

การลดปริมาณมอนอเมอร์ที่หลงเหลือในวัสดุแผ่นฐานจัดฟันที่มีส่วนประกอบพื้นฐาน
เป็นเมทิลเมทาคริเลตโดยการแช่น้ำในอ่างอัลตราโซนิก



นางสาวปจิตมา ไทยธรรมยานนท์

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สาขาวิชาทันตกรรมจัดฟัน ภาควิชาทันตกรรมจัดฟัน

คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

RESIDUAL MONOMER REDUCTION IN THE MMA-BASED ORTHODONTIC BASE-
PLATE MATERIALS BY WATER IMMERSION IN ULTRASONIC BATH

Miss Pajima Thaitammanon



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

Department of Orthodontics

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ปจิมมา ไทยธรรมยานนท์ : การลดปริมาณมอนอเมอร์ที่หลงเหลือในวัสดุแผ่นฐานจัดฟันที่มี ส่วนประกอบพื้นฐานเป็นเมทิลเมทาคริเลตโดยการแช่น้ำในอ่างอัลตราโซนิก (RESIDUAL MONOMER REDUCTION IN THE MMA-BASED ORTHODONTIC BASE-PLATE MATERIALS BY WATER IMMERSION IN ULTRASONIC BATH) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ทญ. จินตนา ศิริชุมพันธ์, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: รศ. ชัยรัตน์ วิวัฒน์วรพันธ์, 82 หน้า.

วัตถุประสงค์: เพื่อเปรียบเทียบปริมาณมอนอเมอร์ที่ตกค้างระหว่างวัสดุอคริลิกกับอโรพลาสท์ และเพื่อเปรียบเทียบปริมาณมอนอเมอร์ที่ตกค้างในวัสดุแผ่นฐานจัดฟันชนิดบ่มด้วยตัวเอง ระหว่างวิธีการลด ปริมาณมอนอเมอร์ที่แตกต่างกัน

วัสดุและวิธีการ: เตรียมชิ้นงานรูปแผ่นกลมขนาด 3x50 มิลลิเมตร จำนวน 96 ชิ้นงาน โดยใช้วัสดุ ออโรคริล (เดนทาลูม, ประเทศเยอรมัน) และอโรพลาสท์ (เวอเทคซ์, ประเทศเนเธอร์แลนด์) ทำตามคำแนะนำ ของบริษัทผู้ผลิต และตามขั้นตอนไอเอสโอ 20795-2 (2013) แบ่งชิ้นงานแต่ละยี่ห้อออกเป็นแปดกลุ่ม (กลุ่มละ 6 ชิ้นงาน) กลุ่มที่ I ไม่ผ่านกระบวนการใด เป็นกลุ่มควบคุม กลุ่มที่ II และ III เป็นกลุ่มแช่น้ำอุณหภูมิห้อง (25 องศา เซลเซียส) เป็นเวลา 24 และ 72 ชั่วโมง ตามลำดับ กลุ่มที่ IV-VIII เป็นกลุ่มที่แช่น้ำอุณหภูมิ 50 องศาเซลเซียสใน อ่างอัลตราโซนิกเป็นเวลา 3, 5, 10, 15 และ 20 นาที ตามลำดับ วัดปริมาณมอนอเมอร์ที่ตกค้างโดยใช้เครื่องโคร มาโทกราฟีของเหลวสมรรถนะสูง วิเคราะห์ข้อมูลโดยใช้การวิเคราะห์ความแปรปรวนแบบสองทาง ตามด้วย การวิเคราะห์ความแปรปรวนแบบทางเดียวและการทดสอบของทูกีเอชเอสดีที่ระดับนัยสำคัญ 0.05

ผลการทดลอง: ในกลุ่ม II-VIII กลุ่มออโรคริล มีปริมาณมอนอเมอร์ที่ตกค้างน้อยกว่ากลุ่ม อโรพลาสท์อย่างมีนัยสำคัญทางสถิติ ในกลุ่มออโรคริล ปริมาณมอนอเมอร์ที่ตกค้างในกลุ่ม II-VIII น้อยกว่า กลุ่ม I อย่างมีนัยสำคัญทางสถิติ ขณะที่กลุ่ม VIII น้อยกว่ากลุ่ม II,III อย่างมีนัยสำคัญทางสถิติในกลุ่ม อโรพลาสท์ ปริมาณมอนอเมอร์ที่ตกค้างในกลุ่ม I และ II ไม่มีความแตกต่างอย่างมีนัยสำคัญทางสถิติ แต่ใน กลุ่ม III มีปริมาณมอนอเมอร์ที่ตกค้างน้อยกว่ากลุ่ม I อย่างมีนัยสำคัญ ปริมาณมอนอเมอร์ที่ตกค้างในกลุ่ม VI, VII และ VIII ไม่มีความแตกต่างอย่างมีนัยสำคัญทางสถิติ แต่ในกลุ่ม VIII มีปริมาณมอนอเมอร์ที่ตกค้างน้อยกว่า กลุ่ม VI อย่างมีนัยสำคัญ

สรุปผลการทดลอง: ในวิธีการลดมอนอเมอร์วิธีเดียวกัน ปริมาณมอนอเมอร์ที่ตกค้างในวัสดุอคริลิก ต่ำกว่าวัสดุอโรพลาสท์ การแช่น้ำอุณหภูมิ 50 องศาเซลเซียสในอ่างอัลตราโซนิก เป็นเวลา 10-20 นาที ลด ปริมาณมอนอเมอร์ที่ตกค้างในวัสดุแผ่นฐานอะคริลิกจัดฟันได้ใกล้เคียงหรือดีกว่าการแช่น้ำที่อุณหภูมิห้องเป็น เวลา 24 และ 72 ชั่วโมง อย่างไรก็ตามใช้เวลาน้อยกว่า

ภาควิชา ทันตกรรมจัดฟัน

ลายมือชื่อผู้ผลิต

สาขาวิชา ทันตกรรมจัดฟัน

ลายมือชื่อ อ.ที่ปรึกษาหลัก

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ลายมือชื่อ อ.ที่ปรึกษาร่วม

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PAJIMA THAITAMMAYANON: RESIDUAL MONOMER REDUCTION IN THE MMA-BASED
ORTHODONTIC BASE-PLATE MATERIALS BY WATER IMMERSION IN ULTRASONIC BATH.
ADVISOR: ASSOC. PROF. CHINTANA SIRICHOMPUN, CO-ADVISOR: ASSOC. PROF. CHAIRAT
WIWATWARRAPAN, 82 pp.

Objective: To compare the levels of residual monomer between Orthocryl and Orthoplast; and to compare the levels of residual monomer in self-cured orthodontic base-plate materials among the different reduction methods.

Materials and methods: A total of 96 disc specimens (3x50 mm) were prepared from Orthocryl[®] (Dentaurum, Germany) and Orthoplast[®] (Vertex, The Netherlands), according to the instructions of the manufacturers and ISO 20795-2 (2013). The specimens from each brand were divided into eight groups (6 specimens per group). Group I were left untreated as controls. Groups II and III were immersed in the-room-temperature (25°C) water for 24 and 72 hours, respectively. Groups IV-VIII were immersed in 50°C water of an ultrasonic bath for 3, 5, 10, 15 and 20 minutes, respectively. The level of residual monomer was determined by using high performance liquid chromatography. Data were analyzed by a two-way ANOVA, followed by a one-way ANOVA Tukey's HSD *post hoc* test at 0.05 significant level.

Results: In group II-VIII, the Orthocryl groups showed a statistically significantly lower residual monomer level than the Orthoplast groups. In the Orthocryl groups, the levels in group II-VIII were significantly lower than group I, while the level in group VIII was significantly lower than those in groups II, III. In the Orthoplast groups, no significant differences existed between group I and II, but the level in group III was significantly lower than that in groups I. The levels in group VI, VII and VIII were not significantly differences with those in groups III, but the level in group VIII was significantly lower than that in groups VI.

Conclusion: In the same monomer reduction method, the levels of residual monomer in Orthocryl were lower than those in Orthoplast. Water immersion at 50°C in an ultrasonic bath for 10-20 minutes reduced the amount of residual monomer in an orthodontic acrylic base-plate material, which was similar to or better than water immersion for 24 and 72 hours at room temperature. However, less time was required.

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Student's Signature

Advisor's Signature

Co-Advisor's Signature

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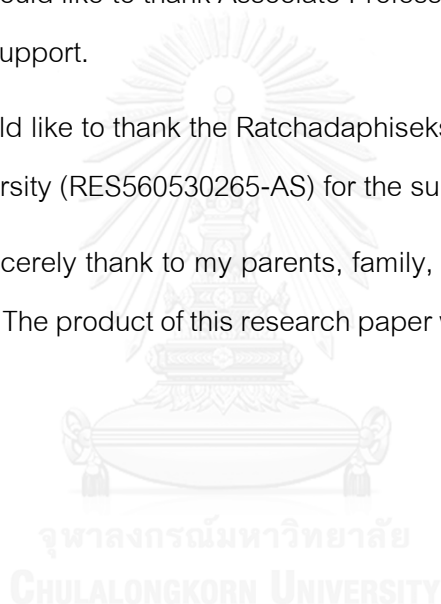
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CHAPTER I

INTRODUCTION

Background and Rationale

Based on methacrylates, polymeric materials have been widely used in the construction of both active and passive removable orthodontic appliances for many years. Orthodontic appliances are used for newborn cleft lip and palate patients, space maintenance, tipping teeth, overbite reduction, thumb deterrent, block-movements, and retention after treatment. The appliances are used in children, adult and older patients. Moreover, the base plate of orthodontic appliance was kept in contact with oral mucosa for a long time during treatment.

Polymerization of the orthodontic acrylic resin is an additional reaction that requires the activation of an initiator, such as benzoyl peroxide, and is then decomposed by the addition of a chemical activator, such as dimethyl-p-toluidine. Curing process is followed by conversion of methyl methacrylate (MMA) to polymethyl methacrylate (PMMA). During polymerization reaction of acrylic resins, not all the monomers are converted into polymers. Some unreacted monomers called residual monomers are therefore left.

When the orthodontic base plate contacts with saliva and mucosa, residual monomer will be leached from the acrylic resin to the oral environment. The residual monomer can cause local and systemic reactions such as erythema, necrosis, pain and burning sensation. Allergy from the residual monomer is varied among each patient (1, 2). In Orthodontics, Goncalves et al reported on a 60-year-old woman who had an allergic reaction to the residual MMA after the insertion of a retainer for one month (1).

It has been reported that residual monomer in dental acrylic resin has deleterious effects on many of its properties, such as water sorption, hardness, flexural strength, dimensional stability, tensile strength, and biocompatibility. Consequently, it is desirable to reduce the residual MMA content in the dental acrylic resin to as low a level as possible,

prior to an insertion in the patient's mouth. It has been recommended that the residual monomer content of autopolymerized acrylic resins could be reduced by an immersion in water for 24 hours before usage (3). However, this method takes a long time for the reduction of residual monomer.

Ultrasonic bath has been used in cleaning the dental instrument. Several studies found that ultrasonic bath was used to extract chemical substance and nutrient in industry (4, 5). However, the effectiveness of ultrasonic bath in promoting residual monomer reduction in orthodontic acrylic resin has not been investigated.

The aim of this study is to compare the level of residual methyl methacrylate monomer of self-cured orthodontic base-plate materials after water immersion at room temperature and after water immersion in ultrasonic bath.

Research Questions

1. Do the levels of residual monomer in Orthocryl differ from those in Orthoplast?
2. Do the levels of residual monomer in self-cured orthodontic base-plate materials by different reduction methods differ among each other?

Objectives

1. To compare between the levels of residual monomer in Orthocryl and those in Orthoplast.
2. To compare the levels of residual monomer in self-cured orthodontic base-plate materials among the different reduction methods.

Research Hypotheses

1. H_0 : The levels of residual monomer in Orthocryl do not significantly differ from those in Orthoplast at 0.05 significant level.

H_A : The levels of residual monomer in Orthocryl significantly differ from those in Orthoplast at 0.05 significant level.

2. H_0 : The reduction methods do not significantly affect the levels of residual monomer in self-cured orthodontic base-plate materials at 0.05 significant level.

H_A : A minimum of one reduction method significantly affects the level of residual monomer in self-cured orthodontic base-plate materials at 0.05 significant level.

Limitations

1. This research is an experimental study *in vitro*.
2. Specimens are prepared by the spray-on technique to imitate the orthodontic laboratory procedure.
3. Temperature of ultrasonic bath is set at 50°C.
4. Distilled water is used for the water immersion process.

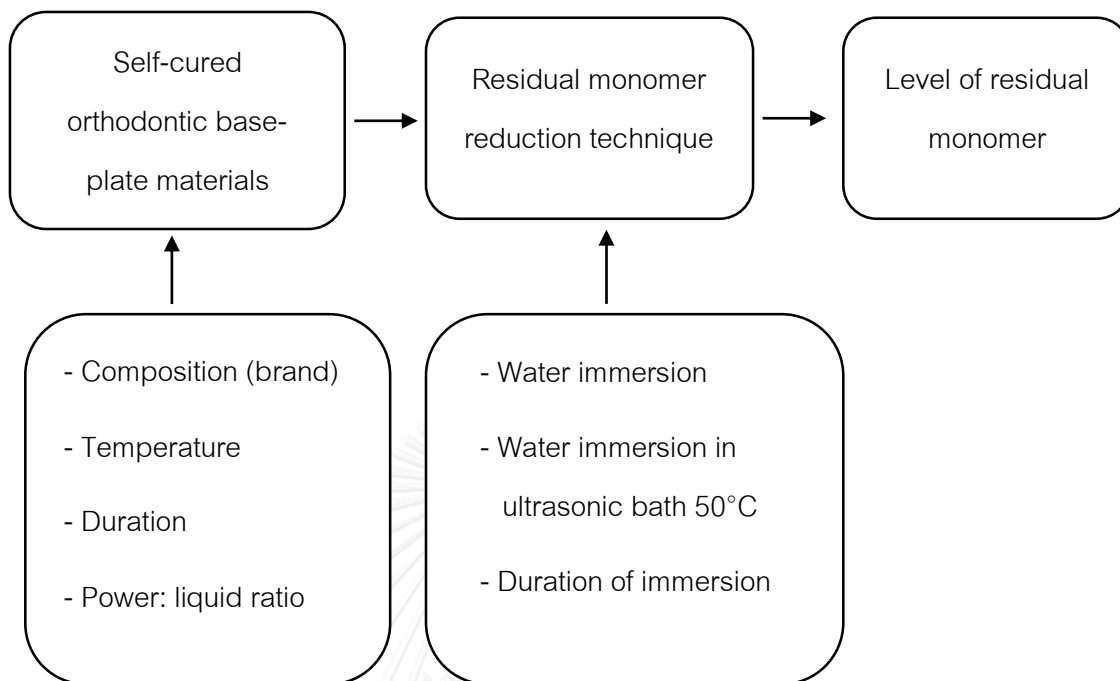
Expected Benefits and Applications

1. Less time for reducing the level of residual monomer in the orthodontic base-plate.
2. Faster and safer delivery of a removable orthodontic appliance to patients.

Research Design

An experimental study

Conceptual Framework



CHAPTER II

LITERATURE REVIEW

Acrylic resins

Acrylic resins are derivatives of ethylene and vinyl group in their structural formula.

The acrylic resins used in dentistry are the esters of: (6)

1. Acrylic acid, $\text{CH}_2=\text{CHCOOH}$
2. Methacrylic acid, $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOH}$

Most of removable orthodontic appliances are made of acrylic resins. The properties of materials are not ideal but present a compromise between physical properties on the one hand and ease of use and cost on the other (7).

Methyl methacrylate (MMA) (Figure 1)

Liquid monomer, MMA, is mixed with the PMMA, which is supplied in the form of powder. The monomer partially dissolves the polymer to form a plastic dough-like material. MMA is a clear and transparent liquid at room temperature with the following physical properties: (8)

Molecular weight	=	100
Melting point	=	-48°C
Boiling point	=	100.8 °C
Density	=	0.945 g/ml at 20 °C
Heat of polymerization	=	12.9 kcal/mol

MMA exhibits a high vapor pressure and is an excellent organic solvent. Although the polymerization of the MMA can be initiated by ultraviolet, visible light, or heat, it is commonly polymerized in dentistry by the use of a chemical initiator (8).

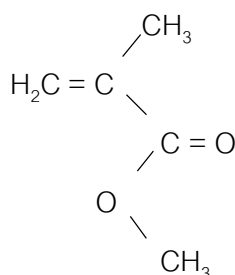


Figure 1 Methyl methacrylate molecule

Polymethyl methacrylate (PMMA) (Figure 2)

PMMA is a transparent resin of water-like clarity that is transparent to light in the visible and ultraviolet ranged down to a wavelength of 250 nm. It is a hard resin with a Knoop hardness number of 18 to 20 KHN. It possesses an approximate tensile strength of 60 MPa and a density of 1.19 g/cm³. Its modulus of elasticity is approximately 2400 MPa.

The main advantage of PMMA is ease of processing. Although it is a thermoplastic resin, it is usually moulded by mixing the MMA monomer with the polymer powder. The monomer plasticizes the polymer to dough-like consistency and can then be easily moulded initially in the mold space.

Like all acrylic resins, PMMA exhibits a tendency to absorb water by imbibition. Its non-crystalline structure possesses a high internal energy. Thus, molecular diffusion can occur in the resin because less activation energy is required (8).

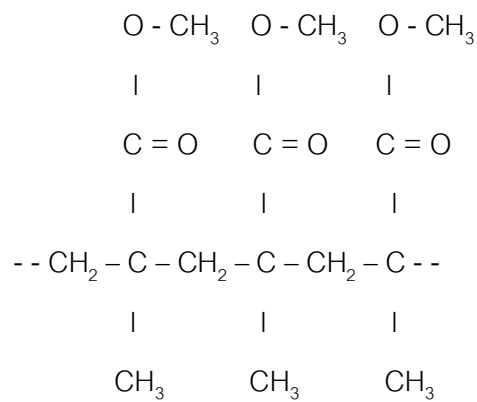


Figure 2 Chemical structure of PMMA

Chemically activated acrylic resins

The chemically activated acrylic resins polymerize at room temperature. They are also known as 'self-curing', 'cold cure' or 'autopolymer' resins. In cold cured acrylic resins, the chemical initiator (benzoyl peroxide) is activated by another chemical (dimethyl-p-toluidine), which is present in the monomer. Therefore, the fundamental difference between the heat- and the self-cure resins is the method of activating benzoyl peroxide (9).

Composition of chemically activated acrylic resins

Powder

- PMMA and other copolymers (5%): dissolved by monomer
- Benzoyl peroxide: Initiator
- Compounds of mercuric sulfide, cadmium sulfide: Opacifiers
- Dibutyl phthalate: Plasticizer
- Dyed organic fillers and inorganic particles like glass fibres or beads: Fillers

Liquid

- MMA monomer: Dissolves polymer
- Dimethyl-p-toluidine: Activator
- Dibutyl phthalate: Plasticizer
- Glycol dimethacrylate (1% to 2%): Cross linking agent
- Hydroquinone (0.006%): Inhibitor

Chemical stages of polymerization: The polymerization reaction can be summarized as follows: (10)

Powder (polymer) + liquid (monomer) → polymer + heat (exothermic reaction)

When the powder and liquid components are mixed, the tertiary amine causes decomposition of benzoyl peroxide, producing free-radicals. Polymerization is then initiated. MMA dissolves into the PMMA beads, forming a material of doughy consistency.

Concurrently, dimethyl-p-toluidine comes in contact with benzoyl peroxide, generating benzyl free-radicals that go on to initiate the chemical reaction for free-radical additional polymerization. Polymerization in the two-part chemical-cured acrylic resin is free-radical addition across an aliphatic C=C double bond. As polymerization progresses, the amount of aliphatic C=C double bond decreases. Often, the reaction is not complete, so there will be a finite amount of monomer (MMA) remaining in the chemical-cured acrylic. The polymerization is never as complete as that of the heat-curing type; self-cured resin usually contains 3%-5% residual monomer, in comparison with approximate 0.2%-0.5% free monomer found in resin processed in boiling water (9).

Self-cured resins have some advantages, such as an excellent esthetic property, easily fabrication and repair. Its disadvantages are lack of color stability, shrinkage, presence of residual monomer, and cracking or crazing. However, the problem of residual

monomer has not totally been solved. Because of the subsequent oxidation of the tertiary amine, the color stability of the self-cured resins is inferior to that of the heat-curing type. The condition can be minimized by adding certain stabilizing agents to prevent such oxidation. In addition, the polymerization may be consummated by the use of a more stable activators.

Manipulation technique for removable orthodontic appliances

In orthodontics, the manipulation technique for preparation of removable orthodontic appliances is the spray-on technique, in which the polymer is saturated by its monomer. Whilst the doughing technique, in which liquid and powder are mixed together, is widely utilized in prosthodontics.

Goncalves et al (11) found that the level of residual monomer in the doughing technique was not significantly different from the spray-on technique at the initial time and at 24 hours after preparation. However, Ica et al (12) found that the residual monomer release rate in acrylic resin prepared with the doughing technique was higher than that with the spray-on technique. Therefore, the spray-on technique is recommended for preparation of the orthodontic base plates.

Residual monomer

During the polymerization reaction of acrylic resins, not all the monomers are converted into polymers; therefore, some unreacted monomers called residual monomers are left. The concentration of residual monomer is varied dependent on the methods and the conditions of polymerization (13-18).

Some researchers have also reported that self-cured acrylic resin has higher levels of residual monomer, when compared to the heat-cured acrylic resin (19, 20). Baker et al (3) detected higher amounts of residual MMA in the saliva of subjects wearing dentures made from self-cured resins, when compared to those made from heat-cured resins. Moreover, Sadamori et al (21) also reported that residual monomer content in

acrylic dentures could be detected for up to several years after usage. While it appeared that most of the residual monomer was lost after about five years, a complete loss of the residual monomer content may take many more years.

Effects of residual monomer

1. Allergy

Removable orthodontic appliances were used for many months or years and kept in contact with the oral mucosa for a long period. The residual monomer of acrylic resins and its dilution have been widely investigated. During the first 24 hours after polymerization, the presence of unreacted residual monomer has been indicated. MMA is considered an allergen and can cause local adverse reactions, such as erythema, edema, a burning sensation, fissures, necrosis, pain (22, 23), and even some systemic reactions (24), such as labial edema, difficulty in swallowing, chronic urticaria, and hypersalivation (22, 25).

Generally, allergic reactions to acrylic are local manifestations, but clinical presentations can be differed. For orthodontic acrylic resin, Goncalves et al (1) reported an allergic reaction to MMA self-curing acrylic resin during orthodontic treatment in a 60-year-old woman patient, after an orthodontic retainer had been inserted. A localized hypersensitive reaction on the palate, hypersalivation, a bitter taste in the mouth, and difficulty swallowing were revealed. For prosthesis acrylic resin, Ruiz Genao et al (23) mentioned labial edema in a case of an allergy to MMA, after an insertion of the prosthesis. Moreover, 22 patients suffering from burning mouth syndrome and five cases showing an allergy to MMA, as well as a high residual monomer concentration in their dentures, were reported (26).

Many studies agree that residual monomer releasing into the oral environment is a main cause of allergic reactions. Therefore, orthodontists should try to reduce the residual monomers from the self-cured orthodontic base-plate materials. It is essential to keep minimal levels of residual monomer.

2. Mechanical properties

The level of residual monomer in the acrylic resin is related to the mechanical properties. Dogan et al (27) found a positive correlation between water sorption and residual monomer. The residual monomer can cause voids in acrylic resin. When residual monomer leaches out, water molecules can penetrate the void and act as a plasticizer. Thus, water molecules push the polymer chains further apart. Consequently, the secondary chemical-bonding forces (van der Waals forces) between the polymer chains decrease. As a result, the mechanical properties of polymers are reduced. Moreover, several studies demonstrated that mechanical properties were improved when the amount of residual monomer reduced (13, 28).

Method for reduction of residual monomer

1. Polymerization method

The amount of residual monomer is left in the polymer dependent on polymerization temperature and time. Different polymerization techniques have been proposed to decrease the residual monomer of auto-polymerizing acrylic resins. Kedjarune et al (29) observed a reduced amount of residual monomer when polymerization time was extended. Similarly, Dogan et al (27) found that the level of residual monomer, in auto-polymerizing resins, decreased with an increase in temperature when the curing time is kept constant. Bayraktar et al (14) concluded that the lowest overall residual MMA content was obtained from auto-polymerizing specimens, followed by an additional polymerization in water at 60°C and storing in distilled water at 37°C for at least one day. Vallittu et al (30) also stated that increasing the polymerization temperature (from 30°C to 60°C) for the auto-polymerizing acrylic resins decreased the residual MMA content of the polymer (from an average of 4.6 wt% to 3.3 wt%). It was also demonstrated in which only the polymerization temperature was varied that the amount of residual monomer in auto-polymerizing resins decreased as the temperature increased.

2. Post-polymerization method

- Immersion in water at room temperature

Generally, water immersion is a simple method for reducing the residual monomer of acrylic resin. Stafford et al (19) studied the loss of residual monomer from 6 pieces of orthodontic acrylic resins. They found high levels of residual monomer in orthodontic resins and a rapid loss of residual monomer in the first 24 hours after immersing of the specimens in water. Similarly, Basker et al (31) showed that water immersion reduced the residual monomer content in an orthodontic resin. According to Baker et al (3) recommended that auto-polymerizing appliances should be immersed in water for 24 hours before an insertion to patient.

- Immersion in water at 60°C

The residual monomer in acrylic resin may be reduced by a further polymerization at the free-radical sites, which could be achieved following a period of immersion in hot water.

Bural et al (32) investigated the effect of post-polymerization heat-treatments on the residual monomer concentration, the degree of conversion and an *in vitro* cytotoxicity of auto-polymerizing acrylic repair resin. They concluded that the post-polymerization heat-treatment of auto-polymerizing acrylic repair resin by an immersion in water at 60°C for 30 minutes is clinically recommended to improve the degree of conversion, while reducing the leaching residual MMA.

- Microwave post-polymerization treatment

Microwave post-curing was described as a method of reducing the level of residual monomer in the acrylic resin. Urban et al (33) found that the amount of residual monomer of relining acrylic resins after a post-polymerization treatment by water-bath and by microwave irradiation was reduced. Moreover, the mechanical properties and biocompatibility of the relining and denture base materials could be improved.

Cimpan et al (34) also studied the effect of microwave heating on the residual monomer level in an auto-polymerizing resin used in the repair of prostheses. They found that the specimens subjected to microwave irradiation after 20-minutes of auto-polymerization showed a reduced level of residual monomer, when compared with those undergoing other polymerization methods. A similar finding was observed by Patil et al (28) It was found that the residual monomer content of denture liner relining resin decreased and the flexural strength increased significantly with the application of microwave irradiation, by the use of different time/power combinations. The specimens with the lowest residual monomer content were the similar specimens which presented with the highest flexural strength. Microwave post-polymerization irradiation was then concluded to effectively increase the flexural strength of denture liner (at 650 W for 5 min) by reducing the residual monomer content via a further polymerization at free-radical sites. However, no study reports that the post-curing method by microwave reduces the amount of residual monomer in orthodontic acrylic resins.

Ultrasonic cleaning

Ultrasonic cleaning uses high frequency sound waves to create cavitation bubbles in a liquid. These cavitation bubbles release energy which have a scrubbing effect on contaminants adhering to substrates like metals, glass, and ceramics. This action also penetrates blind holes, cracks, and recesses, and is able to thoroughly remove all traces of contamination tightly adhering or embedded onto solid surfaces.

Ultrasonic cleaners work by the two principle mechanisms as follows: (35)

1. Cavitation

Cavitation is a process where the constructive interference of sonic energy causes the formation of rarefiable bubbles in the cleaning liquid. When these microscopic bubbles implode, they produce microscopic jets of liquid that can impinge on the surface of parts to be cleaned.

Cavitation is generated through at least three steps: nucleation, growth, and violent collapse or implosion. The impact energy caused by implosion of the gas bubble hits the surface of the object to be cleaned, interacting both physically and chemically.

2. Acoustic streaming

In acoustic streaming, bulk movement of the liquid occurs. Contaminants that get removed from the surface are carried away by acoustic streaming, and hence are prevented from re-attaching to the surface.

Cavitation and acoustic streaming work together in all forms of ultrasonic cleaning, but the relative contribution of each is a function of frequency. At low ultrasonic frequencies, cavitation is very strong and dominates the cleaning process. At high ultrasonic frequencies, cavitation bubbles are very small, but acoustic streaming velocities can be very high. At high frequencies, acoustic streaming thus dominates the cleaning process and less cleaning occurs due to cavitation.

At present, no study reports the ultrasonic use for reducing the amount of residual monomer in orthodontic acrylic resins.

CHAPTER III

RESEARCH METHODOLOGY

Materials and methods

Research equipment

Materials

1. Two brands of self-cured orthodontic resins:
 - Orthocryl[®] (Dentaurum, Germany; Figure 3)
 - Orthoplast[®] (Vertex, The Netherlands; Figure 4)
2. Hydroquinone (HQ)
3. Tetrahydrofurane (THF)
4. Methanol
5. Methyl methacrylate (MMA)
6. Sheet of polyester film
7. Standard metallographic grinding papers (P500,P1200)

Apparatus

1. Circular stainless steel mould according to ISO 20795-2:2013 (Figure 5)
2. Pressure cooker
3. Polishing machine (Figure 6)
4. Digital Calliper (Mitutoyo Corporation Japan)
5. Ultrasonic bath (FZ 40 KHz., VGT-1990 QTD, China; Figure 7)
6. Magnetic stirring bars and apparatus
7. Centrifuge (Beckman, USA)

8. High-performance liquid chromatography (Shimadzu, Japan; Figure 8)
9. Closable one-mark volumetric glass flasks
10. Volume Pipette
11. Closable glass tubes



Figure 3 Orthocryl® (Dentaurum, Germany)



Figure 4 Orthoplast® (Vertex, The Netherlands)



Figure 5 A stainless steel mould
(a diameter of 50 ± 1 mm and a depth of 3.0 ± 0.1 mm)



Figure 6 Polishing machine

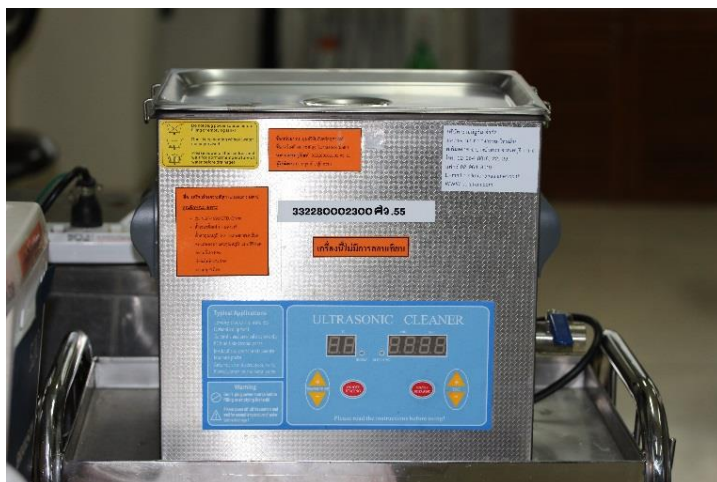


Figure 7 Ultrasonic bath (FZ 40 KHz., VGT-1990 QTD, China)



Figure 8 High-performance liquid chromatography

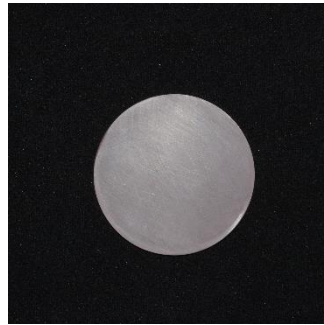


Figure 9 A disc specimen

(a diameter of 50 ± 1 mm and a thickness of 2.0 ± 0.1 mm)

Variables

Independent variables

- Brands: Orthocryl[®] (Dentaurum, Germany), Orthoplast[®] (Vertex, The Netherlands)
- Residual monomer reduction technique

Dependent variable

- The levels of residual monomer

Sample size

The sample size estimation formula for testing mean of two independent populations

$$n = \frac{(\sigma_1^2 + \sigma_2^2) (z_\alpha + z_\beta)^2}{(\mu_1 - \mu_2)^2}$$

Using the data based on the previous study that determined the residual monomer content in acrylic orthodontic resins after storage in water (19) ($\sigma_1=0.24$, $\sigma_2=0.31$, $\mu_1=3.5$, $\mu_2=2.73$) at 0.05 significant level ($\alpha=0.05$) and statistical of 0.80

($\beta=0.20$), the calculated sample size is three. However, most of the studies on residual monomer reduction set the sample size at 6 specimens per group (33, 36). Therefore, the sample size per group in this study was also set at six to make the results comparable with other studies.

Research methodology

Study for powder: liquid ratio for each specimen

Twelve specimens were manufactured from orthodontic base-plate materials; six specimens from Orthocryl[®] (Dentaurum, Germany) and six specimens from Orthoplast[®] (Vertex, Netherlands). All specimens were prepared in the laboratory room temperature ($23^{\circ}\pm 2^{\circ}\text{C}$), by using a spray-on technique, which was the technique used for preparing removable orthodontic appliances. According to the manufacturer's instruction, the time for manufacturing each specimen was less than nine minutes. According to the ISO 20795-2(2013), each specimen was manufactured as follows:

1. Thin layer of orthodontic base-plate powder (PMMA) was applied into a stainless steel mould (a diameter of 50 mm and a depth of 3.0 ± 0.1 mm; Figure 5). Orthodontic base-plate liquid (MMA) was then dropped into the powder. The powder and the liquid were applied, until the mould was filled up. Before the mould was covered with the upper flat cover, orthodontic base-plate material was covered with a polyester film
2. In order to fabricate each specimen, the orthodontic base-plate powder and liquid were weighed with an analytical balance and recorded. The powder and liquid of all specimens were then calculated for means and for setting the powder: liquid ratio of the experiment.

Preparation of experimental specimen discs

A total of 96 experimental specimen discs were manufactured from orthodontic base-plate materials; 48 specimens from Orthocryl[®] and 48 specimens from Orthoplast[®]. All specimens were prepared in the laboratory environment at room temperature ($23\pm 2^{\circ}\text{C}$) by using a spray-on technique. According to the manufacturer's instruction, the time for manufacturing each specimen was less than nine minutes. According to the ISO 20795-2(2013), each specimen was manufactured as follows (in Diagram 1):

1. The set ratio of powder: liquid was used. A thin layer of orthodontic base-plate powder (PMMA) was applied into a stainless steel mould (a diameter of 50 mm and a depth of 3.0 ± 0.1 mm). Orthodontic base-plate liquid (MMA) was then dropped into the powder. The powder and the liquid were applied, until the mould was filled up. Before the mould was covered with the upper flat cover, orthodontic base-plate material was covered with a polyester film.

2. The mould was put in a pressure cooker. The specimen was polymerized following the manufacturers' instructions (2.2 bar at 45°C 20 minutes for Orthocryl and 2.5 bar at 55°C 20 minutes for Orthoplast). Each specimen was then put in a zipped plastic bag and kept in a freezer at -20°C . If the specimen were stored in a freezer (below -18°C), the monomer content remains constant for several months, according to ISO 20795-2(2013).

3. Prior to grinding, the specimen was kept in the dark of a laboratory environment at $23^{\circ}\pm 2^{\circ}\text{C}$ for 24 ± 5 hours. The metallographic grinding papers (P500) were used in turn to wet-grind material equally from both sides of the specimen disc. The periphery of the specimens was ground against the $15\mu\text{m}$ (P1200) grain metallographic grinding paper, until the entire periphery was abraded and smooth.

4. The specimens were checked with a digital caliper to ensure that each specimen had a diameter of 50 ± 1 mm and a thickness of 2.0 ± 0.1 mm (Figure 9) and that the top and bottom surfaces were flat. Each specimen was then put in a zipped plastic bag and kept in a freezer at -20°C .

All experimental specimens (N = 96) were divided into eight groups as follows:
(Diagram 2)

Group I (n = 12): the control group

Group II (n = 12): water immersion for 24 hours

Group III (n = 12): water immersion for 72 hours

Group IV (n = 12): water immersion in ultrasonic bath, 50°C for 3 minutes

Group V (n = 12): water immersion in ultrasonic bath, 50°C for 5 minutes

Group VI (n = 12): water immersion in ultrasonic bath, 50°C for 10 minutes

Group VII (n = 12): water immersion in ultrasonic bath, 50°C for 15 minutes

Group VIII (n = 12): water immersion in ultrasonic bath, 50°C for 20 minutes

Preparation of solutions for extraction monomer

Preparation of solutions for extraction monomer was as follows: (Diagram 3)

1. Tetrahydrofurane solution (Solution A)

0.02 g Hydroquinone (HQ) was weighed approximately into a 1l one-mark volumetric glass flask. Tetrahydrofurane (THF) was added until the total volume was 1l.

2. Methanol solution (Solution B)

0.02 g Hydroquinone (HQ) was weighed approximately into a 1l one-mark volumetric glass flask. Methanol was added until the total volume was 1l.

3. Methanol/tetrahydrofurane solution (Solution C)

One volume part of solution A and four volume parts of solution B were mixed.

Preparation of calibration solutions for high-performance liquid chromatography (HPLC)

Preparation of calibration solutions for HPLC was as follows: (Diagram 4)

1. MMA (approximately 6 mg, 60 mg, 150 mg, 300 mg and 400 mg) was weighed into five separated one-mark volumetric 5 ml glass flasks.
2. Solution C was added until the total volume was 5 ml.
3. 100 μ l of each calibration solution was transferred into separate 10 ml one-mark volumetric glass flask, solution C was added until the total volume was 10 ml.
4. The mass of MMA was recorded for each individual calibration solution and the final concentrations (μ g per ml) were calculated.
5. The calibration solutions were injected into the HPLC system.
6. A calibration graph was drawn by plotting the peak area of monomer in the calibration solution against the respective concentrations of MMA expressed in μ g per ml.

Methods for extraction of monomer

Methods for extraction of monomer were as follows: (Diagram 5)

1. Each specimen disc was broken into pieces to make them be small enough to pass through the neck of a 10 ml volumetric glass flask. Samples of approximately 650 mg were accurately weighed with an analytical balance and recorded for three groups. Each group of specimen was applied into separate one-mark 10 ml volumetric glass flask.
2. Tetrahydrofurane solution (A) was added into the volumetric flask until the total volume is 10 ml. A magnetic stirring bar was added to each one-mark volumetric glass flask to agitate the sample solutions at room temperature for 72 ± 2 hours.
3. A separate volumetric pipette was used to transfer 2 ml aliquot of each previously prepared sample solution to one-mark 10 ml volumetric flask. To precipitate

the dissolved polymer, methanol solution (B) was added to each sample solution to a total volume of 10 ml.

4. A separate volumetric pipette was used to transfer 5 ml of the polymer and monomer containing slurry from each of the 10 ml flask to centrifuge tubes. The slurry was centrifuged at $3000 g_n m/s^2$ for 15 minutes.

5. A separate volumetric pipette was used to transfer a 3 ml aliquot of each centrifuged solution to separate glass tubes.

No remaining polymer in the solution was determined. The solution appeared clear when the beam of light is directed vertically through the glass tube containing the solution. If the solution did not appear clear, methanol solution (B) was added until solution appears clear. The volume of the methanol solution (B) was record. The slurry was centrifuged at $3000 g_n m/s^2$ for 15 minutes again (process No. 4)

6. When the solution appears clear, the residual monomer content was determined by means of the HPLC method.

High-performance liquid chromatography (HPLC) test

HPLC equipment and operating conditions were as follows:

- Column – 4-5 mm internal diameter, 100 mm length with octadecyl silanized 10 mm particles
- Mobile phase - 66% methanol, 34% deionized distilled water
- Flow rate - 0.8 ml/min
- Detection - UV 205 nm
- Temperature - Constant room temperature

Determination of the percentage of methyl methacrylate

1. The clear solution from the upper portion of the centrifuged solution was introduced into the injector of the HPLC system. The HPLC was operated until all components were completely eluted.
2. The concentration of MMA monomers in the sample solutions, c_{MMA} ($\mu\text{g/ml}$) was determined using a linear regression equation obtained from a calibration graph.
3. The following equation was used to calculate total amount of MMA monomer in the sample solution, m_{MMA} (μg):

$$m_{\text{MMA}} = \left[c_{\text{MMA}} \times \left(\frac{10}{2} \right) \times 10 \right]$$

Note - $\times \frac{10}{2}$: Solution B was added to a 2 ml aliquot of the sample solution until a total volume of 10 ml is achieved.

- $\times 10$: The volume of the original sample solution was 10 ml.

4. This value was used to calculate the weight percentage of the residual MMA by the following equation:

$$\text{Residual monomer (\% mass fraction)} = (m_{\text{MMA}} / m_{\text{sample}}) \times 100$$

m_{MMA} = Total quantity of MMA in the sample solution (μg)

m_{sample} = The mass of sample solution (μg)

Diagram 1 Preparation of experimental specimen discs

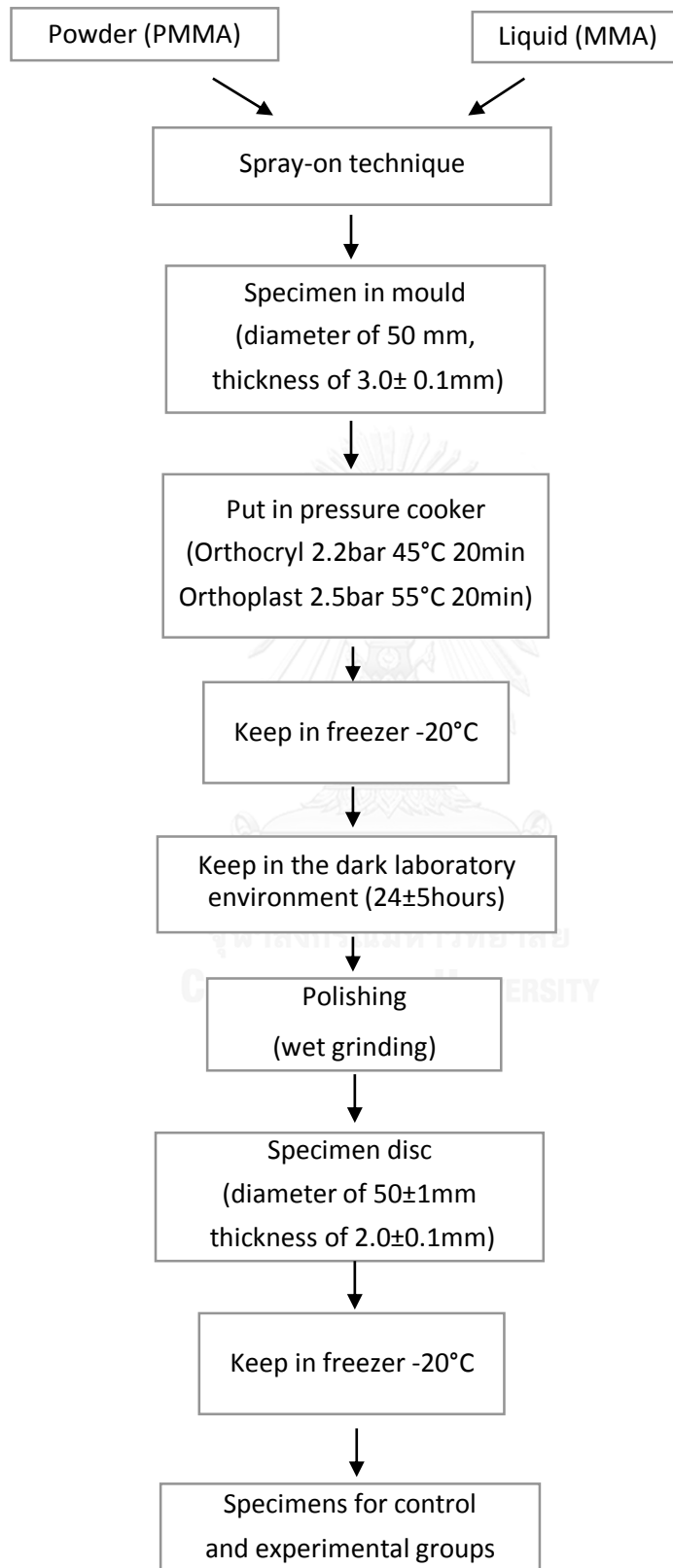


Diagram 2 Categorization of control and experimental groups

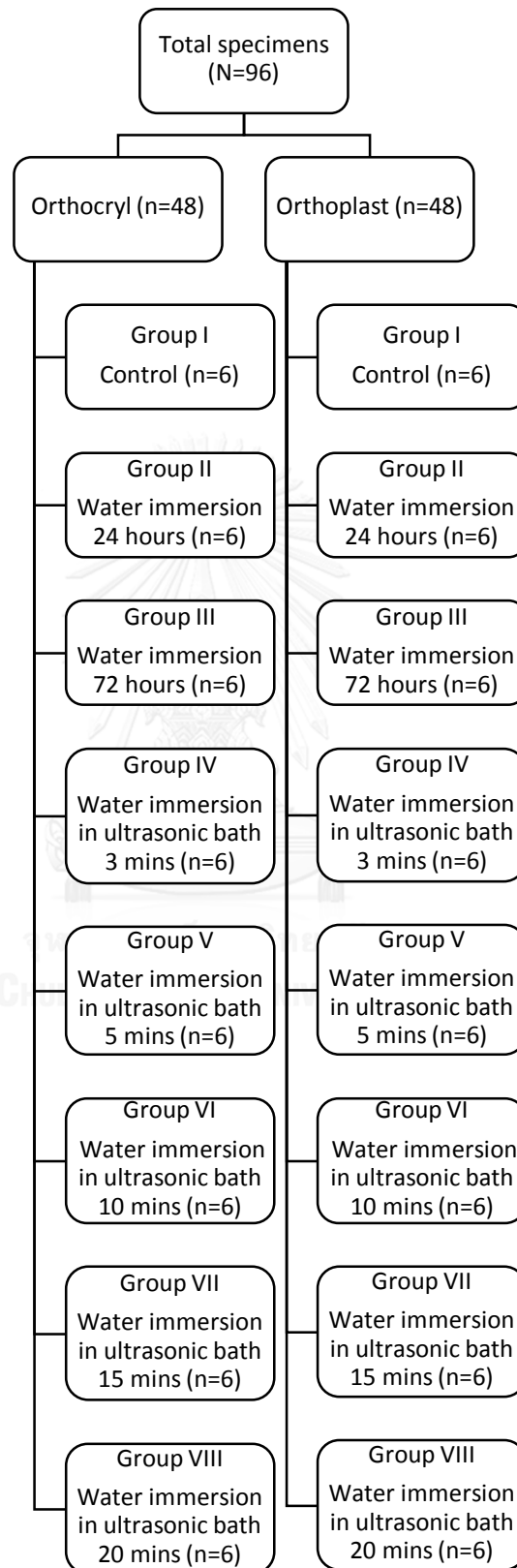


Diagram 3 Preparation of solutions for extraction monomer

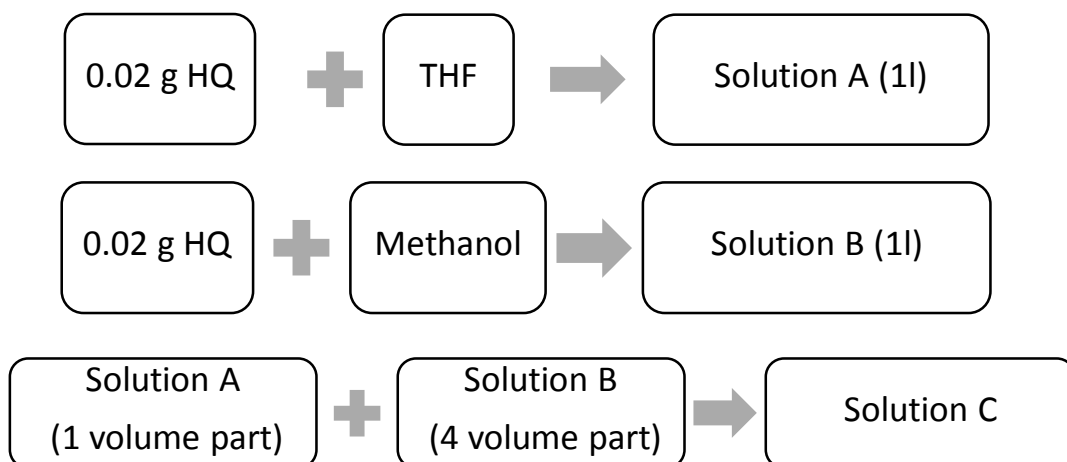


Diagram 4 Preparation of calibration solutions

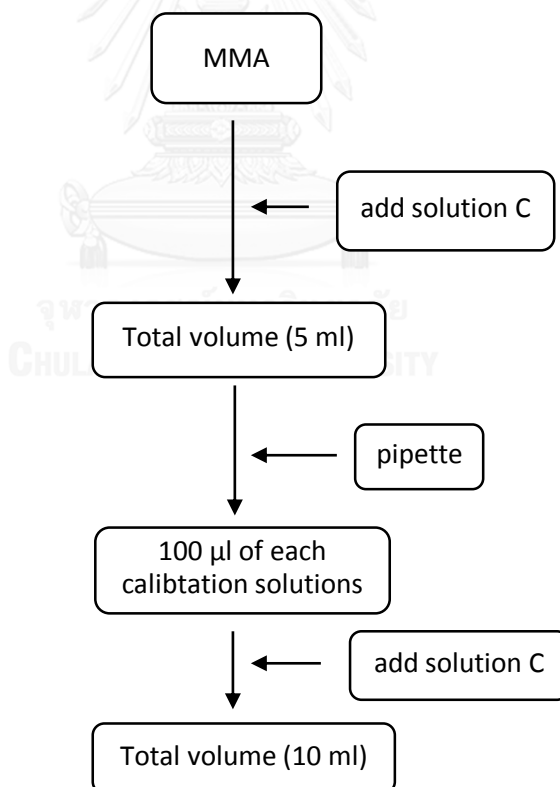


Diagram 5 Method of extraction monomer

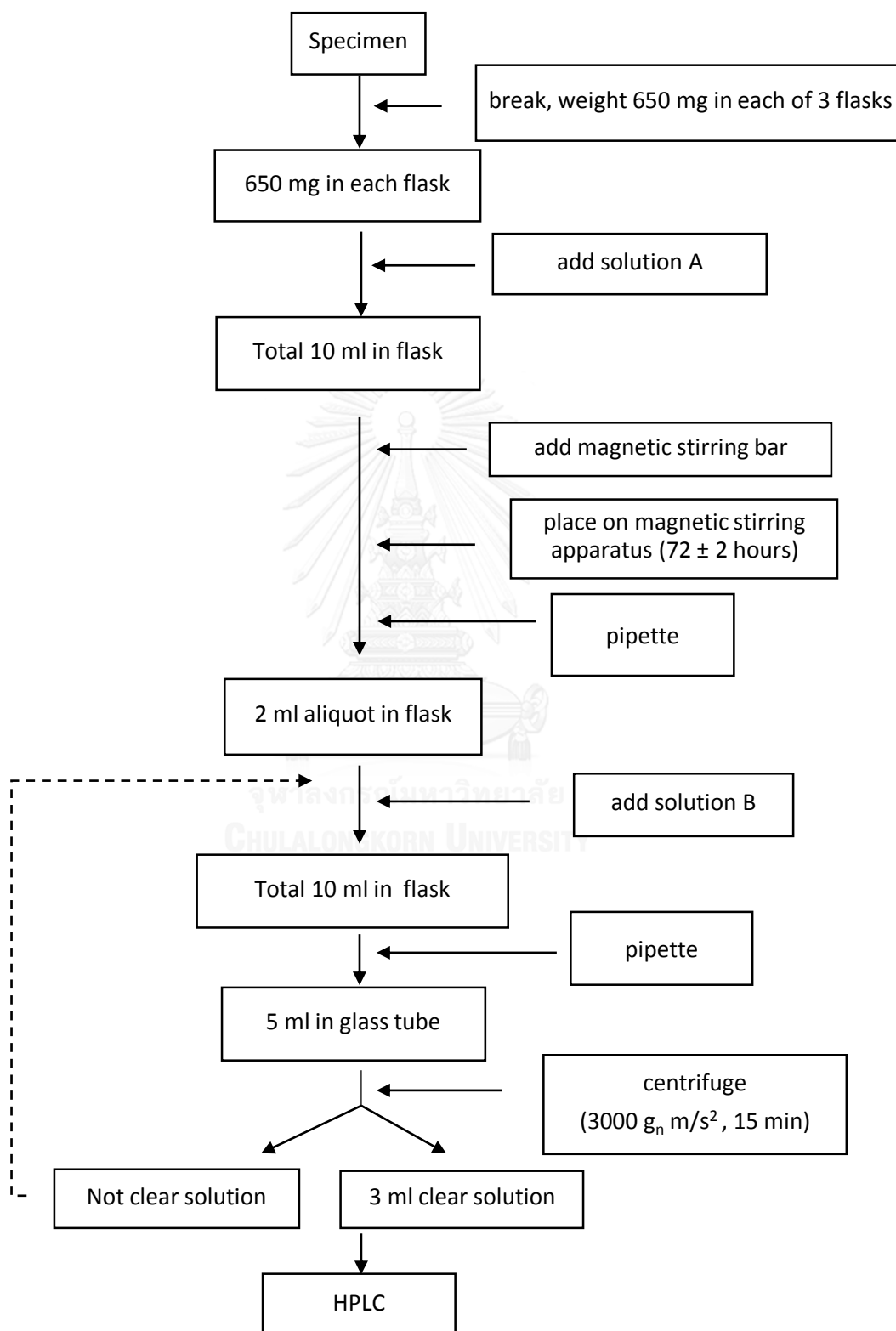
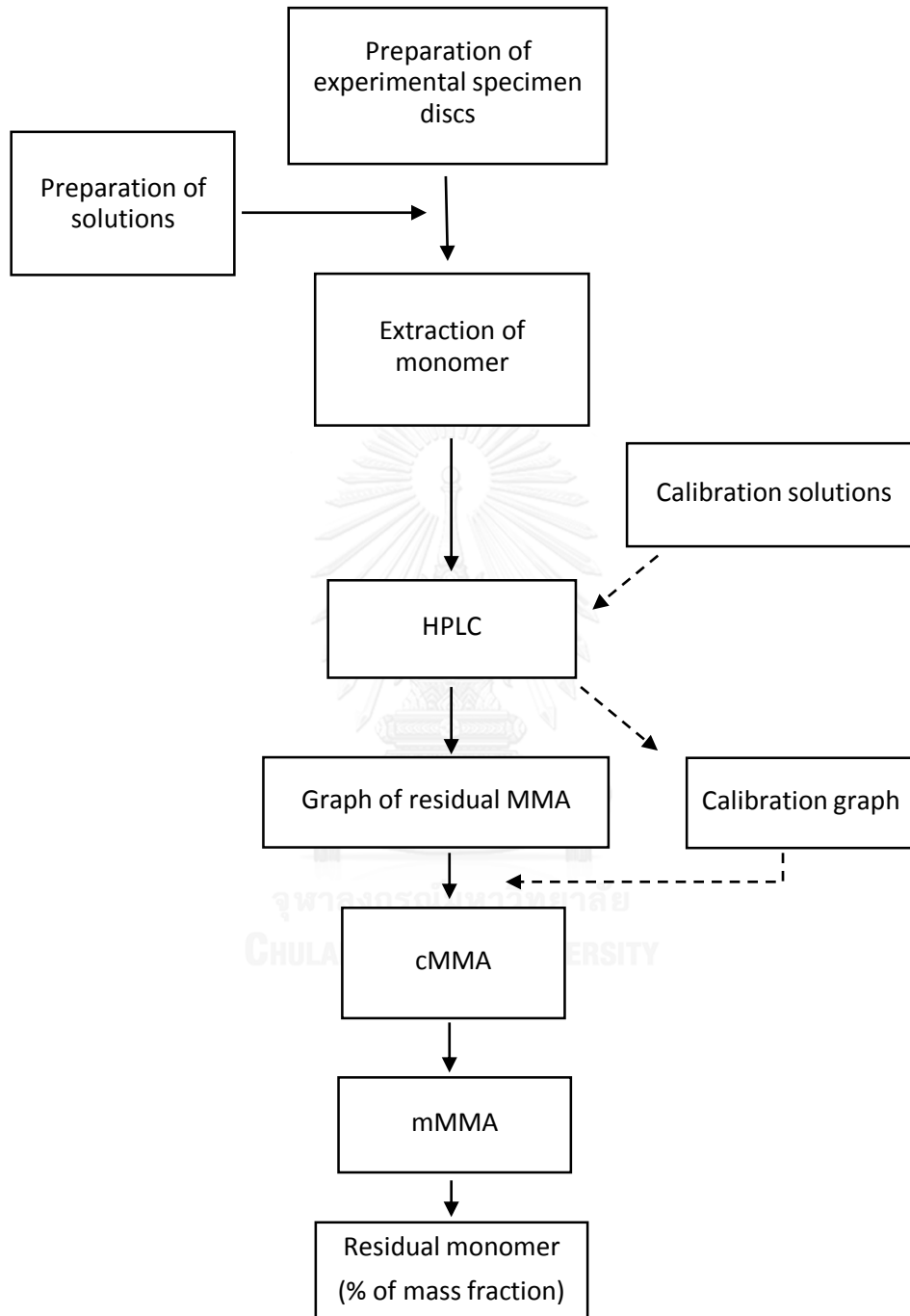


Diagram 6 Summary of experimental methods



Data analysis

Data was analyzed as follows:

1. According to ISO 20795-2:2013, nine sample solutions from three specimens were analyzed for pass/fail determinations of the level of residual MMA monomer. The level of residual monomer must not exceed 5% by mass fraction.

- If seven (or more) of the sample solutions did not exceed 5% by mass fraction, the test was read "Pass".

- If five or six of the sample solutions did not exceed 5% by mass fraction, the new specimen discs were made and the test was repeated. If eight (or more) of the subsequent sample solutions did not exceed 5% by mass fraction, the test was read "Pass".

- If four (or fewer) of the sample solutions did not exceed 5% by mass fraction, the test was read "Fail".

2. Statistical analyses at 0.05 significant levels were performed with an SPSS program for Windows version 17.0.

2.1 The percentage of residual monomer of all specimens in each group was separately tested for normal distribution with Kolmogorov-Smirnov test.

2.2 The percentage of residual monomer of all specimens in each group was analyzed with a two-way ANOVA, followed by a one-way ANOVA Tukey's HSD *post hoc* test.

CHAPTER IV

RESULTS

Means and standard deviations of the residual monomer concentrations of the Orthocryl and Orthoplast were presented in Table 1. Figure 10 shows that the level of residual monomer after water immersion for 24 and 72 hours. Figure 11 shows that the level of residual monomer after water immersion at 50°C in an ultrasonic bath at different time.

The highest residual monomer content was found in the Orthocryl control group (group I), while the group of Orthocryl water immersion at 50°C in an ultrasonic bath for 20 minutes (group VIII) showed the lowest amount of residual monomer.

By using a Komogolov-Smirnov test (Table 4 in appendix), the normal distributions of the data were illustrated. Therefore, the parametric statistics was applied by a two-way ANOVA, followed by a one-way ANOVA Tukey's HSD *post hoc* test.

According to a two-way ANOVA (Table 2 and Table 5 in appendix), both brands of self-cured orthodontic base-plate materials and the residual monomer reduction methods affected the level of residual monomer ($p < 0.05$). A minimum of one reduction method significantly affected the level of residual monomer in self-cured orthodontic base-plate materials ($p < 0.05$). There was an interaction between the variables. It meant that the effect of brand was dependent on the residual monomer reduction method ($p < 0.05$).

The level of residual monomer in the group of water immersion for 24 hours to the group of water immersion at 50°C in an ultrasonic bath for 20 minutes (group II-VIII) were significantly lower than that in the control group (group I) ($p < 0.05$). No significant differences existed between the groups of water immersion for 24 and 72 hours (group II and III) ($p > 0.05$). The level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 20 minutes (group VIII) was significantly lower than those in the groups of water immersion for 24 and 72 hours (groups II and III) ($p < 0.05$). However, no significant differences existed between the group of water immersion for 72 hours (group

III) and the group of water immersion at 50°C in an ultrasonic bath for 15 minutes (group VII) ($p>0.05$), but the level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 15 minutes (group VII) was significantly lower than that in the group of water immersion for 24 hours (groups II) ($p<0.05$).

No significant differences was observed among the groups of water immersion at 50°C in an ultrasonic bath for 10 minutes, water immersion for 24 and 72 hours (group VI, II and III) ($p>0.05$), but the level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 15 minutes (group VII) was significantly lower than that in the group of water immersion at 50°C in an ultrasonic bath for 10 minutes (groups VI) ($p<0.05$). The level of residual monomer in the groups of water immersion at 50°C in an ultrasonic bath for 3 and 5 minutes (group IV and V) were significantly higher than those in the groups of water immersion for 24 and 72 hours (groups II and III) ($p<0.05$), but these groups were shown to possess significantly lower residual monomer contents than in the control group (group I) ($p<0.05$).

According to a one-way ANOVA (Table 1 and Table 6 in appendix), the results were shown below.

In the Orthocryl groups

The levels of residual monomer in the group of water immersion for 24 hours to the group of water immersion at 50°C in an ultrasonic bath for 20 minutes (group II-VIII) were significantly lower than that in the control group (group I) ($p<0.05$). The level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 20 minutes (group VIII) was significantly lower than those in the groups of water immersion for 24 and 72 hours (group II and III) ($p<0.05$).

The levels of residual monomer in the groups of water immersion at 50°C in an ultrasonic bath for 10 and 15 minutes (group VI and VII) were not significantly differences with those in the groups of water immersion for 24 and 72 hours (group II and III) ($p>0.05$),

but the level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 15 minutes (group VII) was significantly lower than that in the group of water immersion at 50°C in an ultrasonic bath for 10 minutes (groups VI) ($p < 0.05$). The level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 3 and 5 minutes (group IV and V) were significantly higher than those in the groups of water immersion for 24 and 72 hours (groups II and III) ($p < 0.05$).

In Orthoplast groups

No significant differences existed between the control group (group I) and the group of water immersion for 24 hours (group II) ($p > 0.05$), but the level of residual monomer in the group of water immersion for 72 hours (group III) was significantly lower than that in the control group (group I) ($p < 0.05$). The levels of residual monomer in the groups of water immersion at 50°C in an ultrasonic bath for 10, 15 and 20 minutes (group VI, VII and VIII) were not significantly differences with those in the group of water immersion for 72 hours (groups III) ($p > 0.05$), but the level of residual monomer in the group of water immersion at 50°C in an ultrasonic bath for 20 minutes (group VIII) was significantly lower than that in the group of water immersion at 50°C in an ultrasonic bath for 15 minutes (groups VII) ($p < 0.05$).

The level of residual monomer in the groups of water immersion at 50°C in an ultrasonic bath for 3 and 5 minutes (group IV and V) were significantly higher than those in the group of water immersion for 72 hours (groups III) ($p < 0.05$), but these groups were shown to possess significantly lower residual monomer contents than the control group (group I) and the group of water immersion for 24 hours (group II) ($p < 0.05$).

Comparison between Orthocryl and Orthoplast groups

In the control group (group I), there was no significant difference between the Orthocryl and the Orthoplast group ($p > 0.05$).

In the same reduction monomer method, the Orthocryl groups showed a statistically significantly lower residual monomer contents than the Orthoplast groups ($p < 0.05$).



Table 1 Means (wt%) and standard deviations of residual monomer concentration of the MMA-based orthodontic base-plate materials (n = 6)

Group	Reduction method	Orthocryl (n=6)	Orthoplast (n=6)
I	Control	3.31±0.18 ^a	3.27±0.10 ^a
II	Water immersion 24 hours	2.31±0.13 ^{e,f}	3.06±0.09 ^{a,b}
III	Water immersion 72 hours	2.34±0.11 ^{e,f}	2.75±0.04 ^{c,d}
IV	Water immersion at 50°C in an ultrasonic bath 3 minutes	2.70±0.22 ^d	3.08±0.06 ^{a,b}
V	Water immersion at 50°C in an ultrasonic bath 5 minutes	2.78±0.17 ^{c,d}	3.14±0.03 ^{a,b}
VI	Water immersion at 50°C in an ultrasonic bath 10 minutes	2.42±0.13 ^e	2.98±0.10 ^{b,c}
VII	Water immersion at 50°C in an ultrasonic bath 15 minutes	2.10±0.20 ^{f,g}	2.73±0.13 ^{c,d}
VIII	Water immersion at 50°C in an ultrasonic bath 20 minutes	2.04±0.08 ^g	2.56±0.08 ^{d,e}

a,b,c,d,e,f,g Means with the same lowercase letter are not significantly different at $p < 0.05$ by a one-way ANOVA with Tukey's HSD *post hoc* test.

Table 2 Means (wt%) and standard deviations of residual monomer concentration of the MMA-based orthodontic base-plate materials (n = 12)

Group	Reduction method	Orthocryl and Orthoplast (n=12)
I	Control	3.29±0.14 ^a
II	Water immersion 24 hours	2.69±0.41 ^c
III	Water immersion 72 hours	2.54±0.23 ^{c,d}
IV	Water immersion at 50°C in an ultrasonic bath 3 minutes	2.89±0.25 ^b
V	Water immersion at 50°C in an ultrasonic bath 5 minutes	2.96±0.22 ^b
VI	Water immersion at 50°C in an ultrasonic bath 10 minutes	2.70±0.31 ^c
VII	Water immersion at 50°C in an ultrasonic bath 15 minutes	2.41±0.37 ^{d,e}
VIII	Water immersion at 50°C in an ultrasonic bath 20 minutes	2.30±0.28 ^e

^{b,c,d,e} Means with the same lowercase letter are not significantly different at $p < 0.05$ by a two-way ANOVA with Tukey's HSD *post hoc* test.

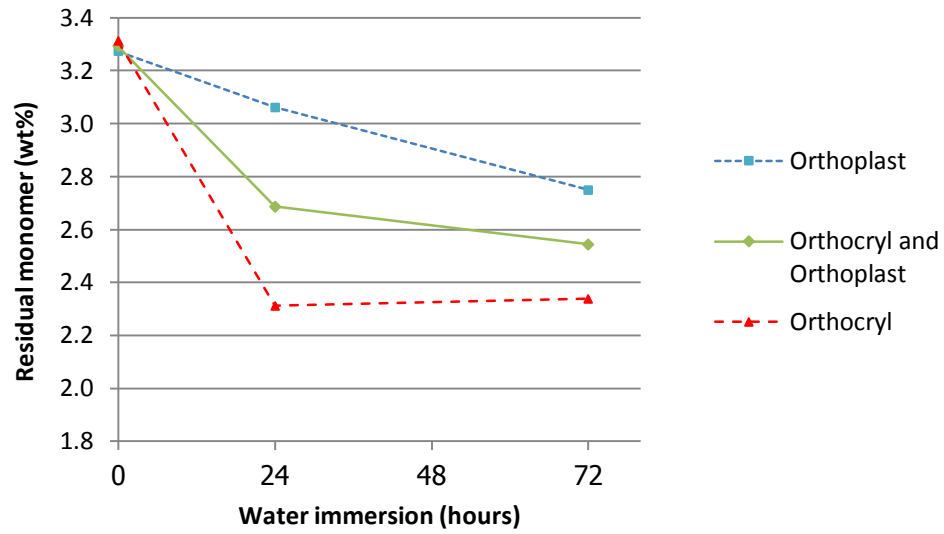


Figure 10 The level of residual monomer after water immersion

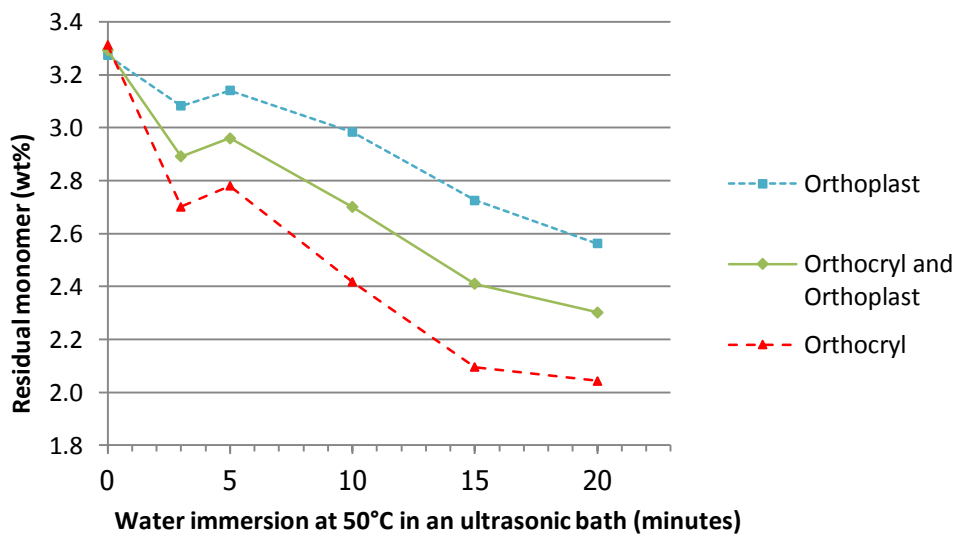


Figure 11 The level of residual monomer after water immersion at 50°C in an ultrasonic bath at different time

CHAPTER V

DISCUSSION AND CONCLUSION

Discussion

Among several orthodontic literatures, few studies have examined the amount of residual monomer in orthodontic acrylic materials (11, 19). Consequently, this study investigated into the effects of water immersion in an ultrasonic bath on the amount of residual monomer from two different orthodontic acrylic resins.

The adverse effects of residual monomer are allergy and physical properties. Allergic reactions to acrylic are local manifestations, but clinical presentations can be differed. Goncalves et al (2006) reported an allergic reaction to MMA self-curing acrylic resin during an orthodontic treatment in a 60-year-old woman patient, after an orthodontic retainer had been inserted. A localized hypersensitive reaction on the palate, hypersalivation, a bitter taste in the mouth, and difficulty swallowing were revealed (1). The level of residual monomer in the acrylic resin was related to the physical properties. Dogan et al (1995) found a positive correlation between water sorption and residual monomer. The residual monomer can cause voids in acrylic resin. When residual monomer leaches out, water molecules can penetrate the void and act as a plasticizer (27). Thus, water molecules push the polymer chains further apart. Consequently, the secondary chemical-bonding forces (van der Waals forces) between the polymer chains decrease. As a result, the mechanical properties of polymers were reduced. Several studies demonstrated that mechanical properties were improved, when the amount of residual monomer reduced (13, 28).

According to ISO 20795-2 (2013), the residual monomer concentration of orthodontic base polymers should not exceed 5 wt%. In this study, each specimen complied with the stated requirement. Despite the fact that ISO 1567 was mentioned as a reference (37), the levels of residual monomer was limited to a 4.5 wt% for self-curing acrylic resins. Harrison and Hugget (1992) referred British Standard Specifications for self-curing orthodontic resins with a 3.5 wt% as a limit for the levels of residual monomer

(38). However, Goncalves et al (2006) found an allergic reaction to the self-curing acrylic resin of an orthodontic retainer base plate, although the levels of residual monomer were below the international standards (1). Therefore, it is desirable to reduce the residual MMA content in the acrylic resin to as low a level as possible, prior to an insertion in the patient's mouth.

HPLC, which is a well-established method for the determination of residual monomer in dental acrylic resins (20, 39, 40), is non-destructive, enables simultaneous analysis of various substances, and provides correct estimates of the level of residual monomer in acrylic resins (41). The HPLC was thus used in this study to determine the level of residual monomer.

In Orthocryl group, statistical analysis of the results revealed that the levels of residual monomer after water immersion for 24 and 72 hours were significantly less than that of the untreated group (or control group). These results were concurrent with those reported by Stafford and Brook (1985) in that the residual monomer loss rapidly in the first 24 hours of water immersion. The amount of residual monomer in the orthodontic resins fall in time occurs by two mechanisms: the continued polymerization of the monomer and the leaching out of the monomer (42).

In Orthocryl groups, no significant differences of residual MMA monomer existed between groups of water immersion 24 and 72 hours. When the specimen was immersed in water, the residual monomer leached out. The voids in the specimen were filled with water molecules by an inward diffusion. Both the outward leakage of the residual monomer and the inward diffusion of water are time-dependent processes. In addition, the level of residual monomer within the specimen changes over time until equilibrium is reached (43).

The level of residual monomer in the groups of water immersion at 50°C in an ultrasonic bath for 3 to 20 minutes (group IV-VIII) were significantly lower than that in the control group (group I) ($p < 0.05$). With the reduction residual monomer methods, the level of residual monomer after water immersion at 50°C in an ultrasonic bath was reduced,

probably because of the microscopic jets of liquid produced by the cavitations. The jets were able to enhance residual monomer to leach out from the surface of specimen and, thus, some polymerizations were additionally provided. Moreover, immersion in hot water (50°C) enhanced a more rapid diffusion of residual monomer into water and increased polymerization at the sites of active radicals (20, 44).

Regarding the specimens immersed at 50°C in an ultrasonic bath (Figure 11), the levels of residual monomer were decreased with time. This was due to the release (into water) of the residual monomer located near the surface of the specimen. On the contrary, those entrapped in the inner layers were not able to release into water within a short period of time. In addition, water immersion at 50°C in an ultrasonic bath for a period of longer than 20 minutes might cause a greater reduction in the level of residual monomer.

Water immersion in an ultrasonic bath does not normally change the dimensional stability of materials. However, its effects on the physical properties need some further investigations.

The residual monomer content of autopolymerized acrylic resins could be reduced by an immersion in water for 24 hours before usage (3, 19), but a long time for reducing the residual monomer is needed. On the other hand, water immersion in an ultrasonic bath spends less time to reduce the level of residual monomer in the orthodontic base-plate materials.

The levels of residual monomer in Orthocryl were significantly lower than those in Orthoplast in the same reduction residual monomer condition. This may be influenced by the particle size and the polymeric composition of the powder, as well as by each brand's concentrations of initiator and accelerator (19).

Since this study was processed *in vitro*, some more realistic results can then be obtained by some further studies in an oral environment. In addition, comparison of different commercial brands of orthodontic base-plate materials and different temperature of water are suggested.

Conclusion

In the same monomer reduction method, the levels of residual monomer in Orthocryl were lower than those in Orthoplast.

Water immersion at 50°C in an ultrasonic bath for 10-20 minutes reduced the amount of residual monomer in an orthodontic acrylic base-plate material, which was similar to or better than water immersion for 24 and 72 hours at room temperature. However, less time was required.



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APPENDIX



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APPENDIX

$$f(x) = (2.1923 \cdot 10^7)x + 649581$$

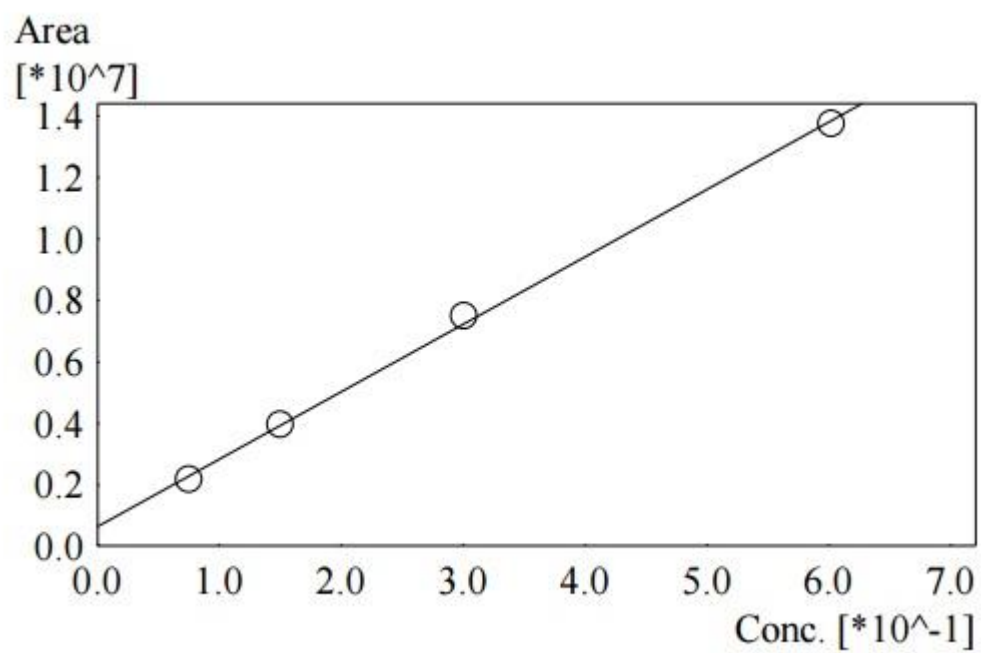


Figure 12 Standard calibration curve

Table 3 Experimental data in the Orthocryl groups

Control	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.424	3.260
		2	0.462	3.548
		3	0.476	3.655
	2	4	0.468	3.596
		5	0.437	3.356
		6	0.413	3.174
	3	7	0.397	3.051
		8	0.451	3.463
		9	0.435	3.346
	4	10	0.445	3.421
		11	0.495	3.803
		12	0.415	3.192
	5	13	0.385	2.959
		14	0.392	2.974
		15	0.404	3.102
	6	16	0.41	3.148
		17	0.429	3.298
		18	0.421	3.237

Table 3 Experimental data in the Orthocryl groups (continued)

Water immersion 24 hours	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.280	2.151
		2	0.281	2.160
		3	0.278	2.138
	2	4	0.287	2.205
		5	0.308	2.367
		6	0.295	2.266
	3	7	0.284	2.180
		8	0.281	2.161
		9	0.296	2.276
	4	10	0.314	2.414
		11	0.299	2.298
		12	0.284	2.184
	5	13	0.317	2.434
		14	0.315	2.422
		15	0.309	2.376
	6	16	0.332	2.553
		17	0.330	2.538
		18	0.321	2.465

Table 3 Experimental data in the Orthocryl groups (continued)

Water immersion 72 hours	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.311	2.392
		2	0.304	2.338
		3	0.298	2.290
	2	4	0.286	2.197
		5	0.321	2.469
		6	0.278	2.135
	3	7	0.315	2.419
		8	0.308	2.367
		9	0.318	2.446
	4	10	0.279	2.144
		11	0.291	2.236
		12	0.276	2.121
	5	13	0.307	2.360
		14	0.313	2.404
		15	0.305	2.344
	6	16	0.324	2.492
		17	0.324	2.489
		18	0.315	2.420

Table 3 Experimental data in the Orthocryl groups (continued)

Ultrasonic bath 50°C, 3 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.385	2.958
		2	0.389	2.987
		3	0.389	2.987
	2	4	0.373	2.864
		5	0.383	2.944
		6	0.320	2.462
	3	7	0.387	2.974
		8	0.384	2.950
		9	0.373	2.869
	4	10	0.329	2.529
		11	0.343	2.634
		12	0.336	2.584
	5	13	0.321	2.467
		14	0.321	2.467
		15	0.320	2.458
	6	16	0.307	2.357
		17	0.343	2.636
		18	0.328	2.519

Table 3 Experimental data in the Orthocryl groups (continued)

Ultrasonic bath 50°C, 5 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.380	2.919
		2	0.375	2.883
		3	0.376	2.887
	2	4	0.395	3.037
		5	0.393	3.022
		6	0.402	3.090
	3	7	0.373	2.867
		8	0.372	2.859
		9	0.332	2.553
	4	10	0.360	2.765
		11	0.347	2.668
		12	0.353	2.712
	5	13	0.360	2.767
		14	0.340	2.610
		15	0.341	2.622
	6	16	0.344	2.645
		17	0.340	2.615
		18	0.327	2.513

Table 3 Experimental data in the Orthocryl groups (continued)

Ultrasonic bath 50°C, 10 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.333	2.560
		2	0.342	2.630
		3	0.326	2.507
	2	4	0.326	2.505
		5	0.321	2.466
		6	0.309	2.373
	3	7	0.322	2.475
		8	0.328	2.519
		9	0.326	2.505
	4	10	0.330	2.538
		11	0.314	2.413
		12	0.312	2.396
	5	13	0.295	2.269
		14	0.300	2.306
		15	0.299	2.299
	6	16	0.296	2.276
		17	0.289	2.219
		18	0.290	2.227

Table 3 Experimental data in the Orthocryl groups (continued)

Ultrasonic bath 50°C,15 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.277	2.129
		2	0.244	1.876
		3	0.242	1.858
	2	4	0.298	2.290
		5	0.322	2.472
		6	0.315	2.422
	3	7	0.286	2.199
		8	0.279	2.146
		9	0.293	2.253
	4	10	0.282	2.167
		11	0.270	2.076
		12	0.288	2.214
	5	13	0.244	1.875
		14	0.238	1.827
		15	0.225	1.729
	6	16	0.279	2.145
		17	0.275	2.112
		18	0.254	1.950

Table 3 Experimental data in the Orthocryl groups (continued)

Ultrasonic bath 50°C,20 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.256	1.969
		2	0.242	1.860
		3	0.248	1.906
	2	4	0.287	2.204
		5	0.273	2.097
		6	0.265	2.034
	3	7	0.264	2.029
		8	0.263	2.022
		9	0.284	2.183
	4	10	0.271	2.082
		11	0.265	2.038
		12	0.261	2.005
	5	13	0.252	1.937
		14	0.262	2.012
		15	0.266	2.045
	6	16	0.271	2.083
		17	0.268	2.061
		18	0.288	2.211

Table 4 Experimental data in the Orthoplast groups

Control	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.428	3.291
		2	0.427	3.284
		3	0.432	3.320
	2	4	0.432	3.322
		5	0.452	3.474
		6	0.475	3.647
	3	7	0.413	3.174
		8	0.417	3.201
		9	0.380	2.922
	4	10	0.423	3.248
		11	0.412	3.166
		12	0.402	3.091
	5	13	0.413	3.176
		14	0.428	3.291
		15	0.413	3.176
	6	16	0.446	3.428
		17	0.442	3.398
		18	0.427	3.278

Table 4 Experimental data in the Orthoplast groups (continued)

Water immersion 24 hours	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.400	3.073
		2	0.404	3.106
		3	0.405	3.113
	2	4	0.397	3.053
		5	0.394	3.031
		6	0.392	3.013
	3	7	0.407	3.131
		8	0.399	3.067
		9	0.410	3.153
	4	10	0.417	3.203
		11	0.408	3.133
		12	0.418	3.210
	5	13	0.410	3.148
		14	0.387	2.976
		15	0.384	2.949
	6	16	0.385	2.958
		17	0.380	2.919
		18	0.377	2.897

Table 4 Experimental data in the Orthoplast groups (continued)

Water immersion 72 hours	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.363	2.787
		2	0.366	2.813
		3	0.358	2.750
	2	4	0.365	2.805
		5	0.361	2.773
		6	0.355	2.727
	3	7	0.352	2.705
		8	0.354	2.718
		9	0.371	2.853
	4	10	0.366	2.811
		11	0.363	2.787
		12	0.348	2.672
	5	13	0.369	2.836
		14	0.356	2.738
		15	0.351	2.699
	6	16	0.344	2.646
		17	0.350	2.691
		18	0.347	2.666

Table 4 Experimental data in the Orthoplast groups (continued)

Ultrasonic bath 50°C, 3 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.401	3.078
		2	0.397	3.053
		3	0.398	3.055
	2	4	0.412	3.163
		5	0.408	3.133
		6	0.410	3.148
	3	7	0.382	2.935
		8	0.401	3.086
		9	0.385	2.958
	4	10	0.389	2.986
		11	0.448	3.446
		12	0.391	3.002
	5	13	0.396	3.040
		14	0.400	3.072
		15	0.390	2.994
	6	16	0.400	3.067
		17	0.417	3.197
		18	0.401	3.075

Table 4 Experimental data in the Orthoplast groups (continued)

Ultrasonic bath 50°C, 5 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.411	3.164
		2	0.409	3.147
		3	0.403	3.102
	2	4	0.405	3.109
		5	0.399	3.062
		6	0.409	3.140
	3	7	0.405	3.114
		8	0.409	3.146
		9	0.405	3.114
	4	10	0.419	3.218
		11	0.410	3.152
		12	0.414	3.180
	5	13	0.408	3.137
		14	0.408	3.136
		15	0.411	3.160
	6	16	0.415	3.180
		17	0.401	3.084
		18	0.414	3.173

Table 4 Experimental data in the Orthoplast groups (continued)

Ultrasonic bath 50°C,10 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.380	2.924
		2	0.388	2.986
		3	0.380	2.923
	2	4	0.383	2.948
		5	0.423	3.238
		6	0.388	2.986
	3	7	0.431	3.305
		8	0.386	2.963
		9	0.401	3.075
	4	10	0.379	2.917
		11	0.360	2.772
		12	0.377	2.901
	5	13	0.391	3.009
		14	0.400	3.076
		15	0.386	2.971
	6	16	0.380	2.921
		17	0.380	2.915
		18	0.376	2.890

Table 4 Experimental data in the Orthoplast groups (continued)

Ultrasonic bath 50°C, 15 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.375	2.884
		2	0.378	2.907
		3	0.378	2.907
	2	4	0.366	2.811
		5	0.361	2.773
		6	0.367	2.819
	3	7	0.342	2.631
		8	0.335	2.572
		9	0.336	2.579
	4	10	0.384	2.950
		11	0.361	2.775
		12	0.345	2.652
	5	13	0.336	2.584
		14	0.365	2.806
		15	0.344	2.642
	6	16	0.336	2.585
		17	0.339	2.603
		18	0.337	2.592

Table 3 Experimental data in the Orthoplast groups (continued)

Ultrasonic bath 50°C,20 minutes	Specimens	Samples	C (mg/ml)	RM (wt%)
	1	1	0.345	2.651
		2	0.343	2.637
		3	0.347	2.668
	2	4	0.339	2.607
		5	0.347	2.669
		6	0.335	2.577
	3	7	0.328	2.520
		8	0.358	2.748
		9	0.335	2.573
	4	10	0.333	2.560
		11	0.323	2.481
		12	0.335	2.575
	5	13	0.328	2.518
		14	0.315	2.420
		15	0.325	2.500
	6	16	0.323	2.484
		17	0.320	2.461
		18	0.320	2.461

Statistical analysis

Table 5 Normality test

One-Sample Kolmogorov-Smirnov Test		RM
N		96
Normal Parameters ^{a,b}	Mean	2.7234
	Std. Deviation	.40881
Most Extreme Differences	Absolute	.092
	Positive	.048
	Negative	-.092
Kolmogorov-Smirnov Z		.898
Asymp. Sig. (2-tailed)		.395

a. Test distribution is Normal.

b. Calculated from data.



Table 6 A two-way ANOVA

Tests of Between-Subjects Effects

Dependent Variable:RM

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	14.593 ^a	15	.973	60.624	.000
Intercept	712.043	1	712.043	44370.369	.000
brand	4.802	1	4.802	299.213	.000
Method	8.611	7	1.230	76.653	.000
brand * Method	1.181	7	.169	10.510	.000
Error	1.284	80	.016		
Total	727.920	96			
Corrected Total	15.877	95			

a. R Squared = .919 (Adjusted R Squared = .904)



Multiple Comparisons

RM

Tukey HSD

(I) Method2	(J) Method2	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
control	water 24 h	.6058*	.05172	.000	.4449	.7668
	water 72 h	.7483*	.05172	.000	.5874	.9093
	ultrasonic 3 mins	.4008*	.05172	.000	.2399	.5618
	ultrasonic 5 mins	.3325*	.05172	.000	.1715	.4935
	ultrasonic 10 mins	.5925*	.05172	.000	.4315	.7535
	ultrasonic 15 mins	.8825*	.05172	.000	.7215	1.0435
	ultrasonic 20 mins	.9900*	.05172	.000	.8290	1.1510
water 24 h	control	-.6058*	.05172	.000	-.7668	-.4449
	water 72 h	.1425	.05172	.122	-.0185	.3035
	ultrasonic 3 mins	-.2050*	.05172	.004	-.3660	-.0440
	ultrasonic 5 mins	-.2733*	.05172	.000	-.4343	-.1124
	ultrasonic 10 mins	-.0133	.05172	1.000	-.1743	.1476
	ultrasonic 15 mins	.2767*	.05172	.000	.1157	.4376
	ultrasonic 20 mins	.3842*	.05172	.000	.2232	.5451
water 72 h	control	-.7483*	.05172	.000	-.9093	-.5874
	water 24 h	-.1425	.05172	.122	-.3035	.0185
	ultrasonic 3 mins	-.3475*	.05172	.000	-.5085	-.1865
	ultrasonic 5 mins	-.4158*	.05172	.000	-.5768	-.2549
	ultrasonic 10 mins	-.1558	.05172	.065	-.3168	.0051
	ultrasonic 15 mins	.1342	.05172	.173	-.0268	.2951
	ultrasonic 20 mins	.2417*	.05172	.000	.0807	.4026

ultrasonic 3 mins	control	-.4008*	.05172	.000	-.5618	-.2399
	water 24 h	.2050*	.05172	.004	.0440	.3660
	water 72 h	.3475*	.05172	.000	.1865	.5085
	ultrasonic 5 mins	-.0683	.05172	.888	-.2293	.0926
	ultrasonic 10 mins	.1917*	.05172	.009	.0307	.3526
	ultrasonic 15 mins	.4817*	.05172	.000	.3207	.6426
	ultrasonic 20 mins	.5892*	.05172	.000	.4282	.7501
ultrasonic 5 mins	control	-.3325*	.05172	.000	-.4935	-.1715
	water 24 h	.2733*	.05172	.000	.1124	.4343
	water 72 h	.4158*	.05172	.000	.2549	.5768
	ultrasonic 3 mins	.0683	.05172	.888	-.0926	.2293
	ultrasonic 10 mins	.2600*	.05172	.000	.0990	.4210
	ultrasonic 15 mins	.5500*	.05172	.000	.3890	.7110
	ultrasonic 20 mins	.6575*	.05172	.000	.4965	.8185
ultrasonic 10 mins	control	-.5925*	.05172	.000	-.7535	-.4315
	water 24 h	.0133	.05172	1.000	-.1476	.1743
	water 72 h	.1558	.05172	.065	-.0051	.3168
	ultrasonic 3 mins	-.1917*	.05172	.009	-.3526	-.0307
	ultrasonic 5 mins	-.2600*	.05172	.000	-.4210	-.0990
	ultrasonic 15 mins	.2900*	.05172	.000	.1290	.4510
	ultrasonic 20 mins	.3975*	.05172	.000	.2365	.5585
ultrasonic 15 mins	control	-.8825*	.05172	.000	-1.0435	-.7215
	water 24 h	-.2767*	.05172	.000	-.4376	-.1157
	water 72 h	-.1342	.05172	.173	-.2951	.0268
	ultrasonic 3 mins	-.4817*	.05172	.000	-.6426	-.3207

	ultrasonic 5 mins	-0.5500*	.05172	.000	-0.7110	-0.3890
	ultrasonic 10 mins	-0.2900*	.05172	.000	-0.4510	-0.1290
	ultrasonic 20 mins	.1075	.05172	.437	-0.0535	.2685
ultrasonic 20 mins	control	-0.9900*	.05172	.000	-1.1510	-0.8290
	water 24 h	-0.3842*	.05172	.000	-0.5451	-0.2232
	water 72 h	-0.2417*	.05172	.000	-0.4026	-0.0807
	ultrasonic 3 mins	-0.5892*	.05172	.000	-0.7501	-0.4282
	ultrasonic 5 mins	-0.6575*	.05172	.000	-0.8185	-0.4965
	ultrasonic 10 mins	-0.3975*	.05172	.000	-0.5585	-0.2365
	ultrasonic 15 mins	-0.1075	.05172	.437	-0.2685	.0535

Based on observed means.

The error term is Mean Square(Error) = .016.

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



RM

Tukey HSD^{a,b}

Method2	N	Subset				
		1	2	3	4	5
ultrasonic 20 mins	12	2.3025				
ultrasonic 15 mins	12	2.4100	2.4100			
water 72 h	12		2.5442	2.5442		
water 24 h	12			2.6867		
ultrasonic 10 mins	12			2.7000		
ultrasonic 3 mins	12				2.8917	
ultrasonic 5 mins	12				2.9600	
control	12					3.2925
Sig.		.437	.173	.065	.888	1.000

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = .016.

a. Uses Harmonic Mean Sample Size = 12.000.

b. Alpha = 0.05.



Table 7 A one-way ANOVA

ANOVA

RM

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	14.593	15	.973	60.624	.000
Within Groups	1.284	80	.016		
Total	15.877	95			



Multiple Comparisons

RM

Tukey HSD

(I) Method	(J) Method	Mean Differenc e (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
orthocryl control	orthocryl water24h	1.00000*	.07314	.000	.7413	1.2587
	orthocrylwater72h	.97333*	.07314	.000	.7146	1.2321
	orthocryl ultra3min	.61000*	.07314	.000	.3513	.8687
	orthocryl ultra5min	.53167*	.07314	.000	.2729	.7904
	orthocryl ultra10min	.89500*	.07314	.000	.6363	1.1537
	orthocryl ultra15min	1.21667*	.07314	.000	.9579	1.4754
	orthocryl ultra20min	1.26833*	.07314	.000	1.0096	1.5271
	orthoplast control	.03833	.07314	1.000	-.2204	.2971
	orthoplast water24h	.25000	.07314	.069	-.0087	.5087
	orthoplast water72h	.56167*	.07314	.000	.3029	.8204
	orthoplast ultra 3 min	.23000	.07314	.139	-.0287	.4887
	orthoplast ultra 5 min	.17167	.07314	.591	-.0871	.4304
	orthoplast ultra 10 min	.32833*	.07314	.002	.0696	.5871
	orthoplast ultra 15 min	.58667*	.07314	.000	.3279	.8454
	orthoplast ultra 20 min	.75000*	.07314	.000	.4913	1.0087
orthocryl water24h	orthocryl control	-1.00000*	.07314	.000	-1.2587	-.7413
	orthocrylwater72h	-.02667	.07314	1.000	-.2854	.2321
	orthocryl ultra3min	-.39000*	.07314	.000	-.6487	-.1313
	orthocryl ultra5min	-.46833*	.07314	.000	-.7271	-.2096
	orthocryl ultra10min	-.10500	.07314	.986	-.3637	.1537
	orthocryl ultra15min	.21667	.07314	.209	-.0421	.4754
	orthocryl ultra20min	.26833*	.07314	.034	.0096	.5271
	orthoplast control	-.96167*	.07314	.000	-1.2204	-.7029
	orthoplast water24h	-.75000*	.07314	.000	-1.0087	-.4913

	orthoplast water72h	-.43833*	.07314	.000	-.6971	-.1796
	orthoplast ultra 3 min	-.77000*	.07314	.000	-1.0287	-.5113
	orthoplast ultra 5 min	-.82833*	.07314	.000	-1.0871	-.5696
	orthoplast ultra 10 min	-.67167*	.07314	.000	-.9304	-.4129
	orthoplast ultra 15 min	-.41333*	.07314	.000	-.6721	-.1546
	orthoplast ultra 20 min	-.25000	.07314	.069	-.5087	.0087
orthocrylwater72h	orthocryl control	-.97333*	.07314	.000	-1.2321	-.7146
	orthocryl water24h	.02667	.07314	1.000	-.2321	.2854
	orthocryl ultra3min	-.36333*	.07314	.000	-.6221	-.1046
	orthocryl ultra5min	-.44167*	.07314	.000	-.7004	-.1829
	orthocryl ultra10min	-.07833	.07314	.999	-.3371	.1804
	orthocryl ultra15min	.24333	.07314	.088	-.0154	.5021
	orthocryl ultra20min	.29500*	.07314	.011	.0363	.5537
	orthoplast control	-.93500*	.07314	.000	-1.1937	-.6763
	orthoplast water24h	-.72333*	.07314	.000	-.9821	-.4646
	orthoplast water72h	-.41167*	.07314	.000	-.6704	-.1529
	orthoplast ultra 3 min	-.74333*	.07314	.000	-1.0021	-.4846
	orthoplast ultra 5 min	-.80167*	.07314	.000	-1.0604	-.5429
	orthoplast ultra 10min	-.64500*	.07314	.000	-.9037	-.3863
	orthoplast ultra 15min	-.38667*	.07314	.000	-.6454	-.1279
	orthoplast ultra 20 min	-.22333	.07314	.171	-.4821	.0354
orthocryl ultra3min	orthocryl control	-.61000*	.07314	.000	-.8687	-.3513
	orthocryl water24h	.39000*	.07314	.000	.1313	.6487
	orthocrylwater72h	.36333*	.07314	.000	.1046	.6221
	orthocryl ultra5min	-.07833	.07314	.999	-.3371	.1804
	orthocryl ultra10min	.28500*	.07314	.017	.0263	.5437
	orthocryl ultra15min	.60667*	.07314	.000	.3479	.8654
	orthocryl ultra20min	.65833*	.07314	.000	.3996	.9171
	orthoplast control	-.57167*	.07314	.000	-.8304	-.3129
	orthoplast water24h	-.36000*	.07314	.000	-.6187	-.1013
	orthoplast water72h	-.04833	.07314	1.000	-.3071	.2104
	orthoplast ultra 3 min	-.38000*	.07314	.000	-.6387	-.1213
	orthoplast ultra 5 min	-.43833*	.07314	.000	-.6971	-.1796

	orthoplast ultra 10min	-.28167*	.07314	.020	-.5404	-.0229
	orthoplast ultra 15min	-.02333	.07314	1.000	-.2821	.2354
	orthoplast ultra 20min	.14000	.07314	.862	-.1187	.3987
orthocryl ultra5min	orthocryl control	-.53167*	.07314	.000	-.7904	-.2729
	orthocryl water24h	.46833*	.07314	.000	.2096	.7271
	orthocrylwater72h	.44167*	.07314	.000	.1829	.7004
	orthocryl ultra3min	.07833	.07314	.999	-.1804	.3371
	orthocryl ultra10min	.36333*	.07314	.000	.1046	.6221
	orthocryl ultra15min	.68500*	.07314	.000	.4263	.9437
	orthocryl ultra20min	.73667*	.07314	.000	.4779	.9954
	orthoplast control	-.49333*	.07314	.000	-.7521	-.2346
	orthoplast water24h	-.28167*	.07314	.020	-.5404	-.0229
	orthoplast water72h	.03000	.07314	1.000	-.2287	.2887
	orthoplast ultra 3 min	-.30167*	.07314	.008	-.5604	-.0429
	orthoplast ultra 5 min	-.36000*	.07314	.000	-.6187	-.1013
	orthoplast ultra 10min	-.20333	.07314	.302	-.4621	.0554
	orthoplast ultra 15min	.05500	.07314	1.000	-.2037	.3137
	orthoplast ultra 20min	.21833	.07314	.199	-.0404	.4771
	orthocryl ultra10min	orthocryl control	-.89500*	.07314	.000	-1.1537
orthocryl water24h		.10500	.07314	.986	-.1537	.3637
orthocrylwater72h		.07833	.07314	.999	-.1804	.3371
orthocryl ultra3min		-.28500*	.07314	.017	-.5437	-.0263
orthocryl ultra5min		-.36333*	.07314	.000	-.6221	-.1046
orthocryl ultra15min		.32167*	.07314	.003	.0629	.5804
orthocryl ultra20min		.37333*	.07314	.000	.1146	.6321
orthoplast control		-.85667*	.07314	.000	-1.1154	-.5979
orthoplast water24h		-.64500*	.07314	.000	-.9037	-.3863
orthoplast water72h		-.33333*	.07314	.002	-.5921	-.0746
orthoplast ultra 3 min		-.66500*	.07314	.000	-.9237	-.4063
orthoplast ultra 5 min		-.72333*	.07314	.000	-.9821	-.4646
orthoplast ultra 10min		-.56667*	.07314	.000	-.8254	-.3079
orthoplast ultra 15min		-.30833*	.07314	.006	-.5671	-.0496
orthoplast ultra 20min	-.14500	.07314	.827	-.4037	.1137	

orthocryl ultra15min	orthocryl control	-1.21667*	.07314	.000	-1.4754	-.9579
	orthocryl water24h	-.21667	.07314	.209	-.4754	.0421
	orthocrylwater72h	-.24333	.07314	.088	-.5021	.0154
	orthocryl ultra3min	-.60667*	.07314	.000	-.8654	-.3479
	orthocryl ultra5min	-.68500*	.07314	.000	-.9437	-.4263
	orthocryl ultra10min	-.32167*	.07314	.003	-.5804	-.0629
	orthocryl ultra20min	.05167	.07314	1.000	-.2071	.3104
	orthoplast control	-1.17833*	.07314	.000	-1.4371	-.9196
	orthoplast water24h	-.96667*	.07314	.000	-1.2254	-.7079
	orthoplast water72h	-.65500*	.07314	.000	-.9137	-.3963
	orthoplast ultra 3 min	-.98667*	.07314	.000	-1.2454	-.7279
	orthoplast ultra 5 min	-1.04500*	.07314	.000	-1.3037	-.7863
	orthoplast ultra 10min	-.88833*	.07314	.000	-1.1471	-.6296
	orthoplast ultra 15min	-.63000*	.07314	.000	-.8887	-.3713
	orthoplast ultra 20min	-.46667*	.07314	.000	-.7254	-.2079
orthocryl ultra20min	orthocryl control	-1.26833*	.07314	.000	-1.5271	-1.0096
	orthocryl water24h	-.26833*	.07314	.034	-.5271	-.0096
	orthocrylwater72h	-.29500*	.07314	.011	-.5537	-.0363
	orthocryl ultra3min	-.65833*	.07314	.000	-.9171	-.3996
	orthocryl ultra5min	-.73667*	.07314	.000	-.9954	-.4779
	orthocryl ultra10min	-.37333*	.07314	.000	-.6321	-.1146
	orthocryl ultra15min	-.05167	.07314	1.000	-.3104	.2071
	orthoplast control	-1.23000*	.07314	.000	-1.4887	-.9713
	orthoplast water24h	-1.01833*	.07314	.000	-1.2771	-.7596
	orthoplast water72h	-.70667*	.07314	.000	-.9654	-.4479
	orthoplast ultra 3 min	-1.03833*	.07314	.000	-1.2971	-.7796
	orthoplast ultra 5 min	-1.09667*	.07314	.000	-1.3554	-.8379
	orthoplast ultra 10min	-.94000*	.07314	.000	-1.1987	-.6813
	orthoplast ultra 15min	-.68167*	.07314	.000	-.9404	-.4229
	orthoplast ultra 20min	-.51833*	.07314	.000	-.7771	-.2596
orthoplast control	orthocryl control	-.03833	.07314	1.000	-.2971	.2204
	orthocryl water24h	.96167*	.07314	.000	.7029	1.2204
	orthocrylwater72h	.93500*	.07314	.000	.6763	1.1937

	orthocryl ultra3min	.57167*	.07314	.000	.3129	.8304
	orthocryl ultra5min	.49333*	.07314	.000	.2346	.7521
	orthocryl ultra10min	.85667*	.07314	.000	.5979	1.1154
	orthocryl ultra15min	1.17833*	.07314	.000	.9196	1.4371
	orthocryl ultra20min	1.23000*	.07314	.000	.9713	1.4887
	orthoplast water24h	.21167	.07314	.241	-.0471	.4704
	orthoplast water72h	.52333*	.07314	.000	.2646	.7821
	orthoplast ultra 3 min	.19167	.07314	.400	-.0671	.4504
	orthoplast ultra 5 min	.13333	.07314	.901	-.1254	.3921
	orthoplast ultra 10min	.29000*	.07314	.014	.0313	.5487
	orthoplast ultra 15min	.54833*	.07314	.000	.2896	.8071
	orthoplast ultra 20min	.71167*	.07314	.000	.4529	.9704
orthoplast	orthocryl control	-.25000	.07314	.069	-.5087	.0087
water24h	orthocryl water24h	.75000*	.07314	.000	.4913	1.0087
	orthocrylwater72h	.72333*	.07314	.000	.4646	.9821
	orthocryl ultra3min	.36000*	.07314	.000	.1013	.6187
	orthocryl ultra5min	.28167*	.07314	.020	.0229	.5404
	orthocryl ultra10min	.64500*	.07314	.000	.3863	.9037
	orthocryl ultra15min	.96667*	.07314	.000	.7079	1.2254
	orthocryl ultra20min	1.01833*	.07314	.000	.7596	1.2771
	orthoplast control	-.21167	.07314	.241	-.4704	.0471
	orthoplast water72h	.31167*	.07314	.005	.0529	.5704
	orthoplast ultra 3 min	-.02000	.07314	1.000	-.2787	.2387
	orthoplast ultra 5 min	-.07833	.07314	.999	-.3371	.1804
	orthoplast ultra 10 min	.07833	.07314	.999	-.1804	.3371
	orthoplast ultra 15 min	.33667*	.07314	.002	.0779	.5954
	orthoplast ultra 20 min	.50000*	.07314	.000	.2413	.7587
orthoplast	orthocryl control	-.56167*	.07314	.000	-.8204	-.3029
water72h	orthocryl water24h	.43833*	.07314	.000	.1796	.6971
	orthocrylwater72h	.41167*	.07314	.000	.1529	.6704
	orthocryl ultra3min	.04833	.07314	1.000	-.2104	.3071
	orthocryl ultra5min	-.03000	.07314	1.000	-.2887	.2287
	orthocryl ultra10min	.33333*	.07314	.002	.0746	.5921

	orthocryl ultra15min	.65500*	.07314	.000	.3963	.9137
	orthocryl ultra20min	.70667*	.07314	.000	.4479	.9654
	orthoplast control	-.52333*	.07314	.000	-.7821	-.2646
	orthoplast water24h	-.31167*	.07314	.005	-.5704	-.0529
	orthoplast ultra 3 min	-.33167*	.07314	.002	-.5904	-.0729
	orthoplast ultra 5 min	-.39000*	.07314	.000	-.6487	-.1313
	orthoplast ultra 10 min	-.23333	.07314	.124	-.4921	.0254
	orthoplast ultra 15 min	.02500	.07314	1.000	-.2337	.2837
	orthoplast ultra 20 min	.18833	.07314	.430	-.0704	.4471
orthoplast ultra 3 min	orthocryl control	-.23000	.07314	.139	-.4887	.0287
	orthocryl water24h	.77000*	.07314	.000	.5113	1.0287
	orthocrylwater72h	.74333*	.07314	.000	.4846	1.0021
	orthocryl ultra3min	.38000*	.07314	.000	.1213	.6387
	orthocryl ultra5min	.30167*	.07314	.008	.0429	.5604
	orthocryl ultra10min	.66500*	.07314	.000	.4063	.9237
	orthocryl ultra15min	.98667*	.07314	.000	.7279	1.2454
	orthocryl ultra20min	1.03833*	.07314	.000	.7796	1.2971
	orthoplast control	-.19167	.07314	.400	-.4504	.0671
	orthoplast water24h	.02000	.07314	1.000	-.2387	.2787
	orthoplast water72h	.33167*	.07314	.002	.0729	.5904
	orthoplast ultra 5 min	-.05833	.07314	1.000	-.3171	.2004
	orthoplast ultra 10 min	.09833	.07314	.993	-.1604	.3571
	orthoplast ultra 15 min	.35667*	.07314	.001	.0979	.6154
orthoplast ultra 20 min	.52000*	.07314	.000	.2613	.7787	
orthoplast ultra 5 min	orthocryl control	-.17167	.07314	.591	-.4304	.0871
	orthocryl water24h	.82833*	.07314	.000	.5696	1.0871
	orthocrylwater72h	.80167*	.07314	.000	.5429	1.0604
	orthocryl ultra3min	.43833*	.07314	.000	.1796	.6971
	orthocryl ultra5min	.36000*	.07314	.000	.1013	.6187
	orthocryl ultra10min	.72333*	.07314	.000	.4646	.9821
	orthocryl ultra15min	1.04500*	.07314	.000	.7863	1.3037
	orthocryl ultra20min	1.09667*	.07314	.000	.8379	1.3554
	orthoplast control	-.13333	.07314	.901	-.3921	.1254

	orthoplast water24h	.07833	.07314	.999	-.1804	.3371
	orthoplast water72h	.39000*	.07314	.000	.1313	.6487
	orthoplast ultra 3 min	.05833	.07314	1.000	-.2004	.3171
	orthoplast ultra 10 min	.15667	.07314	.733	-.1021	.4154
	orthoplast ultra 15 min	.41500*	.07314	.000	.1563	.6737
	orthoplast ultra 20 min	.57833*	.07314	.000	.3196	.8371
orthoplast ultra 10 min	orthocryl control	-.32833*	.07314	.002	-.5871	-.0696
	orthocryl water24h	.67167*	.07314	.000	.4129	.9304
	orthocrylwater72h	.64500*	.07314	.000	.3863	.9037
	orthocryl ultra3min	.28167*	.07314	.020	.0229	.5404
	orthocryl ultra5min	.20333	.07314	.302	-.0554	.4621
	orthocryl ultra10min	.56667*	.07314	.000	.3079	.8254
	Orthocryl ultra15min	.88833*	.07314	.000	.6296	1.1471
	orthocryl ultra20min	.94000*	.07314	.000	.6813	1.1987
	orthoplast control	-.29000*	.07314	.014	-.5487	-.0313
	orthoplast water24h	-.07833	.07314	.999	-.3371	.1804
	orthoplast water72h	.23333	.07314	.124	-.0254	.4921
	orthoplast ultra 3 min	-.09833	.07314	.993	-.3571	.1604
	orthoplast ultra 5 min	-.15667	.07314	.733	-.4154	.1021
	orthoplast ultra 15 min	.25833	.07314	.051	-.0004	.5171
	orthoplast ultra 20 min	.42167*	.07314	.000	.1629	.6804
	orthoplast ultra 15 min	orthocryl control	-.58667*	.07314	.000	-.8454
orthocryl water24h		.41333*	.07314	.000	.1546	.6721
orthocrylwater72h		.38667*	.07314	.000	.1279	.6454
orthocryl ultra3min		.02333	.07314	1.000	-.2354	.2821
orthocryl ultra5min		-.05500	.07314	1.000	-.3137	.2037
orthocryl ultra10min		.30833*	.07314	.006	.0496	.5671
orthocryl ultra15min		.63000*	.07314	.000	.3713	.8887
orthocryl ultra20min		.68167*	.07314	.000	.4229	.9404
orthoplast control		-.54833*	.07314	.000	-.8071	-.2896
orthoplast water24h		-.33667*	.07314	.002	-.5954	-.0779
orthoplast water72h		-.02500	.07314	1.000	-.2837	.2337

	orthoplast ultra 3 min	-.35667*	.07314	.001	-.6154	-.0979
	orthoplast ultra 5 min	-.41500*	.07314	.000	-.6737	-.1563
	orthoplast ultra 10 min	-.25833	.07314	.051	-.5171	.0004
	orthoplast ultra 20 min	.16333	.07314	.672	-.0954	.4221
orthoplast ultra 20 min	orthocryl control	-.75000*	.07314	.000	-1.0087	-.4913
	orthocryl water24h	.25000	.07314	.069	-.0087	.5087
	orthocrylwater72h	.22333	.07314	.171	-.0354	.4821
	orthocryl ultra3min	-.14000	.07314	.862	-.3987	.1187
	orthocryl ultra5min	-.21833	.07314	.199	-.4771	.0404
	orthocryl ultra10min	.14500	.07314	.827	-.1137	.4037
	orthocryl ultra15min	.46667*	.07314	.000	.2079	.7254
	orthocryl ultra20min	.51833*	.07314	.000	.2596	.7771
	orthoplast control	-.71167*	.07314	.000	-.9704	-.4529
	orthoplast water24h	-.50000*	.07314	.000	-.7587	-.2413
	orthoplast water72h	-.18833	.07314	.430	-.4471	.0704
	orthoplast ultra 3 min	-.52000*	.07314	.000	-.7787	-.2613
	orthoplast ultra 5 min	-.57833*	.07314	.000	-.8371	-.3196
	orthoplast ultra 10 min	-.42167*	.07314	.000	-.6804	-.1629
	orthoplast ultra 15 min	-.16333	.07314	.672	-.4221	.0954

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

RM

Tukey HSD^a

Method	N	Subset for alpha = 0.05						
		1	2	3	4	5	6	7
orthocryl ultra20min	6	2.0433						
orthocryl ultra15min	6	2.0950	2.0950					
orthocryl water24h	6		2.3117	2.3117				
orthocrylwater72h	6		2.3383	2.3383				
orthocryl ultra10min	6			2.4167				
orthoplast ultra 20 min	6			2.5617	2.5617			
orthocryl ultra3min	6				2.7017			
orthoplast ultra 15 min	6				2.7250	2.7250		
orthoplast water72h	6				2.7500	2.7500		
orthocryl ultra5min	6				2.7800	2.7800		
orthoplast ultra 10 min	6					2.9833	2.9833	
orthoplast water24h	6						3.0617	3.0617
orthoplast ultra 3 min	6						3.0817	3.0817
orthoplast ultra 5 min	6						3.1400	3.1400
orthoplast control	6							3.2733
orthocryl control	6							3.3117
Sig.		1.000	.088	.069	.199	.051	.733	.069

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 6.000.

VITA

Miss Pajima Thaitammayanon was born on 21th May 1986. She graduated her Doctor of Dental Surgery from Chulalongkorn University in 2009. After graduation, she worked at Nong Ya Plong hospital and Tha Yang hospital as a general practitioner for 1 year and 2 year, respectively. In 2013, she started her Master degree at Chulalongkorn University in Orthodontic department and continued ever since.

