

# Influence of Microwave Polymerization Method and Thickness on Porosity of Acrylic Resin

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## Abstract

**Purpose:** This study evaluated the influence of polymerization cycle and thickness of maxillary complete denture bases on the porosity of acrylic resin.

**Materials and Methods:** Two heat-activated denture base resins—one conventional (Clássico) and one designed for microwave polymerization (Onda-Cryl)—were used. Four groups were established, according to polymerization cycles: A (Onda-Cryl, short microwave cycle), B (Onda-Cryl, long microwave cycle), C (Onda-Cryl, manufacturing microwave cycle), and T (Clássico, water bath). Porosity was evaluated for different thicknesses (2.0, 3.5, and 5.0 mm; thicknesses I, II, and III, respectively) by measurement of the specimen volume before and after its immersion in water. The percent porosity data were submitted to Kruskal–Wallis for comparison among the groups.

**Results:** The Kruskal–Wallis test detected that the combinations of the different cycles and thicknesses showed significant differences, and the mean ranks of percent porosity showed differences only in the thinnest (2.0 mm) microwave-polymerized specimens (A = 53.55, B = 40.80, and C = 90.70). Thickness did not affect the results for cycle T (I = 96.15, II = 70.20, and III = 82.70), because porosity values were similar in the three thicknesses.

**Conclusions:** Microwave polymerization cycles and the specimen thickness of acrylic resin influenced porosity. Porosity differences were not observed in the polymerized resin bases in the water bath cycle for any thickness.

Poly(methyl methacrylate) has been widely used as a denture base material since the late 1930s. It is by no means a completely ideal material, and its advantages and disadvantages are well established; however, it is important to select an appropriate resin for the chosen method of processing to obtain the best results.<sup>1</sup>

The polymerization of denture base resin by microwave energy has been studied for more than three decades. The advantages of polymerizing denture base resin by microwave energy are a greatly reduced polymerization time,<sup>2</sup> a cleaner method of processing,<sup>3</sup> and a denture base with superior adaptation to the dental cast.<sup>4</sup> Several reports evaluating the effect of microwave energy on the porosity of denture base resin have been published.<sup>5-11</sup> Porosity of denture base resin continues to be one of the undesirable characteristics of poly(methyl methacrylate). This has been attributed to a variety of factors including the following: air entrapped during mixing, monomer contraction during polymerization, monomer vaporization associated with the exothermic reaction, and the presence of residual monomer.<sup>12,13</sup>

Ilbay et al<sup>14</sup> reported that the porosity was superior in acrylic resin cured by microwave energy at a higher power. Compagnoni et al<sup>6</sup> found no significant differences in porosity among denture base resin specimens polymerized with three different cycles of microwave energy. They also reported the porosity found in the microwave-polymerized denture base resin tested was similar in porosity to the heat-polymerized resin tested. In addition, it is well documented that porosities

Group (cycle)	Material	Manufacturer	Polymerization cycle
A	Onda-Cryl, clear microwave-polymerized denture base resin	Artigos Odontológicos Clássico Ltd, SP, Brazil	Microwave oven: 500 W for 3 min
В	Onda-Cryl, clear microwave-polymerized denture base resin	Artigos Odontológicos Clássico Ltd	Microwave oven: 90 W for 13 min $+$ 500 W for 90 s
С	Onda-Cryl, clear microwave-polymerized denture base resin	Artigos Odontológicos Clássico Ltd	Microwave oven: 320 W for 3 min + 0 W for 4 min + 720 W for 3 min*
Т	Clássico, clear heat-polymerized denture base resin	Artigos Odontológicos Clássico Ltd	Conventional water bath: 74°C for 9 h

\*According to manufacturer's recommendation.

were found in thick sections of microwave-cured denture resins when the thickness exceeded 3 mm.<sup>15-17</sup>

Reitz et al<sup>7</sup> showed that the frequency and size of porosity in thick specimens could be reduced to 30% by a longer polymerization time at a lower wattage setting on the microwave oven. Yannikakis et al<sup>10</sup> noted severe porosity in thicker areas of conventional resin specimens that underwent microwave polymerization, but found no significant porosity in the resin designed specifically for this technique.

Wolfaardt et al<sup>13</sup> investigated the occurrence and nature of porosity in a heat-cured denture resin and confirmed that the factors responsible for the generation of porosity were far more active in thick sections of the denture resin than in thin sections. According to Tager,<sup>18</sup> porosity is a property of solids that relates to their structure and is expressed in the presence of voids (pores) between separate grains, layers, crystals, and other elements of a coarse structure of a solid. This definition emphasizes the fact that the concept of porosity can be applied to solids, and that pores are spaces not between molecules, but between super molecule structures. Tager also supports Dubin's definition where pores are described as "voids or spaces in solids." Jerolimov et al<sup>19</sup> affirmed that in evaluation of porosity, the various thicknesses and geometries of a denture affect the efficiency of dissipation of exothermic heat, which influences the generation of porosity. Several other studies<sup>5,14,16,20-22</sup> have evaluated porosity of resin and discussed techniques for accomplishing microwave polymerization. They conclude it is important to control temperature accurately and ensure correct timing to minimize porosity when microwave polymerization is used.

Lack of porosity is essential for retaining a smooth, clean, polishable surface. The objective of this study was to investigate the porosity of a complete acrylic resin maxillary denture base with varying thicknesses polymerized with different polymerization cycles. There were two hypotheses for this study. First, the porosity of a denture base resin does not depend on the polymerization cycle. Second, denture base resin thickness does not influence the porosity, regardless of polymerization cycles used.

## **Materials and methods**

Four cycles were used to prepare the denture base resins specimens (Table 1). These specimens were processed in three thicknesses (2.0, 3.5, and 5.0 mm).

## **Specimen fabrication**

The maxillary denture bases were used as specimens for porosity analysis. The specimens were formed on a selected clinical maxillary gypsum cast without undercuts. Sheets of baseplate wax (Epoxiglass, Ind. Com. de Produtos Químicos, Diadema, SP, Brazil) were adapted and sealed onto three casts. To ensure uniformity of thickness proposed (2.0, 3.5, and 5.0 mm), the thickness of each maxillary waxed-up trial denture base was measured within its extension using a millimeter periodontal probe that read up to 0.5 mm. To reproduce maxillary waxedup denture bases for each thickness, a silicone mold (RTV-3120, Reforplás Ind. Com. Ltda., Cubatão, SP, Brazil) was obtained from the first maxillary waxed-up trial denture base. One hundred and twenty wax trial denture bases were obtained from these three RTV silicone molds, 40 for each thickness. Hard wax (Epoxiglass) was melted and poured into the silicone mold, and the cast was positioned against it. After 30 minutes, the cast and the waxed-up trial denture base were removed from the mold. The waxed-up specimens were invested in conventional metal flasks for cycle T and in fiberglass-reinforced plastic flasks for the microwave polymerization cycles (A, B, and C), with dental stone (Herodent, Vigodent S/A Ind. Com., Rio de Janeiro, Brazil). After the investing material had set, the wax was removed, and the denture base resins were mixed according to the manufacturer's recommendations. The denture resin was packed into the mold. The specimens in groups A, B, and C were processed with Onda-Cryl denture base resin. A conventional heat-polymerized resin (Clássico) was used to prepare the specimens within the control group (T). A pneumatic press (Delta, Delta Máquinas Especiais, Vinhedo, SP, Brazil) was used for trial packing the denture base resin initially at 1500 psi and, finally, at 3500 psi maintained for 30 minutes. Cycle T specimens were polymerized in an automatic polymerization tank (Termotron P-100, Termotron Equipamentos, Piracicaba, SP, Brazil). For the microwave polymerization method, a domestic microwave oven with a rotating table was used (Continental AW-30, Bosh Eletrodomésticos, Manaus, AM, Brazil).

## **Porosity analysis**

One hundred and twenty denture base resin specimens were processed. One-third of the specimens were 2.0 mm (thickness I), one-third were 3.5 mm (thickness II), and the other third were 5.0 mm (thickness III). For each thickness, 40 acrylic

resins specimens were divided into four cycles (Table 1), consisting of ten specimens each. The 2.0- and 3.5-mm thickness specimens were chosen to represent the common dimensions of a denture base. The 5.0-mm specimens represented thicker denture bases.<sup>10</sup>

All specimen groups were bench cooled overnight before deflasking. After deflasking, the excess resin was trimmed from all specimens with a bur (Maxi-Cut, Lesfils de August Malleifer SA, Ballaiguest, Switzerland). The porosity was determined using techniques reported previously<sup>6,18,23</sup> using polymers and water. The method used in the present study was supported by Tager,<sup>18</sup> who reported that the porosity of a sorbet is estimated quantitatively by total pore volume (W<sub>0</sub>). According to Tager, the most common method of estimating W<sub>0</sub> is the classical sorption method and mercury porosimetry.

The classical sorption method was used in this study. After processing, the specimens were dried in a desiccator (Corning Brazil, Indústria Comércio Ltd, SP, Brazil) containing silica gel under a vacuum. They were weighed daily by an analytical balance (Sartorius, Sartorius AG, Göttingen, Germany) capable of measuring to 0.0001 g until a constant mass was reached, indicating a state of equilibrium. Weights of thicknesses I, II, and III specimens equalized after 10, 20, and 20 days, respectively. With specimens dried, two weights were made: one with the specimen in air and the other with the specimen immediately immersed in distilled water. Afterwards, the specimen groups were stored in distilled water at 37°C in a thermostatically controlled incubator (Fanem, Fanem Ltd, SP, Brazil). These were then weighed at regular intervals until a constant mass indicating a state of water saturation was reached. The weights of thicknesses I, II, and III specimens equalized after 15, 25, and 35 days, respectively. After these periods, the specimens were removed from the water, excess water was removed by blotting with filter paper, and the specimens were weighed. Again, two weights were made: one in air and the other with the specimen immediately immersed in distilled water.

After the mass registers of the specimens dried, and after absorption or desorption of distilled water, the porosity calculations were made using the following equations:<sup>18,23</sup>

$$V_d = \frac{m_d - m'_d}{\rho_w} \tag{1}$$

$$V_s = \frac{m_s - m'_s}{\rho_w} \tag{2}$$

$$\% \text{ Porosity} = \frac{(V_s - V_d) \times 100}{V_d}$$
(3)

where  $V_d$  = dried specimen volume;  $m_d$  = mass of dried specimen in air;  $m'_d$  = mass of dried specimen in water;  $\rho_w$  = density of water;  $V_s$  = volume of the specimen saturated by water;  $m_s$  = mass of saturated specimen in air; and  $m'_s$  = mass of saturated specimen in water. In Equations (1) and (2), the volumes were determined using  $\rho_w$  = 1000 Kg/m<sup>3</sup>. Having solved these equations, the porosity could be calculated by the volume of saturated specimen minus the dried specimen volume divided into the dried specimen, and multiplied by 100 to produce total percent porosity value for each specimen (Equation 3).



Figure 1 Mean ranks of porosity for all polymerization cycles (A, B, C, and T) in the different thicknesses (I, II, and III) of acrylic resin specimens.

In the methodology of the present study, the porosity calculation was based on mass and volume of each specimen before and after its immersion in water, and the density of the water.<sup>5,6,18</sup> The analysis of porosity was executed using two factors (polymerization cycle and specimen thickness); the polymerization cycle was analyzed with four levels, and the specimen thickness was analyzed with three levels. The Kruskal–Wallis test for nonparametric variables was performed, and the level of significance was alpha = 0.05.

## Results

The Kruskal–Wallis test showed that the combination of the cycles and the thicknesses presented significant differences (H = 47.459; df = 14; p = 0.000). The Kruskal–Wallis test uses the mean ranks of percent porosity, which is graphically represented in Figure 1. To identify the groups of experiments with significantly different mean ranks, or with differences in relation to the thickness, cycle of polymerization, or both, multiple nonparametric comparisons had to be carried out. The results of these comparisons were separated in two sets, for comparisons between mean ranks of different cycles within the same thickness (Table 2) and for comparisons between mean ranks of different thicknesses within the same polymerization cycle (Table 3).

The mean data and significant differences of percent porosity for all groups are presented in Table 4.

Comparing different cycles within the same thickness (Table 2) revealed that the porosity in 2.0-mm specimens (thickness I) was significantly lower for cycles A and B than cycles C and T. All the thickness II specimens presented similar results, except between cycles A and T, where A exhibited highest porosity. Within thickness III, all the specimens presented similar results, except between cycles C and T, where T exhibited highest porosity.

The comparison between different thicknesses within the same polymerization cycle (Table 3) demonstrated that thicknesses I and III had equivalent behavior in cycle A; thickness II showed the highest percent of porosity. In cycle B, thickness II continued presenting greater percent porosity compared with thickness I, but similar porosity to thickness III. In cycle C, thicknesses I and II presented similar results with percent porosity higher than thickness III. For cycle T, the thickness did not affect the results—porosity values were similar in the three thicknesses.

Cycle Т Thickness Cvcle Mean Rank А В С L А 53 55 \_ n.s \* \* В 40.80 \* С 90.70 n.s Т 96.15 Ш A 106.80 n.s. n.s. \* В 82.65 n.s. n.s. С 87.75 n.s Т 70.20 \_ Ш А 67.65 n.s. n.s. n.s. В 71.00 n.s. n.s С 43 45 \* Т 82.70 \_

 Table 2
 Nonparametric multiple comparisons between mean ranks of porosity of different cycles within the same thickness

n.s. = not significant; \*0.01  $\leq \alpha \geq 0.05$ .

## Discussion

This study evaluated the influence of polymerization cycle of maxillary complete denture bases with several thicknesses on the porosity of acrylic resin. The results indicate that polymerization cycle and denture base thickness both have an influence on porosity. These findings are similar to those of previous studies,<sup>7,8,10,13,16,17</sup> which demonstrated that the presence of pores also depends on the specimen thickness, not only on the polymerization cycle.

In the present study, the influence of microwave polymerization cycle on the porosity of acrylic resin was only observed in the thinnest (thickness I) specimens. Thickness I specimens polymerized with cycles A and B showed significantly lower porosity than resins polymerized with cycle C; however, thickness II and III specimens demonstrated similar results regardless of the microwave polymerization cycle.

 
 Table 3
 Nonparametric multiple comparisons between mean ranks of porosity of different thicknesses within the same polymerization cycle

	Thickness		Thickness		
Cycle		Mean Rank	I	II	
A	I	53.55	_	*	n.s.
	Ш	106.80		-	*
	111	67.65			-
В	I	40.80	-	*	n.s.
	Ш	82.65		-	n.s.
	III	71.00			-
С	I	90.70	-	n.s.	*
	Ш	87.75		-	*
	111	43.45			-
Т	I	96.15	-	n.s.	n.s.
	II	70.20		-	n.s.
	111	82.70			-

n.s. = not significant; \*0.01  $\leq \alpha \geq 0.05$ .

**Table 4** Mean percent porosity (s.d.) for each group

Thickness	Cycle A	Cycle B	Cycle C	Cycle T
I	0.56 (± 0.12)	0.51 (± 0.27)	1.09 (± 0.28)	1.51 (± 0.06)
11	1.20 (± 0.95)	0.79 (± 1.05)	1.23 (± 0.18)	0.45 (± 0.30)
Ш	0.83 (± 0.22)	1.57 (± 0.46)	-0.43 (± 0.04)	1.62 (± 1.24)

The porosity of all specimens using cycle T was similar for all three thicknesses; however, for microwave polymerization cycles (A, B, and C), porosity showed different results at different thicknesses. Bafile et al,<sup>5</sup> Compagnoni et al,<sup>6</sup> and Lai et al<sup>21</sup> did not find a significant difference in porosity between microwave polymerization cycles and conventional water bath cycle. The lack of agreement between those studies and the present study may be explained by the different shapes and sizes used in each.<sup>19</sup>

It is also important to emphasize that for the same polymerization cycle, the percent porosity was lower for the greatest thickness (III). These results are not in accordance with several authors<sup>7,10,13</sup> who observed more porosity in thick sections of acrylic resin specimens. According to Craig and Powers,<sup>24</sup> solid material can absorb water by a diffusion process. For this to occur, it is necessary that leaching of the liquids of the polymer occurs. The water may then occupy these empty spaces, called pores; however, in thicker specimens, the level of residual monomer is always higher in the central region than nearer the surface.<sup>19</sup> This may explain the contrasting results.

In this method of porosity analysis, the higher water absorption of acrylic resin specimens led to a higher percentage of porosity. According to the literature,<sup>18</sup> a similar profile to our results shows that the material is in a glassy state and presents a uniform distribution of pores. Tager<sup>18</sup> reported, "Polymers exist not only in a solid state, but also in a rubber-like state, and therefore the question of the competence of using the concepts 'pore' and 'porosity' naturally arises. It is quite evident that these concepts are fully applicable to solid polymers (glassy and crystalline), whose voids practically do not change in time."

A negative value of mean percent porosity in cycle C, thickness III was observed in this study (Table 4). This can be explained by the mechanism of the transport of liquids in the interior of polymer.<sup>23,25</sup> The monomer is hydrophilic, and with the aid of storage in distilled water at  $37^{\circ}$ C in a thermostatically controlled incubator, it can be eliminated for the interior of the water. Therefore, there was not enough time for the diffusion of the water from the interior of the occupied spaces into the interior of the pores. So, the V<sub>s</sub> was inferior to the V<sub>d</sub> and, consequently, percent porosity was negative.

The two hypotheses of this study must be rejected, because both the polymerization cycle and the thickness of the acrylic resin base influence the generation of pores. The first hypothesis of this study, that the porosity of a denture base resin depends on the polymerization cycle, was accepted only on the microwave-polymerized specimens. The second hypothesis, that the denture base resin thickness influences the porosity, must be rejected for cycle T only. A limitation of this study is that only one denture base resin was polymerized by microwave energy. Furthermore, although the method used for the analysis of porosity is objective, further research should be conducted to evaluate how these results apply to other studies with comparable methods for the analysis of porosity in polymers. The clinical implications of this study suggest that microwave polymerization can produce denture base resins with varied levels of porosity, depending on the resin base thickness.

## Conclusions

Within the limitations of this in vitro study, the following conclusions were drawn:

- 1. The influence of microwave polymerization cycle on porosity of acrylic resin appears only on the thinnest (2.0 mm—thickness I) specimens.
- 2. Group T (water bath polymerization cycle) presented similar porosity results for specimens of all tested thicknesses.

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