

# Microwave Disinfection: Cumulative Effect of Different Power Levels on Physical Properties of Denture Base Resins

Plinio M. Senna, DDS, MSc,<sup>1</sup> Wander Jose Da Silva, DDS, MSc, PhD,<sup>1</sup> Fernanda Faot, DDS, MSc, PhD,<sup>2</sup> & Altair Antoninha Del Bel Cury, DDS, MSc, PhD<sup>1</sup>

<sup>1</sup> Department of Prosthodontics and Periodontology, Piracicaba Dental School, State University of Campinas, Piracicaba, Brazil
<sup>2</sup> Department of Prosthodontics, School of Dentistry, Federal University of Pelotas, Brazil

### Keywords

Microwaves; disinfection; dentures; acrylic resin.

#### Correspondence

Plinio M. Senna, Piracicaba Dental School—Prosthodontics and Periodontology, Av. Limeira, 901 PO Box 52, Piracicaba São Paulo 13414-903, Brazil. E-mail: psenna3@fop.unicamp.br

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### Abstract

**Purpose:** This study evaluated the cumulative effects of different microwave power levels on the physical properties of two poly(methylmethacrylate) (PMMA) denture base resins.

Materials and Methods: Eight sets of four PMMA specimens each (two polymerized in a water bath and two using microwave energy) were immersed in beakers containing 200 ml of distilled water. Each beaker was subjected to microwave irradiation for 3 minutes at a power level of 450,630, or 900 W. The surface roughness, surface hardness, linear stability, flexural strength, elastic modulus, impact strength, and fractographic properties were evaluated after either 6 or 36 simulated disinfection cycles. The data were statistically analyzed using ANOVA and the Tukey post hoc test ( $\alpha = 0.05$ ). **Results:** The polymerization method did not influence any property (p > 0.05) except linear stability. The surface roughness (p < 0.001) and hardness (p = 0.011) increased after 36 irradiation cycles at 630 or 900 W. The resin polymerized using microwave energy exhibited greater linear distortion (p = 0.012), and there was a cumulative effect on linear stability for both resins (p < 0.001). No significant change (p > 0.05) was observed in flexural strength; however, the elastic modulus decreased (p = 0.008) after 36 disinfection cycles. The impact strength and crack propagation angles displayed no significant differences (p > 0.05). **Conclusion:** Within the limitations of this study, it can be concluded that microwave disinfection at 450 W to 630 W for 3 minutes is safe for PMMA.

Due to the potential for the development of denture stomatitis,<sup>1</sup> the presence of *Candida* spp. biofilms on dentures must be controlled. Microwave energy has been proposed as an effective, clean, and low-cost disinfection method.<sup>2,3</sup> However, the effectiveness of this procedure is maximized when the denture is immersed in water,<sup>4</sup> and this may expose the resin to high temperatures. Temperatures exceeding 71°C may cause distortion of the PMMA polymer matrix due to relaxation of internal stresses acquired during polymerization.<sup>5</sup>

The effect of microwave irradiation and the resulting increase in temperature on the microstructure and physical properties of PMMA resin is still unclear, since there are no standard irradiation protocols and assessment methodologies in the literature.<sup>6</sup> The parameters affecting the final temperature of the water are the water volume, exposure time, and irradiation power. This study employed a 200 ml volume, as this is sufficient to submerge a complete denture.<sup>7,8</sup> Exposure time has been defined as the shortest time required for an effective disinfection. A 3 minute exposure at 450 W to 650 W promotes disinfection<sup>4,9</sup> while minimizing the polymer shrinkage observed after 6 minutes of exposure.<sup>6,8</sup>

Regarding irradiation power, Thomas and Webb<sup>10</sup> reported that lower power levels should be harmless to the resins due to the exposure to lower final temperatures; however, it has not been conclusively determined whether varying this parameter produces different effects on the physical properties of the PMMA resin.<sup>9</sup> It has also not yet been determined whether PMMA resin specifically formulated for microwave polymerization is more resistant to irradiation than PMMA polymerized using the conventional water-bath method. Furthermore, available studies have not taken into consideration the fact that the disinfection is performed several times, and the cumulative effect of irradiation on the resin properties has been neglected.

In light of the need to establish a safe and effective home denture disinfection protocol that does not affect biomechanical performance, the aim of this study was to evaluate the cumulative effect of different microwave power levels on physical properties of two PMMA resins. The surface roughness, surface hardness, linear stability, flexural strength, elastic modulus, impact strength, and fractographic properties were evaluated after 6 and 36 simulated microwave disinfection cycles.

# **Materials and methods**

### **Experimental design**

This in vitro study was randomized and blinded with respect to microwave irradiation power, with substrate type (microwave or hot water bath polymerization) and number of disinfection cycles as factors. Variables included surface roughness, surface hardness, linear stability, flexural strength, elastic modulus, impact strength, and fracture propagation angle.

Two PMMA resins, one polymerized in a water bath, the other using microwave energy, were used to fabricate specimens according to the manufacturer's instructions. Disks (30 mm diameter  $\times$  5 mm thick) were used for evaluation of the surface roughness and hardness, and rectangular specimens measuring 64  $\times$  10  $\times$  3.3 mm<sup>3</sup> (in accordance with ISO 1567:1999<sup>11</sup>) were fabricated for use in linear stability, flexural strength, and elastic modulus evaluations. Rectangular specimens measuring 65  $\times$  10  $\times$  2.5 mm<sup>3</sup> were fabricated for impact strength testing and fractographic analysis.<sup>12</sup>

Four specimens (two of each resin) were immersed in 200 ml of distilled water in each of eight beakers. Each beaker was subjected individually to microwave irradiation for 3 minutes at 450, 630, or 900 W (Fig 1). A set of control specimens was not irradiated. The mean of the two specimens in each beaker was calculated for each resin, leading to a sample size of 8 for each resin in each property. The sample size was selected based on preliminary tests, which demonstrated an adequate power (80%) for detecting statistically significant differences. The irradiation process was repeated 6 or 36 times to simulate cleaning three times weekly for 15 or 90 days. One set of eight beakers was irradiated for 1, 2, and 3 minutes, and the water temperature was measured with a digital thermometer for correlation with the mechanical results.

### **Specimen preparation**

The specimens were prepared according to the manufacturer's instructions at room temperature  $(25 \pm 1^{\circ}\text{C} \text{ and } 50 \pm 5\%$  relative humidity). For each specimen, metal master patterns were individually invested with high-viscosity silicone (Zetalabor; Zhermack S.p.A, Badia Polesine, Rovigo, Italy) in a plastic flask for microwave-polymerized PMMA resin (OndaCryl, Classico Artigos Odontológicos Ltda, São Paulo, Brazil) or a metallic flask for heat-water-polymerized PMMA resin (Lucitone 550, Dentsply International Inc., York, PA). The PMMA resins were mixed in accordance with the manufacturer's instructions and packed into the silicone molds at the doughy stage.

The polymerization of the hot-water resin was performed by placing the metallic flask in a polymerization unit (Termotron P-100; Termotron Equipamentos Ltd, Piracicaba, Brazil) filled with water at 74°C for 9 hours. For microwave polymerization, the plastic flask was placed inside a domestic microwave oven

at 2450 MHz/ 900 W (AW-42; Continental, Manaus, Brazil). The material was irradiated for 3 minutes at 360 W, allowed to stand for 4 minutes, then irradiated for another 3 minutes at 810 W. The flasks were bench cooled for 2 hours.

The specimens were removed from the flasks, trimmed, and finished using abrasive paper (320, 400, and 600 grit, Carbimet; Buehler, Lake Bluff, IL) in a polishing machine (Model APL-4; Arotec, São Paulo, Brazil). Specimens intended for surface hardness testing received an additional polish using 1200-grit abrasive paper followed by 1 and 3  $\mu$ m diamond paste (Extec Corp, Enfield, CT) on a cotton disk (Extec Corp.). The specimens were ultrasonically cleaned (Thornton T 740, Thornton-Inpec Eletrônica LTDA, Vinhedo, Brazil) for 20 minutes, then immersed in distilled water at 37°C for 48 ± 2 hours for monomer release before testing.<sup>13</sup>

### Surface roughness test

The surface roughness was measured using a profilometer (Surfcorder SE1700; Kosaka Laboratory Ltd., Tokyo, Japan) with a resolution of 0.01 mm, calibrated at a specimen length of 0.8 mm, 2.4 mm percussion of measure, and stylus velocity of 0.5 mm/s. Three readings were obtained from each specimen, and the mean value was calculated.<sup>14</sup>

### Surface hardness test

The surface hardness of the resin discs was measured using a 25 g load for 5 seconds in a microhardness tester (Shimadzu HMV-2000, Kyoto, Japan) equipped with a Knoop indenter. Five indentations 100  $\mu$ m apart were made on each specimen, and the results were averaged to obtain the hardness value for the specimen (kg/mm<sup>2</sup>).<sup>15</sup>

# Linear stability, flexural strength, and elastic modulus tests

The same specimens were used to determine these three parameters. The linear stability was evaluated first, followed by flexural strength. The elastic modulus was obtained from the results of the flexural strength test.

Five linear measurements were performed over the length of the specimen, and the average was calculated. The measurements were performed using an optical microscope at a magnification of  $120 \times (UHL VMM-100$ -BT; Renishaw, Gloucestershire, UK) connected to digital measurement equipment (Quadra-Chek 200; Metronics Inc., Mississauga, Canada) with a resolution of 0.001 mm. The linear distortion was calculated from the ratio between the initial length and measurements obtained after irradiation (6 or 36 cycles) for each specimen and expressed as a percentage.<sup>16</sup>

The flexural strength (MPa) and elastic modulus (MPa) were determined using a three-point bending test in a universal testing machine (Instron Model 4467, Instron Industrial Products, Grove City, PA) calibrated with a 500 kgf load cell. The crosshead speed was 5 mm/min. The flexural testing device consisted of a central loading plunger and two polished cylindrical supports (3.2 mm diameter, 10.5 mm long). The distance between the centers of the supports was 50 mm. The compressive force was applied perpendicular to the center of the



Figure 1 Experimental design.

specimens until there was a deviation in the load-deflection curve and specimen fracture occurred.<sup>11</sup>

### Impact strength test and fractographic analysis

Impact strength tests were performed using an impact testing machine (AIC – EMIC, São José dos Pinhais, Brazil) employing the Charpy method. The specimens were horizontally positioned with a distance of 40 mm between the two fixed supports and broken by a pendulum with an energy of 0.5 J.<sup>12</sup>

After impact testing, the fractures of the broken specimens were classified as brittle or ductile by visual inspection in accordance with the looseness of the specimen material. The fragments were analyzed using a stereomicroscope (Leica MZ 6, Leica Geosystems, Heerbrugg, Switzerland) at  $40 \times$  magnification to identify the fracture origin. The fracture interface of both specimen fragments were recorded using a digital camera (SCC-131, Samsung; Seoul, South Korea) to determine the angle of fracture propagation using AUTOCAD 2010 software (AutoDesk, Inc., San Rafael, CA).<sup>12</sup>

### **Microwave irradiation**

To account for loss in potency after the initial use,<sup>17</sup> the microwave oven was preheated before the start of its use by microwaving 1 l of distilled water for 2 minutes at maximum power. Each beaker was placed on the center of the carousel and irradiated for 6 or 36 3-minute cycles at one of the three power levels tested. After each cycle, the specimens were immediately washed with room temperature tap water  $(23 \pm 1^{\circ}C)$  and stored at  $95 \pm 5\%$  relative humidity for 2 hours before the next irradiation cycle.

### **Statistical analysis**

Analyses were performed using SAS software (SAS Institute Inc., version 9.0, Cary, NC) at a 5% level of significance. The normality of error distribution and the degree of nonconstant variance were verified for the means of the mechanical tests. The resin polymerization method effects were analyzed using one-way ANOVA. Surface roughness and hardness data were evaluated using ANOVA for repeated measures.



Figure 2 Surface roughness (mean ± SD) of PMMA specimens after microwave disinfection cycles by different power levels (n = 8).

Two-way ANOVA was performed to analyze flexural and impact strength considering power and number of cycles as study factors. Tukey's HSD was used for post hoc ANOVA analysis.

### Results

No resin properties except linear stability were affected by the method of polymerization (p > 0.05). The surface roughness of specimens submitted to 36 cycles at 630 or 900 W exhibited increased roughness compared with those irradiated at 450 W (p < 0.001; Fig 2). The surface hardness (p = 0.011) increased after 36 irradiation cycles at 630 or 900 W (p = 0.011; Table 1).

The polymerization method influenced the linear stability of the resins, with the resin polymerized using microwave energy experiencing greater linear distortion (p = 0.012). Regardless of the polymerization method, the 900 W power level produced greater linear distortion (p < 0.001). There was also a significant

**Table 1** Surface hardness (mean  $\pm$  SD) of PMMA specimens (n = 8) arranged by power level and number of microwave disinfection cycles

		Knoop hardness				
Polymerization method	Power (W)	Baseline	6 cycles	36 cycles		
Water bath	450	19.4 ± 1.4 (Aa)	18.6 ± 0.9 (Aa)	19.3 ± 0.6 (Aa)		
	630	$19.9 \pm 1.2$ (Aa)	$19.8 \pm 1.2$ (ABa)	$19.9 \pm 0.7$ (Aa)		
	900	$19.7 \pm 0.7$ (Aa)	$19.5 \pm 0.3$ (Ba)	$20.7 \pm 0.8$ (Ba)		
Microwave	450	$18.9 \pm 0.7$ (Aa)	$19.1 \pm 0.5$ (Aa)	$19.1 \pm 0.5$ (Aa)		
	630	$19.8 \pm 1.2$ (Aa)	$19.1 \pm 1.0$ (Aa)	$20.6\pm0.9~(Bb)$		
	900	$19.5 \pm 0.8$ (Aa)	$20.3 \pm 0.7$ (Ba)	$20.1 \pm 0.9$ (Ba)		

Different uppercase letters indicate statistically significant differences between powers; different lowercase letters indicate statistically significant differences between number of cycles (p < 0.05, Tukey test). No significant difference was found between polymerization methods (p > 0.05).

difference between the number of cycles (p < 0.001), with greater distortion appearing after 36 cycles (Table 2).

No significant differences were observed in flexural strength (p > 0.05); however, the elastic modulus was progressively reduced after disinfection, with a greater reduction in specimens irradiated at 900 W (p = 0.008; Table 3). There were no significant differences in impact strength or angle of fracture propagation for either resin (p > 0.05; Table 3).

The water temperature curve is presented in Figure 3. Temperatures above 71°C appeared after 1.5 minutes at 900 W, 2 minutes at 630 W, and 2.5 minutes at 450 W.

# Discussion

The effect of microwave disinfection on the physical properties of PMMA have previously been evaluated.<sup>6,16,18-22</sup> However, variations in the irradiation regimen (with respect to time,

**Table 2** Linear distortion (%; mean  $\pm$  SD) dependence on polymerization method, power, and number of disinfection cycles (n = 8)

Polymerization method	Power (W)	Baseline	6 cycles	36 cycles
Water bath	450	0 (—,a)	$0.09 \pm 0.07$ (A,a)	0.29 ± 0.20 (A,b)
	630	0 (-,a)	$0.11 \pm 0.05$ (A,a)	$0.30 \pm 0.16$ (A,b)
	900	0 (—,a)	$0.25 \pm 0.20$ (B,b)	$0.79 \pm 0.28$ (B,c)
Microwave (*)	450	0 (-,a)	$0.09 \pm 0.05$ (A,a)	$0.47 \pm 0.39$ (A,b)
	630	0 (-,a)	$0.12 \pm 0.10$ (A,a)	$0.63 \pm 0.35$ (A,b)
	900	0 (-,a)	$0.71 \pm 0.18$ (B,b)	$1.46 \pm 0.35$ (B,c)

Different uppercase letters indicate statistically significant differences between power levels; different lowercase letters indicate statistically significant differences between number of cycles; \*indicates the presence of statistically significant differences between polymerization methods (p < 0.05, Tukey test).

**Table 3**Flexural strength, elastic modulus, impact strength, crack propagation angle (mean  $\pm$  SD), and frequency of fracture types according to thepower level and number of disinfection cycles (n = 8)

D.L:							Fractures	
method	Power (W)	Cycles	strength (MPa)	Elastic modulus (MPa)	strength (J)	angle	Fragile	Intermediate
Water bath	Baseline		$88.9 \pm 8.8$ (Aa)	2011.9 ± 98.3 (Aa)	0.21 ± 0.03 (Aa)	$51.3 \pm 5.1$ (Aa)	75.0% (6)	25.0% (2)
	450	6	$88.9\pm7.2~\text{(Aa)}$	1937.2 ± 131.7 (Ab)	$0.20\pm0.03$ (Aab)	$53.0\pm8.1~\text{(Aa)}$	62.5% (5)	37.5% (3)
		36	$89.2\pm5.2~\text{(Aa)}$	1877.4 ± 192.4 (Ab)	$0.19\pm0.04$ (Ab)	$54.1 \pm 8.2$ (Aa)	75.0% (6)	25.0% (2)
	630	6	$89.9\pm5.0~\text{(Aa)}$	$1892.9 \pm 120.4$ (Bb)	$0.19\pm0.03$ (Aab)	$49.4\pm7.2~\text{(Aa)}$	75.0% (6)	25.0% (2)
		36	$91.8 \pm 4.4$ (Aa)	1832.1 ± 158.8 (Bb)	$0.18 \pm 0.05$ (Ab)	$47.1\pm4.0~\text{(Aa)}$	87.5% (7)	12.5% (1)
	900	6	$90.3\pm8.5~\text{(Aa)}$	$1758.3 \pm 247.1$ (Bb)	$0.19\pm0.03$ (Aab)	$47.7\pm4.7~\text{(Aa)}$	62.5% (5)	37.5% (3)
		36	$95.1\pm8.1~\text{(Aa)}$	$1556.1 \pm 245.3$ (Bb)	$0.17\pm0.05$ (Ab)	$52.5\pm6.8~\text{(Aa)}$	100.0% (8)	0.0% (0)
Microwave	Baseline		$87.6\pm4.2~\text{(Aa)}$	$2033.48 \pm 297.$ (Aa)	$0.22\pm0.03$ (Aa)	$50.1\pm6.4~\text{(Aa)}$	87.5% (7)	12.5% (1)
	450	6	$88.7\pm8.9~\text{(Aa)}$	$1948.2 \pm 214.2$ (Aa)	$0.21 \pm 0.03$ (Aa)	$49.6\pm3.9~\text{(Aa)}$	100.0% (8)	0.0% (0)
		36	$90.3\pm8.3~\text{(Aa)}$	1713.5 ± 262.7 (Ab)	$0.19 \pm 0.05$ (Aa)	$49.1\pm5.0~\text{(Aa)}$	75.0% (6)	25% (2)
	630	6	$88.2\pm10.6~\text{(Aa)}$	$1959.5 \pm 187.4$ (Aa)	$0.20\pm0.04$ (Aa)	$48.4\pm7.6~\text{(Aa)}$	62.5% (5)	37.5% (3)
		36	$91.1\pm6.0~\text{(Aa)}$	$1691.5 \pm 158.9$ (Ab)	$0.20\pm0.06$ (Aa)	$49.8\pm8.2~\text{(Aa)}$	75.0% (6)	25.0% (2)
	900	6	$88.6\pm6.4~\text{(Aa)}$	$1740.7 \pm 230.5$ (Ba)	$0.20\pm0.05$ (Aa)	$51.3\pm5.8~\text{(Aa)}$	87.5% (7)	12.5% (1)
		36	$93.7\pm5.4~\text{(Aa)}$	$1600.9 \pm 159.0$ (Bb)	$0.18\pm0.04$ (Aa)	$54.2\pm4.3~\text{(Aa)}$	87.5% (7)	12.5% (1)

Different uppercase letters indicate statistically significant differences between power levels; different lowercase letters indicate statistically significant differences between number of cycles (p > 0.05, Tukey test). No difference was observed between polymerization methods (p > 0.05).



**Figure 3** Water temperature immediately after 1, 2, and 3 minutes of microwave irradiation (n = 8).

power, and water volume) and the small number of disinfection cycles have led to contradictory conclusions regarding the safety of microwave disinfection for removable prostheses. The use of different powers in this study had the objective of evaluating whether lower power levels would be safer for the resin, as reported by Thomas and Webb.<sup>10</sup> The 450<sup>9</sup> and 630 to 650 W<sup>4,9</sup> power levels were effective against *Candida* spp. biofilms using the same exposure times; however, no studies have been conducted to evaluate the relative efficacy of both power levels. Therefore, the 450, 630, and 900 W power levels were tested, with the highest level simulating the denture wearer mistakenly using a full-power setting. The cumulative effect of the power setting was assessed after 36 irradiation cycles, simulating disinfection three times per week for 90 days.<sup>2</sup>

There have been no studies examining the effect of microwave disinfection regimens on the impact resistance and crack propagation of PMMA-based polymers. Evaluation of crack propagation behavior enables the identification of any intrinsic effect the microwave disinfection protocol has on the PMMA polymer. Also, no previous study evaluated PMMA polymers polymerized by different processes. It should be noted that the specimens were stored in a high-humidity environment between disinfection cycles and between evaluation tests so the specimens would not experience aging due to water immersion.  $^{\rm 23}$ 

Micro-organism adhesion is related to the surface roughness. In the present study, the surface roughness increased after 36 cycles at all power levels, but the values were still clinically acceptable to avoid biofilm accumulation.<sup>24</sup> The increased roughness can be attributed to the irradiation power, corroborating a previous study by Machado et al<sup>19</sup> using two disinfection cycles of 650 W for 6 minutes. Although the specimens were protected by gauze, the extended manipulation might have contributed to the increased roughness.

The surface hardness is directly associated with the integrity of the material and its resistance to degradation by chemical, thermal, or mechanical action. In the present study, higher surface hardness values were observed after 36 cycles, but this finding was not statistically significant. The slight increase in hardness may be explained by the fact that the resins in this study were thermally polymerized, resulting in a low extramonomer conversion during the additional polymerization process.<sup>25</sup> Previous studies also reported no changes in surface hardness after five 650 W 3 minute cycles,<sup>18</sup> or after two<sup>21</sup> and seven cycles<sup>6,19</sup> of 650 W for 5 minutes.

In the present study, the PMMA resin polymerized using microwave energy exhibited significantly larger dimensional changes. Microwave irradiation can promote the rearrangement of polymer chains, and the greater distortion observed in specimens undergoing microwave polymerization may be explained by the nature of the polymerization process. The fast polymerization reaction may trap stresses within the polymer matrix, which are then released during the microwave disinfection cycle.

PMMA resin irradiated in the dry state may distort up to 0.03%.<sup>16</sup> However, when immersed in water, Basso et al<sup>26</sup> reported distortion values of up to 0.5%, with a limit of 1% considered clinically acceptable. In the present study, the 450 and 630 W power levels caused less distortion than the 900 W level, independent of the polymerization method. The linear distortion after 36 900-W cycles in the microwave-polymerized PMMA resin was 1.46%, above the clinical limit of 1%.<sup>26</sup> Considering the risk of severe damage to the denture, it is important to provide exact instructions to the patient regarding the microwave power settings.

Prosthesis fractures may occur due to stress concentrations, increased flexing of the material during mastication, or a sudden drop onto a hard surface.<sup>27,28</sup> In the present study, no significant changes were observed in the flexural strength despite the potential for the microwaves to rearrange the polymer chains and affect the mechanical strength of the resin.<sup>29</sup> The results are in agreement with previous studies, which also reported no differences<sup>16,21</sup> even after five disinfection cycles.<sup>18</sup> However, the elastic modulus was significantly lower after 36 cycles for specimens treated at 900 W, indicating increased flexibility of the specimens.

In the present study, no significant changes in impact resistance or crack propagation angle were evident (Table 3). The average fracture propagation angles obtained for all groups were higher than those reported for a purely brittle polymer structure,<sup>12,30</sup> indicating that the microstructural behavior was progressive crack evolution, perhaps influenced by the repeated heating treatments; however, the limit of the material resistance to masticatory forces in terms of elastic deformation and crack propagation has not been clearly described.

Although four specimens were simultaneously irradiated to simulate the resin mass of a denture, this simulation is far from the clinical condition, considering that conventional dentures have irregular shape and thickness. Additional studies over longer periods should be conducted with the purpose of assessing material degradation and loss of structural properties.

## Conclusion

Within the limitations of this study, it can be concluded that microwave disinfection for 3 minutes at 450 W to 630 W is safe for PMMA polymers.

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