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Factors Influencing the Color Stability of Nano-Filled Composite Resin

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Abstract

Objective: The impact of whitening toothpastes on the color stability of nano-filled composite resin is not known exactly. The aim of this in vitro study was to evaluate the color stability of nano-filled composite resin that was submitted to different staining solutions and daily tooth brushing simulations with conventional or hydrogen peroxide-containing whitening toothpaste.

Methods: Nano-filled composite resin (Filtek Ultimate) was used to fabricate disc-shaped specimens. The specimens were stored for 1 hour/day in 3 different staining solutions: artificial saliva (control), coffee, and red wine. The specimens were also mechanically brushed with conventional or hydrogen peroxide-containing whitening toothpaste for 30 minutes. Color changes were recorded at baseline and after exposure. Data were analyzed using 2-way analysis of variance and Tukey's honestly significant difference tests.

Results: Mechanical brushing succeeded in reducing color change values significantly ($P < .05$). The type of toothpaste did not significantly affect the color change values ($P > .05$). Red wine affected color change values the most, followed by coffee ($P < .05$).

Conclusion: The immersion of nano-filled composite resin in coffee and red wine caused a significant color change. As a result of mechanical brushing, color recovery was achieved within the acceptability threshold in specimens immersed in coffee.

Keywords: Color change, color recovery, composite resin, discoloration, toothpaste

INTRODUCTION

Nowadays, tooth-colored restorative materials are frequently used in clinical applications as patient awareness and expectations in the field of aesthetic dentistry have increased. Ensuring color match with healthy dental tissues in restorative procedures and maintaining the color of the restoration throughout its clinical use is critical to successful and long-lasting treatment. Properties of composite restorations such as color stability, surface roughness, and surface gloss are of great importance in their aesthetic performance. Many studies have shown that composite restorations change color over time, and this problem is one of the main reasons for replacing anterior composite restorations.[1] It is reported that composite resins change color with time because of extrinsic and intrinsic factors.[2] Extrinsic factors cause staining of the material as a result of absorption or adsorption of colorant pigments from the oral environment. The color stability of composite resins is found to vary with diet (pigment exposure) and oral hygiene habits.[3,4] Intrinsic factors are related to the composition of the composite resin, including conversion rate, filler particle ratio and type, inhibitor and initiator systems, and resin matrix composition.[5]

The organic matrix structure of composite resins and the characteristic properties of inorganic particles directly affect the susceptibility of composite resins to discoloration. The monomer ratio of the organic matrix, its chemical structure, and the amount of water absorption determine the intensity of staining.[6] On the other hand, the classification of composite resins is based on inorganic particle type and size, due to the great influence of inorganic particle amounts of restorative materials on physical properties. Composite resins, which are widely used today, are divided into 3 main categories as micro-filled,

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micro-hybrid, and nano-filled (nano-composites and nano-hybrid composite resins).[7] Micro-filled composites have superior aesthetic properties with their good polishability, but besides, their mechanical properties are insufficient. Micro-hybrid composites are widely used due to their optimum mechanical and physical properties and good polishability. In nano-filled composites, the good mechanical properties of hybrid composites and the superior polishing properties of micro-filled composites are combined. Nano-hybrid composites are hybrid composites containing glass particles and prepolymerized nanoparticles of 40-50 nm.[8]

CIELAB color system was defined by the Commission Internationale de l'Eclariage (the International Commission on Illumination) in 1976. It is the most suitable system for making different color measurements. Color assessment using this method is widely accepted in dental research.[9-16] In Munsell and CIELAB color systems, the location of a color is determined by 3 coordinates. Brightness, hue, and saturation in the Munsell color system are defined as L*, a*, and b* in the CIELAB color system. L* stands for lightness and takes a value between 0 and 100. 0 represents black, and 100 represents white. a* denotes saturation (chroma) on the red–green axis, and b* denotes saturation (chroma) on the yellow–blue axis. In the CIELAB system, the ΔE value reports the amount of color difference observed in color measurements of the same object at 2 different time points.[17] The CIELAB values are related to the color perception of the human eye. The detectable or unacceptable color difference is expressed by the ΔE value equal to or above the specified threshold value. Given the ideal observing conditions, most observers could easily detect the color differences when the ΔE value was greater than 2.0.[18] In addition, it has been reported that color differences cannot be observed in cases where the ΔE value is below 1.0.[18] Studies have reported that the human eye can perceive ΔE values greater than or equal to 2.7 under clinical conditions.[9]

In the field of dentistry, composite resins are widely used because they meet aesthetic expectations and can be applied in minimally invasive applications.[19] In recent years, patients' desire for lighter shaded teeth has increased with the importance of aesthetics. Dental bleaching is a treatment applied under the control of a dentist. However, there are also over-the-counter bleaching products that can be used without the dentist's supervision. Over-the-counter bleaching products whiten teeth at lower costs than professional treatments applied by the dentist.[20] Whitening toothpastes remove discoloration by mechanical and chemical mechanisms. Today, the use of whitening toothpastes, which is the most easily accessible product among these products, has also increased. Although it is thought that whitening toothpastes, which are frequently used today, are expected to be effective in color recovery on the surface of composite resin restorations, the number of studies on this subject is still scarce. The aim of this in vitro study was to evaluate the color stability of nano-filled composite resin that was submitted to different staining solutions and mechanical brushing simulation with conventional or hydrogen peroxide-containing whitening toothpaste. The null hypothesis was that neither the use of toothpaste nor the type of staining solution would affect the color change.

Main Points

- The nano-filled composite resin underwent significant color change when aged in coffee and red wine. Due to brushing, the color change of the specimens immersed in the coffee remained below the acceptable threshold.
- The type of toothpaste had no significant impact on color recovery.
- The results of the present study demonstrate the significance of good oral hygiene in the maintenance of tooth-colored restorations.

METHODS

Study Design

The present laboratory study was designed to investigate factors influencing the color stability of nano-filled composite resin. The factors investigated were immersion in various staining solutions (1 hour per day for 5 days) at 3 levels: artificial saliva (as a control), coffee, and red wine; simulated mechanical brushing at 3 levels: no brushing (control), brushing with conventional toothpaste, and brushing with hydrogen peroxide-containing whitening toothpaste. The experimental design yielded 9 groups (n=10). The flowchart of the study design and materials used are shown in Figure 1 and Table 1, respectively.

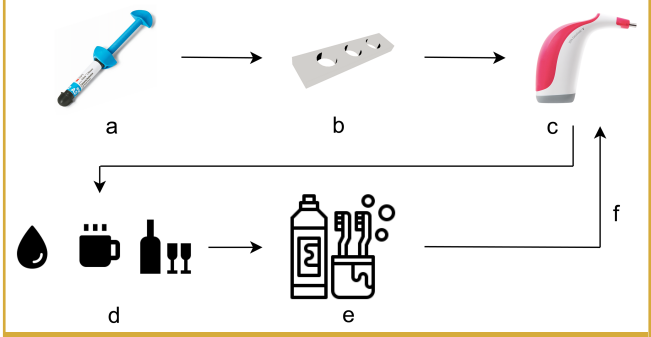

Figure 1. Flowchart of the study design. (a) Composite resin used. (b) Fabrication of composite resin discs. (c) Color measurement for baseline values. (d) Staining protocol: artificial saliva (control), coffee, and red wine. (e) Mechanical brushing protocol: no brushing (control), brushing with conventional toothpaste, and brushing with hydrogen peroxide-containing whitening toothpaste. (f) Final color measurement for calculating color change.

Table 1. Materials Used

Material	Type	Composition
Filtek Ultimate Universal Restorative (3M ESPE, St. Paul, MN, USA). Lot: #NE19736	Nano-filled composite resin	Organic matrix: Bisphenol-A glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA), poly(ethylene glycol) dimethacrylate (PEGDMA), ethoxylated bisphenol-A-glycidyl methacrylate (Bis-EMA), camphorquinone. Filler particles: 20 nm silica particles, 4-11 nm zirconium particles; 72.5% by weight.
Colgate Total 12 (Colgate-Palmolive Co., New York, NY, USA)	Conventional toothpaste	Glycerin, hydrated silica, aqua, sodium lauryl sulfate, arginine, aroma, zinc oxide, cellulose gum, benzyl alcohol, poloxamer 407, zinc citrate, tetrasodium pyrophosphate, cocamidopropyl betaine, sodium fluoride (1450 ppm F^-), sodium saccharin, xanthan gum, phosphoric acid, sucralose, CI 77891.
Colgate Optic White Expert (Colgate-Palmolive Co., New York, NY, USA)	Hydrogen peroxide-containing whitening toothpaste	Glycerin, propylene glycol, calcium pyrophosphate, PEG/PPG-116/66 copolymer, polyvinylpyrrolidone (PVP), PEG-12, pentasodium triphosphate, sodium lauryl sulfate, silica, aroma, sodium monofluorophosphate, disodium pyrophosphate, sodium saccharin, hydrogen peroxide, limonene.

Specimen Preparation

The nano-filled composite resin specimens (Filtek Ultimate Universal Restorative; 3M ESPE, St. Paul, MN, USA) were prepared by placing composite resin into a Teflon mold (8 × 2.1 mm). Transparent polyester strips and 1-mm-thick glass coverslips were used on both surfaces of Teflon molds and gentle finger pressure was applied to these glass coverslips to obtain flat sample surfaces. Specimens were photopolymerized on both sides over glass coverslips with an LED (Valo Grand; 1000 mW/cm^2; Ultradent Products, South Jordan, Utah, USA) for 20 seconds.

As a result of the power analysis executed with the G*Power software (version 3.1; University of Dusseldorf, Germany) program to determine the number of samples in the study, the number of samples determined for 80% power and α: 0.05 was determined as 10 for each group. Ninety disc-shaped specimens were prepared. Afterward, the surface of specimens was smoothed under water with the help of silicon carbide abrasives with 600-, 800-, 1200-, 2500-, and 4000-grit under water. The thickness of the specimens was measured using a digital caliper to ensure a final thickness of 2 mm. All specimens were cleaned for 10 minutes with ultrasonic cleaner and were then placed in artificial saliva for 24 hours at 37°C to ensure complete polymerization.

Staining and Mechanical Brushing Protocol

After the initial color measurements were made, each material group was randomly divided into 3 in accordance with the staining solutions: artificial saliva (distilled water, 1 mM $CaCl_2$, 50 mM KCl, 2 mM KH_2PO_4, and 0.01% NaN_3, pH adjusted to 7 with 1 M KOH; control group), coffee (Nescafe Gold, Single bags; Nestle, Switzerland), and red wine (DLC Cabernet Sauvignon-Merlot; 14.5% alcohol by volume; Doluca, Tekirdag, Turkey). The coffee was prepared by dissolving 2 g of coffee granules in 200 mL of boiled distilled water for 7 minutes in line with the recommendations of the manufacturer and left to cool at room temperature. The wine used in the study was freshly opened during the experiment and carefully shaken to prevent sediment build-up before use. The specimens in coffee and red wine groups were immersed in staining solution for 1 hour, mechanically brushed for 30 minutes, and kept in artificial saliva at 37°C for 22.5 hours. The specimens in the artificial saliva group (as control) were maintained in artificial saliva at 37°C for 23.5 hours and then submitted to the mechanical brushing protocol for 30 minutes. Staining solutions were renewed every day.

After immersion in the staining solutions, the specimens were allocated into 3 subgroups: no brushing (control), brushing with conventional toothpaste (Colgate Total 12; Colgate-Palmolive Co., New York, NY, USA), and brushing with hydrogen peroxide-containing whitening toothpaste (Colgate Optic White Expert; Colgate-Palmolive Co., New York, NY, USA). The specimens were subjected to a brushing simulation using a mechanical device, which was originally made for chewing simulation (MOD dental, Esetron, Ankara, Turkey), with modifications to serve as a mechanical brushing simulator. Mechanical brushing simulations were operated under 4.5 N loading at 0.6 Hz (36 cycles per minute for 30 minutes) using toothbrushes (TePe Supreme; Soft-bristled; TePe, Malmo, Sweden). A toothpaste slurry (1 : 3 ratio) was prepared with distilled water and renewed every 10 minutes.[21]

The samples were kept in artificial saliva until the next exposure cycle. The staining and mechanical brushing cycle was applied for 5 days and color measurements of all samples were repeated at the end of the fifth day.

COLOR STABILITY MEASUREMENT

The color measurements were carried out using Easy Shade V (VITA Zahnfabrik H. Rauter GmbH & Co, Bad Sackingen, Germany) under D65 standard lighting conditions against a

standard white background according to the CIELAB system. The specimens were rinsed for 10 seconds and gently dried. The clinical spectrophotometer was calibrated with supplied calibration block following the manufacturer's instructions prior to the measurements. The color difference (ΔE) was determined using a formula based on the L*, a*, and b* values between the same specimen baseline and after the staining and mechanical brushing cycle:

$$\Delta E = \sqrt{(L_1 - L_0)^2 + (a_1 - a_0)^2 + (b_1 - b_0)^2}$$

The acceptability threshold level was considered as ΔE = 2.7.[9]

Statistical Analysis

The statistical analysis was executed using statistical software (GraphPad Prism; GraphPad Software, San Diego, Calif, USA). The data distribution was examined with the Shapiro–Wilk test. The ΔE values were analyzed by 2-way analysis of variance (ANOVA) with staining solution and toothpaste type as the main factors. Tukey's honestly significant difference test was conducted for pair-wise comparisons. A *P*-value of < .05 level was considered statistically significant.

RESULTS

The results of the 2-way ANOVA analysis are shown in Table 2. Two-way ANOVA test results indicated that staining solution (factor 1) and mechanical brushing (factor 2) had a statistically significant influence on the ΔE values ($P < .001$). Additionally, statistically significant interaction between these 2 factors was detected ($P < .001$) (Table 3).

The ΔE values in coffee and red wine were noticeably higher compared with those in artificial saliva ($P < .05$). Moreover, the ΔE values of the specimens immersed in red wine were significantly higher than those of the specimens kept in coffee. In comparison to the control group with no brushing, the ΔE values were found to be significantly lower in both groups in which specimens were submitted to brushing protocol. It is observed that the type of toothpaste is not significantly important for the color recovery effect of mechanical brushing ($P > .05$).

Table 2. Mean and Standard Deviation of Color Change (ΔE ± SD) Values of Nano-Filled Composite Resin After Submitting Different Staining Solutions and Daily Tooth Brushing Simulation

Immersion Solution	Mechanical Brushing Simulation		
	No Brushing	Conventional Toothpaste	Whitening Toothpaste
Control	$0.54 \pm 0.06^{c, A}$	$0.45 \pm 0.08^{c, A}$	$0.43 \pm 0.06^{c, A}$
Coffee	$3.77 \pm 0.43^{b, A}$	$2.34 \pm 0.3^{b, B}$	$2.14 \pm 0.33^{b, B}$
Red wine	$7.64 \pm 0.65^{a, A}$	$3.44 \pm 0.55^{a, B}$	$3.25 \pm 0.53^{a, B}$

Mean values represented with same superscript uppercase letters (row; among brushing simulations) or lowercase letters (column; among immersion solutions) are not significant according to pairwise test ($P > .05$).

The ΔE values of the specimens stained with coffee and subjected to the mechanical brushing protocol were below the clinically acceptable threshold. However, although the ΔE values were greatly reduced as a result of brushing, the final ΔE values of the red wine-immersed specimens were found above the acceptability threshold (Figure 2).

DISCUSSION

Ensuring color match and preserving the color of the restorative material during clinical use is critical to achieving esthetic outcomes. Color change over time is one of the major disadvantages of composite resin restorations, and this can indicate the need for restoration renewal. This in vitro study aimed to evaluate the color stability of nano-filled composite resin that was submitted to different staining solutions and mechanical brushing simulation with conventional or hydrogen peroxide-containing whitening toothpaste. Under the conditions of this study, the effects of the staining solutions on the color change of composite resin varied depending on the staining solution used. Additionally, mechanical brushing with toothpaste significantly reduced ΔE values and contributed to the color recovery. Therefore, the null hypothesis that "neither the use of toothpaste nor the type of staining solution would affect the color change" was rejected.

Studies evaluating color stability mostly examine the effects of coffee and red wine consumption.[10,16,20,22] It was found that consumers drank a cup of coffee in 15 minutes and the daily consumption was equivalent to 3 cups.[16] In another study examining the effects of mouthwashes on the color stability of aesthetic restorative materials, staining solutions were applied for 2 minutes daily.[23] Another study reported that the oral environment is exposed to hot beverages for a total of 1 minute while consuming.[24] If the prepared samples are kept in the staining solutions at once, the mechanical brushing simulation cannot be performed and the staining solution may form a residue on the outer surface of the composite resin.[16] Therefore, in this study, the specimens were kept in coffee and red wine for 1 hour, then submitted to mechanical brushing for 30 minutes, and kept in artificial saliva for 22.5 hours, and this cycle was repeated for 5 days.

In the field of dentistry, the CIELAB color system is favored in many studies for the determination of ΔE value and is cited in the literature as the most commonly used color measurement system in the literature.[9-16] The color difference becomes important when it is recognized by the human eye. The human eye has a limited capacity to perceive color differences. Given the ideal observing conditions were provided, most observers could easily detect the color differences when the ΔE value was greater than 2.0.[24] In addition, it has been reported that color differences cannot be observed in cases where the ΔE value is below 1.0.[24] Studies have reported that the human eye can detect ΔE values

Table 3. Two-Way ANOVA Results of Color Change (ΔE) Values

Source	Type III Sum of Squares	df	Mean Square	F	Significance
Corrected model	417.118[a]	8	52.140	301.416	.000
Intercept	640.000	1	640.000	3699.797	.000
Brushing	78.091	2	39.046	225.720	.000
Staining	278.093	2	139.046	803.817	.000
Brushing × staining	60.934	4	15.233	88.064	.000

[a]$R^2 = 0.968$ (adjusted $R^2 = .964$).

greater than or equal to 2.7 under clinical conditions.[9] For standardization purposes, clinical spectrophotometer device was used to determine ΔE values in the present study. Digital color measurement devices have been developed in order to minimize potential errors in the detection of ΔE values, detect color differences that cannot be detected with the naked eye, and make objective measurements.[25]

Color pigments in most liquids can cause discoloration of the composite resin since composite resin materials can absorb water.[6,10] Water absorption is mainly related to the polymer content in the resin matrix. Excessive water absorption causes expansion and plasticization of the composite resin. This creates microcracks or interfacial voids at the interface between the filler and the organic matrix, resulting in stain penetration and color change in the composite resin. Coffee has yellow coloring pigments that interact with the organic matrix of the composite resin and these pigments are responsible for the staining ability of coffee.[26] Red wine is another staining beverage with an acidic pH, water-soluble pigments, and alcohol content such that it has been associated with its staining ability.[27,28] According to the results of the present study, the ΔE values of composite resin specimens after immersing in coffee (ΔE = 3.77 ± 0.43) and red wine (ΔE = 7.64 ± 0.65) range outside of the acceptability threshold (ΔE = 2.7). This result is consistent with other studies suggesting that coffee and red wine cause a significant color change in composite resins.[12,16,20]

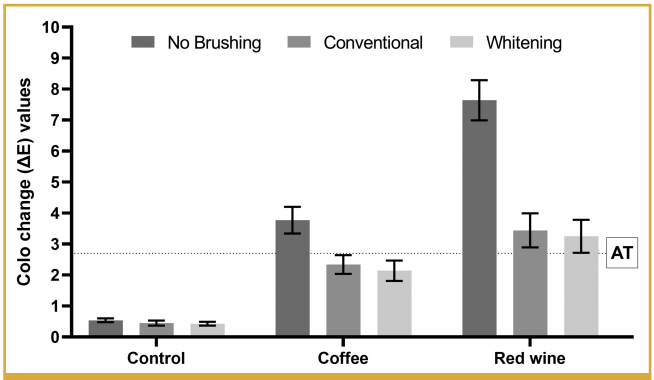

Figure 2. Color change values (ΔE) of nano-filled composite resin according to immersion media and mechanical brushing simulation at the end of the fifth day. Acceptability threshold (AT) = 2.7.

The results of the present study revealed that the highest ΔE values were observed for red wine immersion. It is believed that this result is caused by the alcohol and tannins in the composition of red wine. The red wine used in the study contains 14.5% alcohol by volume. Many studies have been reported that ethanol causes softening by removing structures such as unreacted monomers, oligomers, and linear polymers on the composite resin surface.[29,30] The decrease in hardness on the surface facilitates the absorption of the pigments in wine by the resin structure and causes more color change.[31] According to the results of the study, it was determined that the specimens kept in the coffee solution also showed ΔE values above the acceptability threshold (ΔE = 2.7). In a study evaluating the effect of coffee on the color stability of composite resins, it was reported that the yellow colorants in coffee have low polarity and thus have the ability to adhere to the surface and penetrate deeper.[32] Previous research reported that composite resins immersed in coffee, coke, and red wine showed ΔE values above the clinical limits, and red wine caused the most staining, similar to our study.[29]

In a study in which 2 different composite resins were first immersed in coffee and red wine solutions and then brushed with different toothpastes, it was reported that the color of both composite materials was close to the initial color after the brushing process and there was no clinically significant difference between the whitening efficacy of toothpastes.[21] Similarly, in the present study, toothpastes were found to be effective in the color recovery, but there was no significant difference compared to each other. A recent study examined the color recovery efficiency of whitening toothpastes, conventional toothpastes, and 2 different whitening agents on composite resins stained in a coffee. As a result of the study, it was reported that there was no statistically significant difference between the groups in ΔE values before and after brushing. Additionally, it was concluded that effective tooth brushing was more important than the type of toothpaste used.[33] In a previous study, the color change values of composite resins were recorded at the end of 1, 7, 30, and 90 days after brushing with conventional and whitening toothpastes. It was found that the color changes of the composite resins were not clinically detectable at all time points. Thus, consistent with the results of the current study, the tested toothpastes showed no significant difference.[34]

Conflicting results may be encountered in studies evaluating color stability. In the methodology of some studies, no polishing protocol was applied on the specimen surfaces after restoration. If the composite resin surfaces are not polished after polymerization, the oxygen inhibition layer cannot be removed and will cause extrinsic pigments to accumulate more intensely. Therefore, in our study, mechanical polishing was applied to the specimen surfaces in a way that imitates the clinical conditions. On the other hand, composite resins with smaller particle-sized fillers could explain nano-filled composite resin's lower susceptibility for extrinsic staining.[35] On the contrary, in some studies, it has been found that composites with small particle size do not always show less color change, and rather than the particle size, the filler ratio and organic matrix content of composite resins affect color stability.[21]

It is noteworthy to point out a limitation that the present laboratory study was designed to investigate 2 factors: staining solutions and mechanical brushing. Other factors such as thermal fluctuations in the oral environment, salivary clearance, and biofilm adhesion may also influence the color stability of composite resins. Additionally, the evaluation period in the present study was short. A longer experimental setup may lead to different results, which should be taken into account for further studies.

Within the limitations of this study, it was found that immersing nano-filled composite resins in coffee and red wine caused a significant color change. As a result of the mechanical brushing, color recovery was achieved within the acceptability threshold on specimens immersed in coffee. Although the color change was still clinically distinguishable, mechanical brushing reduced the effect of red wine staining on nano-filled composite resins. In addition, the results of the present study underscore the significance of oral hygiene habits in maintaining tooth-colored restorations.

Ethics Committee Approval: No ethical approval was required for this article since it does not contain any studies with human participants or animals performed by the author.

Informed Consent: For this type of study, formal consent is not required.

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