

Tensile bond strength

Levene Statistic	df1	df2	Sig.
9.326	11	108	.000

ANOVA

Tensile bond strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3484.034	11	316.730	228.762	.000
Within Groups	149.531	108	1.385		
Total	3633.564	119			



Multiple Comparisons

Dependent Variable: Tensile bond strength

	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Dunnett T3	Yamahachi control	Yamahachi MMA	.026000	.032438	1.000	-.10204	.15404
		Yamahachi MF-MA	-.025000	.043839	1.000	-.20511	.15511
		Yamahachi Bonding	-6.911000*	.558344	.000	-9.44091	-4.38109
		Yamahachi MMA + Bonding	-11.259000*	.745485	.000	-14.63799	-7.88001
		Yamahachi MF-MA + Bonding	-15.831000*	.747084	.000	-19.21725	-12.44475
		Trubyte control	.347000*	.023383	.000	.25069	.44331

	Trubyte MMA	.357000*	.021450	.000	.26312	.45088
	Trubyte MF-MA	.366000*	.021213	.000	.27218	.45982
	Trubyte Bonding	-6.946000*	.171335	.000	-7.71669	-6.17531
	Trubyte MMA + Bonding	-9.553000*	.380105	.000	-11.27376	-7.83224
	Trubyte MF-MA + Bonding	-6.938000*	.248682	.000	-8.06133	-5.81467
Yamahachi	Yamahachi control	-.026000	.032438	1.000	-.15404	.10204
MMA	Yamahachi MF-MA	-.051000	.046001	1.000	-.23623	.13423
	Yamahachi Bonding	-6.937000*	.558518	.000	-9.46682	-4.40718
	Yamahachi MMA + Bonding	-11.285000*	.745615	.000	-14.66392	-7.90608
	Yamahachi MF-MA + Bonding	-15.857000*	.747214	.000	-19.24318	-12.47082
	Trubyte control	.321000*	.027221	.000	.20657	.43543
	Trubyte MMA	.331000*	.025580	.000	.21792	.44408
	Trubyte MF-MA	.340000*	.025382	.000	.22692	.45308
	Trubyte Bonding	-6.972000*	.171901	.000	-7.74253	-6.20147
	Trubyte MMA + Bonding	-9.579000*	.380361	.000	-11.29964	-7.85836
	Trubyte MF-MA + Bonding	-6.964000*	.249073	.000	-8.08717	-5.84083
Yamahachi	Yamahachi control	.025000	.043839	1.000	-.15511	.20511
MF-MA	Yamahachi MMA	.051000	.046001	1.000	-.13423	.23623
	Yamahachi Bonding	-6.886000*	.559296	.000	-9.41544	-4.35656
	Yamahachi MMA + Bonding	-11.234000*	.746198	.000	-14.61263	-7.85537
	Yamahachi MF-MA + Bonding	-15.806000*	.747796	.000	-19.19189	-12.42011
	Trubyte control	.372000*	.040133	.000	.19682	.54718
	Trubyte MMA	.382000*	.039038	.000	.20695	.55705
	Trubyte MF-MA	.391000*	.038909	.000	.21591	.56609

	Trubyte Bonding	-6.921000*	.174412	.000	-7.69143	-6.15057
	Trubyte MMA + Bonding	-9.528000*	.381502	.000	-11.24815	-7.80785
	Trubyte MF-MA + Bonding	-6.913000*	.250812	.000	-8.03565	-5.79035
Yamahachi	Yamahachi control	6.911000*	.558344	.000	4.38109	9.44091
Bonding	Yamahachi MMA	6.937000*	.558518	.000	4.40718	9.46682
	Yamahachi MF-MA	6.886000*	.559296	.000	4.35656	9.41544
	Yamahachi MMA + Bonding	-4.348000*	.930934	.013	-8.04740	-.64860
	Yamahachi MF-MA + Bonding	-8.920000*	.932216	.000	-12.62510	-5.21490
	Trubyte control	7.258000*	.558066	.000	4.72795	9.78805
	Trubyte MMA	7.268000*	.557988	.000	4.73791	9.79809
	Trubyte MF-MA	7.277000*	.557979	.000	4.74690	9.80710
	Trubyte Bonding	-.035000	.583306	1.000	-2.56664	2.49664
	Trubyte MMA + Bonding	-2.642000	.674812	.059	-5.34570	.06170
	Trubyte MF-MA + Bonding	-.027000	.610519	1.000	-2.58766	2.53366
Yamahachi	Yamahachi control	11.259000*	.745485	.000	7.88001	14.63799
MMA +	Yamahachi MMA	11.285000*	.745615	.000	7.90608	14.66392
Bonding	Yamahachi MF-MA	11.234000*	.746198	.000	7.85537	14.61263
	Yamahachi Bonding	4.348000*	.930934	.013	.64860	8.04740
	Yamahachi MF-MA + Bonding	-4.572000*	1.055000	.022	-8.71544	-.42856
	Trubyte control	11.606000*	.745276	.000	8.22690	14.98510
	Trubyte MMA	11.616000*	.745218	.000	8.23687	14.99513
	Trubyte MF-MA	11.625000*	.745211	.000	8.24586	15.00414
	Trubyte Bonding	4.313000*	.764359	.009	.93737	7.68863
	Trubyte MMA + Bonding	1.706000	.836283	.860	-1.74942	5.16142

	Trubyte MF-MA + Bonding	4.321000*	.785323	.009	.93551	7.70649
Yamahachi	Yamahachi control	15.831000*	.747084	.000	12.44475	19.21725
MF-MA + Bonding	Yamahachi MMA	15.857000*	.747214	.000	12.47082	19.24318
	Yamahachi MF-MA	15.806000*	.747796	.000	12.42011	19.19189
	Yamahachi Bonding	8.920000*	.932216	.000	5.21490	12.62510
	Yamahachi MMA + Bonding	4.572000*	1.055000	.022	.42856	8.71544
	Trubyte control	16.178000*	.746876	.000	12.79164	19.56436
	Trubyte MMA	16.188000*	.746818	.000	12.80161	19.57439
	Trubyte MF-MA	16.197000*	.746811	.000	12.81060	19.58340
	Trubyte Bonding	8.885000*	.765920	.000	5.50213	12.26787
	Trubyte MMA + Bonding	6.278000*	.837710	.000	2.81586	9.74014
	Trubyte MF-MA + Bonding	8.893000*	.786842	.000	5.50036	12.28564
Trubyte control	Yamahachi control	-.347000*	.023383	.000	-.44331	-.25069
	Yamahachi MMA	-.321000*	.027221	.000	-.43543	-.20657
	Yamahachi MF-MA	-.372000*	.040133	.000	-.54718	-.19682
	Yamahachi Bonding	-7.258000*	.558066	.000	-9.78805	-4.72795
	Yamahachi MMA + Bonding	-11.606000*	.745276	.000	-14.98510	-8.22690
	Yamahachi MF-MA + Bonding	-16.178000*	.746876	.000	-19.56436	-12.79164
	Trubyte MMA	.010000	.012202	1.000	-.04037	.06037
	Trubyte MF-MA	.019000	.011780	.981	-.03067	.06867
	Trubyte Bonding	-7.293000*	.170425	.000	-8.06406	-6.52194
	Trubyte MMA + Bonding	-9.900000*	.379696	.000	-11.62097	-8.17903
	Trubyte MF-MA + Bonding	-7.285000*	.248056	.000	-8.40862	-6.16138
	Yamahachi control	-.357000*	.021450	.000	-.45088	-.26312

Trubyte	Yamahachi MMA	-.331000*	.025580	.000	-.44408	-.21792
MMA	Yamahachi MF-MA	-.382000*	.039038	.000	-.55705	-.20695
	Yamahachi Bonding	-7.268000*	.557988	.000	-9.79809	-4.73791
	Yamahachi MMA + Bonding	-11.616000*	.745218	.000	-14.99513	-8.23687
	Yamahachi MF-MA + Bonding	-16.188000*	.746818	.000	-19.57439	-12.80161
	Trubyte control	-.010000	.012202	1.000	-.06037	.04037
	Trubyte MF-MA	.009000	.007219	1.000	-.01951	.03751
	Trubyte Bonding	-7.303000*	.170170	.000	-8.07419	-6.53181
	Trubyte MMA + Bonding	-9.910000*	.379582	.000	-11.63103	-8.18897
	Trubyte MF-MA + Bonding	-7.295000*	.247881	.000	-8.41872	-6.17128
Trubyte MF- MA	Yamahachi control	-.366000*	.021213	.000	-.45982	-.27218
	Yamahachi MMA	-.340000*	.025382	.000	-.45308	-.22692
	Yamahachi MF-MA	-.391000*	.038909	.000	-.56609	-.21591
	Yamahachi Bonding	-7.277000*	.557979	.000	-9.80710	-4.74690
	Yamahachi MMA + Bonding	-11.625000*	.745211	.000	-15.00414	-8.24586
	Yamahachi MF-MA + Bonding	-16.197000*	.746811	.000	-19.58340	-12.81060
	Trubyte control	-.019000	.011780	.981	-.06867	.03067
	Trubyte MMA	-.009000	.007219	1.000	-.03751	.01951
	Trubyte Bonding	-7.312000*	.170140	.000	-8.08320	-6.54080
	Trubyte MMA + Bonding	-9.919000*	.379568	.000	-11.64004	-8.19796
	Trubyte MF-MA + Bonding	-7.304000*	.247861	.000	-8.42773	-6.18027
Trubyte Bonding	Yamahachi control	6.946000*	.171335	.000	6.17531	7.71669
	Yamahachi MMA	6.972000*	.171901	.000	6.20147	7.74253
	Yamahachi MF-MA	6.921000*	.174412	.000	6.15057	7.69143

	Yamahachi Bonding	.035000	.583306	1.000	-2.49664	2.56664
	Yamahachi MMA + Bonding	-4.313000*	.764359	.009	-7.68863	-.93737
	Yamahachi MF-MA + Bonding	-8.885000*	.765920	.000	-12.26787	-5.50213
	Trubyte control	7.293000*	.170425	.000	6.52194	8.06406
	Trubyte MMA	7.303000*	.170170	.000	6.53181	8.07419
	Trubyte MF-MA	7.312000*	.170140	.000	6.54080	8.08320
	Trubyte MMA + Bonding	-2.607000*	.415906	.002	-4.34976	-.86424
	Trubyte MF-MA + Bonding	.008000	.300567	1.000	-1.19526	1.21126
Trubyte	Yamahachi control	9.553000*	.380105	.000	7.83224	11.27376
MMA +	Yamahachi MMA	9.579000*	.380361	.000	7.85836	11.29964
Bonding	Yamahachi MF-MA	9.528000*	.381502	.000	7.80785	11.24815
	Yamahachi Bonding	2.642000	.674812	.059	-.06170	5.34570
	Yamahachi MMA + Bonding	-1.706000	.836283	.860	-5.16142	1.74942
	Yamahachi MF-MA + Bonding	-6.278000*	.837710	.000	-9.74014	-2.81586
	Trubyte control	9.900000*	.379696	.000	8.17903	11.62097
	Trubyte MMA	9.910000*	.379582	.000	8.18897	11.63103
	Trubyte MF-MA	9.919000*	.379568	.000	8.19796	11.64004
	Trubyte Bonding	2.607000*	.415906	.002	.86424	4.34976
	Trubyte MF-MA + Bonding	2.615000*	.453282	.002	.79170	4.43830
Trubyte MF-	Yamahachi control	6.938000*	.248682	.000	5.81467	8.06133
MA +	Yamahachi MMA	6.964000*	.249073	.000	5.84083	8.08717
Bonding	Yamahachi MF-MA	6.913000*	.250812	.000	5.79035	8.03565
	Yamahachi Bonding	.027000	.610519	1.000	-2.53366	2.58766
	Yamahachi MMA + Bonding	-4.321000*	.785323	.009	-7.70649	-.93551

Yamahachi MF-MA + Bonding	-8.893000*	.786842	.000	-12.28564	-5.50036
Trubyte control	7.285000*	.248056	.000	6.16138	8.40862
Trubyte MMA	7.295000*	.247881	.000	6.17128	8.41872
Trubyte MF-MA	7.304000*	.247861	.000	6.18027	8.42773
Trubyte Bonding	-.008000	.300567	1.000	-1.21126	1.19526
Trubyte MMA + Bonding	-2.615000*	.453282	.002	-4.43830	-.79170

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



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