ORIGINAL RESEARCH

Color Stability of Microhybrid and Nanofilled Composite Resins: Effect of Surface Sealant Agents Containing Different Filler Content

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Abstract

Aim: To evaluate the effect of surface sealants containing different filler content on the color stability of microhybrid and nanofilled composite resins.

Materials and methods: The materials evaluated as study groups were comprised a nanofilled composite resin (Filtek Ultimate, 3M ESPE) and a microhybrid composite resin (Filtek Z250, 3M ESPE). Forty-five disc-shaped specimens (10 mm × 2 mm) were prepared from each composite resin. Each study group was divided into three subgroups: control, G-Coat Plus, and Fortify Plus ($n = 15$). The baseline color values ($L^*a^*b^*$) of each specimen were measured using a spectrophotometer according to the CIE $L^*a^*b^*$ color scale. Then, the specimens were immersed in red wine for a period of 3 hours per day for 15 days (3 hours/day × 15 days). After the immersion period, the color values ($L^*a^*b^*$) of each specimen were measured again. The ΔL^*, Δa^*, and Δb^* values and the color change value (ΔE) were calculated. The data were statistically analyzed with two-way ANOVA and Duncan tests ($p = 0.05$).

Results: All the composite resin groups demonstrated/indicated much more color changes after immersion in red wine ($\Delta E > 3.3$). The greater ΔE values were observed with the groups applied surface sealants than the control groups ($p < 0.05$). Fortify Plus further increased the ΔE values of both composite resins than G Coat Plus ($p < 0.05$). Filtek Ultimate showed higher ΔE values than Filtek Z250 in all the subgroups ($p < 0.05$).

Conclusion: The surface sealants regardless of the filler content negatively influenced the color change of the composite resins after immersed in red wine. The microhybrid composite resin had better color stability than the nanofilled composite resin.

Clinical significance: There is no favorable effect of using surface sealants on composite resins to prevent discoloration; besides, the sealants can also increase the color alteration.

Keywords: Composite resin, Discoloration, Filler, Spectrophotometry, Surface sealant.

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Introduction

In recent years, the composite resins have become the most frequently used dental restorative materials, mainly because of the increased esthetic demands of patients, improvements in formulations, and simplification of adhesive procedures.[1–3] All of the esthetic restorative materials should simulate the natural tooth in color, translucency, and texture.[1] The materials must also maintain color stability for a long time period.[2] Despite improvements in formulations of the composite resins, the discoloration is still one of the most common reasons for the replacement of composite resin restorations.[4] Therefore, the color stability is regarded as the essential parameter for clinical behavior of restorative materials.[5] The discoloration of composite resins may be formed due to intrinsic and extrinsic reasons. Intrinsic factors can occur depending on the alterations of the resin matrix, filler, particle size and volume, silane coating, and type of photoinitiator. Extrinsic discoloration is mainly resulted by colorants contained in beverages and foods via adsorption and absorption.[1,2,5] The resin matrix type and characteristics of the filler particles have a direct impact on the surface smoothness and susceptibility to extrinsic staining of the composite resins.[6,7] Currently, two categories of composite resins, microhybrid and nanofilled composite resins, have been widely used in restorative dentistry.[3]

The clinical appearance of a composite resin restoration depends mostly on the quality of finishing and polishing procedures.[3,8] The filler particles and resin matrix of composite resins differ in terms of microhardness, so they are not abraded to the same degree during finishing and polishing stages.[8,9] The irregularities and microcavities on the surface of composite resins occur based on especially the size of filler particles after polishing procedures.[8] The surface defects and irregularities induce staining on the surface of composite restorations.[9,10]

A common approach applied to overcome the color change in composite resins is the application of surface sealants. The surface sealants are polymerizable materials that include low-molecular-weight monomers (Bis-GMA, UDMA, and TEGDMA) and photoinitiators.[11] Recently, filler particles have also added into some

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surface sealants to improve the mechanical properties.[12–15] The surface sealants that have a low viscosity and high wetting ability are used to seal microporosity on the surface of composite resins, thus the marginal integrity, surface luster, and abrasion resistance can improve.[16,17] The application of surface sealants may also affect the absorption of colorant pigments and contribute to the color stability of composite resins.[10,12,16] There are several studies that examine the effects of surface sealants on color stability. While some of these studies have indicated that the application of surface sealants provided less discoloration,[9,10,12,16,18] some of them have reported that the surface sealants did not alter the color stability of composite resins.[11,14,17,19–21] It was also stated that the surface sealants dramatically increased the staining of the composite resins.[13,15,22] In a study, it was concluded that the adverse effect of surface sealants on the color stability could be changed by the presence of filler particles in the sealants.[22] Consequently, the effect of surface sealants containing different filler content on the composite resins is not clear.

Therefore, the objective of the study was to determine the effect of surface sealants containing different filler content on the color stability of microhybrid and nanofilled composite resins. The null hypotheses tested were the following: (1) the surface sealants with different filler content would not affect the color stability of microhybrid and nanofilled composite resins, and (2) the color stability of microhybrid and nanofilled composite resins would not be different.

Materials and Methods

Two composite resins and two surface sealants were used in the present study. The composite resins were a nanofilled composite resin Filtek Ultimate (3M ESPE, St. Paul, MN, USA) and a microhybrid composite resin Filtek Z250 (3M ESPE, St. Paul, MN, USA) in the A2 shade (Table 1). The surface sealants were a nanofilled surface sealant G-Coat Plus (GC, Tokyo, Japan) and a microfilled surface sealant Fortify Plus (Bisco, Schaumburg, IL, USA). The materials are listed in Table 1 together with the composition, name of manufacturer, and lot number.

Specimen Preparation

Forty-five disk-shaped specimens, 10 mm in diameter and 2 mm in thickness, from each composite resin were prepared using a Teflon mold (Fig. 1). After the composite resins were inserted into the Teflon mold, a polyester strip (Mylar strip; SS White Co., Philadelphia, PA, USA) was pressed onto the mold surface with a glass plate to extrude excess material and obtain a flat surface. The composite materials were polymerized for 20 seconds using a LED light-curing unit (Smartlite Focus, 1,000 mW/cm^2, Dentsply, Milford, DE, USA) according to the manufacturer's instructions. The power of the light-curing unit was verified by a radiometer after every 10 specimens. The light-curing unit was placed perpendicular to the specimen surface, and the distance between the light source and the specimen was standardized using a 1 mm glass slide. After the light curing, the specimens were removed from the mold and stored in distilled water at 37°C for 24 hours to ensure complete polymerization. The top surfaces of all the specimens were then sequentially polished under dry conditions for 30 seconds with graded series (coarse, medium, fine, and extra fine) of a multistep polishing system (Sof-Lex; 3M ESPE, St. Paul, MN, USA, Lot: N587386). After each polishing step, all the specimens were thoroughly rinsed with water and air-dried to remove debris. One single operator performed all of the polishing treatments, trying to simulate clinical finishing and polishing procedure.

The specimens prepared from each composite resin were randomly divided into three subgroups ($n = 15$): control, G-Coat Plus, and Fortify Plus. A control group of each material received no surface sealant application after polishing procedures. In the G-Coat Plus subgroups, G-Coat Plus was applied on one surface of the specimens using a micro-tip applicator and light-cured for 20 seconds according to manufacturer's instructions. In the Fortify Plus subgroups, one surface of the specimens was conditioned for 15 seconds with 37% phosphoric acid gel, followed by rinsing with copious amounts of water and drying. One thin layer of Fortify Plus was applied to the surface, gently air-thinned, and light-cured for 10 seconds according to manufacturer's instructions.

Baseline Color Assessment

Baseline color measurements were done from one surface of all the specimens according to the CIE $L^*a^*b^*$ color space system over a white background using a digital camera connected to a LED spectrophotometer (Spectroshade; MHT Optic Research AG, Niederhasli, Switzerland). Measurements were repeated three times for each specimen, and the mean values of the L^*, a^*, and b^* data were calculated. In G-Coat Plus and Fortify Plus subgroups, the color was measured over the surface sealant-applied surfaces. The color of the composite discs was assessed in the Commission Internationale de l'Eclairege $L^*a^*b^*$ (CIELAB) color space system. The CIELAB system is a chromatic value color space that measures the value and chroma on three coordinates: L^*—the lightness of the color measured from black ($L^* = 0$) to white ($L^* = 100$); a^*—color in the red ($a^* > 0$) and green ($a^* < 0$) dimension; and b^*—color in the yellow ($b^* > 0$) and blue ($b^* < 0$) dimension.

Table 1: Composition of the materials according to the manufacturers' data

Materials	*Classification*	*Monomer composition*	*Filler type*	*Filler size*	*Lot no.*
Filtek Ultimate (3M ESPE, St. Paul, MN, USA)	Nanofilled composite	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA	Zirconia, silica	20 nm	N817010
Filtek Z250 (3M ESPE, St. Paul, MN, USA)	Microhybrid composite	Bis-GMA, UDMA, Bis-EMA	Zirconia, silica	0.6 µm	N758399
G-Coat Plus (GC, Tokyo, Japan)	Nanofilled surface sealant	Urethane methyl methacrylate, methyl methacrylate, phosphoric ester monomer	Silicon dioxide	40 nm	1710031
Fortify Plus (Bisco, Schaumburg, IL, USA)	Microfilled surface sealant	Bis-EMA, UDMA	Amorphous silica	0.4 µm	1500000592

Bis-GMA, bisphenol A-glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate

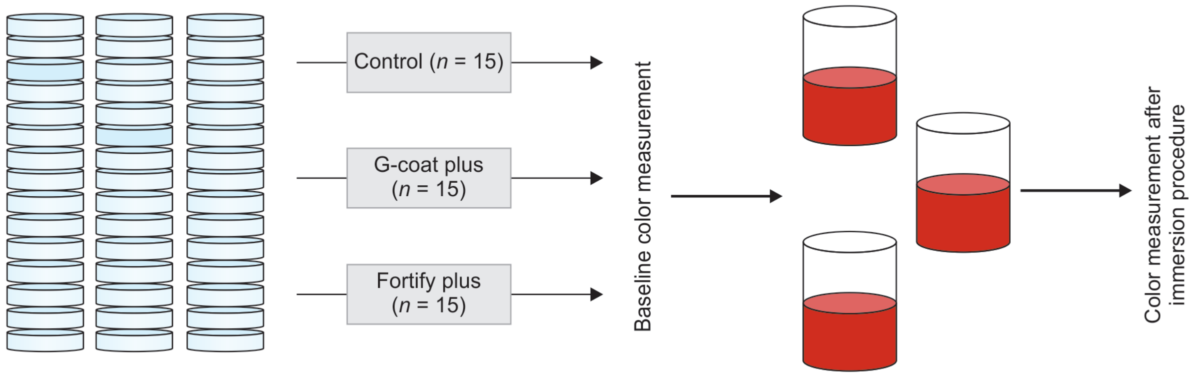

Fig. 1: Schematic illustration of the study design and methodology

Immersion of Specimens in the Staining Solution

In this study, red wine was used as a staining solution. The specimen surfaces without color measurement were covered with wax before the staining procedure. All the specimens were immersed in red wine for 3 hours/day, following which the specimens were stored in distilled water at 37°C until the next day immersion. The distilled water was changed daily. The specimens were blotted with a blotting paper during transfers from red wine to distilled water. All the specimens were immersed in red wine for a total period of 3 hours/day for 15 days.

Assessment of Color Change

The color measurements were performed after the immersion period in the same manner as the baseline measurement. All the specimens were wiped dry using a tissue paper. The L^*, a^*, and b^* values of each specimen after the immersion period were measured thrice from one surface of the specimen over a white background using the spectrophotometer. The mean L^*, a^*, and b^* values were recorded after all the measurements. The mean ΔL^*, Δa^*, and Δb^* values were calculated by computer. The color difference, ΔE, was calculated from the mean ΔL^*, Δa^*, and Δb^* values for each specimen using the following formula:

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

Statistical Analysis

Statistical analyses were performed with the SPSS Program, version 20.0 (Statistical Package for the Social Sciences; SPSS, Chicago, IL, USA). The data were analyzed using the two-way analysis of variance (ANOVA) to evaluate the effects of composite resin type and surface sealants on color change, and the Duncan test was used to determine the differences between groups. All statistical analyses were determined at a 0.05 level of significance.

Results

The mean ΔE values and standard deviations for the test groups were summarized in Table 2. The composite resins demonstrated far greater ΔE values than clinically perceptible discoloration values ($\Delta E > 3.3$) at all the test groups. The application of both surface sealants significantly increased ΔE values of the composite resins ($p < 0.05$). Fortify Plus further increased the ΔE values of both composite resins than G Coat Plus ($p < 0.05$). Filtek Ultimate showed higher ΔE values than Filtek Z250 in all the subgroups ($p < 0.05$).

The values of ΔL^*, Δa^*, and Δb^* for all the groups were presented in Table 3. There was a significant difference in ΔL^* and Δa^* values of the composite resins between control and Fortify Plus groups ($p < 0.05$). The positive ΔL^* indicates that the specimens became lighter, whereas the negative ΔL^* indicates that the specimens became darker. The negative ΔL^* values were observed in all the groups. The negative Δa^* indicates a shift toward green color, whereas the positive Δa^* indicates a shift toward red color. There was a significant difference in Δb^* values of the composite resins between control and G Coat Plus groups ($p < 0.05$). The positive Δb^* indicates a shift toward yellow color, while the negative Δb^* denotes a shift toward blue color. The negative Δb^* values were observed in all the groups.

Based on the results of this study, the ΔE, ΔL^*, Δa^*, and Δb^* changes for all the materials in all the subgroups were clinically nonperceptible.

Discussion

Color is one of the most important factors to obtain optimum esthetics for composite resin restorations.[23,24] The major disadvantage of composite resins is color instability, which may cause the replacement of the restorations.[24] The color stability of composite resins is related to different factors such as chemical composition of the material, depth of polymerization, finishing/polishing procedures, and coloring agents.[23] The resin matrix and filler particles of the composite resins directly affect the surface roughness and susceptibility to extrinsic staining.[2,25] Surface roughness has a significant impact on the staining of composite resins.[25] The application of surface sealants containing filler

Table 2: Means and standard deviations of color changes (ΔE) for the composite resin and surface sealant groups after the staining procedure

	ΔE		
	Filtek ultimate	*Filtek Z250*	$p^{\dagger}$
Control	17.67 ± 2.32^a	12.98 ± 1.74^a	0.011
G Coat Plus	20.73 ± 1.35^b	17.07 ± 1.94^b	0.048
Fortify Plus	35.07 ± 5.62^c	30.07 ± 6.15^c	0.006
$p^{\ddagger}$	0.000	0.000	

Same small superscript letter indicates no statistical difference in the column
$p^{\dagger}$, significance levels of the composite resins for each subgroup
$p^{\ddagger}$, significance levels of the subgroups of each composite resin

Table 3: Means and standard deviations of color coordinates (ΔL^*, Δa^*, Δb^*) for the composite resin and surface sealant groups after the staining procedure

	Filtek ultimate			*Filtek Z250*		
	ΔL^*	Δa^*	Δb^*	ΔL^*	Δa^*	Δb^*
Control	-17.21 ± 2.35^a	-0.68 ± 0.61^a	-2.69 ± 2.87^a	-12.55 ± 1.56^a	1.07 ± 0.32^a	-2.94 ± 1.29^a
G Coat Plus	-17.07 ± 1.27^a	-0.38 ± 0.25^a	-11.75 ± 0.69^b	-14.51 ± 1.74^a	0.91 ± 0.24^a	-8.93 ± 1.01^b
Fortify Plus	-34.61 ± 5.74^b	2.63 ± 1.99^b	-3.89 ± 2.24^a	-27.79 ± 4.36^b	7.03 ± 9.55^b	-3.49 ± 1.72^a
p	0.000	0.000	0.000	0.000	0.005	0.000

Same small superscript letter indicates no statistical difference in the column
p^*, significance levels of the subgroups of composite resins for each color coordinate

particles may provide better resistance to staining by filling surface defects on the composite resins and reducing surface roughness of the composite resins.[10,13,18] However, the results of this study demonstrated that the immersion procedure induced discoloration of the composite resins and the surface sealant application dramatically caused more staining of the composite resins.

Discoloration can be evaluated by visual or instrumental techniques.[1] The color evaluation by instrumental techniques including colorimetry, spectrophotometry, and digital image analysis ensures to avoid all bias due to human eye evaluation.[1,2,5] The spectrophotometry has been reported to be a reliable technique in dental material studies.[2,23] The CIE $L^*a^*b^*$ color system used in this study is a recommended method for dentistry since it characterizes color based on human perception.[1–3,5] The system indicates the color according to three spatial coordinates: L^*, a^*, and b^*, where L^* represents the brightness (value) of a shade, a^* represents the amount of red-green color, and b^* represents the amount of yellow-blue color. The color measurements are made in the $L^*a^*b^*$ coordinate, and the color changes are calculated as ΔE.[1–3,5] It has been reported that the human eye could not detect ΔE values of less than 1.5, and $\Delta E \leq 3.3$ was the critical value for the visual perception of the restorative materials.[7] In the present study, the far greater ΔE values were observed than 3.3 for all the test groups. The present study indicated that both of the composite resins showed significantly increased ΔE values with the application of the surface sealants. There are controversial results of previous studies related to this subject in the literature. Some of these studies have reported that the surface sealants could improve the color stability of the composite resins.[9,10,12,16,18]

As discussed in some of previous studies, application of surface sealants could decrease the color stability of composite resins, which might be a possible side effect of sealant application.[13,15,22] In a study, it was concluded that the use of surface sealants dramatically increased the staining of the microhybrid composite resin restorations after immersion in coffee.[22] The authors attributed this to the surface sealants that did not contain filler particles. However, it was also stated that the surface sealants containing filler particles did not prevent staining of the composite resins.[13–15,19] In a previous study, it was indicated that the nanofilled surface sealant, G Coat Plus, increased the surface roughness and discoloration values of the microhybrid and nanofilled composite resins after ultraviolent ageing.[15] Another study was reported that the microfilled surface sealant, Fortify Plus, did not prevent staining of the microhybrid composite resin Filtek Z250 after immersion in coffee, furthermore the surface sealant also stained.[13] In this study, Fortify Plus further increased ΔE values of the composite resins than G Coat Plus. Therefore, the hypothesis that the surface sealants with different filler content would not affect the color stability of microhybrid and nanofilled composite resins must be rejected.

The resin matrix type of the surface sealants could affect staining susceptibility as well as the filler content.[10,13] The presence of more hydrophilic comonomers in the resin sealants could be responsible for discoloration due to absorbing water and other colorant fluids.[17,22] Fortify Plus has higher amount of Bis-EMA (10–40%) and UDMA (20–50%) in its composition, which are the main components of the organic matrix and this may lead to this material being more susceptible to staining.[17] Additionally, 37% phosphoric acid etching before Fortify Plus application in accordance with manufacturer's instruction may result in greater staining by increasing surface roughness of the composite resins. Another factor that affects the performance of surface sealants is the sealant thickness over the composite resin.[9,11,17] The composite resin can become more susceptible to absorb colorant as a result of increasing the thickness of the surface sealant applied.[17] In this study, only one layer of the sealant was applied on the composite resins. However, it was not possible to standardize the sealant thickness due to the flowable characteristic and moistening ability of these materials.

It is emphasized that the effect of the surface sealant on the composite resin is more dependent on surface properties of the composite resin than the sealant.[17] The filler particle size of a composite resin has a significant effect in mechanical and surface properties of the composite resins.[1,2] The wear of composite resins can induce debonding of the fillers from the resin matrix, thereby this may result in increasing the surface roughness and forming a surface susceptible to extrinsic staining.[2] It can be expected that a nanofilled composite resin with a smaller particle size will have a smoother surface and will retain less surface stains.[2] In a previous study, it was concluded that the nanofilled composite resins had more color stability than the microhybrid composite resins after the 6 month evaluation period.[1] On the contrary, another study was reported that the nanofilled composite resin showed more color change than the microhybrid composite resin after immersion in different beverages.[2] In the present study, the nanofilled composite resin Filtek Ultimate demonstrated more discoloration than the microhybrid composite resin Filtek Z250. This can be due to the composition of the resin matrix. The resin matrix has also a major effect in the color stability of composite resins.[1] The discoloration is modulated by the conversion rate and chemical characteristics of the resin matrix.[2,3] The composite resins containing TEGDMA in their composition release larger amounts of monomers in the aqueous media than Bis-GMA- and UDMA-based composite resins, resulting in greater color alteration.[26] In previous studies, greater discoloration was obtained from the composite resins that contain

TEGDMA, which might be responsible for the high-water absorption and discoloration rates.[3,27] In our study, Filtek Ultimate, which includes TEGDMA, showed higher staining susceptibility than Filtek Z250, not containing TEGDMA. Consequently, the hypothesis that the color stability of microhybrid and nanofilled composite resins would not be different must be rejected.

In methodology, the color stability of the composite resins was evaluated after immersion in red wine. In previous studies, the staining of composite resins by certain colored solutions, such as coffee, tea, red wine, and other beverages, and the color stability after aging in different solutions have been evaluated.[5,23,24,27] But it was stated that red wine had the highest staining capacity.[23,24,27] The immersion time in red wine was specified 3 hours a day during 15 days for the present study. In a previous study, it was stated that this immersion procedure could be considered equivalent to a longer duration of exposure to stains *in vivo*.[6] In this study, the immersion procedure into red wine induced great color changes of the composite resins. According to the results of the present study, it could be noted that the surface sealants increased staining of the composite resins. The results of this study should be evaluated considering the limitations of the *in vitro* study. In this study, the only one immersion media was used. The immersion time and procedure can also affect the color change. These results can change under different conditions such as other immersion medias, immersion procedures, composite resins, and surface sealants. Moreover, the oral environment is dynamic and different from *in vitro* conditions; hence, further clinical studies are also needed to investigate the effect of surface sealants on the color stability.

Conclusion

Within the limitations of the present study, the results indicate that the immersion procedure used in the study significantly affected the color alteration of the composite resins. The application of surface sealants containing different filler content was not efficient to provide protection against the staining of microhybrid and nanofilled composite resins. The microhybrid composite resin demonstrated less color change than the nanofilled composite resin. The performance of surface sealants on the composite resin restorations can be evaluated with further laboratory studies under different conditions and clinical trials.

Clinical Significance

The surface sealants with different filler content have no ability of preventing staining of the composite resins. The sealants dramatically cause more discoloration in both nanofilled and microhybrid composite resins. In clinical practice, patients should be aware of the staining effect of red wine if consumed for a longer period of time.

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