

# The Effect of Curing Units and Staining Solutions on the Color Stability of Resin Composites

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## Clinical Relevance

The color stability of resin composites did not vary based on the type of light curing unit. The effect of staining solutions on color changes in resin composites was resin composite and immersion time-dependent.

## SUMMARY

**This study investigated the effects of two different light curing units and two staining solutions on the color stability of a hybrid composite and a nanohybrid composite after different immersion periods. Thirty disk-shaped specimens (10 mm in diameter, 2-mm thick) were fabricated for each of the resin composites, Clearfil AP-X and Filtek Supreme. The specimens were randomly**

**divided into two groups according to the curing unit used: Group I specimens (n=15) were cured with a quartz-tungsten-halogen (QTH) light for 40 seconds, and Group II specimens (n=15) were cured with a light-emitting diode (LED) unit in standard mode for 40 seconds. The specimens were incubated in 100% humidity at 37°C for 24 hours. Then, the baseline color values ( $L^*$ ,  $a^*$ ,  $b^*$ ) of each specimen were measured with a spectrophotometer according to the CIELab color scale. After baseline color measurements, five randomly selected specimens from each group (Groups I and II) were immersed in one of two staining solutions (tea or coffee) or distilled water (control). After 1, 7 and 30 days of immersion, the color values of each specimen were remeasured and the color change value ( $\Delta E^*_{ab}$ ) calculated. Color changes caused by immersion in tea and coffee for 30 days were only perceptible in the Clearfil AP-X specimens cured with QTH or LED. In the Filtek Supreme specimens, coffee perceptibly stained the teeth after all immersion periods and tea stained after 30 days. Polymerization with QTH or LED did not cause any significant difference in the color stability of**

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**Clearfil AP-X or Filtek Supreme. While there were no significant differences between staining solutions in the Clearfil AP-X specimens cured with LED after one and seven days of storage and one day of storage in the QTH cured specimens, significant differences were observed between water and coffee after seven days of storage. In the Filtek Supreme specimens cured with QTH or LED, there were statistically significant differences between the staining solutions after one and seven days of storage. After 30 days of storage, no significant difference was found between tea and coffee in either resin composite cured with QTH or LED. The effect of the staining solutions (tea, coffee) on color changes in composites was immersion time and resin-material dependent.**

## INTRODUCTION

Resin composite restorations have begun to constitute a significant portion of dentists' routine practice due to patients' great demand for an esthetic appearance. Color plays an important role in obtaining natural-looking results. However, the discoloration of composite restorations occurs from time to time, and clinicians generally need to replace the restorations.<sup>1-2</sup>

The color stability of resin composites is affected by several extrinsic and intrinsic factors.<sup>3</sup> External discoloration can be the result of dietary and smoking habits, bad oral hygiene and adsorption or absorption of water-soluble stains throughout the resin matrix.<sup>4-6</sup> The composition and size of the filler particles affect surface smoothness and susceptibility to extrinsic staining.<sup>7</sup>

Intrinsic factors, such as the resin matrix of composites<sup>8-9</sup> and incomplete polymerization,<sup>10-11</sup> have a considerable influence on color stability. This is usually attributable to chemical degeneration of the filler-resin bond and solubility of the resin matrix.<sup>3,9</sup> Adequate polymerization of resin composites is important to ensure optimum physico-mechanical properties. The effectiveness of polymerization is not only dependent upon the chemistry of the material and the filler particle size, but also on the light curing units, including spectral distribution, exposure time and intensity.<sup>12-13</sup>

Today, there are a range of light-curing units, such as conventional quartz-tungsten-halogen (QTH), light-emitting diode (LED), plasma arc (PAC) and laser. Although the most common light-curing unit is the QTH light, it has some drawbacks, including deterioration of the bulb, reflector or filter and a decrease in light output with time.<sup>14-15</sup> A decrease in light output will cause a low degree of monomer conversion, which will affect the color stability of the composite.<sup>16</sup> The extent of polymerization depends on the conversion percent or the ratio of double bonds converted to single bonds. Composites with a higher percent conversion have greater mechanical properties, greater wear resistance and better color stability.<sup>17</sup> LED curing units were developed to overcome the problems inherent with halogen units. These curing units produce a narrow band of wavelengths, around 470 nm, which coincides with the optimum absorption wavelength for camphorquinone photoinitiator.<sup>18-19</sup>

Despite the increase in the use of LED curing units, research comparing resin composite color change associated with halogen and LED curing lights is generally limited. Therefore, this study evaluated the influence of two different light-curing units (QTH and LED) and staining solutions (tea and coffee) on the color stability of Clearfil AP-X (hybrid composite) and Filtek Supreme (nanohybrid composite) after three immersion periods (1, 7 and 30 days).

## METHODS AND MATERIALS

A hybrid resin composite, Clearfil AP-X (Kuraray, Osaka, Japan), and a nanohybrid resin composite, Filtek Supreme (3M ESPE, St Paul, MN, USA) of A2 shade, were used (Table 1). Thirty disk-shaped specimens from each resin composite, 10 mm in diameter and 2 mm thick, were prepared in a polytetrafluoroethylene ring covered with a celluloid matrix and glass slides. The specimens were randomly divided into two groups according to the curing unit used: Group I specimens (n=15) were cured with a QTH light (Hilux, Benlioglu, Ankara, Turkey) for 40 seconds having a light intensity of 450 mW/cm<sup>2</sup>, and Group II specimens (n=15) were cured with an LED unit (Elipar Freelight, 3M ESPE) in standard mode for 40 seconds having a light intensity of 400 mW/cm<sup>2</sup> from both the top and bottom (Table 2). The output of the curing units was

Table 1: Composition of the Resin Composite Materials Used

Resin Composite	Filler Weight (%)	Filler Volume (%)	Filler Type	Filler Size	Monomer Composition
<b>Clearfil AP-X</b> (Kuraray Co, Osaka, Japan) Batch #00746A	86	70	Silanated barium glass and silanated colloidal silica	0.1-15 µm	Bis-GMA, TEGDMA
<b>Filtek Supreme</b> (3M, ESPE, St Paul, MN, USA) Batch #3AG	78.5	58-60	Silica, zirconium oxide, aluminum oxide	5-20 nm with 20 nm silica filler	Bis-GMA, TEGDMA, Bis-EMA, UDMA

checked with a radiometer (Kerr, Demetron, Orange, CA, USA). The distance between the light and specimen was standardized by use of a 1-mm glass slide. No polishing techniques were used to avoid modification of the surfaces, which may have influenced the results. The specimens were incubated in 100% humidity at 37°C for 24 hours. Then, the baseline color values ( $L^*$ ,  $a^*$ ,  $b^*$ ) of each specimen were measured with a spectrophotometer (Minolta CM-3600d, Minolta Co, Osaka, Japan) according to the Commission Internationale d'Eclairage (CIE Lab). The spectrophotometer was calibrated with a standard white card before each group of specimens was measured, and the card was used as a background when measuring all specimens. The measurements were repeated twice in each sample and the mean values were calculated.

After taking the baseline color measurements, five randomly selected specimens from each group (Groups I and II) were immersed in one of two staining solutions (tea or coffee) or distilled water (control) for 1, 7 or 30 days. The tea solution (Yellow Label, Lipton, Rize, Turkey) was prepared by immersing two prefabricated tea bags (2 x 2 g) into 250 ml of boiling water for three minutes. To prepare the coffee solution, 5 g of instant coffee (Nescafe Classic, Nestle, Istanbul, Turkey) was poured into 250 ml of boiling water. After stirring for one minute, the solution was filtered through filter paper. The solutions were prepared daily.

After 1, 7 and 30 days of immersion, the color values of each specimen were remeasured, and the color change value ( $\Delta E^*_{ab}$ ) was calculated according to the following formula:<sup>20</sup>

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

where  $L^*$  represents lightness,  $a^*$  green-red ( $-a$  = green;  $+a$  = red) and  $b^*$  blue-yellow ( $-b$  = blue;  $+b$  = yellow). Before each measurement, the immersed specimens were rinsed with water for one minute and dried with absorbent paper.

Statistical Analysis

After the different immersion periods, a Mann-Whitney U-test was used to compare the color changes among the resin composites in the three different solutions.

To compare the effects of different light curing units (QTH and LED) on color change in each resin composite, a Kruskal-Wallis test was conducted.

A Kruskal-Wallis multiple comparison test was used to compare the staining solutions after each immersion period.

RESULTS

Table 3 presents the mean and standard deviations of color change values ( $\Delta E_{ab}$ ) in Clearfil AP-X and Filtek Supreme after polymerization with different light curing units and after immersion in the different staining solutions for different periods of time. Staining was considered visually perceptible when the  $\Delta E_{ab}$  value was greater than 3.3.<sup>21</sup> In the Clearfil AP-X specimens cured

Table 2: Light Curing Units Used			
Light-curing Unit	Manufacturer	Curing Mode	Curing Profile
Hilux (Quartz-Tungsten-Halogen)	Benlioglu Dental, Ankara, Turkey	Standard	450 mW/cm <sup>2</sup> (40 seconds)
Elipar Freelight (LED)	3M, ESPE, St Paul, MN, USA	Standard	400 mW/cm <sup>2</sup> (40 seconds)

Table 3: Mean Values and Standard Deviations of Color Change Values ( $\Delta E_{ab}$ )					
Resin Composite	Curing Units	Immersion Period	Staining Solutions		
			Water	Tea	Coffee
Clearfil AP-X	QTH	1 day	2.16(0.65)	2.34(0.91)	3.11(0.37)
		7 days	1.25(0.48)	1.93(0.84)	2.70(0.60) <sup>c</sup>
		30 days	2.08(0.53)	3.93(0.45) <sup>a</sup>	5.04(0.99) <sup>d</sup>
	LED	1 day	1.18(0.73)	2.45(1.21)	1.91(0.66) <sup>e</sup>
		7 days	1.65(0.53)	2.17(1.00)	2.47(0.74) <sup>f</sup>
		30 days	1.85(0.94)	4.82(1.04) <sup>b</sup>	4.25(0.31) <sup>g</sup>
Filtek Supreme	QTH	1 day	1.00(0.62)	2.49(0.54)	4.20(0.79) <sup>h</sup>
		7 days	1.33(0.18)	2.44(0.69)	5.50(0.83) <sup>c</sup>
		30 days	1.49(0.53)	8.10(2.28) <sup>a</sup>	7.95(1.59) <sup>d</sup>
	LED	1 day	0.60(0.14)	2.14(0.19)	3.56(0.58) <sup>e</sup>
		7 days	1.17(0.40)	2.24(0.71)	4.26(0.39) <sup>f</sup>
		30 days	1.54(0.62)	8.51(1.13) <sup>b</sup>	7.11(0.97) <sup>g</sup>
<sup>a</sup> Indicates clinically perceptible values ( $\Delta E_{ab}>3.3$ ). Same superscript letters in the same column indicate statistical significance (p<0.05).					

with QTH or LED, only color changes caused by immersion in tea and coffee for 30 days were perceptible ( $\Delta E_{ab} > 3.3$ ). There was no perceptible change in color for the rest of the groups. Coffee perceptibly stained the Filtek Supreme specimens (cured with QTH or LED) for all immersion periods. Color changes in the Filtek Supreme specimens caused by tea immersion for 30 days were perceptible.

Significant differences were observed between the QTH cured Filtek Supreme and Clearfil AP-X specimens immersed in tea for 30 days ( $p=0.009$ ) and those immersed in coffee for seven days ( $p=0.009$ ) and 30 days ( $p=0.028$ ). When comparing LED cured Filtek Supreme and Clearfil AP-X specimens, there were significant differences in all immersion periods in coffee (1 day  $p=0.016$ , 7 days  $p=0.009$ , 30 days  $p=0.009$ ) and in tea for 30 days of immersion ( $p=0.009$ ).

No significant differences in color changes were found between QTH and LED cured Clearfil AP-X and Filtek Supreme specimens immersed in different staining solutions for different periods.

There were no significant differences between staining solutions after one day of immersion of the Clearfil AP-X specimens cured with QTH or LED. Significant differences were only observed between water and coffee in the QTH cured Clearfil AP-X specimens; whereas, no difference was seen between the staining solutions in LED cured Clearfil AP-X specimens after seven days of immersion. Significant differences were observed between water and tea and water and coffee; whereas no difference was seen between coffee and tea after 30 days of immersion (Table 4).

In the Filtek Supreme specimens cured with QTH or LED, there were significant differences among all staining solutions after one and seven days of immersion. After 30 days of immersion, while differences between water and tea and water and coffee were significant, the difference between tea and coffee was not (Table 4).

## DISCUSSION

The color stability of a resin composite is related to the resin matrix, dimensions of filler particles, depth of polymerization and staining agents.<sup>6,8-9,11,16,21-22</sup> The resin composite should be adequately polymerized to achieve optimal mechanical and optical properties. An under-polymerized resin composite can produce undesirable effects, such as water absorption and solubility of the unreacted monomers,<sup>23</sup> making it is more susceptible to staining.

In this study, polymerization with QTH or LED did not cause any significant difference in the color stability of Clearfil AP-X or Filtek Supreme. Recent studies have shown that surface hardness obtained with LED light, which is evidence of an adequate degree of polymerization on the surface, was comparable to that from QTH curing lights.<sup>24-25</sup> Since the degree of conversion has been reported to influence the discoloration of composites,<sup>16</sup> a surface with a lower degree of polymerization can exhibit increased discoloration. In this study, the light intensity of QTH was 18000 mJ/cm<sup>2</sup> and LED was 16000 mJ/cm<sup>2</sup>. Because these light intensities were similar, the samples might have been polymerized to equal degrees.

In a study determining color changes in a composite cured with halogen, high intensity halogen, PAC and LED curing units after two years, the highest color changes were recorded from the specimens cured with PAC light.<sup>26</sup> Similar to the findings in the current study, no difference was observed between halogen and LED cured specimens.

When evaluating the effects of staining solutions on the color stability of Clearfil AP-X and Filtek Supreme, significantly higher discoloration was found on Filtek Supreme after 30 days of storage in tea and coffee. The composite color changes were related to their compositions. Clearfil AP-X is a hybrid composite with a particle size range of 0.1-15  $\mu$ m, with filler loading 70% by volume. Filtek Supreme is a nanofilled composite, with filler loading 58-60% by volume and a particle size of

Table 4: Kruskal-Wallis Multiple Comparison Test Results for  $\Delta E_{ab}$  in Terms of the Staining Solutions

Immersion Period	Staining Solutions	Clearfil AP-X		Filtek Supreme	
		QTH	LED	QTH	LED
1 Day	Water-Tea	-	-	+	+
	Water-Coffee	-	-	+	+
	Tea-Coffee	-	-	+	+
7 Days	Water-Tea	-	-	+	+
	Water-Coffee	+	-	+	+
	Tea-Coffee	-	-	+	+
30 Days	Water-Tea	+	+	+	+
	Water-Coffee	+	+	+	+
	Tea-Coffee	-	-	-	-

+ indicates statistically significant difference ( $p < 0.05$ ).

- indicates no statistically significant difference ( $p > 0.05$ ).



0.6-1.4  $\mu\text{m}$ . The composition and size of the filler particles affect surface smoothness and the susceptibility for extrinsic staining.<sup>7</sup> Therefore, it can be expected that Filtek Supreme, with a smaller particle size, will have a smoother surface and will retain less surface stains than rough surfaces. However, Filtek Supreme, which had been subjected to tea and coffee solutions, had the highest color change. This could be related to the difference in filler volumes of the two resin composites. Filtek Supreme, with a lower filler content, might have absorbed more water at the matrix-filler interface. The absorbed water causes filler-matrix debonding or hydrolytic degradation of the filler.<sup>27</sup> This finding is in accordance with previous studies concluding that the composites with the lowest filler contents had poor color stability.<sup>28-29</sup>

Filtek Supreme is both BisGMA- and BisEMA-based. BisEMA, which is an ethoxylated version of BisGMA, is highly hydrophobic, as it does not contain any unreacted hydroxyl groups on the main polymer chain.<sup>30</sup> Therefore, it might have been expected that Filtek Supreme would have been more resistant to staining. Yap and others<sup>31</sup> found that BisEMA-based composites were highly resistant to the degradation effect of food-simulating liquids. However, in the current study, it was surprising that Filtek Supreme exhibited significant color changes. Ertas and others<sup>32</sup> evaluated the discoloration of different types of resin composites exposed to different drinks. Similar to the findings of the current study, they found that the nanohybrids Filtek Supreme and Grandio demonstrated more color changes than the posterior composite P60 and hybrid composite Z250. Ertas and others<sup>32</sup> attributed these results to the agglomerated particles and nanoclusters present in Filtek Supreme. They stated that these particles were less color-resistant than the zirconia-silica micron-sized fillers present in Z250 and P60, which might have been due to the former's relatively high water sorption character.

Villalta and others<sup>33</sup> investigated the effects of two staining solutions (coffee and wine) and three bleaching systems on color changes in a nanocomposite (Filtek Supreme) and a microhybrid composite (Esthet-X). In agreement with the current study, the nanocomposite had a greater color change than the microhybrid composite after staining.

In the current study, the results show that the effect of coffee on color change in Filtek Supreme and Clearfil AP-X was similar to tea after 30 days of immersion. In a study by Güler and others,<sup>34</sup> tea, coffee, cola, red wine and cherry juice were used as staining agents to evaluate the stainability of five provisional restorative materials, and it was found that red wine caused the highest color difference. Contrary to the results of the current study, coffee was found to stain more than tea.

Many studies also reported that coffee causes more discoloration than tea.<sup>32,35</sup> The discrepancy between previous studies and the current study could be attributed to the immersion periods. As tea and coffee are the most frequently consumed drinks, the authors wanted to observe their effects on long-term usage.

In many studies, the values of  $\Delta E_{ab}$  that were higher than 3.3 were considered clinically unacceptable.<sup>21,36</sup> In this study, the color changes of Clearfil AP-X exceeded a  $\Delta E_{ab}$  value of 3.3 only after 30 days of immersion in tea and coffee solutions. Filtek Supreme specimens demonstrated perceptible color changes after immersion in coffee for all immersion periods and after 30 days of immersion in tea. Therefore, the authors can conclude that immersion time is a critical factor in the color stability of resin composites. This finding is in agreement with a study by Yannikakis and others,<sup>37</sup> who reported that color changes became more intensive as immersion time increased.

In the current study, the two resin composites, Filtek Supreme and Clearfil AP-X, exhibited acceptable color changes after immersion in water for all immersion periods. Vichi and others<sup>36</sup> tested the influence of water exposure on the color stability of three structurally different resin-based composites, Tetric Ceram, Spectrum TPH and Z100. They found that Tetric Ceram remained completely within an acceptable shade shift after aging. They attributed these results to the different nature of the matrix and filler particle dimensions. In another study evaluating color and translucency changes in a hybrid and a microfilled composite after light exposure with and without water storage, both composites that were investigated showed color changes which were within the clinically acceptable range. No significant differences in  $\Delta E_{ab}$  values were found between the resin composites.<sup>38</sup>

## CONCLUSIONS

Under the conditions of this study, the following conclusions can be made:

1. Polymerization with QTH or LED did not cause any significant difference in the color stability of a hybrid composite, Clearfil AP-X or the nanofill composite, Filtek Supreme.
2. Clearfil AP-X appeared to be more color stable than Filtek Supreme, and coffee and tea appeared to stain equally. The effect of staining solutions (tea, coffee) on color changes in composites might be immersion-, time- and resin-material dependent.

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