

# Effect of Postpolymerization Method on the Color Stability of Composite Resins Submitted to Ultraviolet Aging



Paulo Henrique dos Santos, DDS, MSc, PhD\*  
Fernando Isquierdo de Souza, DDS\*\*  
Ana Paula Albuquerque Guedes, DDS, MSc\*\*\*  
Sabrina Pavan, DDS, MSc, PhD\*\*\*\*

*The aim of this research was to evaluate the effect of postpolymerization method on the color stability of resin-based composites. Samples of direct and indirect restorative materials were polymerized with two photo-curing units (Visio photo-curing oven system and LED Elipar Freelight 2). All samples were submitted to an initial chromatic analysis using a spectrometer and submitted to ultraviolet-accelerated artificial aging. The direct material showed less color change than the indirect material, independent of the photo-activation method used. Samples photo cured with the LED system showed less change than those photo cured with the Visio system. The postpolymerization oven did not improve the color stability of direct and indirect resin-based composites. (Int J Periodontics Restorative Dent 2012;32:e95–e100.)*

\*Assistant Professor, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University–UNESP, Araçatuba, SP, Brazil.

\*\*Graduate Student, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University–UNESP, Araçatuba, SP, Brazil.

\*\*\*Postgraduate Student, Department of Restorative Dentistry, Araçatuba School of Dentistry, São Paulo State University–UNESP, Araçatuba, SP, Brazil.

\*\*\*\*Assistant Professor, Department of Dentistry, Adamantina School of Dentistry, FAI, Adamantina, SP, Brazil.

Correspondence to: Dr Paulo Henrique dos Santos, Rua Jose Bonifacio, 1193, Araçatuba, SP, Brazil 16015-050; fax: 55 18 36363245; email: paulosantos@foa.unesp.br.

The increasing demand for esthetic restorative treatment has led restorative material industries and researchers all over the world to seek an ideal restorative material that satisfies the requirements of beauty as well as biologic and mechanical principles. Technologic advances in materials science have resulted in the development of dental composites with different formulations, such as ormocer (organically modified ceramics) and nanofilled materials. Some of the advantages of dental composites are less polymerization shrinkage, better surface polishing and finishing,<sup>1,2</sup> lower cost, and simpler manipulation when compared with ceramics.<sup>3</sup>

These composites differ from direct restorative composites by presenting a higher degree of conversion and a more homogenous polymerization.<sup>4</sup> Over the past few years, a new generation of indirect resins has been developed called ceromers (ceramic optimized polymers), with higher inorganic particle density in comparison with traditional composites.<sup>5</sup> These materials have a postpolymerization system that

**Table 1** Materials used

Composite	Batch no.	Resinous matrix	Filler
Sinfony (3M ESPE)	177130	Aliphatic and cycloaliphatic monomers (UDMA)	50 wt% of filler: aluminum glass and silica
Filtek Z-250 (3M ESPE)	5RF	bis-GMA, UDMA, and bis-EMA	84.5 wt% of filler: zirconium/silica

UDMA = urethane dimethacrylate; bis-GMA = bisphenol glycidyl methacrylate; bis-EMA = ethoxylated bisphenol-A dimethacrylate.

generally results in greater flexural strength and possess a wear rate similar to that of the dental structure.<sup>5-8</sup>

Color change is the main reason for replacing prosthetic restorations.<sup>9</sup> There are two types of discoloration described in the literature: exogenous, caused by roughness and surface changes resulting in dental plaque accumulation and pigmentation by staining agents, and endogenous, a change of intrinsic color in deep portions of the composite resin caused by chemical and physical reactions resulting from exposure to heat and ultraviolet (UV) radiation.<sup>10</sup> According to Lu and Powers,<sup>11</sup> artificial aging in a climatic chamber increased the conditions of temperature, humidity, and UV light. These extreme conditions could cause irreversible endogenous discoloration in the material.<sup>12</sup>

Thus, color stability is an important parameter for direct and indirect esthetic restorations and is fundamental to the clinical success of restorative treatment. Moreover, the efficiency of the postpolymerization method to improve color stability is still a concern in restorative procedures. In this study, two

null hypotheses were tested: there is no difference between direct and indirect composite resin in relation to color stability and there is no difference in the color stability between a direct photo-curing unit and a photo-curing oven used to cure indirect resin-based composite materials (postpolymerization system).

### Method and materials

In this study, two resin-based composites were used: one indicated for the direct (Filtek Z-250) and the other for the indirect restorative technique (Sinfony) (Table 1).

#### Preparation of specimens

Samples of both composites were fabricated using a metal matrix 1.2-mm thick and 1.5 mm in diameter, polymerized according to the following techniques (n = 10):

- Photo-curing oven system: Composites were prepolymerized for 5 seconds with the Visio Alfa halogen light-curing

unit (400 mW/cm<sup>2</sup>, 3M ESPE) and postpolymerized using the Visio Beta Vario system (3M ESPE) containing four fluorescent bulbs (9 W each) under vacuum for 15 minutes

- Light-emitting diode (LED) photo-curing unit: Composites were photo cured using Elipar Freelight 2 (1,200 mW/cm<sup>2</sup>, 3M ESPE) for 20 seconds

Samples were polished using an automatic polishing machine (APL-4, Arotec) with 360-, 600-, and 1,200-grit abrasive papers under water irrigation and cleaned using an ultrasonic cleaner (model 2210, Branson Ultrasonic) with distilled water for 2 minutes at the end of the polishing process.

#### Color analysis and UV aging

All samples were submitted to initial chromatic analysis using a visible UV reflections spectrometer (UV-2450, Shimadzu), and color was evaluated using the Commission Internationale de l'Eclairage L\*a\*b\* system, consisting of axes a\* and b\* (a\*, red-green proportion; b\*,

**Table 2 Two-way analysis of variance**

	df	Sum of squares	Mean square	F	P
Composite	1	28.2491634	28.2491634	92.1668	.00001
Photo-activation method	1	20.4561182	20.4561182	66.7409	.00001
Composite × method	1	1.5043060	1.5043060	4.9080	.03123
Residue	36	11.0340187	0.3065005		

**Table 3 Mean color change after UV aging**

Photo-activation method	Color change*	
	Filtek Z-250	Sinfony
Visio photo-curing oven system	3.27 (0.56) <sup>A,a</sup>	5.33 (0.38) <sup>A,b</sup>
LED Elipar Freelight 2	2.22 (0.55) <sup>B,a</sup>	3.52 (0.67) <sup>B,b</sup>

\*Different capital letters in columns and lowercase letters in rows denote significant differences ( $P < .05$ ).

yellow-blue proportion) and  $L^*$  (luminosity), which is perpendicular to the  $a^*b^*$  plane. With this system, each color can be specified with the coordinates  $L^*$ ,  $a^*$ , and  $b^*$ .<sup>13,14</sup>

After the initial chromatic analysis, samples were submitted to an artificial UV aging process. Accelerated aging was performed in an aging chamber (QUV Accelerated Weathering Tester, Q-Panel) simulating extreme environmental conditions. The aging process consisted of alternating periods of UV light and condensation, with 8 hours of UV light at 60°C and 4 hours of condensation at 40°C and humidity (50% to 100%) produced using a UVB lamp (UVB-313, Q-Lab). The

color analysis was performed after the aging period, corresponding to 384 hours (128 hours of condensation, 256 hours of UVB exposure)

Color stability was determined by computing the difference ( $\Delta E$ ) between the coordinates obtained from samples before and after the aging procedure. Total color change ( $\Delta E$ ) is usually used to differentiate color and is calculated as follows:  $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ .

Data were submitted to two-way analysis of variance (ANOVA) and the Tukey test ( $\alpha = .05$ ). The two factors of variation analyzed were type of composite resin and method of photo-activation.

## Results

Table 2 illustrates the results of the two-way ANOVA, which shows that for both factors studied as well the interaction between them, there was a significant difference between the groups ( $P < .05$ ).

The results in Table 3 indicate that Filtek Z-250 showed less color change ( $\Delta E$ ,  $3.27 \pm 0.56$ ) in comparison with Sinfony indirect composite ( $\Delta E$ ,  $5.33 \pm 0.38$ ) for the Visio photo-curing oven system ( $P < .05$ ). Similarly, Filtek Z-250 resin showed less color change ( $\Delta E$ ,  $2.22 \pm 0.55$ ) when compared with Sinfony ( $\Delta E$ ,  $3.52 \pm 0.67$ ) when photo-activation was per-

formed with the Elipar Freelight 2 system ( $P < .05$ ). For Filtek Z-250, the Visio photo-curing oven system showed greater color change ( $\Delta E$ ,  $3.27 \pm 0.56$ ) when compared with the Elipar Freelight 2 ( $\Delta E$ ,  $2.22 \pm 0.55$ ) ( $P < .05$ ). Similar results were obtained with indirect Sinfony resin, in which the Visio system ( $\Delta E$ ,  $5.33 \pm 0.38$ ) showed greater color change than Elipar Freelight 2 ( $\Delta E$ ,  $3.52 \pm 0.67$ ) ( $P < .05$ ).

## Discussion

The two null hypotheses were rejected. There was a difference between direct and indirect composite resin in relation to color stability as well as a difference in the color stability between the direct photo-curing unit (LED) and photo-curing oven used to cure indirect resin-based composite material (postpolymerization system). Some aspects could have contributed to discoloration, including the photoinitiator systems contained in the composite resin, organic matrix and polymerization inhibitors,<sup>15</sup> photo-curing units, time of irradiation, and degree of conversion achieved by the composite.<sup>16,17</sup>

The results showed that Filtek Z-250 direct restorative material obtained better color stability than Sinfony indirect composite resin for both photo-activation methods. Kakaboura et al<sup>16</sup> showed a low degree of conversion for Sinfony restorative material in comparison with other indirect composite resins, achieving 66% with the Visio photo-curing system. As illustrated

in Table 1, the composites exhibited different formulations, with Sinfony showing the highest organic matrix proportion and, consequently, fewer load filler particles. Composite resins with a lower inorganic particle content tend to present poor color stability.<sup>14</sup> Samra et al<sup>18</sup> also showed that composites with a lower filler content showed the worst discoloration.

According to Janda et al,<sup>19</sup> camphoroquinone is the most common photoinitiator used in dental composite resins, and although presented in small quantities in the composition, its influence on material color is significant. According to these authors, the poor polymerization could not convert the camphoroquinone completely, leaving yellow traces in the resin coloration. The aging process could cause oxidation of the amines required in the photo-activation of materials,<sup>11</sup> causing discoloration that tends to go from yellow to brown.<sup>19</sup>

Several authors have reported that composite resins allow water sorption at the matrix or the matrix-filler load interface,<sup>20-22</sup> causing hydrolytic degradation. Wozniak et al,<sup>23</sup> using scanning electron microscopy, observed that composite resins exposed to UV light presented high surface roughness with exposed filler in addition to microfractures on the surface, a phenomenon that could cause significant color change irrespective of the photo-activation method.

In addition to the polymerization time and the type of monomer,<sup>21</sup> the type of curing light

should also be considered.<sup>24</sup> The distribution of wavelength and light intensity may be two of the most important factors in the properties of resin materials after polymerization. Correr et al<sup>17</sup> found that LED curing units emit a narrow wavelength (450 to 490 nm), reaching the highest absorption rate of camphoroquinone (468 nm). Moreover, the high specific energy density of this photo-curing unit is another advantage of using these camphoroquinone/amine resin material systems. The degree of conversion depends on energy density, since low energy density generates composites with a low degree of conversion and mechanical properties.<sup>17</sup> These aspects could explain the higher color stability of the composites photo cured using the LED Elipar Freelight 2 system.

The Visio system consists of an initial curing by a halogen photo-curing unit (Visio Alfa) and a final processing in a vacuum chamber with four fluorescent lamps (Visio Beta Vario). Polymerization under vacuum contributes to polymerization with less superficial porosity.<sup>16</sup> The increase in the degree of conversion would act on important mechanical properties of the composite resins, decreasing the solubility, improving dimensional stability, minimizing color change, and contributing to the biocompatibility of the material.<sup>25</sup> Incompletely polymerized composite resin shows reduced mechanical properties and greater susceptibility to discoloration.<sup>26</sup> According to Dietschi et al,<sup>27</sup> the additional

polymerization is not effective for all resin-based materials and could be compensated by cross-linking reactions extending for 1 week after initial polymerization. Significant differences were also found for two second-generation laboratory-processed resin-based composites.<sup>16</sup> Samra et al<sup>18</sup> showed no direct correlation between post-polymerization system and greater color stability. Matsumura et al<sup>28</sup> described the distance between the light source and the material surface as one of the critical factors of photo-curing units. This difference in the distance between hand and laboratory units could affect the depth of polymerization. Based on the results of the present study, the postpolymerization procedure was not able to improve the color stability of resin-based composites, showing values of color change considered clinically unacceptable.

Chromatic changes with  $\Delta E$  values between 0 and 2 would be imperceptible clinically, but those between 2 and 3 would be clearly perceptible.<sup>29</sup> Ruyter et al<sup>30</sup> considered  $\Delta E$  values close to 3.3 as clinically acceptable. The main limitation of this study was that the extreme climatic simulation could not be directly translated to the intraoral situation.<sup>11</sup> Powers et al<sup>31</sup> showed that after 300 hours of accelerated aging, composite resin materials had color parameter changes similar to those observed clinically in a 24-month study. Clinically, other factors such as humidity, temperature, and conditions of oral cavity biochemistry, as well as a

possible combination of these factors, could cause the degradation of composite resins over a long period of time.<sup>23</sup>

## Conclusion

According to the results and within the limitations of this study, it can be concluded that the smallest color changes were obtained using the LED Elipar Freelight 2 system when compared with the postpolymerization system using fluorescent lamp and vacuum. Filtek Z-250 direct restorative resin-based composite showed higher color stability when compared with the Sinfony indirect restorative resin-based material for both photo-activation systems.

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