



Original Article

Mechanical and physical properties of an MMA-based orthodontic base-plate material after ultrasonic treatment in water

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Abstract

Background/objectives: To evaluate the mechanical and physical properties of a self-cured methyl methacrylate (MMA)-based orthodontic base-plate material after 50°C water immersion in ultrasonic bath and conventional monomer reduction methods.

Materials and methods: Twenty-four rectangular and twenty-four disc shaped specimens of an MMA-based orthodontic base-plate material were prepared, and randomly divided into four groups (n=6): Group I, untreated controls; Groups II and III, immersed in room temperature water for 24 and 72 hours respectively; and Groups IV immersed in 50°C water in an ultrasonic bath (100 Watts, 40 kHz) for 10 minutes. Their flexural strength, flexural modulus, microhardness, water sorption, and water solubility were measured. The data was analyzed by a one-way analysis of variance, followed by a *post hoc* Tukey's test at a 95% confidence interval.

Results: The flexural strength and flexural modulus of Group IV were significantly higher than those of Group I; however, microhardness, water sorption, and water solubility were not significantly different. Compared to Group I, Groups II and III showed no significant difference in flexural strength, microhardness, water sorption, or water solubility. However, the flexural modulus of Groups II and III were significantly higher than Group I.

Conclusions: After post-polymerization treatment by water immersion at 50°C in an ultrasonic bath for 10 minutes, the mechanical and physical properties of MMA-based orthodontic base-plate material were improved or unchanged.

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Keywords: flexural strength; flexural modulus; microhardness; orthodontic acrylic resin; ultrasonic bath; water solubility; water sorption

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Introduction

Self-cured methyl methacrylate (MMA)-based (acrylic) resins are widely used in dentistry, particularly in an orthodontic treatment. Their advantages over the heat-cured types are the ease of handling, low cost, and rapid curing. Using self-cured acrylic resin, an orthodontic removable appliance can be fabricated with less equipment and within a short period of time.

During the polymerization reaction, the degree of conversion is never complete leading to the presence of unreacted monomer in the polymer network (Fletcher et al., 1983; Vallittu et al., 1995; Lee et al., 2002). Due to their smaller degree of conversion, greater amount of residual monomer can be found in the self-cured than in the heat cured acrylic resin (Peyton, 1975; Lee et al., 2002; Anusavice et al., 2014). The residual monomer in any dental appliances should be eliminated before the delivery visit, as it can leach out to the oral mucosa and cause irritation. Erythema, necrosis, pain, or burning sensation of the oral mucosa has been reported in some patients using an acrylic dental appliance (Nealey and Del Rio, 1969; McCabe and Basker, 1976; Lai et al., 2004; Goncalves et al., 2006; Chaves et al., 2012). In addition, negative effects of the residual monomer on various mechanical and physical properties of acrylic resins have also been reported (Arab et al., 1989; Dogan et al., 1995; Rantala et al., 2003).

To reduce the level of residual monomer, most of manufacturer's instruction suggest to immerse the finished appliance in water for 72 hours before transferring to patients. However, previous study showed that monomer was most released in the first 24 hours of water immersion (Vallittu et al., 1995). Several methods have been introduced in purpose of reducing the time of post-polymerization treatment, including hot water immersion (Tsuchiya et al., 1994; Araújo et al., 2002; Campanha et al., 2006; 2007; 2009; Urban et al., 2010; Bural et al., 2011) and microwave irradiation (Araújo et al., 2002; Campanha et al., 2006; 2007;

Urban et al., 2010). A new method of 50°C water immersion in an ultrasonic bath has been reported to be an effective post-polymerization treatment, due to its reduction of the residual monomer of self-cured MMA-based orthodontic base-plate within a short period of time (Thaitammayanon et al., 2014; Thaitammayanon et al., 2018). However, its effect on the mechanical and physical properties of acrylic resins have not been investigated.

The aim of this study was to investigate the effect of a 50°C water immersion in an ultrasonic bath and conventional monomer reduction methods on some mechanical and physical properties of an MMA-based orthodontic base-plate material, that is, flexural strength, flexural modulus, microhardness, water sorption, and water solubility.

Materials and methods

Twenty-four rectangular and twenty-four disc-shaped specimens were prepared from an MMA-based orthodontic base-plate material (Orthoplast, Vertex-Dental, Soesterberg, The Netherlands) (Table 1). Preparation of the specimens were performed in accordance with the manufacturer's instructions and ISO 20795-2 (2013). The polymer and monomer were mixed in a ratio of 1:2.7 and applied into a stainless steel mold using a spray-on technique. Polymer powder was poured into stainless steel mold and monomer liquid was dropped into the powder until powder texture disappeared, this procedure was repeated until the mold was full. Each mold was placed in a pressure cooker with a 250 kPa pressure with nitrogen atmosphere at 55°C for 20 minutes.

The specimens were wet ground by P500, P1000 and P1200 silicon carbide (SiC) metallographic grinding papers (TOA, Bangkok, Thailand), respectively. A digital caliper (ABSOLUTE Digimatic, Mitutoyo Corporation, Kanagawa, Japan) was used to

confirm whether dimension of each specimen fulfilled the requirement, 64 mm × 10 ± 0.2 mm × 3.3 ± 0.2 mm for a rectangular specimen and 50 ± 1 mm diameter × 0.5 ± 0.1 mm thickness for a disc-shaped specimen. The specimens of each configuration were divided into four groups (Table 2); n=6 for each group. Each specimen was hung in the center of water bath or ultrasonic bath (VGT 1990 QTD, Guangdong GT Ultrasonic Industrial Co., Shenzhen, China, 100 Watts, 40 kHz) when submit to corresponding treatment to ensure that the post-polymerization treatment of each group affect the specimen on all surfaces.

Flexural strength and flexural modulus tests

According to ISO 20795-2 (2013), before 3-point flexural tests, the width and height of the rectangular specimens were measured three times along their long axis at each three equal spaces. They were then stored in 37 ± 1°C water for 50 ± 2 hours. Each specimen was removed from water, immediately laid on a support (50 ± 0.1 mm apart), immersed in a water bath at 37 ± 1°C, and loaded in a universal testing machine (Shimadzu EZ-S, Shimadzu, Kyoto, Japan). The force on the loading plunger was constantly increased with a displacement rate of 5 ± 1 mm/minute until the specimen was fractured. Flexural strength (FS; MPa) and flexural modulus (FM; MPa) were calculated using the following equations:

$$FS = \frac{3Fl}{2bh^2} \text{ and } FM = \frac{F_1l^3}{4bh^3d}$$

Where

- F = the load (N) at fracture
- F₁ = the load (N) at a point in the straight line portion of the load/deflection curve
- l = the distance between supports (mm)
- b = mean of specimen width (mm), measured immediately prior to water storage
- h = mean of specimen height (mm), measured immediately prior to water storage
- d = the deflection (mm) at load F₁

Vickers microhardness test

After the flexural tests, fragments of each broken specimen were collected for a Vickers microhardness (VHN) test using a Vickers hardness tester (Future-tech FM-810, Future-tech, Kanagawa, Japan) under 25 gf (245 mN) load for 30 seconds (Neves et al., 2013). Mean VHN value of each specimen was calculated from five measurements.

Water sorption and water solubility test

Water sorption and water solubility test were performed according to ISO 20795-2 (2013). Disc-shaped specimens were placed in a rack inside a first desiccator containing fresh dried silica gel and

Table 1: Chemical composition of MMA-based orthodontic base-plate materials.

Brand	Components	Compositions	Manufacturer	Batch numbers
Orthoplast (OP)	Powder	Polymethyl methacrylate > 99%, accelerator < 1%, color agents < 1%	Vertex-Dental, Soesterberg, The Netherlands	B4-957
	Liquid	Methyl methacrylate > 95%, ethylene glycol dimethacrylate < 5%		14003860

Table 2: Means and standard deviations of tested mechanical and physical properties.

Group	Treatment		Time	Flexural strength (MPa)	Flexural modulus (MPa)	Microhardness (VHN)	Water sorption (µg/mm ³)	Water solubility (µg/mm ³)
	Water temperature (°C)	Ultrasonic bath						
I	-	-	-	65.36 ± 1.22 ^a	2085.85 ± 12.47 ^a	13.21 ± 0.25 ^a	18.99 ± 0.22 ^a	0.48 ± 0.16 ^a
II	25	-	24 hours	67.27 ± 0.97 ^a	2200.48 ± 16.07 ^c	13.48 ± 0.58 ^a	19.16 ± 0.13 ^a	0.46 ± 0.25 ^a
III	25	-	72 hours	65.25 ± 1.66 ^a	2113.82 ± 27.89 ^b	13.37 ± 0.42 ^a	19.25 ± 0.18 ^a	0.42 ± 0.15 ^a
IV	50	+	10 minutes	69.70 ± 1.44 ^b	2206.25 ± 12.32 ^c	13.62 ± 0.48 ^a	19.28 ± 0.24 ^a	0.51 ± 0.18 ^a

Vertical identical superscript indicates no significant difference.

stored in an oven at 37 ± 1°C for 23 ± 1 hours. The rack was transferred to a second desiccator and kept at 23 ± 1°C for 60 ± 10 minutes. Each specimen was weighed by an analytical balance (Precisa 40SM-200A, Precisa Gravimetrics AG, Zürich, Switzerland) and then returned to the first desiccator in the oven for 23 ± 1 hours. Repeated the process mentioned above until the mass was constant, that is, the mass loss of each specimen did not exceed 0.2 mg. The final mass (m_1) was recorded and the volume (V) of each specimen was calculated from the mean thickness measured five times and from the mean diameter measured three times.

All specimens were immersed in water for 7 days ± 2 hours at 37 ± 1°C. Each of them was then removed from water, dried with a dry towel, waved in the air for 15 ± 1 seconds, and weighed at 60 ± 10 seconds post-removal from water. The mass was recorded and designated as m_2 . The specimens were placed in the first desiccator and processed with the same methods as mentioned for the m_1 measurement. Until the constant mass was reached, the final mass was recorded and designated as m_3 . The m_1 , m_2 and m_3 was calculated into micrograms (µg) and the water sorption (w_{sp}) and water solubility (w_{sl}) were calculated using following equations:

$$w_{sp} = \frac{m_2 - m_3}{V} \quad \text{and} \quad w_{sl} = \frac{m_1 - m_3}{V}$$

Statistical analyses

SPSS for Windows version 22.0 (IBM, New York, NY) was used for statistical analyses. Significant differences in the properties among each group were analyzed with a one-way analysis of variance at a 95% confidence interval, followed by a *post hoc* Tukey's test. The numerical data were reported as means and standard deviations.

Results

Flexural strength and flexural modulus (Table 2)

Group IV specimens presented the highest mean value of flexural strength and flexural modulus, and were significantly higher than other groups ($p < 0.001$). When compared to the controls, the mean values of flexural modulus in Groups II and III were significantly higher ($p < 0.001$ and $p < 0.05$, respectively), without a significant difference in their flexural strength ($p > 0.05$).

Vickers microhardness (Table 2)

When compared to their controls, all experimental groups showed no significant difference in their VHN ($p > 0.05$).

Water sorption and water solubility (Table 2)

When compared to their controls, all experimental groups revealed no significant difference in their water sorption and solubility ($p > 0.05$).

Discussion

The highest amount of residual MMA leaching into water has been shown on the first day (Vallittu et al., 1995); consequently, an immersion of acrylic resin appliance in water should be performed at least 24 hours before delivery to a patient. Water immersion for some long periods of 24 or 72 hours has been reported to effectively reduce the level of residual monomer in an MMA-based orthodontic base-plate material (Thaitammanon et al., 2015).

Ultrasonic treatment was effective for the reduction of the residual monomer level. It has been reported that the treatment could reduce the residual monomer in auto-polymerized acrylic resin and that the treatment with a low frequency of ultrasound was

more effective (Charasseangpaisarn and Wiwatwarrapan, 2015). A reduction of the residual monomer may contribute to the propagation of ultrasonic pressure and cavitation. In addition, an exploding energy from cavitation may cause some further polymerization of the residual monomer.

An improvement in flexural properties of an MMA-based orthodontic base-plate material after an ultrasonic treatment has been revealed in this study and been consistent with that reported previously. An immersion in 55°C water for 10 min has improved the flexural strength of auto-polymerizing hard chairside reline resins (Neves et al., 2013). On the contrary, no improvement in the flexural properties of prosthodontics auto-polymerized acrylic resin has been observed post-immersion in either 50°C water for 60 minutes and 55°C for 10 minutes (Kobnithikulwong and Wiwatwarrapan, 2015).

The residual monomer can cause some deleterious effect on the mechanical and physical properties of acrylic resins, as a result of its plasticizer effect. Post-polymerization treatments have been documented to enhance some mechanical and physical properties of acrylic resin (Urban et al., 2009; Neves et al., 2013; Kobnithikulwong and Wiwatwarrapan, 2015). A water bath immersion as the post-polymerization treatment has been reported to increase both flexural strength and microhardness of hard reline resins (Urban et al., 2009). In the present investigation, the resins VHN remained unchanged after an ultrasonic treatment, despite an increase in their flexural properties. Some difference in the mixing processes may contribute to such discrepant result. When compared to their dough technique, our spray-on method resulted in more porosity, implying some more water migration in the resin. The water in acrylic can act as a plasticizer (Ferracane, 2006; Pedreira et al., 2009), which causes some negative effect on the mechanical and physical properties of acrylic resins. Water immersion before the tests of flexural properties

and microhardness possibly causes a more predominant migration of water on the superficial layer of specimens than in the bulk. Therefore, microhardness shown no improvement while the flexural properties was not affected by plasticizer effect of water.

The effect of post-polymerization treatment on the properties of removable orthodontic appliance resins has been reported (Faltermeier et al., 2007). They used various heat treatment methods to reduce the level of residual monomer and found that the microhardness and water sorption were improved after treatment. Compare to the present study, the microhardness and water sorption did not show an improvement. Although the same type of acrylic resin was used in both studies, the different post polymerization treatment may be the cause of the different results. Using heat treatment induce further polymerization of residual monomer (Eliades et al., 1987; Bural et al., 2011) while using ultrasonic can cause either polymerization or immigration of residual monomer (Charasseangpaisarn and Wiwatwarrapan, 2015). Therefore, using ultrasonic may leave more porosity in polymer network compare to heat treatment. This phenomenon result in water migration as mentioned above.

A positive correlation has been found between residual monomer and water sorption (Dogan et al., 1995). The presence of higher level of residual monomer indicate the higher porosity in polymer mass. This porosity cause the leaching out and replacement of residual monomer by water when immerse in water bath (Braden, 1964; Braden and Wright, 1983; Kalachandra and Turner, 1987; Arab et al., 1989). The present study showed no improvement in water sorption, so the results can be implied that the main action of ultrasonic may act as the promoter of residual monomer migration, which cause unchanged in

porosity, instead of polymerization. When consider the water solubility, which found unchanged in the present study, this result is contrary to previous study which shown the positive correlation between the amount of residual monomer and water solubility (Miettinen et al., 1997; Sideridou et al., 2003; Tuna et al., 2008).

Although the effectiveness of residual monomer reduction should be firstly considered for the post-polymerization treatment, the effect on mechanical and physical properties should not be neglected. In the present study, the mechanical and physical properties of an MMA-based orthodontic base-plate material after treated with ultrasonic bath showed acceptable value when compared to ISO standard. Therefore, ultrasonic treatment may be the favorable method to reduce the level of residual monomer within a short period of time which may be beneficial for the patients who need appliance repairing or before delivering appliances or dentures to the patients in the mobile dental unit practice.

Conclusions

Fifty degree celsius water immersion in ultrasonic bath for 10 minutes showed an improvement of flexural strength and flexural modulus of MMA-based orthodontic base-plate material. However, the microhardness, water sorption and water solubility remained unchanged. While the conventional monomer reduction methods only improved the flexural modulus, but the others were unaltered.

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References

- Anusavice KJ, Shen C, Rawls HR. Phillips' Science of Dental Materials: Elsevier Health Sciences; 2014.
- Arab J, Newton JP, Lloyd CH. The effect of an elevated level of residual monomer on the whitening of a denture base and its physical properties. *J Dent.* 1989;17:189-94.
- Araújo PHH, Sayer C, Giudici R, Poço JGR. Techniques for reducing residual monomer content in polymers: A review. *Polym Eng Sci.* 2002;42:1442-68.
- Braden M. The absorption of water by acrylic resins and other materials. *J Prosthet Dent.* 1964;14:307-16.
- Braden M, Wright PS. Water absorption and water solubility of soft lining materials for acrylic dentures. *J Dent Res.* 1983;62:764-8.
- Bural C, Aktas E, Deniz G, Unlucerci Y, Kizilcan N, Bayraktar G. Effect of post-polymerization heat-treatments on degree of conversion, leaching residual MMA and in vitro cytotoxicity of autopolymerizing acrylic repair resin. *Dent Mater.* 2011;27:1135-43.
- Campanha NH, Pavarina AC, Giampaolo ET, Machado AL, Carlos IZ, Vergani CE. Cytotoxicity of hard chairside reline resins: effect of microwave irradiation and water bath postpolymerization treatments. *Int J Prosthodont.* 2006;19:195-201.
- Charasseangpaisarn T, Wiwatwarrapan C. The effect of various frequencies of ultrasonic cleaner in reducing residual monomer in acrylic resin. *Ultrasonics.* 2015; 63:163-7.
- Chaves CA, Machado AL, Vergani CE, de Souza RF, Giampaolo ET. Cytotoxicity of denture base and hard chairside reline materials: a systematic review. *J Prosthet Dent.* 2012;107:114-27.
- Dogan A, Bek B, Cevik NN, Usanmaz A. The effect of preparation conditions of acrylic denture base materials on the level of residual monomer, mechanical properties and water absorption. *J Dent.* 1995;23:313-8.
- Eliades GC, Vougiouklakis GJ, Caputo AA. Degree of double bond conversion in light-cured composites. *Dent Mater.* 1987;3:19-25.
- Faltermeier A, Rosentritt M, Mussig D. Acrylic removable appliances: comparative evaluation of different postpolymerization methods. *Am J Orthod Dentofacial Orthop.* 2007;131:301 e16-22.
- Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. *Dent Mater.* 2006; 22:211-22.
- Fletcher AM, Purnaveja S, Amin WM, Ritchie GM, Moradians S, Dodd AW. The level of residual monomer in self-curing denture-base materials. *J Dent Res.* 1983;62:118-20.
- Goncalves TS, Morganti MA, Campos LC, Rizzato SM, Menezes LM. Allergy to auto-polymerized acrylic resin in an orthodontic patient. *Am J Orthod Dentofacial Orthop.* 2006;129:431-5.
- International Organization for Standardization. ISO 20795 2: Dentistry base polymers. Part 2: Orthodontic base polymers. Geneva, Switzerland: 2013.
- Kalachandra S, Turner DT. Water sorption of poly-methacrylate networks: Bis-GMA/TEGDM copolymers. *J Biomed Mater Res.* 1987;21:329-38.
- Kobnithikulwong N, Wiwatwarrapan C. Ethanol and ultrasonic wave postpolymerization treatment reduced residual monomers of autopolymerizing acrylic resins and maintained their flexural properties. International Graduate Research Conference 2015; 11 December 2015; Chiang Mai University, THAILAND 2015. p. 151-5.
- Lai YL, Chen YT, Lee SY, Shieh TM, Hung SL. Cytotoxic effects of dental resin liquids on primary gingival fibroblasts and periodontal ligament cells in vitro. *J Oral Rehabil.* 2004;31: 1165-72.

- Lee SY, Lai YL, Hsu TS. Influence of polymerization conditions on monomer elution and microhardness of autopolymerized polymethyl methacrylate resin. *Eur J Oral Sci.* 2002;110:179-83.
- McCabe JF, Basker RM. Tissue sensitivity to acrylic resin. A method of measuring the residual monomer content and its clinical application. *Br Dent J.* 1976;140:347-50.
- Miettinen VM, Vallittu PK, Dozent DT. Water sorption and solubility of glass fiber-reinforced denture polymethyl methacrylate resin. *J Prosthet Dent.* 1997;77:531-4.
- ealey ET, Del Rio CE. Stomatitis venenata: reaction of a patient to acrylic resin. *J Prosthet Dent.* 1969; 21:480-4.
- Neves CB, Lopes LP, Ferrao HF, Miranda JP, Castro MF, Bettencourt AF. Ethanol postpolymerization treatment for improving the biocompatibility of acrylic reline resins. *Biomed Res Int.* 2013;2013:485246.
- Pedreira APRV, Pegoraro LF, de Góes MF, Pegoraro TA, Carvalho RM. Microhardness of resin cements in the intraradicular environment: Effects of water storage and softening treatment. *Dent Mater.* 2009;25:868-76.
- Peyton FA. History of resins in dentistry. *Dent Clin North Am.* 1975;19:211-22.
- Rantala LI, Lastumaki TM, Peltomaki T, Vallittu PK. Fatigue resistance of removable orthodontic appliance reinforced with glass fibre weave. *J Oral Rehabil.* 2003;30:501-6.
- Sideridou I, Tserki V, Papanastasiou G. Study of water sorption, solubility and modulus of elasticity of light-cured dimethacrylate-based dental resins. *Biomaterials.* 2003;24:655-65.
- Thaitammayanon P, Sirichompun C, Wiwatwarrapan C. Comparison of residual monomer in the MMA-based orthodontic base-plate materials before and after water immersion. *CU Dent J.* 2015;38 (Suppl):67-74.
- Thaitammayanon P, Sirichompun C, Wiwatwarrapan C. Ultrasonic treatment reduced residual monomer in methyl methacrylate-based orthodontic base-plate materials. *Dent Oral Craniofac Res.* 2018;4:1-5.
- Thaitammayanon P, Sirichompun C, Wiwatwarrapan C. Residual monomer reduction in the MMA-based orthodontic base plate materials by water immersion in ultrasonic bath [master's thesis]. Bangkok: Chulalongkorn University; 2014.
- Tsuchiya H, Hoshino Y, Tajima K, Takagi N. Leaching and cytotoxicity of formaldehyde and methyl methacrylate from acrylic resin denture base materials. *J Prosthet Dent.* 1994;71:618-24.
- Tuna SH, Keyf F, Gumus HO, Uzun C. The evaluation of water sorption/solubility on various acrylic resins. *Eur J Dent.* 2008;2:191-7.
- Urban VM, Machado AL, Alves MO, Maciel AP, Vergani CE, Leite ER. Glass transition temperature of hard chairside reline materials after post-polymerisation treatments. *Gerodontology.* 2010; 27:230-5.
- Urban VM, Machado AL, Oliveira RV, Vergani CE, Pavarina AC, Cass QB. Residual monomer of reline acrylic resins. Effect of water-bath and microwave post-polymerization treatments. *Dent Mater.* 2007;23:363-8.
- Urban VM, Machado AL, Vergani CE, Giampaolo ET, Pavarina AC, de Almeida FG, et al. Effect of water-bath post-polymerization on the mechanical properties, degree of conversion, and leaching of residual compounds of hard chairside reline resins. *Dent Mater.* 2009;25:662-71.
- Vallittu PK, Miettinen V, Alakuijala P. Residual monomer content and its release into water from denture base materials. *Dent Mater.* 1995;11: 338-42.