Effect of Aging on Discoloration of Two Composite Surface Sealants

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Abstract
***Objectives*:** Composite surface sealants were introduced aiming to prevent or decrease the discoloration and microleakage of composite restorations. This study sought to assess the effect of aging on discoloration of two composite surface sealants.
***Methods*:** In this experimental study, 24 samples were fabricated of Fortify Plus and BisCover LV composite surface sealants (10mm in diameter and 1mm in thickness) and their baseline color coordinates were measured using the CIE L*a*b* system by a reflection spectrophotometer. The samples made of each material (n=12) were randomly divided into 4 groups of 3 and aged in xenon chamber, tea, distilled water and dry ambient environment (control). Color change (ΔE*) was calculated in the reflectance mode. The data were analyzed using two-way ANOVA and t-test ($P<0.05$).
***Results*:** The minimum ΔE* value was 0.95±0.6 belonging to Fortify Plus stored in dry ambient environment while the maximum ΔE* value was 21.85±4 belonging to BisCover LV aged in tea. Two-way ANOVA showed significant differences in ΔE* among the aging protocols ($P<0.001$); the effect of two materials ($P<0.001$) and the interaction effect of the type of material and aging were also significant ($P=0.001$).
***Conclusion*:** It can be concluded that aging affects the discoloration of composite surface sealants. Tea caused the greatest discoloration. There were no significant differences in the color change of the two materials after accelerated aging with xenon.
***Key Words*:** Composite Resins; Tooth Discoloration; Aging
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Introduction

The clinical use of composite resins has significantly increased due to high patient demand for tooth-colored restorations, improved formulations and simplicity of bonding procedures (1). However, staining and discoloration of restorations, which occur as the result of the process of aging overtime, are among the primary reasons for replacement of composite restorations (2). Color change (ΔE*) would be visible to the human eye if it exceeds a certain value. Studies have reported various ΔE thresholds above which, the color change would be perceptible to the human eye. The reported values range from ΔE equal to 1 (3), ≥3.3 (4,5) and ≥3.7 (6). In a previous study, ΔE in the range of 2 to 3 was referred to as perceptible, values from 3 to 8 were moderately perceptible, and values above 8 were markedly perceptible (7). However, in contrast to the statements of the latter study, a general consensus has been reached on the ΔE value of 3.3 to be clinically acceptable and values above it are largely considered unacceptable (8,9).

The process of color perception occurs in the mind of an observer and therefore, it is difficult, if not impossible to measure.

However, some of its related physical parameters can be measured. Color perception is determined by the light source's spectral power distribution, the object's reflectance as well as the spectral response of the eye. This was a basis for the introduction of the CIE system by the Commission Internationale De l'Éclairage in 1931, which is currently the basis of almost all color measurements. Some modifications have been made in this system; however, its basic structure and principles remain unchanged (10).

Surface of composite restorations is prone to wear and discoloration due to aging and light exposure resulting in loss of gloss, yellowing, delamination, crack formation, peeling and decreased tensile strength. Dental restorative materials must be able to withstand wear and light exposure. The process of quality control and certification of materials often includes accelerated weathering and light stability tests, which are widely used for research purposes and provide fast and reproducible results (11). Verified accelerated aging methods are also performed to provide guidance for material selection and to accurately assess and offer an estimate of the aging of new materials (12).

Other than the polishing procedures, sealant agents have also been used to fill the surface and irregularities, enhance stain and wear resistance and prevent microleakage; however, the durability of sealant agents on composite resin restoration is not fully known (13). Use of unfilled resins to cover the surface of composite resin restorations was first proposed 25 years ago. Unfilled resins, also called glaze, are light-polymerizing resins with bis-GMA matrix. These acrylate-based, light-cured products are multifunctional and highly reactive since they generate free radicals and contain high concentrations of photo initiators in their composition. They are also clear, hard, tough and water and wear resistant (14). Composite surface sealants were primarily recommended to improve the optical properties of composite restorations (15-17). Furthermore, their application decreases the polishing time required to obtain a glossy and smooth appearance. It has been reported that surface sealants minimize microleakage at the restoration margins and ensure a lustrous surface (18). Also, a previous study showed that composite surface sealants improved resistance to staining by reducing the surface porosity and enhancing uniform curing of the surface (19); however, further investigations are still required in this respect.

Therefore, this study sought to assess the effect of aging on discoloration of composite surface sealants. The null hypothesis was that aging would not cause discoloration of composite surface sealants.

Methods

A mold 10mm in diameter and 1mm in thickness was used to fabricate 12 resin discs of each of the 2 composite surface sealants namely BisCover LV and Fortify Plus (Bisco Inc., Schaumburg, IL, USA). Table 1 shows the composition of the two composite surface sealants. Unpolymerized resin was injected into the molds, its surface was covered with a transparent Mylar strip (Universal Strips, Odus Dental, Dietikon, Switzerland), pressed between two glass slabs and light cured using a light-curing unit (LED, Radii Plus, SDI, Australia). Fortify Plus was light cured for 10 seconds and BisCover LV for 30 seconds after waiting for 15 seconds in order for the solvent to evaporate. The light intensity was checked to be >1000mW/cm^2 each time before curing. The thickness of the samples was measured by a digital caliper (Mitutoyo, Tokyo, Japan) with an accuracy of 0.001mm at five sites (at the four corners and at the center) to ensure all samples have similar thickness. Samples were stored in distilled water for 24 hours at 37°C (20).

Table 1- Composition of materials used in this study

Composite surface sealants	Composition	LOT NO.	Manufacturer
BisCover LV	Dipentaerythritol pentaacrylate>20% Ethanol>30%	1000008095	Bisco Inc., Schaumburg, IL, USA
Fortify Plus	Urethane dimethacrylate 20-50% Ethoxylated bisphenol A dimethacrylate 10-40% Amorphous silica 20-50%	0800007881	Bisco Inc., Schaumburg, IL, USA

Baseline color was measured using the CIE L* a* b* standard illuminant D65 against a white background by a reflectance portable sphere spectrophotometer (SP64, X-Rite Inc., MI, USA) with an integrating sphere and X-Rite Color iControl Software. Reflection values were recorded in the visible spectral range (360-750 nm). To obtain the baseline color data, each sample was measured three times and the mean value was reported. The CIE L*a*b* color space illustrates all the colors perceptible to the human eye in the visible light range using a three-dimensional representation. L* describes lightness; a* value indicates the color position in the red-green axis and b* indicates the color position in the yellow-blue axis (21). After aging, color change (ΔE*) was calculated using the following equation:

$\Delta E^* = (L1^*-L0^*)2 + (a1^*-a0^*)2 + (b1^*-b0^*)2)1/2$

After baseline measurement, 3 samples of each sealant were aged in an accelerated aging chamber (Xenotest Beta LM, Atlas Material Testing Technology, Vogelsbergstraße, Germany) according to ASTM G155 standards. Xenon light was irradiated at a wavelength longer than 310nm. One surface of each sample was exposed to a controlled irradiance xenon arc filtered through Xenochrome. The black panel temperature was 70˚C (light cycle) and 38˚C (dark cycle), the dry bulb temperature was 47˚C (light cycle) and 38˚C (dark cycle), and the humidity was 50% (light cycle) and 95% (dark cycle). The test cycle was 40 minutes light only, 20 minutes light plus front water spray, 60 minutes light only, and 60 minutes dark plus back water spray. The test was run with a radiant energy of 45W/m^2 and total exposure time of 80 hours that equals 6 months (22, 23).

The remaining samples made of each sealant were individually immersed (n=3) in vials containing 5mL of brewing tea (Golestan, Tehran, Iran) diluted with water half and half and distilled water for 6 days that equals 6 months of clinical service (24) and stored in an incubator at 37˚C. The vials were sealed to prevent evaporation of the solutions, and the solutions were refreshed daily. The control group samples were stored in dry ambient environment.

The mean and standard deviation of all variables were calculated. Two-way ANOVA was used to compare the color change among the aging groups and between the two sealants. In case of presence of a significant difference, t-test was applied for pairwise comparisons of the groups. The Tamhane's post hoc test was used to compare the effect of different aging protocols on each sealant. $P<0.05$ was considered statistically significant.

Results

A total of 24 samples were subjected to color assessment. The minimum ΔE* value was 0.95±0.6 belonging to Fortify Plus stored in dry ambient environment while the maximum ΔE* value was 21.85±4 belonging to Bis Cover LV stored in tea. Table 2 shows the ΔE* values of the two sealants after different aging protocols.

Table 2- The mean (standard deviation) color change of the tested materials after the aging protocols

	Aging protocols			
Materials	**Xenon test**	**Tea**	**Distilled water**	**Dry ambient environment**
Fortify Plus	3.5(0.6)	4.2(0.3)	1.0(0.1)	0.9(0.06)
BisCover LV	9.6(2.7)	21.8(4.2)	10.3(3.8)	3.5(0.3)

Two-way ANOVA showed significant differences in color change among the aging procedures ($P<0.001$) and the interaction effect of the material type and aging was also significant ($P=0.001$). The difference in color change between the materials was also significant ($P<0.001$). Changes in color after artificial aging were observed in all groups ($P<0.05$) except for the Fortify Plus samples in distilled water and dry ambient environment. Irrespective of the type of material, tea produced the highest level of discoloration (ΔE*=21.8±4.2), which was significantly greater than the discoloration caused by the other aging protocols. The lowest ΔE* value belonged to the control group (ΔE*=0.9±0.06). Fortify Plus showed significant color change due to aging in xenon and tea while BisCover LV showed significant color change due to all aging protocols.

Comparison of Fortify Plus and BisCover LV revealed insignificant difference in color change in xenon chamber ($P>0.05$) and significant differences in tea, distilled water and dry ambient environment ($P<0.05$).

Assessment of the effect of different aging protocols on ΔL and Δa of Fortify Plus revealed that ΔL of the tea group was significantly different from the corresponding values in other groups ($P<0.05$). Also, Δa of the distilled water and dry ambient environment groups was significantly different from the corresponding value in xenon group ($P<0.05$, Table 3).

Assessment of the effect of different aging protocols on ΔL and Δa of BisCover LV group revealed no significant difference in Δa values of different aging groups ($P>0.05$); but significant differences were noted in ΔL values among the groups ($P<0.05$). Comparison of ΔL and Δa of Fortify Plus and

BisCover LV samples after different aging protocols showed that ΔL and Δa were significantly different in tea and xenon groups ($P<0.05$). Distilled water and dry ambient environment aging protocols also had a significant difference in ΔL ($P<0.05$).

Table 3- Comparison of ΔL and Δa of Fortify Plus after different aging protocols

Aging environment	***P* value**	***P* value**
	ΔL	Δa
Xenon vs. Tea	0.006	0.457
Xenon vs. Distilled water	0.432	0.046
Xenon vs. Dry ambient	0.838	0.049
Tea vs. Distilled water	0.007	0.112
Tea vs. Dry ambient	0.004	0.084
Distilled water vs. dry ambient	0.987	0.999

Discussion

In the present study, all artificial aging protocols caused color change in the composite surface sealants. Therefore, the null hypothesis was refuted. In our study, a clinical perceptibility threshold of ΔE* = 3.3 was used as reference. Tea caused the greatest color change in both materials compared to accelerated aging and distilled water with ΔE* values between 4.2-21.8. This result is in agreement with the findings of Stober et al, (5) who reported that discoloration with tea was more severe than UV irradiation. Garcia et al, (25) and Chan et al. (26) reported that tea caused more severe discoloration compared with distilled water. Tea contains yellow colorants, which have high polarity. Discoloration by tea is due to the adsorption of polar colorants into the surface of resin materials. Al-Samadani (27) reported that the wine solution caused the most staining. Compatibility of the polymer phase with yellow colorants is probably responsible for adsorption and uptake of colorants by the organic phase of resin materials. Such discoloration of surface sealant observed in vitro may manifest visible discoloration of restorations in the clinical setting, which compromises esthetics, confuses the dentists with regard to the accuracy of their shade selection in the first visit and leads to patient dissatisfaction.

Accelerated aging process has long been used to assess the color stability of restorative materials. The process of accelerated aging involves UV irradiation and alterations in temperature and humidity, simulating long-term exposure to environmental conditions (28). However, this test does not simulate the effect of external stains and focuses only on internal discoloration. In the current study, accelerated aging caused color change in the range of 3.5-9.6. Color change of restorative materials by xenon test has been related to chemical alterations in the initiator system, activators and resins itself (29). Yellowing compounds form as the result of degradation of residual amine and oxidation of residual unreacted carbon double bonds.

The physicochemical properties of resin matrix monomer may also affect staining resistance of resin materials. Pissaia et al. (30) reported that the translucent shade materials experienced higher ΔE changes. According to the manufacturer, Fortify Plus is primarily composed of urethane dimethacrylate (UDMA), ethoxylated bisphenol A diacrylate (EBPAD) and amorphous silica as filler particles. UDMA seems to be less susceptible to staining (31). Filler particles decrease the resin volume and the volume of organic matrix, which results in less water absorption and color change. Using XPS analysis, Schulze et al. (29) observed that following accelerated aging, the concentration of Si and O decreased by 50%. Although UDMA has been identified as the color change resistant monomer, EBPAD might also affect the staining resistance. Doray et al. (19) concluded that EBPAD and fillers have a significant impact on staining in color solutions and improve the staining resistance. According to this study, the surface staining in accelerated aging group could also be due to the surface alterations in Fortify Plus group. BisCover LV contains dipentaerythritol pentaacrylate and ethanol, and is said to have a high abrasion and staining resistance.

The strength and durability of the bond between filler particles and resin matrix play an important role in stainability because unstable and weak bonds lead to failure and increase the susceptibility to color change. Using reflectance analysis, Soares et al. (32) evaluated the effect of staining on chemical properties and light reflection of Fortify Plus and showed that after staining, chemical changes occurred in this sealant; these changes depended on the molecular weight and macromolecular structure of polymer. These findings may also explain the difference in color change in the tea group in our study. However, since the manufacturer does not provide specific details in this respect, separate assessment of the exclusive effect of the above-mentioned parameters is not possible.

Conclusion

Within the limitations of this study, it can be concluded that, in general, all aging protocols affect the discoloration of composite surface sealants. Tea caused the greatest discoloration. There were no significant differences in color change of the two materials after accelerated aging with xenon.

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Conflict of interest: "None Declared"

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