

Color Stability, Surface Roughness, and Surface Porosity of Acrylic Resins for Eye Sclera Polymerized by Different Heat Sources

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Abstract

Purpose: The aim of this study was to evaluate the color stability, surface roughness, and surface porosity of acrylic resins for eye sclera polymerized by different heat sources and submitted to accelerated artificial aging (AAA).

Materials and Methods: Three groups of ten specimens each were formed according to the heat source used during the polymerization cycle: GI—short cycle, GII—long cycle, and GIII—dry-heat oven. The groups were submitted to color spectrophotometry through the CIE L*a*b* system and to surface roughness and porosity analysis using a Surfcorder IF 1700 profilometer. After the tests, specimens were submitted to AAA, with a maximum aging time of 384 hours, corresponding to a year of clinical use. After aging, the color and roughness of each group were assessed.

Results: The results showed that the variability of ΔE was clinically unacceptable for all groups but the method of polymerization was insignificant (p > 0.05) for color change. For roughness, polymerization cycle was significant for the results. GIII (0.23 ± 0.06) presented the highest roughness difference (before and after AAA), statistically significant (p < 0.05) from GII. No statistically significant difference could be found among groups when considering the porosity test.

Conclusion: It may be concluded that irrespective of the type of heat used for polymerization, there was an intense color alteration, to clinically unacceptable levels, when the specimens were submitted to AAA. For the other properties, alterations were less intense.

The absence of an eye can lead to esthetic and functional disproportion, as well as personal and interpersonal disorders.¹ Epithelial tumors originating from the eyelid, conjunctiva, and paranasal sinuses, malignant tumors of the lachrymal gland, intraocular tumors with orbital extension, rare sclerosing pseudotumors, and fungal infections can lead to the loss of an eye.

Research has focused on these concerns in the search for ideal materials and techniques for prosthetic eye rehabilitation, with the aim of recovering aspects of patients' physical esthetics and social function. The goal of an ocular prosthesis is primarily to reconstitute esthetics, maintain muscle tonicity of the upper eyelid (preventing it from shrinking due to lack of function), conduct tears to their physiological ducts (prevent lashes from sticking and drying the conjunctival area), and protect the orbital mucosa from debris and dust.²⁻⁴

Acrylic resin is the material used to manufacture these ocular prostheses,^{5,6} due to its acceptable physical properties. Com-

posed of a polymer powder of methyl methacrylate and a liquid monomer, it has excellent resistance and is easy to color, because of the acrylic translucency, which is an important factor in ocular prostheses. Acrylic resins are the first-choice material in these situations due to their durability, ease of cleaning, reliable mechanical retention, biocompatibility, and acceptable cost. As with all materials, they also present disadvantages for ocular prostheses, such as slight discomfort because of their rigidity, esthetics inferior to flexible materials, and small ulcers caused by the edges.⁷⁻¹²

The acrylic resin polymerization process occurs through activation by different initiators.¹³⁻¹⁶ Thermoactivated resins transfer heat to heated water when submitted to microwave energy. Another source for resin heating would be a dry, temperature-controlled oven.¹⁷ The great problem of heat polymerization of acrylic resins is that the polymerization reaction is exothermic, and the amount of heat involved in the process could affect

the material properties.¹⁸⁻²⁰ It is important to find an ideal material with physical, mechanical, and biological properties for maxillofacial use.

Therefore, the aim of this study was to evaluate the color stability, surface roughness, and surface porosity of acrylic resins for eye sclera polymerized with different polymerization cycles.

Materials and methods

The methodology for preparing the specimens followed a sequence based on the lost-wax casting method. A stainless steel metal matrix with an internal diameter of 1.5 cm and 5.0 mm thickness was put onto clean glass plates that were previously flattened, polished, and carefully lubricated with petroleum jelly. Heated casting wax (Polidental, Cotia, Brazil) was deposited inside the matrix. After it was totally filled, and with little excess, another flat, clean, polished, and lubricated glass plate was put onto the wax and left there until the surface cooled.

After 10 minutes, the glass plate was removed, and the excess wax was carefully removed with a sharp instrument. Afterward, the specimens were removed from the matrix. Ten wax molds were made for each type of polymerization technique. These molds were included in plaster stone using a metal flask (Fig 1). After the stone setting time, the matrix was removed with hot water.

The resin was manipulated in accordance with the manufacturer's recommendations. When the mass reached the plastic phase, the space left by the matrix was filled in with the material. After total condensation of the material, a polyethylene sheet was put on the resin; the flask was closed and submitted to pressure. The flask was opened, the polyethylene sheet was removed from the resin surface, and excess was removed. Twenty specimens were obtained for each polymerization process (Table 1).

Group I was polymerized by the short-cycle polymerization technique, in which water was heated to 74°C, and flasks containing the specimens were immersed in this water for 1.5 hours. Then, water was warmed to 100°C, and the specimens remained in the flasks for one additional hour.

Group II was submitted to the heating process proposed by Anusavice,²¹ the so-called long cycle. In this process, the water is heated to 75°C; the flasks and specimens are immersed in this water for 8 hours. After this time, they are removed from water and maintained at room temperature for gradual cooling to avoid tensions inside the material. In Group III, the dry-heat polymerization technique was used. In this technique, the flasks with specimens were put into an oven and maintained at a constant temperature of 70°C for 72 hours.

The specimens were then sanded with aluminum oxide abrasive papers of decreasing granulation (#320-, #600-, and #1000grit) under cooling, until they reached a thickness of 3.0 mm, checked with a digital pachymeter. These specimens were returned to the flask, and polymerized colorless resin was deposited on the resin. The set was pressed and specimens of each group were submitted to the same polymerization process as previously described.

After finishing the cycles, the specimens were removed from flasks and submitted to finishing and polishing with abrasive papers of decreasing roughness under cooling. The specimens were then subdivided into two groups (n = 10): Group A—specimens submitted to color and roughness tests; Group B—specimens submitted to the porosity test. Separate groups were used because of the need to stain the specimens by the method used, which affects the color.

Initial color spectrophotometry

After fabrication, the specimens were immediately polished and submitted to color measurement using a spectrophotometer (PCB 6807, Byk Gardner, Geretsried, Germany; CIE- L*a*b* system) against a white standard background (Gardner Laboratory, Inc, Bethesda, MD) employing an optical geometry $45^{\circ}/0^{\circ}$. Color was evaluated according to the CIE-L*a*b* color system,²² in which L indicates color luminosity (ranging from 0 black to 100-white); a* indicates the amount of red (positive values) and green (negative values); b* indicates the amount of yellow (positive values); and blue (negative values). The specimens were coupled to the spectrophotometer. Thirty circularshaped LED lamps and ten colors were lit and reached the material surface at a 45° angle of incidence. This beam is reflected at 0° and thus returns to the device, which captures and records the L*, a*, and b* values of each specimen. These measures were considered the initial values.

Initial roughness measurement

The Surfcorder SE 1700 profilometer (Kosakalab, Tokyo, Japan) performed the initial roughness analysis. The profilometer needle was positioned on each specimen and ran a distance of 0.8 mm at a speed of 0.25 mm/s. When the appliance was activated, the needle moved in a single direction and detected



Figure 1 Matrix to manufacture the acrylic resin specimens.

 Table 1
 Materials and polymerization methods tested

Group	Material	Polymerization technique
Group I	Clássico	Short cycle
Group II	Clássico	Long cycle
Group III	Clássico	Dry-heat oven

Clássico Dental Products, Sao Paulo, Brazil.

the surface irregularities. These irregularities correspond to the peaks, valleys, and waves. Thus, a value that constituted the mean roughness (or Ra) of the surfaces was obtained. This value was defined as the mean of the pick-up displacements upward and downward, in relation to a central line. Three readings were made at different points of the surface. The mean of these three measurements was obtained and considered the initial value.

Aging

The specimens were then submitted to accelerated artificial aging (AAA). For this purpose, they were placed in a nonmetal C-UV Accelerated Aging device (Comexim Matérias Primas Ltda, São Paulo, Brazil) under the action of UV light and condensation, which worked in repeated cycles, successively and automatically. The condensation process was produced by exposing one of the specimen surfaces to a warm mixture of airwater saturated steam, while the opposite side of the specimen was used to fix it to the metal plates.

The specimens were then fixed with device plates using silicon and taken to the condensation chamber under the light source at a distance of 50 mm. The operating program was standardized at 4 hours of exposure to UV-B at 50°C and 4 hours of condensation at 50°C with a maximum aging time of 384 hours. Thus, the C-UV could produce degradation that corresponded to a year of clinical use.²³

After the aging process, the specimens were again submitted to color and roughness analysis using the spectrophotometer and profilometer to determine the color (ΔE) and roughness change, respectively. The total color change, ΔE , is commonly used to represent color difference and is calculated by the formula:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

where $\Delta L = L_1$ (before AAA) $-L_2$ (after AAA). Δa and Δb were calculated in the same way. Values of $\Delta E \geq 3.3$ are considered clinically unaceptable.²⁴ Roughness change was calculated subtracting the final value from the initial value of roughness, corresponding to before and after AAA, respectively.

Surface porosity evaluation

After AAA, the specimens were immersed in an India ink solution for 30 minutes and washed under running water. A $10 \times 10 \text{ mm}^2$ area was delimited in the middle of the specimen, where the surface porosity was estimated for visual appearance by

Table 2	Means	(SD) of	ΔL , a,	b,	and	Ε	values
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	Long cycle	Short cycle	Dry-heat oven
ΔL	-1.04 (0.8)	-0.7 (2.3)	-1.07 (2.7)
Δa	-1.1 (1.8)	-0.8 (1.5)	-0.4 (1.6)
Δb	5.9 (1.8)	5.8 (2.3)	4.2 (2.9)
ΔE	6.4 (2.04)	6.6 (1.7)	5.8 (1.8)

For all combinations (p > 0.05).

diffuse reflection of the surface on polarized light microscopy at 60X magnification. The number of pores was obtained by estimation, starting by counting the pores in the delimited area.

Statistical analysis

After calculation of the color-dimension changes (ΔL , Δa , Δb , ΔE), roughness alteration, and the estimated number of surface pores, the measures were calculated for each group studied. These measures were submitted to one-way ANOVA and Tukey test at a significance level of p = 0.05.

Results

Color spectrophotometry

 ΔL , Δa , Δb , and ΔE means comparison showed that there were significant differences in color change for all groups, but among groups there was no statistically significant difference (p > 0.05) for all tested parameters (Table 2). The ΔE variations were high, mainly for the short cycle, followed by the specimens polymerized in long cycles and specimens polymerized in an oven. The means are above 3.3, and therefore, they are considered clinically unacceptable, as defined by Ruyter et al²⁴ and Mutlu-Sagesen et al.²⁵ So, the effect of aging is that it results in clinically unacceptable color change, irrespective of the polymerization method.

Roughness

The mean values of roughness change (before and after AAA) were analyzed and showed that specimens polymerized in long cycles presented a smaller difference in roughness, followed by the specimens polymerized in short cycles. The largest differences in roughness following polymerization were observed in specimens polymerized in the oven. When the means were compared, it was verified that there was a statistically significant difference between specimens polymerized in long cycles, which presented smoother surfaces than those polymerized in the oven (Fig 2).

Surface porosity

The mean values of specimen porosity of each group were analyzed by ANOVA and Tukey tests (95% level of significance). The statistical analysis showed no statistically significant difference among groups (Fig 3).



Figure 2 Roughness difference means (before – after accelerated artificial aging) of specimens polymerized by different cycles.

Discussion

Over the last few years, acrylic resin has been the material of choice for complete denture bases. Due to the good inherent physical properties of this material, it is also used in ocular prostheses.²⁶ Acrylic resin is a light material, with good esthetic properties and relatively low cost. The great disadvantage of this heat-activated material is the exothermic polymerization reaction. Excessive heating can cause surface discoloration, increase the number of surface pores, and hence, decrease the mechanical resistance of the material.²⁷ Acrylic resin for eye sclera used in ocular prostheses should not undergo color alterations, due to the esthetic problems that will cause; neither can it produce a rough surface, which might be responsible for bacterial proliferation and injury to the underlying tissue.

Usually, acrylic resin has been processed in molds under pressure. These molds are put into metal flasks and immersed in water at a controlled temperature for a specific time, a strategy to convert monomers into polymers.²⁸ Several studies have also tested microwave-polymerized acrylic resin and related the pro-



Porosity

Figure 3 Porosity means of specimens polymerized by different cycles. For all combinations (p > 0.05).

cessing method to the material properties.^{6,29} The present study assumes the hypothesis that water used in the polymerization cycle of this material could interfere with important physical properties such as roughness, porosity, and color stability.³⁰⁻³²

According to the in vivo studies of Bollen et al³³ and Quirynen et al,³⁴ a clinically acceptable surface roughness of hard surfaces in the oral environment should not exceed 0.2 μ m. The results of the present study showed that roughness was higher than the recommended value for all groups irrespective of the polymerization cycle, being lower in Group III; however, when analyzing the roughness before and after AAA, it was verified that Group II had a smaller difference than the other groups, with the highest roughness average found in Group III (Fig 2). After AAA, there was an increase in surface roughness. The best performance of Group III can be explained by the absence of water during the processing of the specimens; however, the application of a dry-heat method can evaporate the monomers, reducing the degree of conversion, or creating linear polymeric chains; consequently, more extensive surface degradation could be found.35

The results of the color analysis demonstrated color alteration, irrespective of the polymerization cycle; however, the groups did not differ statistically among themselves. The ΔE means (before and after AAA) were higher than 3.3, demonstrating that acrylic has a propensity for color alteration (Table 2). Although statistical analysis showed no significant difference among specimens, they are considered clinically unacceptable as defined by Ruyter et al,²⁴ because their ΔE was higher than 3.3.

The material porosity was not affected by the polymerization cycle, as there were a high number of pores in all cycles. The most pores were found in Groups II and III, which presented high averages and standard deviations (Fig 3).

Acrylic resin undergoes water sorption during polymerization due to the diffusion process.³⁶ The water molecules interfere in the interlocking of the polymeric chains and alter the physical characteristics of the resultant polymer, which could cause excessive color alteration, thus affecting the long-term clinical success of the ocular prosthesis.

Porosity seems to be related to the polymerization conditions and to exposure to water. The more acrylic resin is exposed to water (Group II), the larger the number of pores will be, confirming the theory that acrylic resin undergoes water sorption from the environment ^{14,37} and has its physical properties altered.

Ogawa and Hasegawa⁶ showed that the increase in dry-heat temperature activates a chemical reaction between monomer and polymer, producing a more complete polymerization, with small number of pores. Conversely, the results of the present study contradict Ogawa and Hasegawa,⁶ because the long cycle (Group II) had the largest temperature increase and the largest porosity average. This can be explained because the long cycle exposes acrylic resin to water for a longer time (8 hours), allowing sorption and increasing the number of pores.²¹ On the other hand, the short cycle (Group I) presented fewer pores than the long cycle (Group II) due to reduced exposure to water.

When the roughness and color stability results were correlated, it was verified that there is no direct relationship between these two properties. The cycle with specimens that had higher roughness values was not the one in which specimens had color alteration, and vice versa (Table 2, Fig 2). Moreover, the mechanism causing porosity alteration is not the same as that causing a color change, as the polymerization method that most altered color was not the same as the one that had the highest mean porosity (Table 2, Fig 3); however, comparing porosity and roughness change, Group III, which presented higher roughness values, also had more porosity (Figs 2 and 3). As regards color stability, the dry-heat oven (Group III) seems to be a feasible alternative for polymerizing heat-activated acrylic resins, as it was the cycle that least affected the property of the material; however, this method demands a long time to perform.

Conclusions

In accordance with the results of this study:

- (1) Regarding color stability, all polymerization cycles presented clinically unacceptable alterations ($\Delta E > 3.3$).
- (2) Regarding surface roughness, the dry-heat oven (Group III) was the polymerization method with the lowest initial roughness; however, AAA interferes in this property.
- (3) Regarding porosity, it was observed that this property was directly affected by water sorption. This suggests that the longer the contact with water, the more pores in the acrylic resin.

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