

# Porosity, residual monomer and water sorption of conventional heat-cured, microwave-cured and cross-linked acrylic resins

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**ABSTRACT** | This study compared the residual monomer release, water sorption and superficial porosity of different resins commonly employed in eye prostheses: heat-cured (HC); microwave-cured (MC) and self-curing cross-linked acrylic resins (SC). Four groups were established: G1, HC / water bath cycle; G2, MC / microwave cycle; G3, HC / microwave cycle; G4, SC. The amount of residual monomer was similar in G1 and G3, lower in G2 and higher in G4. Water sorption was similar in all groups. G2 showed more superficial porosity, and G1 and G3 were similar in this regard. Neither the conventional heat-curing cycle nor the microwave cycle affected the amount of residual monomer or porosity of the conventional heat-cured acrylic resin. Water sorption was not affected by the type of resin or polymerization cycle used. Residual monomer release and porosity were related to the type of resin employed rather than the polymerization cycle they were submitted to.

**DESCRIPTORS** | Acrylic Resins; Dental Materials; Chemical Properties; Ocular Prosthesis; Porosity.

**RESUMO** | **Porosidade, liberação de monômero residual e sorção de água de resinas termoativadas convencionais, resinas termoativadas para microondas e resinas quimicamente ativadas com ligações cruzadas** • Este estudo comparou a liberação de monômeros residuais, a sorção de água e a porosidade superficial de diferentes resinas acrílicas utilizadas na confecção de próteses oculares: ativadas por calor (HC); ativadas por micro-ondas (MC) e quimicamente polimerizáveis (SC). Quatro grupos foram estabelecidos: G1, HC / ciclo em banho aquecido; G2, MC / ciclo em micro-ondas; G3, HC / ciclo em micro-ondas; G4, SC. A quantidade de monômero residual foi similar nos grupos G1 e G3, menor no G2 e maior no G4. A sorção de água foi similar nos quatro grupos. O grupo G2 apresentou maior porosidade superficial, e os grupos G1 e G3 apresentaram porosidades similares. Os ciclos térmicos por banho aquecido e por micro-ondas não influenciaram a quantidade de monômero residual liberado ou a porosidade das resinas acrílicas polimerizadas por calor. A sorção de água não foi influenciada pelo tipo de resina ou pelo ciclo de polimerização utilizado. A liberação de monômero residual e a porosidade estão relacionadas ao tipo de resina utilizada e não ao ciclo de polimerização empregado.

**DESCRITORES** | Resinas Acrílicas; Materiais Dentários; Propriedades Químicas; Prótese Ocular; Porosidade.

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• Received Dec 12, 13 • Accepted May 16, 14  
• DOI <http://dx.doi.org/10.11606/issn.2357-8041.clrd.2014.68723>

## INTRODUCTION

Acrylic resins improved prosthetic treatment in cases of eye loss, which were originally treated with glass prostheses.<sup>1</sup> Ocular prostheses are essential to promote normal craniofacial development in children or to maintain facial symmetry in adults with eye loss.<sup>2,3</sup> Periodical changes of the ocular prosthesis are necessary in cases of eye loss during childhood or youth.<sup>4,5</sup> In addition, the loss of prosthetic fit and possible changes in the color of the prosthesis may increase the number of visits to maxillofacial clinics.

This study was conducted because of the constant search for new techniques for the construction of ocular prostheses that will efficiently meet the needs of patients with eye loss. The release of residual monomer in acrylic resins is an important factor, since it may lead to hypersensitivity to the resin and cause eczema in both the skin and mucosa.<sup>6-8</sup> Methyl-methacrylate ranks 8<sup>th</sup> among the methacrylates that may cause hypersensitivity, accounting for 7.4% of the cases of hypersensitivity to methacrylates.<sup>9</sup>

Water sorption is a feature of acrylic resins that significantly affects their mechanical and dimensional properties. The water pools among the polymers of the acrylic resin by diffusion, and pulls them apart, slightly expanding the resin. This small volume expansion, of approximately 1%, causes a linear expansion of 0.3%, which practically overrides the contraction caused by polymerization of the resin. The micropores that form in the resin, in turn, may lodge bacteria and fungi, favoring plaque formation and hindering proper cleaning of the prosthesis.<sup>10-13</sup>

Comparative analysis of acrylic resins, considering the release of residual acrylic monomer, superficial porosity and water sorption, processed with different polymerization cycles may contribute to the development of new techniques for the construction of ocular prostheses.

## MATERIALS AND METHODS

Fifteen specimens 25 mm in diameter by 4 mm in height were manufactured for each of the following experimental groups:

- Group 1 (G1), conventional heat-cured acrylic resin (Clássico, Artigos Odontológicos Clássico Ltda., Brazil), polymerized in water bath;
- Group 2 (G2), microwave-cured acrylic resin (Onda Cryl, Artigos Odontológicos Clássico Ltda., Brazil), polymerized by microwave energy;
- Group 3 (G3), conventional heat-cured acrylic resin, polymerized by microwave energy; and
- Group 4 (G4), self-curing, cross-linked acrylic resin (Orto Clas, Artigos Odontológicos Clássico Ltda., Brazil), polymerized at room temperature.

The conventional heat-cured acrylic resin was prepared according to the manufacturer's instructions, using 14 mL of the monomer and 42 mL of the polymer. G1 specimens were processed raising the temperature of the water bath to 74°C, maintaining it at this temperature for 120 min. and then raising the water temperature to 100°C and maintaining it at this level for 60 additional min.<sup>14</sup> G2 specimens were prepared using the microwave-cured acrylic resin and plastic flasks, since the resin was polymerized in a microwave oven. The polymerization cycle was carried out using an 800 W microwave oven. Each flask was placed in the center of the oven and heated for 3 min. at 320 W. After resting for 4 min., they were reheated for 3 min. at 720 W. G3 specimens were prepared with conventional heat-cured resin, placed in plastic flasks and polymerized according to the processing cycle described for G2 specimens. G4 specimens were manufactured using self-curing acrylic resin, following the procedures described above. G4 was different from the others with regard to the proportion of monomer and polymer used. The powder/liquid

volume ratio was 2.5:1, according to the manufacturer's instructions.

The specimens were finished using a tungsten carbide bur and a white stone bur, and polished using a horizontal sander at 250 rpm with a sequence of sandpaper discs of different grit sizes—320, 400 and 600—under constant irrigation. They were then cleaned using an ultrasonic bath for 2 min.. From this stage on, all specimens were manipulated using tweezers to avoid contamination.

### Residual monomer

The specimens were placed in amber-glass bottles filled with 10 mL of deionized water, sealed with plastic film, and kept in a sterilizer at 37°C. Different samples of the solutions in which the specimens were kept were collected every 24 hours and placed in 2 mL Eppendorf tubes for future analysis. After the samples were collected, the specimens were rinsed in distilled water for one second and the remaining solution was discarded. The specimens were then placed in the bottles again, and another 10 mL of deionized water was added. This procedure was repeated for 11 days.

The spectrophotometer used for our analysis was calibrated with a solution of known concentration of monomer. The wavelength used was 206 nm. A standard dilution curve was constructed to be compared with the values obtained by the spectrophotometer and to determine the correct concentrations of residual monomer in the samples collected. In order to do this, the stored solutions were transferred to crystal cuvettes which were then used in the spectrophotometer to measure residual monomer concentration.

### Water sorption

The same specimens were used to assess water sorption. They were placed on a paper towel inside a desiccator containing silica gel for dehumidification. After dehydration in a sterilizer for 24 hours

at 37°C, the specimens were weighed on a precision scale. The specimens were then immersed in beakers containing deionized water and kept in the sterilizer for seven days at 37°C. After this period, the specimens were retrieved, softly dried with a paper towel and weighed again on the precision scale.

### Porosity

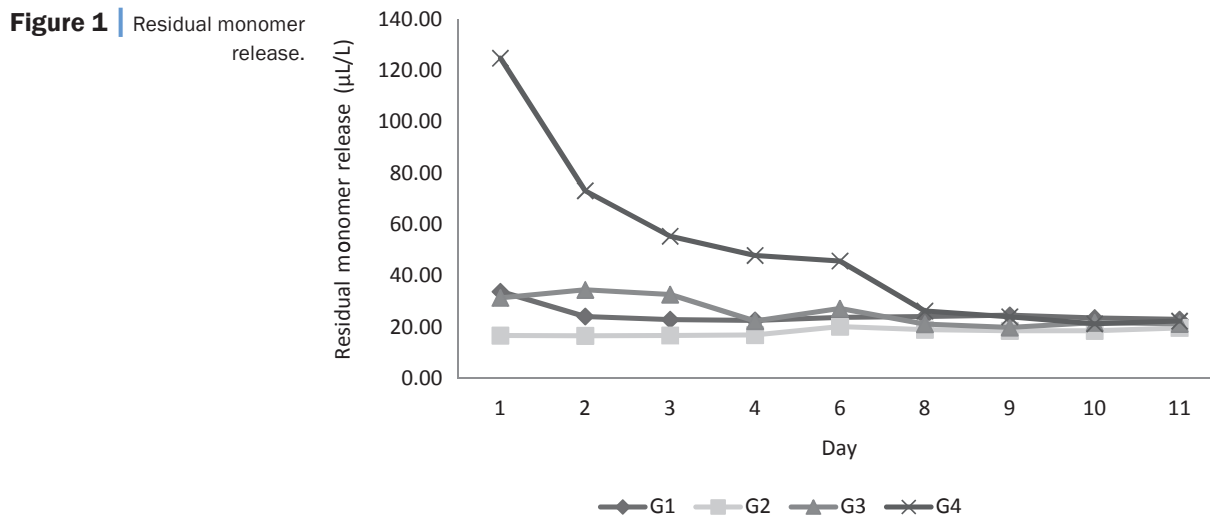
The specimens were immersed in beakers containing gentian violet (1% solution in water) and placed on a lab shaker, where they remained for 30 min.. The immersion in gentian violet aimed at staining the superficial micropores. After removal from the gentian violet solution, the specimens were rinsed in distilled running water for one second and dried with a paper towel. They were then analyzed under a stereoscopic microscope with magnification of 20×. The stained pores were clearly distinguished from the white-colored material of the acrylic resins commonly used in eye prostheses.

A built-in digital camera recorded images of five different areas of each specimen. The images were sent to a computer to be processed by ImageLab 2000® software (Softium Informática Ltda., São Paulo, SP, Brazil). The G4 specimens, constructed using self-curing, colorless, cross-linked acrylic resin, were not analyzed for porosity because the absence of contrast hindered image acquisition. Image processing using the software generated a percentage analysis of the darker areas per mm<sup>2</sup>, which corresponded to the areas stained by gentian violet.

Statistical analysis of the properties assessed was made using split-plot one-way analysis of variance and Tukey's test. The significance level was set at  $p \leq 0.05$ .

## RESULTS

Figure 1 shows that G1 specimens had a greater release of residual monomer in the first 24 hours.



After this period of time, there was a drop of approximately 45% in the release of residual monomer, and this level was maintained until the end of the experiment. G2 specimens showed the lowest release of residual monomer in the first 24 hours, with a level below 20 µL/L throughout the experiment. G3 specimens showed lower release of residual monomer than G1 specimens in the first 24 hours. However, over the same period, this group showed a release of residual monomer twice as high as that showed by G2. G4 specimens showed a greater release of residual monomer in the first 24 hours, three times greater than that showed by G1 and G3, and seven times greater than that showed by G2 (Figure 1).

Statistical analysis of the release of residual monomer by ANOVA and Tukey’s test yielded the following results:

- G1 specimens released the highest amount of residual monomer on day one ( $p = 0.0001$ );
- the amount of residual monomer released by G2 specimens was similar at all time intervals evaluated ( $p = 1$ );
- the amount of residual monomer released by G3 specimens was lower as of day eight ( $p = 0.007$ );
- the amount of residual monomer released by G4 specimens was similar on days three and four,

and from day eight on ( $p = 1$ );

- a significant difference in the release of residual monomer was observed among the groups, except between G1 and G3 (Table 1).

The weights of the specimens (in grams), both dry and hydrated, are shown as mean values and standard deviations. There is no statistical difference among these groups when we analyze the water sorption through the weight of the specimens (Table 2).

The superficial porosity of five areas of each specimen was analyzed. For statistical analysis, however, only three areas were considered, disregarding the most discrepant values to avoid distortions. The results are shown as means and standard deviations, expressed in % of total area. Analysis of the data obtained showed that G2 specimens had higher superficial porosity than G1 and G3 specimens, and that G1 and G3 specimens had similar superficial porosity (Table 3).

## DISCUSSION

The results obtained suggest that the process employed for polymerization of the heat-cured resin, either in conventional water bath or by microwave energy, does not affect the release of residual

**Table 1** | Mean and standard deviation of residual monomer release ( $\mu\text{L/L}$ ). Different letters (uppercase for groups and lowercase for days) show statistically significant difference ( $p < 0.05$ ;  $n = 15$ ).

Groups	Days									Total
	1	2	3	4	6	8	9	10	11	
G1	33.84 $\pm$ 8.31 Aa	24.16 $\pm$ 1.63 Ab	22.91 $\pm$ 1.52 Ab	22.58 $\pm$ 1.14 Ab	23.73 $\pm$ 2.02 ABb	24.12 $\pm$ 1.64 Ab	24.60 $\pm$ 1.67 Ab	23.54 $\pm$ 1.52 Ab	22.98 $\pm$ 1.31 Ab	222.45 $\pm$ 8.14 A
G2	16.73 $\pm$ 0.55 Ba	16.62 $\pm$ 0.20 Ba	16.76 $\pm$ 0.18 Aa	16.90 $\pm$ 0.22 Ba	20.15 $\pm$ 2.28 Ab	18.99 $\pm$ 1.40 Bb	18.56 $\pm$ 0.61 Bb	18.56 $\pm$ 0.61 Cb	19.68 $\pm$ 3.06 Bb	162.94 $\pm$ 4.95 B
G3	31.42 $\pm$ 7.75 Aa	34.55 $\pm$ 9.62 Ca	32.72 $\pm$ 10.86 Ba	22.38 $\pm$ 2.83 Ab	27.20 $\pm$ 9.77 Bb	21.17 $\pm$ 2.11 Cb	19.88 $\pm$ 0.70 Bb	21.72 $\pm$ 2.84 ABb	21.28 $\pm$ 2.30 ABb	244.19 $\pm$ 22.13 A
G4	124.80 $\pm$ 2.73 Ca	73.13 $\pm$ 7.76 Db	55.43 $\pm$ 8.53 Cc	47.93 $\pm$ 6.29 Cc	45.77 $\pm$ 8.52 Cc	26.27 $\pm$ 2.05 Dd	24.01 $\pm$ 4.62 Ad	21.28 $\pm$ 2.15 Be	22.35 $\pm$ 2.39 Ae	440.97 $\pm$ 27.28 C

**Table 2** | Mean and standard deviation of water sorption (grams). No statistically significant difference was observed ( $n = 15$ ).

	G1	G2	G3	G4
Dried	2.021 $\pm$ 0.063	2.041 $\pm$ 0.074	2.144 $\pm$ 0.065	1.947 $\pm$ 0.078
Hydrated	2.037 $\pm$ 0.062	2.056 $\pm$ 0.075	2.155 $\pm$ 0.067	1.970 $\pm$ 0.086
Water sorption	0.016 $\pm$ 0.004	0.015 $\pm$ 0.005	0.011 $\pm$ 0.003	0.023 $\pm$ 0.037

monomer. Other studies have suggested that heat-cured acrylic resins polymerized by microwave energy release a greater amount of residual monomer than those polymerized by heat.<sup>15,16</sup> However, chemical tests have shown that a lower amount of residual monomer is released when the acrylic resin is polymerized by microwave energy,<sup>17</sup> suggesting that such differences might be due to variations in the composition of the different acrylic resins analyzed and the methodology employed in each study.

In the present study, Onda Cryl<sup>®</sup>—an acrylic resin for microwave ovens—polymerized by microwave energy released the least residual monomer. This may be due to the fact that the internal boiling of the acrylic monomer produces a smaller amount of residual monomer after final polymerization.<sup>18</sup> Another possible explanation is the higher powder/liquid ratio used with the Onda Cryl<sup>®</sup> resin, 3:1 in volume, since a high powder/liquid ratio provides a higher-quality resin.<sup>19</sup> The powder/liquid ratio of the Onda Cryl<sup>®</sup> resin is almost twice the ratio

**Table 3** | Mean and standard deviation of porosity (% of the total area). Different letters represent statistically significant difference between groups ( $p < 0.05$ ;  $n = 45$ ).

G1	G2	G3
0.244 $\pm$ 0.079 A	0.456 $\pm$ 0.107 B	0.297 $\pm$ 0.087 A

of the Acron MC<sup>®</sup> resin, which was used most in the studies we reviewed. When both brands were compared, the Onda Cryl<sup>®</sup> resin showed better results.<sup>20</sup>

The amount of residual monomer released by the Orto Clas<sup>®</sup> resin—a self-curing, cross-linked acrylic resin—was at least three times higher than the amount released by the other groups in the first 24 hours.<sup>21</sup> This is in accordance with the fact that self-curing acrylic resins usually produce 3% to 5% of residual monomer, whereas heat-cured ones produce only 0.2% to 0.5%.<sup>22</sup>

The length of the observation period in this study took into consideration the 14 days which are necessary for the release of residual monomer

to become stable.<sup>8,19</sup> However, our results ran only through the 11<sup>th</sup> day, since the release of residual monomer became stable in all groups by that time. The results showed a tendency towards stabilization of the residual monomer release in all groups around the eighth day. The time it takes for the residual monomer to be released is an important factor because each different material and processing technique must receive specific treatment after polymerization in order to obtain an ideal prosthesis.<sup>19</sup> Both the acrylic resins and the polymerization cycles used in this study are considered acceptable for the construction of prostheses.<sup>23</sup>

Water sorption was assessed considering the weight of the specimens in grams, both dry and hydrated. The standard deviation for all specimens when dry was 0.01, indicating that the specimens were adequately manufactured. The four experimental groups yielded similar results regarding water sorption. The absence of a significant difference among the groups means that the three types of resin analyzed in this study showed similar water sorption, regardless of the polymerization cycle to which they were submitted.

The possibility of using microwave energy to polymerize conventional acrylic resins and obtain water sorption properties that comply with ADA (American Dental Association) specifications indicated that water sorption depends on the polymerization cycle used.<sup>22,24,25</sup> In the present study, the polymerization cycle in microwave oven to which the conventional acrylic resin was submitted did not affect water sorption. The lower water sorption of the cross-linked acrylic resin observed in another study<sup>26</sup> was not confirmed in this study.

Similarity in water sorption among the types of acrylic resin analyzed in this study was also found when the conventional acrylic resin was compared with hypoallergenic materials.<sup>27</sup> Although the time that we use to dry off and moisturize the samples could be shorter than necessary, making our results

on the sorption of water among the four groups statistically similar, a study designed to analyze porosity in which the specimens were weighed both when dry and hydrated also found a similarity in weight among the different acrylic resins when hydrated.<sup>28</sup>

Polymerization of acrylic resins is an exothermic reaction, and the increase in temperature can cause the boiling of the reactive monomer, which leads to the formation of bubbles in the resin. When the acrylic portion of a prosthesis is thin, the heat can leave the resin and dissipate in the surrounding cast, preventing the appearance of superficial bubbles.<sup>22</sup> This has led to the study of adjustments made to microwave power and polymerization time, so that resins polymerized by microwave energy show porosity similar to that of conventional resins.<sup>29-31</sup> It has also been observed that the number of flasks and their placement in the microwave oven affect the amount of residual monomer and resin porosity.<sup>32,33</sup>

The method employed in this study considered the polymerization of each flask individually, placed in the center of the microwave oven. This was done to avoid interference caused by the number of flasks or their position during polymerization. The method employed by us to assess superficial porosity is quite accurate, since the computer analysis yields the sum of all the pores on the surface, presented as a percentage of the total area observed. The superficial porosity of the self-curing, colorless, cross-linked resin was not assessed because the lack of contrast made it impossible to acquire an image that could be processed by ImageLab 2000<sup>®</sup> software.

The absence of superficial porosity in heat-cured acrylic resins has already been described,<sup>30</sup> and in the present study this type of resin showed the lowest percentage of superficial porosity, corresponding to 0.24% of the resin surface. The microwave-cured resin showed a higher percentage of superficial porosity than the conventional resin regardless of the polymerization cycle employed,



which is in accordance with previous studies.<sup>29,34</sup> The conventional resin showed greater superficial porosity when polymerized by microwave energy than when polymerized in water bath. However, this difference in superficial porosity was not statistically significant, which is in disagreement with previous studies.<sup>35</sup>

## CONCLUSIONS

The water-bath and microwave cycles did not affect the amount of residual monomer released by or the porosity of the conventional heat-cured acrylic resin. The amount of residual monomer produced varied according to the type of acrylic resin. Water sorption was not affected by the type of resin

or polymerization cycle. Porosity was related to the type of acrylic resin employed rather than the polymerization cycle. The results of this comparative study regarding porosity, amount of residual monomer released and water sorption suggest that conventional heat-cured acrylic resins processed by microwave energy may be used to optimize treatment with an eye prosthesis. Nevertheless, other resin properties should be investigated before this can be recommended for clinical application.

## ACKNOWLEDGMENT

We would like to thank Prof. Dr. Fausto Medeiros Mendes (University of São Paulo) for his support in the statistical analysis.

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