Original Article

Color Stability of Orthodontic Adhesive Resins

Theodore Eliades, DDS, MS, Dr Med, PhD[a]; Christiana Gioka, DDS[a]; Matthew Heim, DDS[a,b]; George Eliades, DDS, Dr Dent[a]; Margarita Makou, DDS, MS, Dr Dent[c]

Abstract: Color alteration of adhesive during treatment and after debonding may be implicated in long-term enamel discoloration. The aim of this study was to assess the color stability of light-cured and chemically cured adhesives subjected to artificial photoaging. Disk-shaped specimens of adhesives were colorimetrically evaluated before and after artificial photoaging using an ISO-recommended protocol. The measurement variable was the color change (ΔE) of adhesives induced by artificial, accelerated photoaging. The ΔE values derived from the two color recordings for the materials at pre- and postaging intervals were statistically analyzed with a one-way analysis of variance (ANOVA), with the adhesive brand serving as a discriminating variable. Differences among groups were further investigated using the Tukey multiple comparisons test ($\alpha = .05$). To establish the statistical significance of the difference of the ΔE values of each adhesive and the ΔE threshold for clinical detection, a paired *t*-test was used ($P = .05$). All adhesives exhibited color change, which in some cases exceeded the clinically detectable color change limit. The extent of the color alterations of aged bonding systems may contribute to enamel discoloration after treatment. (*Angle Orthod* 2004;74:391–393.)

Key Words: Color stability; ΔE, L*a*b*; Adhesive resin; Photoaging

INTRODUCTION

A considerable amount of work has indicated that orthodontic bracket bonding and debonding can lead to adverse effects on tooth enamel. These effects can be identified as enamel loss caused by etching;[1] enamel alterations during fixed orthodontic treatment due to the inhibition of remineralization, leading to decalcification and, possibly, to caries development;[2] and enamel microcracks, scratches, and abrasions induced by the adhesive debonding and cleaning procedures.[3] Apart from the formation of structural and surface defects, the foregoing variables may affect the enamel color, inducing various alterations on the enamel surface, such as white spots.[4]

Despite the extensive evidence for enamel white spot formation associated with orthodontic treatment, the incidence of enamel color changes induced by bonding and debonding procedures has attracted only a limited number of investigations.[5] Enamel color alterations may derive from two sources, ie, the postdebonding resin removal protocols involving grinding with various rotary instruments[6] and the penetration of resin tags into the enamel structure at depths reaching 50 μm.[3] Because resin impregnation in the enamel structure cannot be reversed by debonding and cleaning procedures,[2] enamel discoloration may occur by direct absorption of food colorants and products arising from the corrosion of the orthodontic appliance.[7] The long-term presence of these resin residues in the enamel tags that extend over the middle third of the buccal surface may render the color stability of these materials critical for tooth color.

The hypothesis tested in this study is that photoaging may induce color changes on orthodontic adhesives. The purpose of this investigation was to study the color alteration of orthodontic adhesives using an accelerated artificial photoaging protocol.

MATERIALS AND METHODS

Table 1 lists the materials selected for the study. Six adhesive specimens were prepared from each of the five brands included in the study by filling plastic disks of 16-mm diameter and 1-mm thickness. The inner surface of the rings was lightly pasted with Vaseline to facilitate easy detachment of the adhesive disks. The top and bottom surface of the rings was covered by cellulose strips, which were pressed between glass flats to remove the excess resin. Filled rings were photopolymerized for 20 seconds with a

[a] Biomaterials Laboratory, School of Dentistry, University of Athens, Athens, Greece.

[b] Present address: Department of Orthodontics, School of Dentistry, University of Oklahoma, Oklahoma City, Okla.

[c] Department of Orthodontics, School of Dentistry, University of Athens, Athens, Greece.

Corresponding author: Theodore Eliades, DDS, MS, Dr Med, PhD, 57 Agnoston Hiroon Street, Nea Ionia GR-14231, Athens, Greece (e-mail: teliades@ath.forthnet.gr).

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TABLE 1. The Orthodontic Adhesives Included in the Study

Material	Manufacturer	Polymerization Mode[a]
Concise	Unitek/3M, Minn, USA	Chemically cured
Tranbond XT	Unitek/3M, Minn, USA	LC[a]
Ideal	GAC, NY, USA	LC
Heliosit Orthodontic	Vivadent, Lichtenstein	LC
Enlight	ORMCO, Glendora, CA, USA	LC

[a] LC indicates light cured.

lamp (Trilight, 3M/Espe, Seefeld, Germany) having a tip of 16-mm diameter.

The adhesive resin disks were polished and placed on a tray of standard color background and 75% reflectance. The disks were colorimetrically evaluated with a colorimeter (Microcolor, Data Station, DrLange, Braive Instruments, Leige, Belgium) according to the CIE Lab (Commision Internationale de l'Eclairage, L*, a*, b*)[8] system, using a repeated-measures design ($n = 5$). The CIE L* parameter corresponds to the value or degree of lightness in the Munsell system, whereas the a* and b* coordinates designate positions on red or green (+a* = red, −a* = green) and yellow or blue (+b* = yellow, −b* = blue) axes.

All specimens were subjected to accelerated artificial photoaging with the use of a light-emitting apparatus (Suntest CPS plus, Atlas material testing technology, Geluhausen, Germany), involving exposure of the resin surfaces to a 24-hour continuous irradiation of 50,000 kJ/m^2. This corresponds to an illuminance of approximately 135,000 lux at 400 nm, at 38°C black temperature (BT). This procedure induces aging equivalent to that of exposure to sun irradiation in central Europe for 30 days,[5] which was deemed adequate because the labial surfaces of crowns are not exposed to ambient light during ordinary conditions. Specimens were stored in a dark place throughout the experimental period. After photoaging, a second color determination was performed. Color parameters were averaged for each group, and color differences (ΔE) induced by photoaging were calculated using the following equation:[9]

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5}$$

The ΔE values derived from the two color recordings for the materials at pre- and postaging intervals were statistically analyzed with a one-way analysis of variance (ANOVA), with adhesive brand serving as a discriminating variable. Differences among groups were further investigated using the Tukey multiple comparisons test ($\alpha = .05$). To establish the statistical significance of the difference of the ΔE values of each adhesive and the ΔE threshold for clinical detection, a paired *t*-test was used ($P = .05$).

RESULTS

Table 1 lists the materials included in the study. The ANOVA table for the results obtained is provided in Table 2.

TABLE 2. ANOVA Table for the ΔE Values Obtained[a]

Source of Variation	DF	SS	MS	*F*	*P*[b]
Treatment	4	119.05	29.76	56.2	<.001
Residual	25	13.2	0.52		
Total	29	132.27			

[a] ANOVA indicates analysis of variance.
[b] Values in this column represent the probability that the results could have occurred by random sampling distribution.

TABLE 3. ΔE Differences for the Orthodontic Adhesives Before and After Photoaging ($n = 6$, $\alpha = 0.05$)

Adhesive	ΔE Mean	ΔE SD	Tukey Grouping[a]
ΔE Enlight	8.36	1.44	A
ΔE Concise	4.41	0.48	B
ΔE Heliosit Orthodontic	3.73	0.22	B C
ΔE Transbond XT	3.26	0.47	B C
ΔE Ideal	2.83	0.20	C

[a] Means with same letter are not significantly different at the $\alpha = 0.05$ level.

TABLE 4 Difference Between the ΔE of Adhesives Obtained in this Study and the ΔE Threshold for Clinical Detection (3.7)

Adhesive	ΔE Mean	ΔE SD	ΔE Threshold for Clinical Detection	*P*
Enlight	8.36	1.44	3.7	<.05
Concise	4.41	0.48	3.7	<.05
Heliosit Orthodontic	3.73	0.22	3.7	[a]
Transbond XT	3.26	0.47	3.7	[a]
Ideal	2.83	0.20	3.7	[a]

[a] indicates not significant.

In Table 3, the results of the Tukey multiple comparisons test are given. The materials demonstrated different extents of color change with significant differences among brands, indicating different responses to aging. Table 4 shows the results of the *t*-test used to investigate the significance of difference between the ΔE adhesives and the ΔE for clinical detection of color change.[6,8] This test indicates that only two groups exhibited color alterations above the detection limit.

DISCUSSION

Generally, ΔE values in the range of one unit are considered exact color matches because they cannot be identified by independent observers.[10] Although it has been suggested that differences above two units may indicate color change, most studies set the proposed acceptance limit for color matching to 3.7 units, beyond which the differences are clinically visible.[11] In the present study, the color difference threshold was set at 3.7 units.

In dental polymers, the opacity of the material increases with an increasing refractive index difference between the inorganic particles used as fillers and the organic matrix because of multiple reflection and refraction at the matrix–particle interfaces.[12] In these materials, color changes take place after setting because of the difference in the refractive index between the monomer and the polymer, which makes the material less translucent because of increased light scattering.[13]

The discoloration of polymeric materials may originate from a wide array of sources including: exogenous discoloration arising from the superficial absorption of color pigmentation of food dyes, colored mouth rinses, and plaque; [14–15] endogenous irreversible discolorations attributed to changes in the chemical structure of the material; surface discoloration from absorption or superficial penetration of colorants after chemical degradation of the material surface or discoloration of the outer layers caused by superficial diffusion of hydrophilic colorings; and internal or bulk discoloration derived from the incomplete conversion of photoinitiators and the unconverted C=C bonds.

A factor that also affects the color perception is the specularly reflected light component, a surface roughness-dependent parameter, which is highly sensitive to surface alteration influencing the L* values of the substrate.[16–17] A direct relation has been found for opacity and L* in resin composite and resin-modified glass ionomers.[18] Opacity depends partly on surface roughness because roughly finished surfaces demonstrate a whitish appearance due to increased contribution of surface-localized, random specular reflections.[13] Thus, adhesive removal with the use of rotary instruments may further contribute to color alteration of the resin-infiltrated enamel.

Resin color instability has been attributed to the formation of oxidation byproducts, which contain chromophore groups arising from the addition reaction to the pendant C=C of the cross-linked network.[19] Moreover, for chemically activated systems, oxidation of reactive groups present in amine accelerators and inhibitors, such as tertiary amino or hydroxyl groups, may modify the color at a rate determined by the type of substitution of the aromatic ring.[20] Decomposition of the initiators has been found to be consistent with discoloration linked with changes in b* values toward yellow.[15]

It must be noted that the photoaging protocol used in this study cannot reliably simulate the microenvironmental milieu of the oral cavity. In particular, the long-term resin discoloration attributed to absorption of colorants from the oral environment cannot be estimated and, therefore, this protocol presents a low safety margin in establishing the color alterations of materials.

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