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The Effect of Polymerization Cycles on Color Stability of Microwave-Processed Denture Base Resin

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Abstract

Purpose: This study evaluated the effect of different microwave polymerization cycles on the color changes of a microwave-processed denture base resin after accelerated aging and immersion in beverages.

Materials and Methods: Specimens of light pink acrylic resin were divided into three groups according to polymerization cycle: (A) 500 W for 3 minutes, (B) 90 W for 13 minutes + 500 W for 90 seconds, and (C) 320 W for 3 minutes + 0 W for 4 minutes + 720 W for 3 minutes. Control groups were a heat-processed acrylic resin (T) and a chemically activated denture repair resin (Q). Eight specimens per group were aged in an artificial aging chamber and evaluated at 20, 192, and 384 hours. Another series of 40 specimens per group were immersed in water, coffee, tea, cola, or red wine and evaluated at 1, 12, and 36 days. Color was measured by a spectrophotometer before and after aging or immersion. Color changes (ΔE) were analyzed by ANOVA/Bonferroni *t*-test ($\alpha = 0.05$).

Results: Mean ΔE ($\pm$SD) after 384 hours of accelerated aging were (A) 2.51 ± 0.50; (B) 3.16 ± 1.09; (C) 2.89 ± 1.06; (T) 2.64 ± 0.34; and (Q) 9.03 ± 0.40. Group Q had a significantly higher ΔE than the other groups. Color changes of immersed specimens were significantly influenced by solutions and time, but the five groups showed similar values. Mean ΔE at 36 days were (water) 1.4 ± 0.8; (coffee) 1.3 ± 0.6; (tea) 1.7 ± 0.5; (cola) 1.4 ± 0.7; and (red wine) 10.2 ± 2.7. Results were similar among the five test groups.

Conclusions: Color changes of the microwave-polymerized denture base resin tested were not affected by different polymerization cycles after accelerated aging or immersion in beverages. These changes were similar to the conventional heat-polymerized acrylic resin test, but lower than the repair resin after accelerated aging.

Polymeric dental materials are subject to color changes over time.[1] Color stability of denture base acrylic resins is a concern, as it is associated with esthetic reproduction of oral mucosa. Thus, colorants and other constituents of denture base resins should be stable during denture fabrication. In addition, those components should also resist adverse conditions subsequent to denture wearing.[2]

Discoloration of acrylic resins may be caused by several factors. Intrinsic factors such as degree of conversion and residual monomer can influence color stability. Another possible source of color change is the porosity caused by overheating or insufficient pressure during processing. Colorants may also undergo chemical changes or leach out.[3] Surface roughness, water sorption, and chemical reactivity are other factors associated with discoloration of dental polymeric materials.[4]

Color stability of denture base resins is associated with eating habits. It has been reported that certain beverages, such as tea, coffee, and wine, cause discoloration of acrylic resins.[5] The first deposits to form on complete dentures are usually salivary proteins and food. Subsequent accumulation of biofilm and calculus results in stains that may be difficult to remove.[6] Other extrinsic factors associated with color changes are the effect of cleaning solutions,[7] tobacco, composition of saliva, and denture hygiene habits.[6]

Microwave-processed acrylic resins were introduced in dental practice as an alternative to conventional heat-processed resins. Better accuracy and adaptation in the palatal seal area was found for complete dentures fabricated with microwavable resins.[8] Furthermore, microwave acrylic resins processed in less than 5 minutes show no significant difference in physical

properties when compared with conventional resins.[9-12] A recent study found that microwave-processed specimens showed a significantly higher flexural strength than specimens polymerized by water bath.[13] The polymerization cycle comprised 9 hours at 74°C, and the specimens were composed by the same resins tested in the present study (Clássico, Artigos Odontológicos Clássico Ltda, São Paulo, Brazil). Nevertheless, microwavable acrylic resins exhibit a different composition when compared with heat-processed materials. These differences could result in different discoloration rates over time. May et al[3,14] found that microwave-processed resins are less color stable than conventional materials after accelerated aging.

Despite data from the current literature, it has not been shown whether different microwave polymerization cycles influence color stability of denture base acrylic resins. Previous studies have assessed short cycles, based on microwave processing at 500 W for 3 minutes[14] or 5 minutes.[3] It was previously found that long microwave processing cycles alter some properties of microwavable resins, such as flexural strength.[15] If the properties are influenced by microwave processing cycles, it might be assumed that one cycle would provide the best outcomes. It was supposed that color stability of microwavable acrylic resins would be better when higher wattages and times are used for their polymerization.[14] Nevertheless, one cycle might combine good results with low energy consumption.

The purpose of this study was to investigate the effect of different cycles of microwave energy and time of cure on the color stability of a microwavable denture base resin. In this study, one conventional heat-polymerized denture material and one chemically activated denture repair resin were used as control. The resins were tested after accelerated aging and after immersion in four beverages thought of as staining agents.

Materials and methods

The experimental design included three experimental groups processed with different cycles of microwave energy and different times of cure. Two control groups consisting of heat-polymerized and chemically activated specimens, respectively, were also included (Table 1). Three brands of acrylic resins were used: one specifically designed for microwave polymerization (Onda-Cryl, Artigos Odontológicos Clássico Ltda), another for heat polymerization (Clássico) and another for denture repair (Jet, Artigos Odontológicos Clássico Ltda).

Forty-eight cylindrical specimens (15 mm diameter, 3 mm width) were obtained for each group (n = 240). Metal master patterns were invested in high-viscosity silicone (Zetalabor, Zhermack S.p.A, Badia Polesine, Rovigo, Italy), and supported by type III dental stone (Herodent, Vigodent SA Ind. Com., Rio de Janeiro, Brazil) within flasks. After the investing material had set, the flasks were separated and the master pattern was removed from the silicone mold. Denture base resins were mixed according to the manufacturer's recommendations, and each specimen was made from an individual mix of resin (7 ml of monomer to 21 ml of polymer). The denture resin was allowed to bench cure to the doughy stage in line with the manufacturer's recommendation before packing.

Table 1 Material, polymerization method, and cycle used for each group

Group	Material	Batch no. (monomer/ polymer)	Polymerization method
A	Onda-Cryl	150107/12507.0	Microwave: 3 minutes at 500 W
B	Onda-Cryl	150107/12507.0	Microwave: 13 minutes at 90 W (flask in vertical position) + 90 seconds at 500 W (flask in horizontal position)
C	Onda-Cryl	150107/12507.0	Microwave: 3 minutes at 320 W + 4 minutes at 0 W + 3 minutes at 720 W*
T	Clássico	220207/711070	Water bath: 9 hours at 74°C (control I)
Q	Jet	150107/2090.40	15 minutes at room temperature (control II)

*According to the manufacturer's recommendation.

Having reached the doughy consistency, the resin was packed into the silicone mold. A pneumatic press (PM-2000, Techno Máquinas Ltda, Vinhedo, Brazil) was used for trial packing the microwavable denture base resin initially at 500 kgf and, finally, at 1000 kgf, maintained for 60 minutes. Final packing for the control groups was conducted at 1250 kgf. The specimens in groups A, B, and C were processed with Onda-Cryl denture base resin and polymerized in a microwave oven (ME28S, Electrolux SA, Manaus, Brazil). Specific plastic flasks for microwave use were used. Specimens in group T were polymerized by a conventional heat method with metal flasks in an automatic polymerization water tank (Ribeirão Preto Dental School, Ribeirão Preto, Brazil). Temperature and time were 73°C for 90 minutes, followed by 30 minutes at 100°C. For group Q, no further procedure was carried out after final packing. All specimen groups were bench cooled for a minimum of 5 hours before deflasking. Each specimen was then finished using 200-, 400-, and 600-grit wet/dry sandpaper (Norton, Saint-Gobain Abrasivos Ltd, Guarulhos, Brazil) in a polishing machine (DPU-10, Panambra Ind. e Técn. S.A., São Paulo, Brazil) at 250 rpm for 60 seconds.

Color stability

Color change (ΔE) was measured according to Ma et al.[7] A portable spectrophotometer was employed (Color Guide 45/0, BYK-Gardner Latin America, Santo André, Brazil) and measurement was carried out in the center of each circular specimen. This instrument was used to quantify the tristimulus values and calculate the ΔE from data obtained before and after accelerated aging or immersion of the specimens. After initial color measurements, the specimens were aged or immersed in the solutions.

The magnitude of the total color difference is formulated by a single number ΔE:

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

Table 2 Results for ΔE after accelerated aging

	20 hours			192 hours			384 hours		
Group	Mean ± SD	CI		Mean ± SD	CI		Mean ± SD	CI	
A	1.16 ± 0.31	± 0.21	aA	1.71 ± 0.31	± 0.20	abA	2.51 ± 0.50	± 0.34	bA
B	1.32 ± 0.38	± 0.27	aA	1.76 ± 0.57	± 0.39	abA	3.16 ± 1.09	± 0.75	bA
C	1.34 ± 0.65	± 0.45	aA	2.24 ± 0.74	± 0.51	abA	2.89 ± 1.06	± 0.74	bA
T	1.55 ± 0.15	± 0.10	aA	2.23 ± 0.20	± 0.14	abA	2.64 ± 0.34	± 0.24	bA
Q	6.41 ± 0.65	± 0.45	aB	8.38 ± 1.01	± 0.70	bB	9.03 ± 0.40	± 0.28	bB

Horizontally, means with same lowercase letters are not significantly different. Vertically, means with same uppercase letters are not significantly different (the Bonferroni *t*-test, $P < 0.05$).
SD, standard deviation; CI, confidence interval ($\alpha = 0.05$).

where L* stands for lightness, a* for redness-greenness, and b* for yellowness-blueness.

Accelerated aging

After initial assessment, eight specimens for each group were placed in a weathering chamber (Sistema Acelerado de Envelhecimento para Não Metálicos, Comexim Mat. Primas Ind. e Com. Ltda, São Paulo, Brazil). Each specimen was exposed to the ultraviolet B light spectrum of four TL 40W/12RS lamps (Philips do Brasil Ltda, São Paulo, Brazil). The weathering chamber was set to produce an irradiance level of 80 W/m^2. Specimens were aged by alternating 4 hours of irradiation (dry) and 4 hours of 100% humidity (dark), that is, each aging cycle was 8 hours long. Accelerated aging employed a temperature of 50°C during both stages. The color of the specimens was measured after 40, 192, and 384 hours; this last period corresponds to 10 years of natural aging.[16]

Action of staining agents

The remaining specimens were then stored in different beverages at 37 ± 1°C. Eight specimens per group were stored in 200 ml of each solution, as follows: Solution 1, or (S1), distilled water (negative control); (S2) coffee (Utam, Café Utam S.A., Ribeirão Preto, Brazil), prepared according to the manufacturer; (S3) tea (Lipton Ice Tea Limão, AmBev, Jundiaí, Brazil); (S4) cola soft drink (Coca-Cola, Cia. de Bebidas Ipiranga, Ribeirão Preto, Brazil); and (S5) red wine (Chalise Tinto Seco, Vinícola Salton, Bento Gonçalves, Brazil). According to Guler et al,[4] the average time for consumption of 1 cup of a drink is 15 minutes, and among coffee drinkers, the average consumption of coffee is 3.2 cups per day. Therefore, 24 hours' storage time in coffee simulated consumption for 1 month. To compare results, the same time period was employed for the other solutions. Specimens were then evaluated after 1 day (T1), 12 days (T2), and 36 days (T3).

Statistical analyses

This study comprises two distinct experiments: (1) color stability after accelerated aging and (2) action of staining agents from beverages. Two-way ANOVA with one repeated factor (mixed design) was used to evaluate the effect of groups and time of accelerated aging on color change. Mixed design three-way ANOVA was used to assess the effect of groups, beverage, and immersion time on mean ΔE. The Bonferroni *t*-test was used for post hoc comparisons ($\alpha = 0.05$). All calculations were performed with statistical software (GMC Basic Software, v.2002, Ribeirão Preto Dental School, Ribeirão Preto, Brazil).

Results

Accelerated aging

Mean values and standard deviations of ΔE after accelerated aging are listed in Table 2. An evident association between aging time and color changes can be observed. According to the ANOVA, aging time, groups, and their interaction were significant (Table 3). The chemically activated acrylic resin group (Q) was less color stable than the others, regardless of time. Furthermore, ΔE of group Q was strongly different for each time tested. The other groups showed more stable results, while mean changes were nearly similar regardless of time; however, slight differences were observed. Group T presented a slight color change at 20 hours, and this alteration was slightly higher at the final time. Microwave-processed acrylic resin also suffered minor alterations after 20 hours, regardless of the polymerization cycle.

Action of staining agents

Three-way ANOVA demonstrated that solutions and time of immersion exerted significant influence on color change

Table 3 Mixed design two-way ANOVA for the accelerated aging experiment

Source of variation	SS	df	MS	F	*P*
Between specimens					
Group	669.55	4	167.39	176.41	<0.001*
Error	26.57	28	0.95		
Total	777.66	119			
Within specimens					
Time	57.30	2	28.65	147.24	<0.001*
Group × Time	9.27	8	1.16	5.95	0.004*
Error	14.98	77	0.19		

*Significant at $P < 0.05$.

Table 4 Mixed design three-way ANOVA for the staining experiment

Source of variation	SS	df	MS	F	*P*
Between specimens					
Group	0.74	4	0.19	0.08	0.988
Solution	2829.69	4	707.42	288.87	<0.001*
Group × Solution	49.03	16	3.06	1.25	0.234
Error	428.56	175	2.45		
Total	5534.94	599			
Within specimens					
Time	663.25	2	331.63	435.63	<0.001*
Group × Time	10.13	8	1.27	1.66	0.106
Solution × Time	1250.47	8	156.31	205.33	<0.001*
Group × Solution × Time	36.63	32	1.14	1.50	0.042*
Error	266.44	350	0.76		

*Significant at $P < 0.05$.

(Table 4). Furthermore, results indicated the presence of interaction among groups, solutions, and time of immersion. This implies that the difference in ΔE among the groups could have differed after immersion in each solution and at each time. Figure 1 shows the mean ΔE for the triple interaction. Post hoc comparison followed a simple effects approach and inspected which group contributed to the differences. This was done to improve statistical power as well as to clarify interpretation of results.[17] Mean color change was similar among the five groups, despite a few specific combinations between solutions and time.

Discussion

Differences in mean ΔE and the rate of color change after accelerated aging were observed for the five groups tested. The large ΔE for group Q is intrinsic to the chemically activated acrylic resin system. These materials are cured by the chemical reaction of benzoyl peroxide and an amine activator like dimethyl-*p*-toluidine; however, amines have been reported to produce yellowing upon oxidation.[14] This process causes discoloration after curing and further color change with aging.[18] Previous studies have also reported substantial color instability for autopolymerizing resins used in denture repair and provisional restorations when exposed to accelerated aging.[14,18,19]

Interaction analysis between time and groups showed that heat-polymerized and microwave-processed resin suffered a statistically significant color change after the initial period (20 hours). This implies that group T is as color stable as groups A, B, and C. In other words, differences in the chemical composition of microwavable resins tested do not influence color stability. The monomer formulated for microwave polymerization could contain either a triethylene or tetraethylene glycol dimethacrylate. This modification is necessary for processing at elevated temperatures, due to the low vapor pressure of dimethacrylates.[20] Haselton et al[21] found that a provisional denture resin containing triethylene glycol dimethacrylate presents less color stability than less complex methacrylate resins; however, it contains other components that may be detrimental to its color stability, that is, bis-GMA and urethane dimethacrylate. Similar porosity can be appointed as another reason for similar color stability. A previous study has reported that the Onda-Cryl resin was similar in porosity to the Clássico heat-polymerized resin.[10] May et al[3] found that another microwavable resin showed high rates of color change after accelerated aging, when compared to some conventional heat-polymerized resins. In the present study, there was no difference between these two categories of denture base materials. A possible reason is that the tested materials were obtained from the same manufacturer, thus presenting more similar compositions.

Correlations between clinical conditions and the present results are difficult to establish. Nevertheless, it was stated that color changes of ΔE less than 1.0 are not visually discernible, whereas a clinically acceptable value is less than 3.3.[22] Mean ΔE for the microwave- and heat-processed resins tested were below this second cut-off point following 5 and 10 years' simulation. Previously found differences between those materials present low magnitude and probably no clinical relevance. Furthermore, microwave polymerization cycles did not influence accelerated aging outcomes. An explanation for this similarity is that the three cycles possibly result in a comparable residual monomer level;[3] however, this statement is hypothetical and should be the subject of another investigation.

As far as beverage staining is concerned, it was found that wine had produced strong discoloration. A common characteristic of beverages like wine, tea, and coffee is the presence of tanning agents, which have strong chromogenic potential.[23] It was found in vitro that coffee and tea were able to cause a considerable gloss change in acrylic resin.[24] For composite resins, Guler et al[4] reported that cola and water caused similar discoloration, whereas coffee, tea, and red wine produced greater color change. Different results for coffee and tea could be explained by the presence of filler particles, which could have acted as a retentive factor for staining agents. Differences after immersion for microwave-processed resin and heat-polymerized resin were limited to specific interactions. Thus, it can be considered as clinically insignificant. Furthermore, no polymerization cycle has caused increased staining. It can be pointed out that microwave-processed resin can be employed with any polymerization cycle tested if color stability is a concern.

An important limitation of this study was that beverages were not associated with denture biofilm. The accumulation of biofilm acts as a matrix for the deposition of stains[6,23] and could lead to greater color changes. Nevertheless, it can be inferred that a well-polished and cleansed surface will show minimal discoloration following exposure to coffee, tea, and cola soft drinks. Red wine consumption can lead to additional staining, which may be removed by denture hygiene methods, as found for coffee.[25] Thus, future studies should evaluate the effectiveness of denture cleansers or brushing on stain removal from acrylic resin materials. The association between discoloration by beverages and biofilm or debris on acrylic resin should also be further assessed.

Conclusions

Color changes after accelerated aging for the microwavable acrylic resin tested were not influenced by polymerization

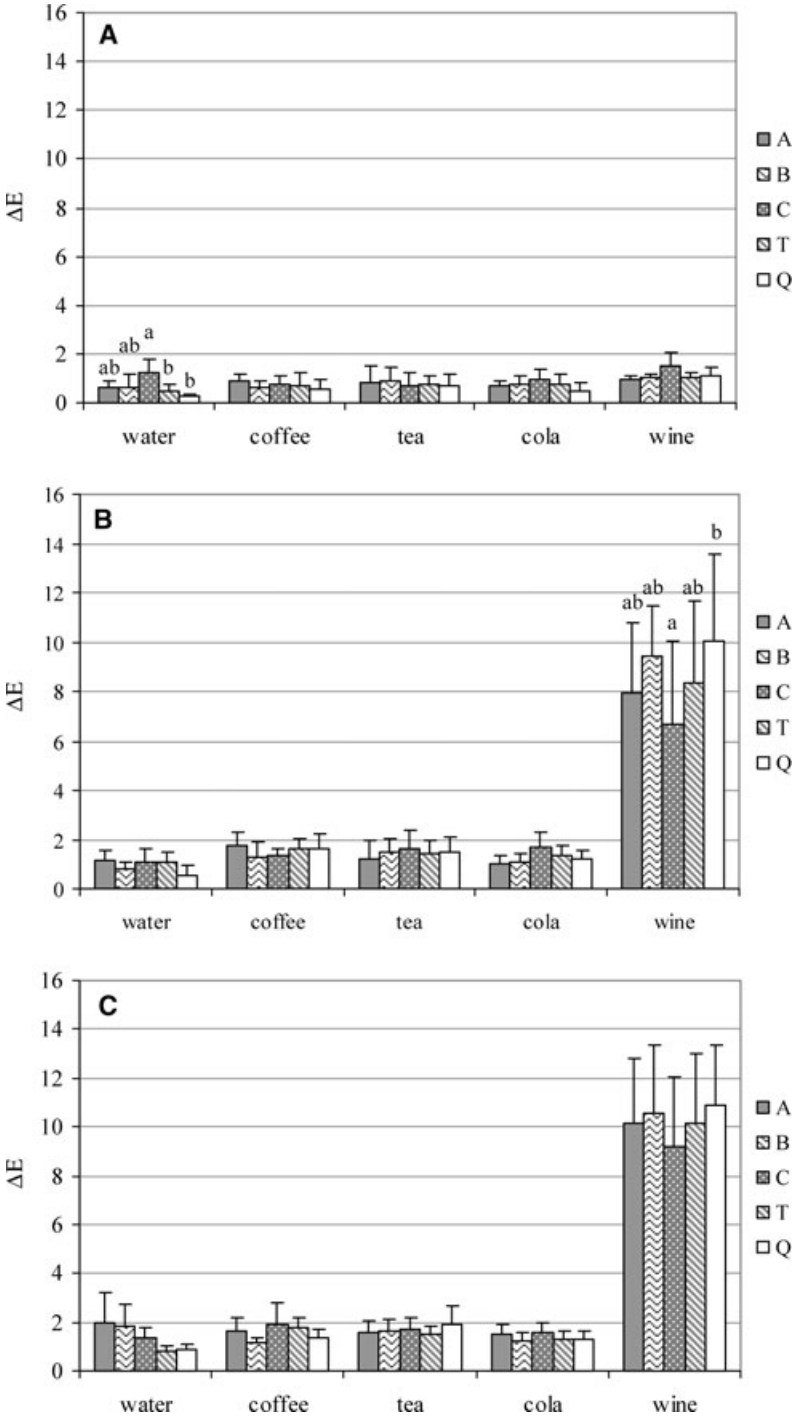

Figure 1 Mean ΔE and standard deviations for the interaction between groups and solutions after (A) 1 day; (B) 12 days; and (C) 36 days of storage in beverages (n = 8 each bar). Where relevant, means under the same lowercase letter are not significantly different (Bonferroni *t*-test, $\alpha = 0.05$).

cycles. Heat-polymerized acrylic resin showed similar changes; however, the repair resin tested was less stable. These findings are possibly due to compositional differences among materials, such as the presence of a chemical activator.

It was also concluded that the three evaluated acrylic resins experienced color changes after immersion in beverages; however, the three microwave polymerization cycles led to similar color stability for the microwavable resin.

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