Effect of Post-Polymerization Microwave Treatment on Mechanical Properties and Dimensional Change of Provisional Self-Cure PMMA

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Abstract

This study evaluated the effect of post-polymerization microwave treatment in dry and wet conditions on the microhardness, flexural strength and dimensional change of PMMA provisional restoration. For all tested parameters, 10 specimens were prepared for 4 groups: 1) Self-cure polymerized PMMA without post-polymerization treatment (Control), 2) Self-cure polymerized PMMA with post-polymerization microwave treatment in dry conditions, 3) Self-cure polymerized PMMA with post-polymerization microwave treatment in wet conditions, and 4) Heat-polymerized PMMA. Specimens were stored 48 hours before testing each property.

The results indicated that post-polymerization microwave irradiation could improve the hardness but not flexural strength of PMMA provisional restoration materials. Post-polymerization with microwave irradiation in wet condition provided the hardness similar to heat-polymerized PMMA, and this group exhibited the highest dimensional change.

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Introduction

The purposes of provisional restorations are for pulp protection, maintaining position of the crown, diagnostic and treatment planning in full mouth rehabilitation cases, designing occlusion, restoring new vertical dimensions, and also for maintaining gingiva contours. Provisional restoration plays an important role in fixed restoration cases, especially in full mouth rehabilitation cases that use provisional restoration for determining function and esthetics before restoring final restorations. Therefore, provisional restoration should resist fractures, have dimensional stability, resist wear, minimize marginal gaps, and resist color change.[1,2]

The most preferable material for provisional restoration is Poly(methyl methacrylate) (PMMA), which is able to be cured in self-cure or heat-cure mode. Heat-cure PMMA attained better physical and mechanical properties compared to the self-cure PMMA, however, it required more time and complicated procedures for fabrication. On the other hand, self-cure PMMA is easier to use and requires a shorter time for setting. Therefore, the self-cure PMMA is more often used, although the properties are inferior to the heat-cure due to the lower degree of conversion. Self-cure PMMA provisional restoration is suitable for short term wearing or where only a few crowns are restored. Nevertheless, full mouth rehabilitation patients have to use provisional restorations long term, and these patients usually have a high bite force. Therefore, the use of conventional self-cure PMMA may not be recommended. Due to the ease of manipulation and short setting time of self-cure PMMA, many studies attempted to improve the mechanical properties of this self-cure material for use as long term provisional restorations or in patients with high masticatory force.

The study of Ruyter *et al*.[3] found that all PMMA materials had residual monomers. The heat-polymerized materials obtained the lowest content of residual monomers. The residual monomers affected the mechanical properties of the polymer.[4,5] Another study by Urban *et al*.[6] compared the residual monomer in hard chair-side reline resin after a water bath and microwave treatment. They found that residual monomers decreased in all treatment groups.

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The water bath treatment method took 10-60 minutes, while the microwave treatment only 3-5 minutes.

Some previous studies found that the flexural strength of the post-polymerization microwave treated hard chair-side reline resin had improved.[7-9] Blagojevic and Murphy[7] indicated that post-polymerization microwave treatment with 600W for 3 minutes not only reduced the residual monomer but also increased the impact strength of the materials. Pavarina *et al*.[10] used microwave irradiation at 650W for 6 minutes for disinfection of hard chair-side reline resin. The specimens were immersed in water and irradiated in a microwave. Besides the disinfection effect, the flexural strength was found to be improved.

For self-cure PMMA provisional restoration material, there is only one study of Ozkomur and Fortes[11] that tested the mechanical properties of two types of self-cure PMMA after post-polymerization microwave treatment without water immersion. It was found that the properties of these two materials improved. It has been noted that the storage conditions in various studies were different. Some studies immerse the PMMA specimens in water, whereas some kept the specimens dry during microwave irradiation. This may affect the properties of the materials in different ways. No study has investigated the effect of microwave treatment conditions on the properties of PMMA materials. Moreover, even with provisional restoration, dimensional stability is the important property of concern. Large size marginal gaps of provisional restorations may lead to easy loss of retention or leakage of the restoration. The prepared provisional restorations should not alter in dimension after fabrication. Ozkomur and Fortes[11] additionally suggested that besides the mechanical properties, the dimensional change of the post-polymerization microwave irradiated PMMA should be investigated further.

The objectives of this study were to evaluate the effect of post-polymerization microwave treatment in dry and wet conditions on the microhardness, flexural strength and dimensional change of PMMA provisional restoration.

Materials and methods

Two polymerization modes, self-cured (Self-cure dentine, Major Prodotti Dentari S.p.a., Moncalieri (TO), Italy) and heat-cured (Major C&B dentine, Major Prodotti Dentari S.p.a., Moncalieri (TO), Italy) PMMA provisional restorative materials were used. The materials were measured and mixed following the manufacturer's instruction. The specimens were prepared in the specific shape for microhardness, flexural strength and dimensional change. For all tested parameters, 10 specimens were prepared for each of the following groups: 1) Self-cure polymerized PMMA without post-polymerization treatment (Control), 2) Self-cure polymerized PMMA with post-polymerization microwave treatment in dry conditions, 3) Self-cure polymerized PMMA with post-polymerization microwave treatment in wet conditions and 4) Heat-polymerized PMMA.

In group 1, the specimens were left for chemical polymerization for 10 minutes before removing from the mold. In group 2, after leaving specimens for chemical polymerization for 10 minutes, the specimens were placed in a microwave (Samsung, ME711K, Samsung Electronics Co.,Ltd., Malaysia) at 600W power for 3 minutes without water immersion (dry condition). In group 3, the specimens were treated the same as group 2 but they were immersed in 100ml distilled water during irradiation (wet condition). In group 4, the mixture of heat-cure PMMA was injected into the mold and then placed into a water bath at 74°C for 8 hours (heat-polymerization). All specimens were examined for defects and were discarded if any defects were observed. The samples in all groups were stored for 48 hours before testing.

Microhardness measurement

Thirty self-cure PMMA 9 mm-high and 3 mm-thick cylindrical blocks were fabricated by silicone molds (Figure 1). After mixing, the material was injected into the molds and pressed with a glass slide and held for 10 minutes for curing. For heat-cure PMMA, the same geometry specimens as for self-curing was fabricated using a type IV die stone mold (Atlas, Boral Prestia Co.,Ltd., Chonburi, Thailand), which was then poured into a small brass flask. The PMMA mixture was injected into the mold, the flask tightened with a screw, and then cured in the water bath at 74°C for 8 hours. After removal, the specimens were submitted to polishing with 600 and 1200 grit silicon carbide paper using rotary grinding machines (Buehler, Lake Bluff, Illinois,

USA) and then finished with 6 and 1 µm diamond paste (Buehler, Lake Bluff, Illinois, USA). After 48 hours storage in water, Vicker hardness was measured using a microhardness testing machine (Mitutoyo, HM-211, Mitutoyo Corporation, Kanagawa, Japan) with a 10 g load for a dwell time of 15 s. Indentations were made at 5 points on the top surface of each specimen: at the center and 2.5 mm from the center peripherally in four directions. The length of the diagonals (µm) of each indentation was measured under 50X magnification. The Vicker Hardness Number (VHN) was calculated and recorded.

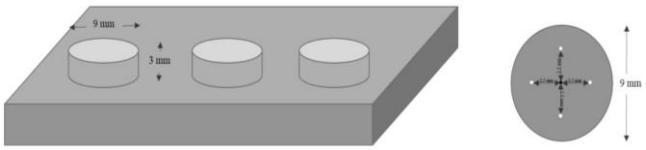

Figure 1. Diagram showing silicone mold for cylinder-shape specimen and measurement points for microhardness test.

Flexural Strength measurement

Thirty self-cure PMMA bar-shaped specimens (2x2x25 mm) as recommended by ADA specification no.27 were fabricated using stainless steel split molds (Figure 2). The mixture of the material was injected into the mold tightly with screws and held for 10 minutes for chemical polymerization. For heat-cure PMMA, 10 bars were fabricated by the same stainless steel molds. After mixing, the material was injected into the mold and tightened. The molding device was then placed in a water bath at 74°C for 8 hours. After removal from the mold, all specimens were observed for the presence of defects and the flawed specimens were excluded. The accuracy of the bar size was verified with a digital micrometer (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan) to ensure the size was not more than ±0.05 mm deviation.

After 48-hours dry storage, the specimens were subjected to a three-point bending test in a universal testing machine (Lloyd instruments, LRX-Plus, AMETEK Lloyd Instrument Ltd., Hamphshire, UK) with a crosshead speed of 0.75 mm/min. The maximum fracture load (N) of each bar was recorded and the flexural strength in MPa was calculated as follows:

$\sigma=3FL/2wd^2$

F is the load (force) at the fracture point (N)

L is the length of the support span

w is width

d is thickness

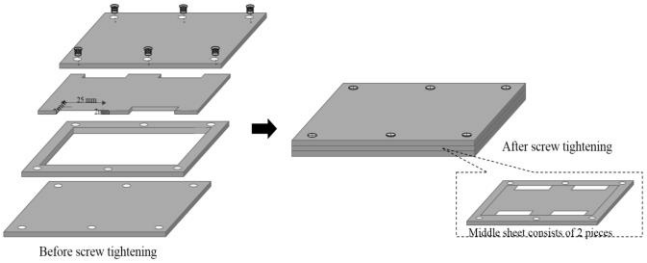

Figure 2. Diagram showing stainless steel mold for bar-shape specimen for flexural strength test.

Dimensional Change

Thirty self-cured and 10 heat-cured disc-shaped specimens, 1 mm in thickness and 30 mm in diameter, were fabricated using a stainless steel mold. The mold composed of 2 parts, a 21-mm high outer ring and a 20-mm high inner metal cylindrical block. On the top surface of the block, the inner circular groove was present with two perpendicular diameter lines 25 mm in length (AB and CD) as shown in (figure 3). For the self-cure formula, the mixture of PMMA was injected into the mold, pressed with a glass slide and held for 10 minutes for curing before removing. For the heat-cure formula, ten discs were fabricated with the same mold and same method except for the curing procedure. After covering with a glass slide, C clamps were used to hold the mold and the specimens were then cured in a water bath at 74°C for 8 hours.

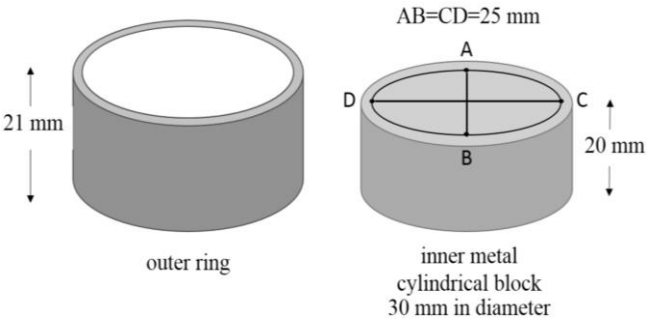

Figure 3. Diagram showing stainless steel mold for disc-shaped specimen and measurement points for dimensional change.

After removing from the mold, the specimens in group 2 and 3 suddenly received post-polymerization microwave treatment. All disc-shaped specimens were subjected to dimensional measurement after removing from

the mold and after dry storage for 48 hours. The perpendicular diameter on the resin, AB and CD, was measured using a measuring microscope (Nikon, MM-400, Nikon Corporation, Tokyo, Japan). The average of AB and CD distance was calculated. Linear dimensional change was identified as the difference between the two tested periods.

Statistical analyses

The collected data were analyzed for normality distribution using the Kolmogorov-Smirnov Test and homogeneity of variance using Levene's test. Normal distribution of the data was indicated, therefore, One-way ANOVA was used to test the significant difference of means for all 3 tested properties. Since Levene's test indicated significant non-homogeneity among the variances of dimensional change data, Dunnett's T3 was therefore used as a post-hoc for multiple comparisons. For VHN and flexural strength data, Tukey's HSD was used for multiple comparison. All statistical testing was performed at a 95% level of confidence.

Results

Vicker Hardness Number (VHN), flexural strength (MPa) and dimensional change (μm) are present in Table 1. One-way ANOVA revealed that there were significant differences of means for at least one pair of the tested groups in all properties (P<0.05).

VHN ranged from 15.18–19.10. The results indicated that hardness of all experimental groups were significantly higher than the control group (P<0.05). Post-polymerization with microwave irradiation in wet conditions and heat-polymerization groups provided the same value of hardness number (P>0.05), 19.10 and 19.08, respectively, and these two groups exhibited higher VHN than that of the group treated with microwave irradiation in dry conditions (16.81).

The flexural strength data showed that heat-polymerization PMMA obtained the highest strength (97.19 MPa) (P<0.05). The flexural strength of the control, microwave irradiation in dry conditions and microwave irradiation in the wet condition group were 82.73, 82.78 and 77.26 MPa, respectively. There were no significant differences among these 3 groups (P>0.05).

For dimensional change testing, the results revealed that all tested groups exhibited shrinkage of the specimens after 48 hours. Heat-polymerized PMMA had a dimensional change of only 5.79 μm in average. The highest shrinkage occurred in the group treated with microwave irradiation with wet conditions (78.86 μm). The self-cure PMMA in the control group shrank to the same level as the microwave irradiation in dry conditions (P>0.05), 52.40 and 53.19 μm, respectively.

	Hardness (VHN)	Flexural strength (MPa)	Dimensional change (μm)
Control	15.18 (0.45)^{A}	82.73 (9.34)^{A}	52.40 (9.06)^{A}
Microwave - dry condition	16.81 (0.61)^{B}	82.78 (7.99)^{A}	53.19 (8.14)^{A}
Microwave - wet condition	19.10 (0.42)^{C}	77.26 (7.87)^{A}	78.86 (10.84)^{B}
Heat-polymerization	19.08 (0.11)^{C}	97.19 (9.15)^{B}	5.79 (1.23)^{C}

Table 1. Mean (SD) of Hardness in VHN, the flexural strength in MPa, and dimensional change in μm.

The same superscripts in each column indicate no significant difference (p>0.05).

Discussion

The study was conducted to search for a method to improve mechanical properties of self-cure PMMA provisional restorative materials without adversely altering the dimensions of the restoration after treatment. Heat-cure PMMA from the same manufacturer was used to be a positive control to ensure that the basic formula of the resin was the same as self-cure except for the polymerization system. Heat-cure PMMA was found to be more completely polymerized than self-cure PMMA. If the self-cure PMMA is improved and provides the hardness and strength comparable to the heat-cure PMMA, self-cure materials can therefore be accepted as long-term provisional restoration materials.

Hardness is the property that relates to the degree of conversion of resin materials. Many studies have measured the hardness to define the level of conversion degree of the polymer. In this experiment, the specimens for hardness testing were stored in distilled water for 48 hours before measurement to imitate oral conditions and wait for complete polymerization. Vallittu *et al*.[12] suggested that the amount of released monomers can be diminished when immersed in

water. The results from the study of Oliveira *et al*.[13] revealed that provisional restoration stored in water did not change hardness values of the materials when compared to the control group. From the results of this study, the surface hardness of the self-cure PMMA provisional restorative materials were found to improve when they received post-polymerization microwave irradiation, both in dry and wet conditions. The present results are in agreement with a previous study which found that microwave irradiation could improve the hardness of PMMA.[11] Microhardness is regularly used to predict the degree of conversion of the polymer materials due to the strong relation between hardness values and the percentage of polymerization degree.[14] In this study, further polymerization might be continued by microwave irradiation. Polarized MMA molecules were induced to align and vibrate in an electric field causing molecules to flip over rapidly generating heat, as the hardness of both post-polymerization groups increased when compared to the control. Especially in the wet condition group, the hardness was highly comparable to the heat-polymerization group. During microwave irradiation in the wet conditions, the high temperature of the water enhanced the diffusion of the remaining residual monomer molecules to the active sites of the polymer chains, thus resulting in further polymerization.

Regarding flexural strength testing, the protocol of storage conditions before testing was adjusted differently from the hardness testing. The specimens were kept dry at room temperature before testing. From our pilot study, it was found that the specimen size changed after immersion in water for 48 hours. The magnitude of dimensional change seemed to be dependent upon the porosity of the specimens. Several studies[15-17] have proved that PMMA has high water absorption. To control the effect of water, which was various in each specimen, the specimens were therefore stored in dry conditions before three-point bending testing. The results indicated that flexural strength of all self-cure PMMA provisional restorative material groups were significantly different from the heat-polymerized PMMA group. There was no significant difference in flexural strength among self-cure PMMA groups. The highest flexural strength attained from heat-polymerized PMMA might have resulted from a long slow curing process that allowed air bubbles to release and form more completed polymerization causing less flaws and porosities inside the specimens.

The flexural strength appeared to have no correlation with hardness values. The group of self-cure PMMA which obtained the highest hardness number (microwave irradiation in wet conditions) did not provide the highest flexural strength. On the other hand, the average flexural strength values of this group (77.26) was lower than those of other self-cure PMMA groups, although no significant difference was indicated. The flexural strength was assumed to be reliant on the flaws and water absorption inside the specimens. Some previous studies found that the strength of the polymer restorative materials did not relate to the hardness.[18,19] Flaws and defects of the specimens greatly affected the strength of the materials. The self-cure PMMA powder and liquid were mixed by hand, therefore entrapping air bubbles inside the mixture. All specimens were initially left for chemical polymerization for 10 minutes at room temperature. Porosities occurred and were restrained inside the specimens. Post-polymerization microwave irradiation might not be effective enough to reduce the defects in the specimens. Even though the specimens were visually examined before testing and the specimens with large size porosity at the outer surface were excluded, porosities inside the PMMA bulk might have initiated the cracks and failures.

Post-polymerization microwave irradiation in the wet condition group tended to have the lowest flexural strength values, although they obtained the highest hardness among the three self-cure PMMA groups. This might be due to the effect of water. Thompson *et al*.[19] studied the effects of the storage medium on maximum flexure stress and found that flexural strength values were greater when stored in air than in water. Water enters the polymer network through porosity and intermolecular spaces. The effect of water is to elute unreacted components and to reduce interchain interactions, as a plasticizing agent[20], which can effect mechanical properties of polymers.

All tested specimens exhibited shrinkage after 48 hours. Shrinkage of the polymer materials basically corresponded to the degree of conversion. The more polymerization takes place, the more shrinkage appears. In this study, dimensional change of the PMMA disc was

measured to evaluate the effect of post-polymerization microwave procedures on the size of the specimens. Unfortunately, heat-cure PMMA discs could not be measured before polymerization because the uncured dough material was too soft to measure. Measurement was performed only after the heat-polymerization process finished and again 48 hours later. The scale of dimensional change was very insignificant because most of the polymerization was completed after heat curing. Only 5.79 μm shrinkage was detected, which might be responsible for dry storage conditions and evaporation of the water content.

The self-cure PMMA groups were left for 10 minute-chemical polymerizations before the first measurement was performed. After microwave irradiation and dry storage for 48 hours, the specimen size was measured once more. The magnitudes of dimensional change were 52.40, 53.19, and 78.86 μm for the control, microwave-dry and microwave-wet conditions, respectively. The amount of dimensional change appeared to relate to the degree of polymerization. Post-polymerization microwave irradiation in wet conditions, which provided the highest hardness value, exhibited highest dimensional change. As mentioned above, water molecules were the medium heated up by microwaves, enhancing the diffusion of remaining residual monomer molecules to the active sites of the polymer chains impacting a polymerization reaction. In the dry condition group, the specimens showed shrinkage at the same level as the control group, which was significantly less than that of the wet condition group. A volumetric shrinkage of approximately 21% occurred during the polymerization of pure methylmethacrylate.[21] In this study, specimens shrank 52–79 μm or only about 0.2–0.3% when compared to the original size. Current ISO standards require a film thickness at the time of seating of no greater than 25 μm for water-based luting cements.[22] However, the film thickness for provisional restoration was variable depending upon the materials, and it was higher than the permanent luting cement.[23,24] Moreover, this study used two-dimension experimental specimens and the specimens were very large (30 mm in diameter) to expressively notify any change. The dimensional changes in all groups were less than 100 μm. This might be acceptable for provisional restoration. The real form of provisional restoration may present less shrinkage, and the clinical dimensional change after using post-polymerization microwave irradiation should be further evaluated.

From the results of this study, post-polymerization microwave irradiation at 600W power for 3 minutes in wet conditions may be suggested for use in clinical practice to fabricate harder PMMA provisional restorations for long term usage. Significant higher hardness could be achieved at the same level as heat-polymerized material although higher flexural strength may not be attained. The dimensional change after microwave irradiation seemed to be acceptable for provisional restoration.

Conclusions

Within the limitations of this study, it can be concluded that post-polymerization microwave irradiation could improve the hardness of PMMA provisional restorations. Microwave irradiation in wet conditions provided hardness comparable to heat-cure PMMA. Flexural strength of self-cure PMMA provisional restorations was inferior to that of heat-cure PMMA. Post-polymerization microwave irradiation did not improve the flexural strength of the materials. Post-polymerization microwave irradiation in wet conditions generated the highest dimensional change on the PMMA disc, but it might be an acceptable level for provisional restoration.

Declaration of Interest

There is no conflict of interest in this study.

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