



Effect of Charcoal-containing Mouthwashes on Color Stability of Nanofill and Microhybrid Composites

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Abstract

Background: Charcoal-containing mouthwashes have become popular in society; however, there is inadequate evidence of their effect on composite restorations' color stability. This study evaluates the effect of two charcoal-containing mouthwashes on the color stability of two composite resins.

Methods: Disk-shaped (6×2 mm) composite resins (Filtek Universal Restorative (FU) and G-ænial Anterior (GC)), each (n=30), were prepared. Baseline color values were measured using a spectrophotometer. After six months of storage in distilled water, randomly assigned samples were subjected to a 12 h immersion in either distilled water (control group), Natural Mouthwash Charcoal, or Colgate Plax White+Charcoal (n= 10). Then, color values were re-measured, and color change (ΔE_{00}) values were computed. Two-way analysis of variance (ANOVA) and Tukey's honestly significant difference (HSD) multiple comparison tests were used to compare mean ΔE_{00} values of composite resin materials ($P < .05$).

Results: ΔE_{00} values of FU exposed to Natural Mouthwash Charcoal were significantly lower compared with other mouthwash groups and the controls. There were no significant differences in ΔE_{00} values among the GC specimens placed in different immersion media ($P > .05$). No statistically significant difference was observed regarding color change between composite restorative materials. ΔE_{00} values for the restorative materials were above the acceptability threshold of 1.8 for all groups.

Conclusion: Charcoal-containing mouthwashes caused unacceptable color changes in aged composites. In most groups, the color change was not significantly different from that in distilled water. The color change of FU immersed in Natural Mouthwash Charcoal was less than that in distilled water. Charcoal-containing mouthwashes can be considered part of daily oral hygiene practices.

Keywords: Charcoal, charcoal-containing mouthwash, color change, color stability, composite resin

INTRODUCTION

Composite resins are commonly chosen in restorative dental treatments due to their highly satisfactory esthetic properties, which allow them to blend seamlessly with natural teeth.¹ Despite their advantages, composite resins are susceptible to discoloration caused by a variety of internal and external factors. Internal factors include the composition of the resin matrix, the size of the filler particles, the extent of the applied load, and the type of photoinitiator used in the resin. External factors contributing to discoloration include the accumulation of plaque on the teeth, consumption of various foods and beverages that may stain the resin, and the use of certain mouthwashes that can alter the color of the restorations.¹⁻³ The discoloration of composite restorations remains a primary reason for their replacement, a process that can be both costly and time-consuming for patients, impacting their overall dental care experience.

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Mouthwashes are used to control biofilm formation, prevent halitosis and caries, and whiten teeth.^{2,4,5} Some mouthwashes may include dyes, detergents, emulsifiers, and organic acids that have the potential to alter the color of composite resin restorations. The absorption of these components into restorative materials can lead to discoloration.^{2,4,6}

Charcoal, a lightweight black carbon and ash residue hydrocarbon, is obtained through a process known as slow pyrolysis. The term slow pyrolysis refers to the elimination of water and other volatile components from carbon-rich materials. Utilizing materials like nutshells, peat, or coconut husks, charcoal is produced by subjecting these substances to heat in an environment devoid of oxygen.⁷ As society becomes increasingly health-conscious, interest in charcoal, used since ancient Greece, has increased again. In this context, many charcoal products have been launched for personal hygiene and dental care.^{7,8} Besides toothpastes and dentifrices, the commercial marketplace has seen charcoal-containing mouthwashes.⁸ Although not scientifically proven, 96% of activated charcoal products on the market claim to bleach teeth successfully.^{9,10} Highly porous activated charcoal compounds have the ability to exchange ions through nanopores. In this way, it can adhere to tooth enamel. It is suggested that its capacity to adsorb stains on the tooth surface can remove coloring agents from the tooth.¹¹ However, the presence of charcoal particles in mouthwashes has the potential to amass in pits, fissures, and dental defects. These particles could also aggregate in the spaces between teeth and dental restorations, resulting in the formation of a dark line surrounding the margins of restorations.¹² This accumulation could have adverse effects on dental aesthetics, potentially requiring the replacement of restorations. While whitening mouthwashes containing charcoal are readily accessible, it is important to note that the safety and effectiveness of charcoal for therapeutic or cosmetic purposes cannot be definitively asserted due to inadequate clinical and laboratory evidence.^{7,8}

Therefore, this study aimed to evaluate the effect of two commercially accessible mouthwashes containing charcoal,

with distilled water serving as the control, on the color stability of two distinct resin composites. The null hypothesis was that there would be no differences in the color of the tested composites after aging in different mouthwashes.

MATERIALS AND METHODS

The composite resin restorative materials and various mouthwashes examined and analyzed in the present study are detailed in Table 1. Distilled water was used as a control solution throughout the experiments. A power analysis was conducted prior to the commencement of the sampling process. The determination of the sample size was carried out utilizing the G Power software (Version 3.1, Heinrich Heine Dusseldorf University, Dusseldorf, Germany) with a 95% confidence level and 95% statistical power, drawing upon findings from previous research.⁶ The requisite sample size for each cohort in the present investigation was established to be a minimum of 9, with an effect magnitude of 0.645. Consequently, 10 specimens were prepared for each group.

Sample Preparation

Sixty disc-shaped specimens were fabricated from two distinct composites, comprising a nanofilled composite resin (FU; Filtek Universal Restorative; 3M ESPE, St. Paul, MN, USA) and a microhybrid composite resin (GC; G-ænial Anterior; GC, Tokyo, Japan) with the A2 shade. The specimens had a diameter of 6 mm and a thickness of 2 mm. The composite resins were placed in a Teflon mold and compressed between glass plates following the application of celluloid tape on the upper surface. A light-emitting diode (LED) curing device (Elipar S10; 3M ESPE, St. Paul, MN, USA) with a light intensity of 1250 mW/cm² was used to polymerize the samples for 10 s as per the manufacturer's guidelines. Post-polymerization, the specimens were immersed in distilled water at 37°C for 24 h to allow complete curing, followed by finishing and polishing using a series of abrasive discs (Sof-Lex; 3M ESPE, St. Paul, MN, USA). The abrasive discs were changed every two specimens in descending order with intermittent motions

Table 1. Materials Used in the Study

Brand	Manufacturer	Chemical Composition	Batch Number
Filtek Universal Restorative	3M, St. Paul, MN, USA	Silane treated ceramic, Aromatic urethane dimethacrylate, Diurethane imethacrylate (DUDMA), Ytterbium fluoride (Ybf3), 1,12-Dodecane dimethacrylate (DDDMA), Silane treated silica, Silane treated zirconia, Water, N,N-Dimethylglycylbenzocaine	NF18742
G-ænial Anterior	GC, Japan	Urethane dimethacrylate (UDMA), dimethacrylateco-monomers, Pre-polymerized fillers (Silica containing, Strontium and lanthanoid, Fluoride containing), Inorganic filler (Silica, Fumed silica)	2012252
Natural Mouthwash Charcoal	The Humble Co. AB, Sweden	Aqua, Glycerin, PEG-40 Hydrogenated castor oil, Betaine, Aroma, Sodium saccharin, Mentha piperita oil, Chlorhexidine digluconate, <i>Lycium barbarum</i> fruit extract, <i>Camellia sinensis</i> extract, Charcoal, <i>Panax ginseng</i> root extract, <i>Aloe barbadensis</i> leaf juice, Sodium fluoride, Citric acid, Sodium benzoate, Potassium sorbate	30033619
Colgate Plax White+Charcoal	Colgate-Palmolive, Poland	Aqua, <i>Bambusa vulgaris</i> shoot extract, Benzyl alcohol, Charcoal powder, Cl 17200, Cl 19140, Glycerin, Polysorbate 20, Propylene glycol, Sodium fluoride, Sodium saccharin, Sorbitol, Tetrapotassium pyrophosphate, Tetrasodium pyrophosphate, Zinc citrate	10197702

DDMA; 1,12-Dodecane dimethacrylate, DUDMA; Diurethane dimethacrylate, UDMA; Urethane dimethacrylate

and rinsed with distilled water. Prior to baseline color measurement, the specimens were immersed in distilled water at 37°C.

Color Measurement of the Composite Resin Samples

Prior to the immersion regimens, the baseline color measurements were conducted in accordance with the Commission Internationale de l'Eclairage (CIE) L*, a*, b* color system relative to a standard illuminant D65 on a white background. Utilizing a reflection spectrophotometer (CM-3600A; Konica Minolta, Osaka, Japan) with a wavelength range of 360 to 740 nm, the measurements were facilitated through the SpectraMagic NX software (Konica Minolta, Tokyo, Japan). Calibration of the spectrophotometer was performed before each measurement session following the guidelines provided by the manufacturer. The acquisition of all values was carried out by a designated operator. The color values (L*, a*, b*) for the upper surface of each specimen were obtained through three measurements, with each sample positioned on the spectrophotometer's measuring head. Upon the completion of three measurements for each sample, the device automatically computed the mean values of L*, a*, and b*, which were subsequently documented. The color assessments were executed at two distinct time points, namely baseline and post-immersion, utilizing the same designated samples.

After conducting the baseline color assessments, the specimens belonging to each respective group were segregated into 3 distinct subcategories. The surfaces of the samples in each group that would not be measured were numbered, thus ensuring that the value measured at the beginning and the value measured after the experiment belonged to the same samples. The samples were submerged in 2 mouthwashes, Natural Mouthwash Charcoal (Natural MW; The Humble Co. AB, Sweden) and Colgate Plax White+Charcoal (Colgate MW; Colgate-Palmolive, Poland), while distilled water served as a control (n=10). Color coordinates were measured before the immersion as a baseline. Samples were stored in distilled water at 37°C (pH=5.8) for 6 months. After aging, the samples were kept in 20 mL solutions for 12 h at 37°C, corresponding to 1 year with 2 min of daily use.¹³ After the completion of the experiments, the samples were rinsed for 10 s with distilled water, gently wiped dry, and final color measurements were performed immediately. The values of color change (ΔE) for the specimens were computed utilizing the CIEDE2000 color difference equation:

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{k_C S_C}\right) \left(\frac{\Delta H'}{k_H S_H}\right)}$$

in which $\Delta L'$, $\Delta C'$, and $\Delta H'$ represent the disparities in lightness, chroma, and hue, respectively. S_L , S_C , and S_H denote weighting functions designed to calibrate the overall color difference to accommodate variations in the position of the

color difference pair within L', a', and b' coordinates. The factors K_L , K_C , and K_H serve as adjustment parameters for specific experimental conditions. R_T functions as a rotational mechanism that considers the interplay between chroma and hue variances in the blue spectrum.^{14,15} The ΔE values were assessed using the 50 : 50% perceptibility threshold and the 50 : 50% acceptability threshold. Specifically, the ΔE_{00} 50 : 50% perceptibility threshold was set at $\Delta E_{00}=0.8$, while the 50 : 50% acceptability threshold was established at $\Delta E_{00}=1.8$, as per the guidelines of a previous study. This approach allows for a standardized evaluation of color differences in terms of what is perceptible and what is considered acceptable.¹⁶

Statistical Analysis

Statistical analyzes were conducted using GraphPad Prism software (GraphPad Software Inc.; San Diego, CA, USA). The assessment of the data distribution's normality was carried out by employing the Shapiro-Wilk test. Two-way ANOVA and Tukey-HSD multiple comparison tests were used to compare the mean ΔE_{00} of composite resin materials. In all tests, $P < .05$ was considered statistically significant.

RESULTS

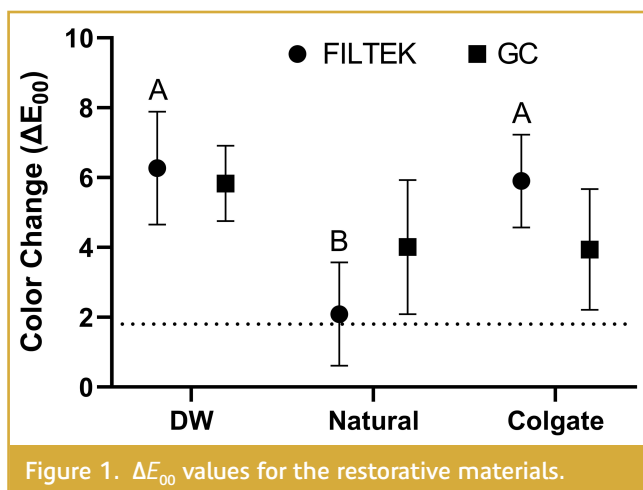
The mean and standard deviations of the ΔE_{00} values of the restorative substances in different immersion solutions are presented in Table 2. According to the two-way ANOVA test, the mouthwash factor had a statistically significant effect on ΔE_{00} values ($P < .001$), while the restorative material type did not significantly affect the color change ($P=.691$). A significant interaction between these two factors was also detected ($P < .001$).

The ANOVA analysis revealed that ΔE_{00} values of FU in Natural MW were significantly lower compared with Colgate MW and the control. The minimum alteration in color was detected when examining FU submerged in a solution of Natural MW. The ΔE_{00} values of the GC specimens immersed in various media did not exhibit any considerable differences ($P > .05$). Furthermore, there was no statistically significant variance noted between composite resin restorative materials regarding color stability. Notably, the ΔE_{00} values for the restorative materials were above the acceptability threshold of 1.8 in all experiments (Figure 1).

Table 2. Mean Color Change (ΔE_{00}) Values and Standard Deviations of Tested Composite Resin Materials

	FILTEK	GC	P-Value
Distilled water (Control)	6.27 (1.62) ^A	5.83 (1.08) ^A	.988
Natural mouthwash	2.09 (1.48) ^B	4.01 (1.92) ^A	.078
Colgate mouthwash	5.90 (1.33) ^A	3.94 (1.73) ^A	.068

Different superscript upper-case letters in the same column imply significant differences among the storing medium according to the pairwise analysis ($P < .05$). The values in the P-value column represent the results of pairwise comparisons between composite resin materials. FU; Filtek Universal Restorative, GC; G-ænial Anterior



DISCUSSION

Direct composite resin restorations are extensively employed in dental practice due to their superior esthetic qualities and ability to closely match the natural appearance of teeth.¹ However, a significant concern arises from the fact that certain mouthwashes can lead to discoloration of these composite restorations, thereby compromising their aesthetic appeal.⁴ This issue is particularly concerning for patients who prioritize the visual aspect of their dental restorations. In this study, it was observed that composites submerged in charcoal-containing mouthwashes exhibited color changes that were deemed clinically unacceptable, highlighting the potential negative impact of these products on the esthetic longevity of composite restorations. Therefore, the null hypothesis was rejected.

Utilization of spectrophotometers is common for the assessment of color modifications due to their ability to eliminate subjective inaccuracies and their reliance on the CIE L*, a*, b* color system.¹⁷ The color of composite resins was assessed in this research through the application of a spectrophotometer. The ΔE_{00} value was determined utilizing the CIEDE2000 formula, which represents an enhancement of the preceding CIELAB formula.¹⁵ This study observed a color change of over 1.8 in the control group and all composite groups immersed in charcoal-containing mouthwash. There was no significant difference between composites. The organic matrices of both composites contain UDMA. Their inorganic structures differ. While FU is a nanofilled composite, GC is a microhybrid composite and contains prepolymerized fillers. The configuration of the composite resin materials, encompassing the resin compositions and filler dimensions, significantly influences the susceptibility of the material to discoloration caused by external factors.¹⁸ A smaller filler size is commonly thought to enhance resistance to staining.¹⁹ Nonetheless, there is also a proposition that hybrid composite resins exhibit superior color steadfastness compared to nano-filled composites.^{18,20} In

this study, no significant difference in color change between nano-filled and microhybrid composites was observed, which is in line with a previous study.²¹ Aluminum oxide discs have been documented to be capable of generating polished surfaces on composite resins.²² In the current investigation, a multi-step polishing approach was implemented.

An unacceptable level of discoloration was observed in the samples after they were immersed in distilled water for a duration of 6 months. Previous studies have indicated that distilled water can lead to internal and external discoloration of resin-based materials. This discoloration is attributed to the hydrophilic properties of the organic matrix within the composite material. Over time, discoloration occurs as a result of water sorption by the material, causing changes in its appearance. This phenomenon underscores the impact of prolonged exposure to distilled water on the esthetic properties of resin-based composites.^{23,24} Water absorption expands and plasticizes composite resin's components, hydrolyzes the silane coupling agents, and allows stains to penetrate.²⁵ Previous research reported that no color differences were observed when composite resins were immersed in distilled water for 24, 48, and 72 h. Thus, it was proposed that water absorption does not change the color of the composite.¹⁹ However, water absorption increases with a longer waiting time.²⁶ The difference in results may be due to the extended storage period in the present investigation. In addition, the degree of conversion can affect water absorption. A low degree of conversion weakens the composite's polymer cross-linking, making it susceptible to water absorption.²⁷ In future studies, the effect of the degree of conversion of the composites used in this research on color stability could be investigated.

In the current study, no difference was seen in the color change of the groups except FU kept in Natural MW, but all groups showed clinically unacceptable color change. Similarly, various investigations have indicated the absence of a notable distinction between the cohorts submerged in distilled water and the composites submerged in mouthwash. Within these investigations, the alteration in color demonstrates variability contingent on the specific composite material or mouthwash utilized. Nevertheless, in contrast to our examination, it was demonstrated in these investigations that the color change occurred at a level considered clinically acceptable.^{19,28-30} The composites in these investigations were not subjected to aging prior to immersion in mouthwash, whereas in our research, we posit that subjecting the composites to a 6-month aging process in distilled water enhances their susceptibility to color alteration.

In the findings of the present study, the color change of the FU immersed in Natural MW was less than that of the groups immersed in distilled water and Colgate MW. To the best of our understanding, there has been no other investigation in the existing literature that has assessed the characteristics of

Natural MW in order to compare them with the findings of the present investigation. Natural MW contains plant-based oils and herbal extracts, as well as charcoal. These substances may have varying impacts on color change in composite materials. In addition, the amount of substances in mouthwashes is also essential. Although publications provide detailed information on mouthwash ingredients, it is often challenging to find information on the quantities of each ingredient listed on the label. While it is possible to contact the product manager to obtain this information, detailed information is often not accessible due to the principle of formula confidentiality.^{31,32} Consequently, further research is necessary to determine which specific substance in the mouthwash is responsible for the observed results.

The present study has certain limitations. The oral environment must be considered when discussing the clinical relevance of the results, as it may differ in several ways from *in vitro* conditions. Factors such as the variety of food, saliva, and their interactions might intensify discoloration. In the oral cavity, the uneven surfaces of composite resin restorations placed in the teeth's posterior pits, fissures, and grooves may not always be perfectly polished. Color change may occur at different levels in these regions. In addition, the immersion period was 12 h in this study, which is equivalent to 1 year of usage, and longer immersion times may be tested in future studies.

CONCLUSION

This *in vitro* investigation found that charcoal-containing mouthwashes caused unacceptable color changes in aged composite materials. However, in most groups, the extent of the color change was not significantly different from that observed in distilled water. Interestingly, the color change in the FU samples immersed in Natural MW was less pronounced compared to those immersed in distilled water. This particular finding suggests that, despite the potential for discoloration, charcoal-containing mouthwashes might still be viable options as part of daily oral hygiene routines.

Availability of Data and Materials: The data that support the findings of this study are available on request from the corresponding author.

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 authors.

Informed Consent: For this type of study, informed consent is not required since it does not contain any studies with human participants or animals.

Peer Review: Externally peer-reviewed.

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Declaration of Interests: The authors have no conflicts of interest to declare.

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