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Color Stability of Dental Composites after Immersion in Beverages and Performed Whitening Procedures

Stabilnost boje dentalnih kompozita nakon uranjanja u napitke i postupaka izbjeljivanja

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Abstract

Objectives: The study aimed to compare the color stability of two different light-cured composites after immersion in three liquids and the effectiveness of 16% carbamide peroxide (CP) in removing the discoloration. **Material and methods:** Color stability of a microhybrid (Z250, 3M ESPE) and nanocomposite (Z550, 3M ESPE) was evaluated after immersion in instant coffee, tea, Coca-Cola, and deionized water as a control group (n=5). Samples were kept in liquids for four hours daily at 37°C for 30 days. Furthermore, 16% CP was applied for the following 14 days, simulating night whitening. A digital spectrophotometer was used for color measurement based on the CIEL*a*b* color coordinates. The color changes (ΔE) were measured at baseline, after immersion in the beverages, and also after the teeth whitening procedure. Mixed and factorial ANOVA followed by Bonferroni's post-hoc test were used for statistical evaluation ($p \leq 0.05$). **Results:** Tested resin composites showed a color change over the acceptability threshold ($\Delta E^* > 3.48$) after immersion in coffee and tea. Nanocomposite reported a significant increase in discoloration in coffee after 30 days ($p < 0.05$). The color of both materials significantly changed ($p < 0.05$) along all three L*a*b* axes in coffee and tea to darker, yellow, and red. Whitening with 16% CP was effective in removing external discoloration in both examined composite materials. **Conclusion:** Coffee and tea induced clinically detectable color changes in dental composites tested, with cumulative effects. Whitening represents an efficient method for the removal of surface discoloration in composite restorations.

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Introduction

Growing concerns regarding dental amalgam toxicity, patient requirements for aesthetic fillings, and a minimally invasive treatment approach resulted in the dominance of composite materials in terms of direct restorative dental treatment. Contemporary researchers focus on improving the physical characteristics and clinical longevity as well as esthetic appearance of existing composite materials, along with the introduction of new technologies. Although the quality of composite fillings has improved in recent years, discoloration after exposure to the oral medium continues to be a challenge for composite materials (1). Prolonged exposure of composites to the oral cavity conditions leads to restoration discoloration and a consequent color discrepancy between tooth and restoration. Discoloration causes patient dissatisfaction and the additional cost for the filling replacement (2).

Methods for removing external dental composite discoloration to achieve esthetics restoration include air polishing,

Uvod

Sve veća zabrinutost zbog toksičnosti dentalnoga amalgama i zahtjeva pacijenata za estetske ispune te minimalno invazivne pristupe liječenju, rezultirali su dominacijom kompozitnih materijala kad je riječ o izravnome restaurativnom dentalnom tretmanu. Suvremeni istraživači usredotočuju se, uz uvođenje novih tehnologija, na poboljšanje fizičkih obilježja i kliničke dugovječnosti te estetskoga izgleda postojećih kompozitnih materijala. Iako se kvaliteta kompozitnih ispuna posljednjih godina poboljšala, promjena boje nakon izlaganja oralnome mediju i dalje je izazov kada se radi o kompozitnim materijalima (1). Dugotrajno izlaganje kompozita uvjetima u usnoj šupljini rezultira promjenom boje restauracije i posljedičnom razlikom u boji između zuba i restauracije. Promjena boje potiče nezadovoljstvo pacijenta i dodatni trošak za zamjenu ispuna (2).

Metode uklanjanja vanjske promjene boje dentalnoga kompozita radi estetskoga izgleda restauracije obuhvaća-

bleaching or whitening, conventional repolishing, or even replacement of existing highly stained composite. Air polishing may produce roughness on the composite surface, even when using mild glycine-based powder (3). Composite color can be restored by repolishing, although this procedure partially removes material from the restoration surface. However, in the case of deep pigment penetration, it is not possible to completely restore the color of the material to its initial shade by polishing. Therefore, bleaching remains a minimally invasive approach for stain removal from direct resin-based composite restorations. The bleaching effect may be obtained either by using a bleaching agent in different concentrations, or various lamps to speed up the process (4). When used properly under the supervision of a dentist, peroxide-based tooth bleaching products are considered safe and effective (5).

With advancements in nanotechnology in recent years, dental composites with nanoparticles were introduced. Microhybrid composites and nanocomposites are the most commonly used materials for direct dental restorations with microscale/nanoscale, individual/clustered nanoscale glass fillers, respectively (6). As claimed, a high surface/volume ratio in nanocomposites results in improved mechanical, thermal, and optical properties (7). To achieve a longer esthetic appearance of the dental filling, it is important to identify a composite material less susceptible to discoloration. The objective of this study was to assess the color stability of two different composite resins in three frequently used solutions - coffee, tea, and Coca-Cola - and determine if bleaching with 16% carbamide peroxide (CP) gel can remove derived discoloration. The first hypothesis of this study was that beverages affect the discoloration susceptibility of the tested composite materials, and the second hypothesis was that bleaching with 16% CP gel would be effective in the color recovery of tested composites.

Material and methods

Resin dental composites used in this study include microhybrid composite (Filtek Z250 (3M ESPE, St. Paul, MN, USA) and nanohybrid composite (Filtek Z550, 3M ESPE, St. Paul, MN, USA) in shades A2. The characteristics of the composites used in this study are listed in Table 1.

ju zračno poliranje, izbjeljivanje, uobičajeno ponovno poliranje ili čak zamjenu postojećeg kompozita s jakim mrljama. Pjeskarenje može potaknuti hrpavost na površini kompozita čak i kada se upotrebljava blagi prah na bazi glicina (3). Boja kompozita može se obnoviti ponovnim poliranjem, iako se tim postupkom djelomično uklanja materijal s površine restauracije. No u slučaju dubokoga prodora pigmenta poliranjem nije moguće potpuno vratiti boju materijala u početnu nijansu. Zato izbjeljivanje ostaje minimalno invazivni pristup u uklanjanju mrlja s izravnih kompozitnih restauracija na bazi smole. Učinak se može postići korištenjem sredstva za izbjeljivanje u različitim koncentracijama ili raznim svjetilkama za ubrzanje procesa (4). Kada se pravilno koriste pod nadzorom stomatologa, proizvodi za izbjeljivanje zuba na bazi peroksida smatraju se sigurnima i učinkovitim (5).

S napretkom u nanotehnologiji posljednjih godina proizvedeni su dentalni kompoziti s nanočesticama. Mikrohibridni kompoziti i nanokompoziti najčešće su korišteni materijali za izravne dentalne nadoknade s mikro/nanomjernim, pojedinačnim/grupiranim staklenim punilima na nanomjери (6). Kako se tvrdi, visok omjer površina/volumen u nanokompozitima rezultira poboljšanim mehaničkim, toplinskim i optičkim svojstvima (7). Za postizanje duljega estetskoga izgleda ispuna zuba važno je identificirati kompozitni materijal manje osjetljiv na promjenu boje. Cilj ove studije bio je procijeniti stabilnost boje dviju različitih kompozitnih smola u trima najčešće korištenim otopinama – kavi, čaju i Coca-Coli – i ustanoviti može li izbjeljivanje 16-postotnim karbamid-peroksidnim (CP) gelom ukloniti nastalu promjenu boje. Prva hipoteza ove studije glasila je da napitici utječu na osjetljivost testiranih kompozitnih materijala na promjenu boje, a druga da bi izbjeljivanje 16-postotnim CP gelom bilo učinkovito u obnavljanju boje testiranih kompozita.

Materijali i metode

Dentalni kompoziti od smole korišteni u ovoj studiji bili su mikrohibridni kompozit (Filtek Z250 (3M ESPE, St. Paul, MN, SAD) i nanohibridni kompozit (Filtek Z550, 3M ESPE, St. Paul, MN, SAD) u nijansama A2. Karakteristike kompozita korištenih u ovoj studiji navedene su u tablici 1.

Table 1 Properties of the materials used in this study
Tablica 1. Svojstva materijala korištenih u ovoj studiji

Product	Manufacturer	Shade	#Batch number	Matrix	Filler particle size in μm	Filler weight in % (wt)	Type
Filtek Z550	3M ESPE, St. Paul, MN, USA	A2	N502352	Bis-GMA, UDMA, Bis-EMA, TEGMA, PEGDMA	20 nm silica, 1-10 μm zirconia/silica particles	82%	Nanohybrid
Filtek Z250	3M ESPE, St. Paul, MN, USA	A2	N535897	Bis-GMA, UDMA, Bis-EMA	0.01-3.5 μm zirconia/silica particles	82%	Microhybrid

Bis-GMA- Bisphenol A-glycidyl methacrylate; UDMA- Urethane dimethacrylate; Bis-EMA- Ethoxylatedbisphenol-A-dimethacrylate; TEGDMA- Triethylene glycol dimethacrylate; PEGDMA - polyethylene glycol dimethacrylate
Bis-GMA-bisfenol A-glicidil metakrilat; UDMA- uretan dimetakrilat; Bis-EMA-etoksiliranibisfenol-A-dimetakrilat; TEGDMA- Trietilen glikol dimetakrilat; PEGDMA - polietilen glikol dimetakrilat

Preparation of Samples

Composite disc samples were prepared using microscopic glass plates covered with Mylar strips and Teflon mold (10 mm in diameter and 2 mm in height). The resin was pressed by a plate to flatten, and it was photopolymerized through the glass and transparent strip from both sides for 20 seconds using a wireless light source with an irradiance of 1200 mW/cm² (Elipar™ FreeLight 2 LED Curing Light, 3M ESPE, St. Paul, MN, USA). The light intensity of the curing lamp was regularly monitored using a radiometer Bluephase Meter (Ivoclar Vivadent, Schaan, Liechtenstein). A total of 40 discs were prepared.

All samples were consequently processed with fine and superfine extra thin (dark orange to yellow) aluminum oxide-impregnated paper discs (Sof-Lex™, 3M ESPE, St. Paul, MN, USA), with average particle size of 24 μm, and 8 μm respectively, embedded in a low-speed handpiece. The abrasive discs were used with reduced speed under dry conditions and light pressure for 10 seconds to standardize the surface and mimic clinical conditions. The polishing disks were discarded after every specimen. After polishing, the samples were initially stored in distilled water at 37°C to complete the polymerization process.

After 24 hours, the samples were randomly divided into three experimental subgroups and the control group (n=5). Three different beverages used in this experiment were instant coffee, green tea, and Coca-Cola. The samples were immersed in these freshly prepared beverages for four hours during the day to simulate high intakes, then rinsed with distilled water, while in the remaining hours they were stored in deionized water. The samples were kept in experimental liquids at 37°C ± 1°C. The procedures were repeated during 30 days. Considering that 24-hour immersion simulates near one month of coffee consumption (8), the performed intermittent staining protocol mimics clinical exposure to these beverages over six months.

Five samples of both composites were kept in deionized water in the incubator at 37°C for 30 days (control group). Distilled or deionized water was previously used as a control group due to minimal color change reported (9, 10, 11).

Preparation of Beverages

The contents of the bag (17.5 g) of instant coffee Nescafe 3in1 Classic (Nestle, Hungary, Kft. Szerenczi Gyara) were poured using 150 ml of boiling water. The solution was stirred and allowed to cool for 10 minutes. Tea solution was prepared by immersing prefabricated tea bag (30 gr) Lipton green tea Nature (Unilever, Belgium) into 200 ml of boiling water. After ten minutes of stirring, the bag was removed. A factory-sealed 0.5-liter Coca-Cola package (Coca-Cola HBC, Sarajevo, B&H) at room temperature was used for the third subgroup.

The pH value of every beverage and pH of bleaching gel was determined at room temperature using a digital pHmeter PHYWE 13702.93 (Göttingen, Germany). Two-point calibration of pHmeter's electrode was performed using standard buffers at pH=4.0 and 7.0. The electrode was immersed in freshly prepared test beverages. A stable pH reading was

Priprema uzoraka

Uzorci kompozitnih diskova pripremljeni su s pomoću mikroskopskih staklenih ploča prekrivenih vrpcoma Mylar u teflonskom kalupu (10 mm u promjeru i 2 mm u visini). Smola je pritisnuta pločom da se izravna i fotopolimerizirana kroz staklo i prozirnu vrpcu s obje strane 20 sekunda iz bežičnoga izvora svjetlosti sa zračenjem od 1200 mW/cm² (Elipar™ FreeLight 2 LED Curing Light, 3M ESPE, St. Paul, MN, SAD). Intenzitet svjetla žarulje za polimerizaciju redovito se pratio radiometrom Bluephase Meter (Ivoclar Vivadent, Schaan, Lihtenštajn). Ukupno je pripremljeno 40 diskova.

Svi uzorci obrađeni su finim i superfinim ekstra tankim (tamnonarančastim do žutim) papirnatim diskovima impregniranim aluminijevim oksidom (Sof-Lex™, 3 M ESPE, St. Paul, MN, SAD), prosječne veličine čestica od 24 μm i 8 μm, umetnut u nasadnik male brzine. Abrazivni diskovi korišteni su pri smanjenoj brzini u suhim uvjetima i lagano ih se pritislo 10 sekunda da bi se standardizirala površina i oponašali klinički uvjeti. Diskovi za poliranje bačeni su nakon svakoga ispoliranoga uzorka. Nakon poliranja uzorci su uronjeni u destiliranu vodu na temperaturi od 37 °C kako bi se završio proces polimerizacije.

Nakon 24 sata uzorci su nasumično podijeljeni u tri eksperimentalne podskupine i kontrolnu skupinu (n = 5). Tri različita pića korištena su u ovom eksperimentu – instant-kava, zeleni čaj i Coca-Cola. Uzorci su bili uronjeni u te svježe pripremljene napitke četiri sata danju da bi se simulirali visoki unosi, zatim su isprani destiliranom vodom, a preostale sate bili su pohranjeni u deioniziranoj vodi. Čuvani su u eksperimentalnim tekućinama na 37 °C ± 1 °C. Postupci su se ponavljali 30 dana. S obzirom na to da 24-satno uranjanje simulira gotovo jednomjesečnu konzumaciju kave (8), provedeni protokol povremenoga bojenja oponaša šestomjesečnu kliničku izloženost tim napitcima.

Pet uzoraka obaju kompozita bilo je u deioniziranoj vodi u inkubatoru na temperaturi od 37 °C tijekom 30 dana (kontrolna skupina). Destilirana ili deionizirana voda prethodno je korištena kao kontrolna skupina zbog minimalne promjene boje (9, 10, 11).

Priprema napitaka

Sadržaj vrećice (17,5 g) instant kave Nescafe 3u1 Classic (Nestle, Mađarska, Kft. Szerenczi Gyara) prelijevan je sa 150 mL kipuće vode. Otopina se miješala i zatim hladila 10 minuta. Otopina čaja pripremljena je uranjanjem vrećice zelenoga čaja (30 gr) Lipton Nature (Unilever, Belgija) u 200 mL kipuće vode. Nakon deset minuta miješanja vrećica je izvađena. Za treću podgrupu korišteno je tvornički zatvoreno pakiranje Coca-Cole od 0,5 litara sobne temperature (Coca-Cola HBC, Sarajevo, BiH).

pH vrijednost svakog napitka i pH gela za izbjeljivanje određivani su na sobnoj temperaturi digitalnim pH-metrom PHYWE 13702.93 (Göttingen, Njemačka). Kalibracija pH-metarske elektrode u dvjema točkama provedena je korištenjem standardnih pufera pri pH = 4,0 i 7,0. Elektroda je uronjena u tek pripremljene ispitne napitke. Stabilno očitavanje pH postignuto je nakon 1 do 2 minute poslije uranjanja i za-

Table 2 Main characteristics of the beverages
Tablica 2. Glavna obilježja pića

Beverage	Main ingredients	pH
Nescafe 3 in 1 Classic	Coffee powder	6.45
Lipton green tea Nature	100% natural, green tea leaves.	6.48
Coca-Cola®	Carbonated water, sugar, caramel color, phosphoric acid, caffeine, natural flavors.	2.26
16% carbamide peroxide VivaStyle®	Glycerin 25-50%, Aqua 10–25%, Urea (Carbamide) Peroxide 16%, Carbomer 5 – 10, Potassium Nitrate 1–5, Sodium Hydroxide 1–5, Aroma 0.1–1.0, EDTA 0.1–1.0, Sodium Saccharin 0.1–1.0	7.00

achieved after 1-2 minutes of immersion, recorded as the pH of the sample. The composition of the immersion solutions (manufacturer's data) and their pH are presented in Table 2.

Composite Samples Bleaching Process

The samples were bleached using 16% carbamide peroxide gel Vivasyle (Ivoclar Vivadent AG, Bendererstrass, Liechtenstein) for seven hours a day over 14-days, mimicking the at-home-night technique. The bleaching agent was carefully removed using a cellulose cloth, rinsed with tap water for one minute, and distilled water consecutively. During the remaining 17 hours, the samples were stored in deionized water at 37°C (12).

Color Evaluation

A Vita Easyshade Compact digital spectrophotometer (Vita Zahnfabrik, Bad Säckingen, Germany) was used for color spectrophotometric determination, previously calibrated with an integrated standard white plate. Color measurements were performed 24 hours after polymerization – on the 7th, 14th, and 30th day of immersion and after whitening. At each measurement, the samples were extracted from deionized water, dried with cellulose wadding, and $L^*a^*b^*$ color parameters were evaluated according to the CIEL^{*} a^*b^* color scale, using white backing. Measurements were repeated three times and the mean L^* , a^* , and b^* values were recorded. The L^* vertical parameter refers to the lightness, while a^* and b^* color coordinates are chromatic axes in the red-green and yellow-blue direction, respectively. Based on the spectrophotometric determination of $L^*a^*b^*$ color values, the total difference between the two shades (ΔE) was calculated. The color shift (ΔE^*_{ab}) was calculated using equation $\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. When the ΔE was higher than the acceptability threshold ($\Delta E^* > 3.48$), it was considered clinically visible, while a value of 1.74 was accepted as the perceptibility threshold (13). The results were statistically analyzed using Statistical Package for the Social Sciences (SPSS) v.20 program package. The values of color changes were analyzed by mixed and factorial ANOVA test ($p \leq 0.05$). Subsequently, Bonferroni-corrected t-tests for independent samples were used for comparison (post-hoc analysis).

Results

The color of composite materials tested in this study changed in coffee and tea beyond the acceptability threshold ($\Delta E > 3.48$) as early as seven days after immersion. After 30

bilježeno je kao pH uzorka. Sastav otopina za uranjanje (podatci proizvođača) i njihov pH nalaze se u tablici 2.

Proces izbjeljivanja kompozitnih uzoraka

Uzorci su izbjeljivani korištenjem 16-postotnoga karbamid-peroksidnoga gela Vivasyle (Ivoclar Vivadent AG, Bendererstrass, Lihtenštajn) sedam sati na dan tijekom 14 dana, oponašajući tehniku *noću kod kuće*. Sredstvo za izbjeljivanje pažljivo je uklonjeno celuloznom krpom, isprano jednu minutu vodom iz slavine i destiliranom vodom uzastopno. Tijekom preostalih 17 sati bili su pohranjeni u deioniziranoj vodi na temperaturi od 37 °C (12).

Procjena boja

Za spektrofotometrijsko određivanje boja korišten je digitalni spektrofotometar Vita Easyshade Compact (Vita Zahnfabrik, Bad Säckingen, Njemačka) koji je prije toga kalibriran integriranom standardnom bijelom pločom. Mjerenja boje obavljena su 24 sata nakon polimerizacije – sedmoga, četrnaestoga i tridesetoga dana od uranjanja i poslije izbjeljivanja. Pri svakom mjerenju uzorci su ekstrahirani iz deionizirane vode i sušeni celuloznom vatom, a parametri boje $L^*a^*b^*$ ocjenjivani su prema ljestvici boja CIEL^{*} a^*b^* koristeći se bijelom podlogom. Mjerenja su ponovljena tri puta i zabilježene su srednje vrijednosti L^* , a^* i b^* . Vertikalni parametar L^* odnosi se na svjetlinu, a koordinate boja a^* i b^* kromatske su osi u crveno-zelenom i žuto-plavom smjeru. Na temelju spektrofotometrijskoga određivanja $L^*a^*b^*$ vrijednosti boja izračunata je ukupna razlika između dviju nijansi (ΔE). Pomak boje (ΔE^*_{ab}) izračunat je s pomoću jednadžbe $\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Kada je ΔE bio veći od praga prihvatljivosti ($\Delta E^* > 3,48$), smatrao se klinički vidljivim, a vrijednost od 1,74 prihvaćena je kao prag perceptibilnosti (13). Rezultati su statistički analizirani u programskom paketu Statistical Package for the Social Sciences (SPSS) v.20. Vrijednosti promjena boje analizirane su mješovitim i faktorijalnim ANOVA testom ($p \leq 0,05$). Nakon toga su za usporedbu (post-hoc analiza) korišteni Bonferronijevi ispravljeni t-testovi za nezavisne uzorke.

Rezultati

Boja kompozitnih materijala testiranih u ovoj studiji promijenila se u kavi i čaju iznad praga prihvatljivosti ($\Delta E > 3,48$) već sedam dana nakon uranjanja. Nakon 30-dnevno-

days of immersion, the smallest value of ΔE_3 was detected for the Z250 composite samples immersed in Coca-Cola, whereas the highest ΔE_3 value was detected for nanocomposite samples immersed in coffee ($\Delta E_3=13.23\pm 2.11$). During water immersion ($\Delta E_3=0.94\pm 0.17$) and Coca-Cola immersion ($\Delta E_3=0.71\pm 0.61$), no significant changes were detected in ΔE values of resin composites of both materials. The values of color difference (ΔE) after immersion in different solutions during the time are shown in Table 3. To determine which of the materials had a higher sensitivity to pigmentation caused by tested solutions, the post-hoc test of ΔE_3 value differences between two tested materials for each of the three solutions was performed by t-test for independent samples with Bonferroni corrected p-value ($p = 0.05/3 = 0.017$). After 30 days of immersion, statistically significant differences in color alterations were caused only by coffee ($p=0.01$). Although a visible color change was found in samples of both materials immersed in tea, the change was not statistically significant ($p=0.19$).

The values of ΔL^* , Δa^* , and Δb^* for tested materials immersed in tested solutions are given in Table 4. For each color dimension (L^* , a^* , and b^*) of both materials (microhybrid and nanocomposite), mixed ANOVA with Time (day one, day 30) as a repeated measure and Beverage (tea, coffee, Coca-Cola) between-groups was computed. Time x Bever-

ga uranjanja najmanja vrijednost ΔE_3 otkrivena je za uzorke kompozita Z250 uronjene u Coca-Colu, a najveća vrijednost ΔE_3 zabilježena je za uzorke nanokompozita uronjene u kavu ($\Delta E_3 = 13,23 \pm 2,11$). Tijekom uranjanja u vodu ($\Delta E_3 = 0,94 \pm 0,17$) i uranjanja u Coca-Colu ($\Delta E_3 = 0,71 \pm 0,61$) nisu otkrivene značajne promjene u vrijednostima ΔE kompozita smole oba materijala. Vrijednosti razlike u boji (ΔE) nakon uranjanja u različite otopine tijekom vremena prikazane su u tablici 3. Kako bi se ustanovilo koji je od materijala osjetljiviji na pigmentaciju prouzročenu ispitivanim otopinama, post-hoc test razlika vrijednosti ΔE_3 između dvaju testiranih materijala za svaku od triju otopina proveden je t-testom za nezavisne uzorke s Bonferronijevom korigiranom p-vrijednošću ($p = 0,05/3 = 0,017$). Nakon 30-dnevnoga uranjanja, statistički značajne razlike u promjenama boje prouzročene su samo kavom ($p = 0,01$). Iako je vidljiva promjena boje pronađena u uzorcima obaju materijala uronjena u čaj, promjena nije bila statistički značajna ($p = 0,19$).

Vrijednosti ΔL^* , Δa^* i Δb^* za ispitane materijale uronjene u ispitane otopine dane su u tablici 4. Za svaku dimenziju boje (L^* , a^* i b^*) oba materijala (mikrohibridni i nanokompozitni) izračunata je mješovita ANOVA s vremenom (prvi dan, 30. dan) kao ponovljenom mjerom i napitkom (čaj, kava, Coca-Cola) između grupa. Interakcija vrijeme x napitak

Table 3 The values of color difference (ΔE) with standard deviations, according to material, solution, and time of measurement
Tablica 3. Vrijednosti razlike u boji (ΔE) sa standardnim odstupanjima prema materijalu, otopini i vremenu mjerenja

Material	Z250				Z550			
	Water	Tea	Coffee	Coca-Cola	Water	Tea	Coffee	Coca-Cola
ΔE_1 (1.- 7. day)		4.49 (± 1.20)	4.55 (± 1.26)	1.79 (± 0.64)		4.50 (± 1.04)	7.61 (± 1.64)	1.25 (± 0.46)
ΔE_2 (1.- 14.day)		6.99 (± 1.04)	6.20 (± 1.42)	1.46 (± 1.38)		5.65 (± 1.66)	10.42 (± 2.46)	1.22 (± 0.83)
ΔE_3 (1.-30.day)	0.94 (± 0.17)	10.23 (± 2.23)	9.36* (± 1.39)	0.71 (± 0.61)	0.91 (± 0.30)	8.46 (± 1.59)	13.23* (± 2.11)	1.80 (± 0.81)
ΔE_4 (after bleaching)		2.37 (± 0.39)	1.59 (± 0.45)	2.48 (± 0.34)		2.73 (± 0.23)	1.92 (± 0.55)	3.12 (± 0.90)

*Statistically significant differences ($p = 0.01$) in a color difference between two tested materials at a specific time

*Statistički značajne razlike ($p = 0,01$) u razlici u boji između dvaju testiranih materijala u određeno vrijeme

Table 4 Means and standard deviations of color change in L^* , a^* , and b^* axis (color change 1st-30th day) of microhybrid and nanocomposite in three different solutions

Tablica 4. Srednje vrijednosti i standardne devijacije promjene boje na osi L^* , a^* i b^* (promjena boje 1. do 30. dana) mikrohibridnog i nanokompozita u trima različitim napitcima

Material	Solution	ΔL	Δa	Δb
Filtek Z250	Tea	7.73 \pm 1.56 ^A	-2.09 \pm 0.45 ^B	-6.35 \pm 1.65 ^C
	Coffee	8.15 \pm 1.40 ^A	-1.57 \pm 0.57 ^B	-4.24 \pm 0.77 ^C
	Coca-Cola	-0.38 \pm 0.36	0.11 \pm 0.28	0.26 \pm 0.36
	Distilled water	-0.45 \pm 0.20	0.67 \pm 0.25	0.05 \pm 0.47
Filtek Z550	Tea	5.83 \pm 1.27 ^A	-1.71 \pm 0.37 ^B	-5.87 \pm 1.06 ^C
	Coffee	9.27 \pm 1.94 ^A	3.36 \pm 0.58 ^B	8.80 \pm 0.99 ^C
	Coca-Cola	-1.50 \pm 0.79	-0.09 \pm 0.19	-0.81 \pm 0.63
	Distilled water	-0.32 \pm 0.87	-0.10 \pm 0.11	0.01 \pm 0.46

ΔL - (brightness) values • (svjetlina) vrijednosti; Δa - (change along red-green axis) values • (promjena duž crveno-zelene osi) vrijednosti; Δb - (change along yellow-blue axis) values • (promjena duž žuto-plave osi) vrijednosti

^A - statistically significant decrease in the value of L^* parameters • statistički značajno smanjenje vrijednosti L^* parametara

^B - statistically significant increase in the value of a^* parameters • statistički značajno povećanje vrijednosti a^* parametara

^C - statistically significant increase in the value of b^* parameters • statistički značajno povećanje vrijednosti b^* parametara

age interaction was significant for all three color dimensions of both materials (all $p < .001$). Post hoc comparisons were performed using t-tests with Bonferroni correction of p-value ($p = 0.05/3 = 0.017$). On day 30, the color shifts at both tested materials were significant along all three L^* , a^* , and b^* axes in coffee and tea ($p < 0.05$). A statistically significant decrease in L^* values (microhybrid: $p < 0.001$ for both tea and coffee; nanocomposite: $p = 0.001$ for tea, and $p < 0.001$ for coffee) pointed to a brightness shift along the white-black axis toward darker. A statistically significant increase in a^* values (microhybrid: $p < 0.001$ for tea, and $p = 0.004$ for coffee; nanocomposite: $p < 0.001$ for both tea and coffee) along the red-green axis indicates that samples immersed in coffee and tea became redder, whereas a significant increase in b^* values (microhybrid: $p = 0.001$ for tea, and $p < 0.001$ for coffee; nanocomposite: $p < 0.001$ for both tea and coffee) for tested materials along the yellow-blue axis indicated a shift towards yellow. An increase in L^* , a^* , and b^* for samples immersed in Coca-Cola has not pointed to any statistical differences in both tested materials (all $p \geq 0.05$).

The efficiency of bleaching in reducing external discoloration from the composite surface (ΔE_4), showed the difference between baseline ΔE values (before discoloration by standing in beverages) and the values after application of 16% CP (Table 3.). After bleaching treatment, ΔE_4 values decreased within acceptable values ($\Delta E_4 < 3.48$) in both tested materials. Factorial ANOVA with Material (microhybrid and nanocomposite) and Beverage (tea, coffee, Coca-Cola) as between groups factors revealed significant differences in ΔE_4 values between materials ($p = 0.03$), with bigger color changes with the nanomaterial Z550 than the microhybrid Z250 after application of 16% CP. A more considerable improvement in color change was observed in the nanocomposite.

Discussion

Aesthetic composite restorations are ordinarily exposed to food and drink effects in the oral environment, resulting in external discoloration. This research was conducted to investigate the selective effect of three heavily consumed beverages worldwide on the color of two different composite materials: coffee, tea, and carbonated drink (Coca-Cola).

Discoloration can be quantified subjectively by visual comparison of color differences or an objective measurement using an instrument. This study used a portable, wireless digital spectrophotometer, with the white-colored plate for the background to determine CIEL*a*b color coordinates. There are several color systems in use, however, the CIEL*a*b* color system has been extensively used in dental research (1, 11, 12). This color-difference formula provided data on overall color change, values of the change across the color coordinates, and also enabled comparisons with previous studies.

In this study, immersion in coffee and tea resulted in unacceptable color alteration of both composite materials. Tested composites showed clinically unacceptable color change after immersion in tea and coffee already after being immersed for seven days (Table 3.). Over time, discoloration progressively increased reaching the highest values at day 30,

bila je značajna za sve tri dimenzije boja obaju materijala (svi $p < .001$). Post hoc usporedbe provedene su 30. dana s pomoću t-testova s Bonferronijevom korekcijom p-vrijednosti ($p = 0,05/3 = 0,017$) te su promjene boje na oba testirana materijala bile su značajne duž sve tri L^* , a^* i b^* osi u kavi i čaju ($p < 0,05$). Statistički značajno smanjenje vrijednosti L^* (mikrohibrid: $p < 0,001$ i za čaj i za kavu; nanokompozit: $p = 0,001$ za čaj i $p < 0,001$ za kavu) upućuje na pomak svjetline duž bijelo-crne osi prema tamnijoj. Statistički značajno povećanje vrijednosti a^* (mikrohibrid: $p < 0,001$ za čaj i $p = 0,004$ za kavu; nanokompozit: $p < 0,001$ i za čaj i za kavu) duž crveno-zelene osi pokazuje da su uzorci uronjeni u kavu i čaj postali crveniji, a značajno povećanje vrijednosti b^* (mikrohibrid: $p = 0,001$ za čaj i $p < 0,001$ za kavu; nanokompozit: $p < 0,001$ i za čaj i za kavu) za testirane materijale duž žuto-plave osi upućuje na pomak prema žutoj boji. Povećanje L^* , a^* i b^* za uzorke uronjene u Coca-Colu nije pokazivalo nikakvu statističku razliku u oba testirana materijala (svi $p \geq 0,05$).

Učinkovitost izbjeljivanja u smanjenju vanjske promjene boje s površine kompozita (ΔE_4), pokazala je razliku između osnovnih vrijednosti ΔE (prije promjene boje stajanjem u napitcima) i vrijednosti nakon primjene 16-postotnoga CP-a (tablica 3.). Poslije tretmana izbjeljivanja vrijednosti ΔE_4 smanjile su na prihvatljivije vrijednosti ($\Delta E_4 < 3,48$) u oba ispitana materijala. Faktorijska ANOVA s materijalom (mikrohibrid i nanokompozit) i pićem (čaj, kava, Coca-Cola) među skupinama otkrila je značajne razlike u vrijednostima ΔE_4 između materijala ($p = 0,03$), s većim promjenama boje kod nanomaterijala Z550 nego kod mikrohibrida Z250 nakon primjene 16-postotnoga CP-a. Značajnije poboljšanje promjene boje uočeno je u nanokompozitu.

Rasprava

Estetski kompozitni nadomjestci obično su u oralnom okruženju izloženi djelovanju hrane i pića, što rezultira vanjskom diskoloracijom. Ovo istraživanje provedeno je da bi se istražio selektivni učinak triju napitaka koji se uvelike konzumiraju diljem svijeta na boju dvaju različitih kompozitnih materijala: kave, čaja i gaziranoga pića (Coca-Cola).

Promjena boje može se kvantificirati subjektivno vizualnom usporedbom razlika u boji ili objektivnim mjerenjem instrumentima. U ovoj studiji korišten je prijenosni, bežični digitalni spektrofotometar s bijelom pločom kao pozadinom za određivanje CIEL*a*b koordinata boje. U upotrebi je nekoliko sustava boja, no CIEL*a*b* sustav boja najčešće se koristi u stomatološkim istraživanjima (1, 11, 12). Ova formula za razliku u boji dala je podatke o ukupnoj promjeni boje i vrijednosti promjene u koordinatama boja, a također je omogućila usporedbe s već objavljenim studijama.

U ovoj je studiji uranjanje u kavu i čaj rezultiralo neprihvatljivom promjenom boje obaju kompozitnih materijala. Ispitani kompoziti pokazali su klinički neprihvatljivu promjenu boje nakon uranjanja u čaj i kavu već poslije sedam dana (tablica 3.). Tijekom vremena se promjena boje progresivno povećavala te je postigla najviše vrijednosti tridesetoga

as demonstrated previously (11, 14). Lower color stability in coffee and tea groups is consistent with previous studies (1, 15, 16, 17). Filtek Z250 showed a higher stainability in tea ($\Delta E_3 = 10.23 \pm$) and coffee ($\Delta E_3 = 9.36 \pm$), while the least color change ($\Delta E_3 = 0.71$) was observed in Coca-Cola. After immersion, nanohybrid composite Z550 displayed color changes of $\Delta E_3 = 8.46 \pm$ (tea), $13.23 \pm$ (coffee) and $1.80 \pm$ (Coca-Cola). Thus, the first part of the study hypothesis was partially confirmed as Coca-Cola color change values were found to be visually undetectable, while coffee and tea produced color changes beyond the acceptability threshold for both materials tested.

Discoloration of samples immersed in coffee and tea is considered to be primarily superficial as a result of surface adsorption of colorants (14) or can be attributed to water absorption with pigments in the resin matrix (1) due to superficial degradation (8). According to Ferracane, the absorption of solvents begins immediately and peaks in one or two months when the polymer network is completely saturated (19). A rapid excretion of unreacted monomers from composite takes place in an aqueous environment. Water molecules enter the composite while unreacted matrix monomers and ions from the filler and activator emerge. Consequently, the superficial resin matrix is softened and discoloration resistance is ultimately reduced (9).

Discoloration assessment in this study was performed after seven, 14, and 30 days. This measurement schedule proved to be rational because the greatest color change occurred in the first seven days of immersion (Table 3.). Based on the assumption that a vast majority of unreacted monomers elute from composite during this period, a residual free space is occupied by water.

During this process of water sorption into the composite resin, the liquid is the carrier of the colorant through the diffusion process (20). Coffee has a strong discoloration effect on teeth and dental composites (10, 14). During coffee roasting, brown-colored high-molecular-weight nitrogenous compounds named melanoidins are produced and they are compounds primarily responsible for this discoloration (17). Other colorants such as tannin or caffeine contribute to staining as they deeply penetrate the composite matrix (14, 21). Furthermore, tea contributes to composite discoloration due to the sorption of tannins (17).

Considering the type of composite material, a significantly higher nanocomposite discoloration was found only in samples immersed in coffee after 30 days ($p < 0.05$). Higher absorption of pigmented beverages in the nanocomposite is consistent with previous studies (9, 22). The difference in discoloration between two composites can be explained by the chemical composition of these materials. This refers to the composition of the organic part and amount of resin matrix since inorganic filler does not absorb water. According to manufacturer's information, Z250 and Z550 have similar matrices in their composition, containing Bis-GMA,

dana, kao što je već pokazano (11, 14). Niža stabilnost boje u skupinama kave i čaja u skladu je s dosadašnjim studijama (1, 15, 16, 17). Filtek Z250 pokazao je veću postojanost u čaju ($\Delta E_3 = 10,23 +$) i kavi ($\Delta E_3 = 9,36 +$), a najmanja promjena boje ($\Delta E_3 = 0,71$) uočena je u Coca-Coli. Nakon uranjanja je nanohibridni kompozit Z550 pokazao promjene boje $\Delta E_3 = 8,46 +$ (čaj), $13,23 +$ (kava) i $1,80 +$ (Coca-Cola). Dakle, prvi dio hipoteze djelomično je potvrđen jer je ustanovljeno da se vrijednosti u promjeni boje koka-kole vizualno ne mogu detektirati, a da su kava i čaj proizveli promjenu boje iznad praga prihvatljivosti za oba ispitana materijala. U prijašnjim istraživanjima zabilježena je veća kompozitna diskoloracija u kavi u usporedbi s čajem (16, 18), a druga su, pak, pokazala suprotno (1, 14). Razlika u rezultatima može se pripisati različitim protokolima uranjanja i testiranim materijalima.

Promjena boje uzoraka uronjenih u kavu i čaj smatra se prvenstveno kao rezultat površinske adsorpcije bojila (14) ili se može pripisati apsorpciji vode s pigmentima u matrici smole (1) zbog površinske degradacije (8). Prema Ferracaneu, apsorpcija otapala počinje odmah i dostiže vrhunac za jedan ili dva mjeseca kada je polimerna mreža potpuno zasićena (19). Brzo izlučivanje nereagiranih monomera iz kompozita događa se u vodenom okruženju. Molekule vode ulaze u kompozit dok iz punila i aktivatora izlaze nereagirani matični monomeri i ioni. Posljedično, površinska smolna matrica omekšana je i otpornost na promjenu boje u konačnici je smanjena (9).

Procjena promjene boje u ovoj studiji provedena je poslije 7, 14 i 30 dana. Takav raspored mjerenja pokazao se racionalnim jer se najveća promjena boje dogodila u prvih sedam dana poslije uranjanja (tablica 3.). Na temelju pretpostavke da velika većina nereagiranih monomera eluira iz kompozita tijekom toga razdoblja, preostali slobodni prostor zauzima voda.

Tijekom toga procesa sorpcije vode u kompozitnu smolu, tekućina je nositelj bojila u procesu difuzije (20). Kava snažno utječe na promjenu boje zuba i dentalnih kompozita (10, 14). Naime, tijekom njezina prženja nastaju smeđe obojeni visokomolekularni dušikovi spojevi pod nazivom melanoidini i oni su uglavnom odgovorni za tu promjenu boje (17). Druge boje, kao što su tanin ili kofein tomu pridonose jer prodiru duboko u kompozitni matriks (14, 21). Nadalje, čaj pridonosi kompozitnoj diskoloraciji zbog sorpcije tanina (17).

S obzirom na vrstu kompozitnoga materijala, značajno veća promjena boje nanokompozita ustanovljena je nakon 30 dana samo u uzorcima uronjenima u kavu ($p < 0,05$). Veća apsorpcija pigmentiranih pića u nanokompozitu u skladu je s već objavljenim studijama (9, 22). Razlika u promjeni boje između dvaju kompozita može se objasniti kemijskim sastavom tih materijala. To se odnosi na sastav organskoga dijela i količinu matriksa smole jer anorgansko punilo ne upija vodu. Prema podacima proizvođača, Z250 i Z550 imaju slične matrice u svojem sastavu koje sadržavaju monomere Bis-GMA, UDMA i Bis-EMA, uz dodatak nanokompozita PEGDMA i TEGDMA. Tekuća kromatografija visoke učinkovitosti (HPLC) potvrdila je da Z250 u svojem sastavu ima količinu hidrofobnoga monomera UDMA u usporedbi s Bis-GMA-

UDMA, and Bis-EMA monomers, with the addition of PEGDMA and TEGDMA to the nanocomposite. High-Performance Liquid Chromatography (HPLC) confirmed that Z250 contains a larger amount of hydrophobic monomer UDMA compared to Bis-GMA and Bis-EMA in its composition (23). Thus, Z250 may be less susceptible to discoloration by the percentage of hydrophobic monomer UDMA in the structure of material. UDMA increases the hydric stability of the composite (15) and demonstrates a lower water sorption and higher resistance to discoloration compared to hydrophilic Bis-GMA (21). Water sorption was attributed to monomers Bis-GMA (21) and TEGDMA in an organic matrix (21, 24). Small hydrophilic molecules of TEGDMA have higher mobility in an aqueous environment; wash out faster than larger, more massive molecules such as Bis-GMA (23). The emptied places of this molecule were occupied by small molecules of water, carrying a pigment. For this reason, any composite must always be adequately polymerized, since higher conversion means less unreacted mobile monomer, less water sorption, and greater color stability (1). Irrespective of the composite resin used, insufficient light-curing of the composite restoration, continued to be a challenge (25). For this reason, the samples in this study were polymerized with a lamp with optimal light intensity confirmed by a radiometer. Several reports have shown that small nanoparticles contribute to a smoother surface and lower discoloration over time due to minimal surface alteration (26). Previously, discoloration of nanomaterials was attributed to the porosity of the glass filler particles (9). The obtained results showed similar color susceptibility of nanocomposite after immersion in beverages compared to conventional microhybrid composite. These results corroborate the findings of a meta-analysis which did not confirm the advantage of nanocomposites in lower staining susceptibility (26).

Previous studies stated that the discoloration of composites might be a consequence of the low pH of beverage (22) causing surface erosion effect and roughening (10). The acidic environment may induce loss of structural ions, which then enable pigments from the solution to penetrate the softened surface. Composite materials tested in this study were immersed in acid solutions of pH values ranging from 2.26 for Coca-Cola to 6.48 for tea (Table 2.). This study showed imperceptible staining for microhybrid composite in Coca-Cola, while nanocomposite in this beverage demonstrated color change beyond the perceptibility threshold (1.74). Minor discoloration in Coca-Cola compared to samples immersed in coffee and tea is in line with the results of previous studies (17, 21). Although this beverage has a highly acidic pH suitable for surface dissolution and polymer softening, low-level discoloration in Coca-Cola solution is attributed to sulfite ammonia caramel (17). Nevertheless, less extensive discoloration may be due to the lack of yellow colorant in this solution (21). The color alteration of tested composites after exposure to deionized water was clinically non-perceptible ($\Delta E < 1$), which is also seen in previous studies (9, 10). The minimal color shift (Table 3) may be due to the water sorption in the organic matrix, as deionized water is colorless (21).

om i Bis-EMA-om (23). Zato Z250 može biti manje osjetljiv na promjenu boje zbog postotka hidrofobnoga monomera UDMA u strukturi materijala. UDMA povećava hidričnu stabilnost kompozita (15) i pokazuje nižu sorpciju vode i veću otpornost na promjenu boje u usporedbi s hidrofilnim Bis-GMA-om (21). Sorpcija vode pripisana je monomerima Bis-GMA (21) i TEGDMA u organskoj matrici (21, 24). Male hidrofilne molekule TEGDMA pokretljivije su u vodenom okružju i ispiru se brže od većih, masivnijih molekula kao što je Bis-GMA (23). Ispražnjena mjesta te molekule zauzele su male molekule vode koje su nosile pigment. Iz tog razloga svaki kompozit uvijek mora biti adekvatno polimeriziran jer veća konverzija znači manje nereagirana monomera, manju sorpciju vode i veću stabilnost boje (1). Bez obzira na korištenu kompozitnu smolu, nedovoljna polimerizacija i dalje je izazov za kompozitne nadomjestke (25). Zbog toga su uzorci u ovom istraživanju polimerizirani svjetiljkom optimalnoga intenziteta potvrđenog radiometrom. U nekoliko izvješća pokazano je da male nanočestice pridonose glatkijoj površini i nižoj diskoloraciji tijekom vremena zbog minimalne promjene površine (26). Prije su se promjene boje nanomaterijala pripisivale poroznosti čestica staklenoga punila (9). Dobiveni rezultati pokazali su sličnu osjetljivost na boju nanokompozita nakon uranjanja u napitke u usporedbi s konvencionalnim mikrohibridnim kompozitima. Ti rezultati potvrđuju nalaze metaanalize u kojoj nije potvrđena prednost nanokompozita u nižoj osjetljivosti na bojenje (26).

U dosadašnjim studijama navedeno je da bi promjena boje kompozita mogla biti posljedica niskoga pH napitka (22), što uzrokuje efekt površinske erozije i hrapavost (10). Kiselina okolina može izazvati gubitak strukturnih iona koji tada omogućuju pigmentima iz otopine da prodru u omekšanu površinu. Kompozitni materijali ispitani u ovoj studiji bili su uronjeni u kisele otopine pH vrijednosti u rasponu od 2,26 za Coca-Colu do 6,48 za čaj (tablica 2.). Ova studija pokazala je neprimjetno bojenje mikrohibridnoga kompozita u kokakoli, dok je nanokompozit u ovom napitku pokazao promjenu boje iznad praga perceptibilnosti (1,74). Manja promjena boje u Coca-Coli, u usporedbi s uzorcima uronjenima u kavu i čaj, u skladu je s rezultatima u dosadašnjim studijama (17, 21). Iako taj napitak ima visok kiseli pH prikladan za površinsko otapanje i omekšavanje polimera, niska promjena boje u otopini Coca-Cole pripisuje se sulfitno-amonijačnoj karameli (17). Ipak, manje opsežna promjena boje može biti i posljedica nedostatka žute boje u otopini (21). Promjena boje ispitanih kompozita nakon izlaganja deioniziranoj vodi bila je klinički neprimjetna ($\Delta E < 1$), što je također istaknuto u već objavljenim studijama (9, 10). Minimalni pomak boje (tablica 3.) može biti posljedica sorpcije vode u organskom matriksu zato što je deionizirana voda bezbojna (21).

Značajna promjena boje kompozita uronjenih u kavu i čaj prikazana je u trima prostornim koordinatama – L^* , a^* , b^* ($p < 0,05$). Za oba ispitana materijala glavni pomak prikazan je u vrijednostima ΔL^* prema crnoj boji, a koordinate a^* i b^* pokazale su značajan odklon na pozitivniju vrijednost (tablica 4.). Posljedično, boja obaju ispitanih materijala u kavi i čaju postala je crvenija, žuća i tamnija. Pozitivne vrijednosti ΔL^* nanokompozita u Coca-Coli upućuju na to da

Significant discoloration of composites immersed in coffee and tea was demonstrated in three spatial coordinates, L^* , a^* , b^* ($p < 0.05$). For both tested materials, the main shift demonstrated in ΔL^* values towards black, while a^* and b^* coordinates showed significant deflection to a more positive value (Table 4.). Consequently, the color of both tested materials in coffee and tea became redder, more yellow, and dark. Positive ΔL^* values of nanocomposites in Coca-Cola indicate that the samples became lighter, which is consistent with the previously reported results (14).

Tooth bleaching has gained popularity in recent years as a non-invasive and affordable method for smile attractiveness enhancement. Previous research reported that every fourth young adult used this method (5). In addition, this study evaluated the suitability of the at-home whitening method in removing discoloration from composite after exposure to stained beverages. Bleaching regained ΔE values within a clinically acceptable threshold ($\Delta E < 3.48$), which is consistent with previous studies (9, 12). Removal of external discoloration from the composite surface using 16% CP proved to be effective on both tested composite materials. Therefore, the second part of the study hypothesis was accepted. Carbamide peroxide decomposes into oxidizing agent hydrogen peroxide that produces very reactive free radicals. These unstable radicals engage in chemical interactions with organic chromophores and oxidize stain molecules. Furthermore, these oxygen species convert the chains within stain into less complex molecules; modify their optical properties resulting in degradation products with lower molecular weights (27). The color has been achieved more efficiently on microhybrid material, likewise in the previous research papers (12). One explanation for this may be the composition, as Z250 contains less resin matrix than nanocomposite. Regarding nanocomposite, it contains a larger amount of resin on the surface; consequently, it is more difficult to remove discoloration by bleaching (12).

It is important to note that this study has certain limitations. Only one shade of composites was evaluated. Moreover, considering a clinical variable, dental restorations are subjected to the effect of the brushing and mastication, saliva components, thermal stress, fluid dynamics, and the adhesion of the biofilm that could also have a role in the process of discoloration. Since this was an *in vitro* study that is unable to simulate complex interactions in the oral cavity, future long-term *in vivo* studies are also needed to assess the color stability of the composites.

Conclusions

In conclusion, this *in vitro* study showed that coffee and green tea caused visually perceptible color changes on the composite resins that continue over time. The resulting color change of the composites presented is due to the chromaticity shift towards yellow, black, and red. Filtek Z250 showed less discoloration, dominantly for samples immersed in coffee. The patients should be informed about the effect of coffee and tea on composite discoloration, and about the cumulative effects on the change of the restoration color.

su uzorci postali svjetliji, što je u skladu s već objavljenim rezultatima (14).

Izbjeljivanje zuba posljednjih je godina postalo popularno kao neinvazivna i pristupačna metoda za povećanje atraktivnosti osmijeha. Prethodno istraživanje pokazalo je da se svaka četvrta mlada odrasla osoba koristila tom metodom (5). Uz to, u ovoj se studiji procjenjivala i prikladnost metode izbjeljivanja kod kuće za uklanjanje diskoloracije s kompozita poslije izlaganja obojenim pićima. Izbjeljivanje je vratilo vrijednosti ΔE unutar klinički prihvatljivoga praga ($\Delta E < 3,48$), što je u skladu s dosadašnjim studijama (9, 12). Uklanjanje vanjske promjene boje s kompozitne površine primjenom 16-postotnoga CP-a pokazalo se učinkovitim pri uporabi obaju ispitanih kompozitnih materijala. Zato je prihvaćen drugi dio hipoteze. Karbamid-peroksid razgrađuje se u vodikov peroksid koji stvara vrlo reaktivne slobodne radikale. Ti nestabilni radikali sudjeluju u kemijskim interakcijama s organskim kromoforima i oksidiraju molekule mrlja. Nadalje, te vrste kisika pretvaraju lance unutar mrlje u manje složene molekule, a modificiraju i njihova optička svojstva, što rezultira proizvodima razgradnje s nižom molekularnom težinom (27). Boja je učinkovitije postignuta na mikrohibridnom materijalu, kao što se ističe i u već objavljenim istraživačkim radovima (12). Jedno od objašnjenja za to može biti sastav jer Z 250 sadržava manje matrice smole od nanokompozita. Kad je riječ o nanokompozitu, on sadržava veću količinu smole na površini, pa je posljedično teže ukloniti diskoloraciju izbjeljivanjem (12).

Važno je istaknuti da ova studija ima određena ograničenja. Ocjenjivana je samo jedna nijansa kompozita. Štoviše, s obzirom na kliničku varijablu, dentalne nadoknade podvrgnute su učinku četkanja i žvakanja, komponenti sline, toplinskome stresu, dinamičnu tekućine i adheziji biofilma koji bi također mogao utjecati na proces diskoloracije. Kako se radilo o studiji *in vitro* koja ne može simulirati složene interakcije u usnoj šupljini, u budućnosti su potrebne dugoročne studije *in vivo* za procjenu stabilnosti boje kompozita.

Zaključci

Zaključno, ova studija *in vitro* pokazala je da kava i zeleni čaj uzrokuju vizualno uočljive promjene boje na kompozitnim smolama koje se nastavljaju tijekom vremena. Promjena boje prikazanih kompozita posljedica je pomaka kromatičnosti prema žutoj, crnoj i crvenoj. Filtek Z 250 pokazao je manje promjene u boji, dominantno za uzorke uronjene u kavu. Pacijente je potrebno obavijestiti o učinku kave i čaja na promjenu boje kompozita te o kumulativnim učincima na promjenu boje restauracije.

Additionally, the bleaching procedure using 16 % CP showed clinically acceptable results as a method for removing external discolorations from the restoration surface. Further research is needed to investigate into the tooth discoloration *in vivo*.

Conflict of interest

The authors declare that they have no conflict of interest.

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Dodatno, postupak izbjeljivanja korištenjem 16-postotnoga CP-a pokazao je klinički prihvatljive rezultate kao metoda za uklanjanje vanjskih diskoloracija s površine restauracije. Potrebna su daljnja istraživanja kako bi se istražila promjena boje zuba *in vivo*.

Sukob interesa

Autori nisu bili u sukobu interesa.

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Doprinosi autora: S. K., M. A. – dizajnirali studiju; S. K., A. DŽ., I. T. – obavili pokus; A. K., L. H. B., A. G. G. – analizirali podatke; M. A., A. K., L. H. B. i A. G. G. – pretraživali literaturu; S. K., I. T., A. DŽ. – napisali rad, uz doprinose svih autora.

Sažetak

Cilj: Željela se usporediti stabilnost boje dvaju različitih svjetlosno polimeriziranih kompozita nakon uranjanja u tri tekućine i učinkovitost 16-postotnoga karbamid-peroksida (CP) u uklanjanju diskoloracije. **Materijali i metode:** Stabilnost boje mikrohibrida (Z250, 3 M ESPE) i nanokompozita (Z550, 3 M ESPE) procijenjena je nakon uranjanja u instant kavu, čaj i Coca-Colu te u deioniziranu vodu kao kontrolnu skupinu ($n = 5$). Uzorci su bili u tekućinama na temperaturi od 37 °C četiri sata na dan tijekom 30 dana. Nadalje, sljedećih 14 dana primijenjen je 16-postotni CP simulirajući noćno izbjeljivanje. Za mjerenje boja korišten je digitalni spektrofotometar na temelju koordinata boja CIEL*a*b*. Promjene boje (ΔE) mjerene su na početku postupka izbjeljivanja zuba, nakon uranjanja u napitke i poslije završenog postupka. Za statističku procjenu korišteni su mješoviti i faktorijalni ANOVA testovi praćeni Bonferronijevim post-hoc testom ($p \leq 0,05$). **Rezultati:** Testirani kompoziti smole pokazali su promjenu boje iznad praga prihvatljivosti ($\Delta E^* > 3,48$) poslije uranjanja u kavu i čaj. Nanokompozit je znatno povećao promjenu boje u kavi poslije 30 dana ($p < 0,05$). Boja obaju materijala značajno se promijenila ($p < 0,05$) duž svih triju L*a*b* osi u kavi i čaju u tamnijem, žutu i crvenu. Izbjeljivanje 16-postotnim CP-om bilo je učinkovito u uklanjanju vanjske diskoloracije u oba ispitivana kompozitna materijala. **Zaključak:** Kava i čaj prouzročili su klinički uočljive promjene boje u ispitanim dentalnim kompozitima s kumulativnim učincima. Izbjeljivanje je učinkovita metoda za uklanjanje diskoloracije s površine kompozitnih nadomjestaka.

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References

- Garoushi S, Lassila L, Hatem M, Shembesh M, Baady L, Salim Z, Vallittu P. Influence of staining solutions and whitening procedures on discoloration of hybrid composite resins. *Acta Odontol Scand.* 2013;71(1):144-150.
- Schroeder T, da Silva PB, Basso GR, Franco MC, Maske TT, Cenci MS. Factors affecting the color stability and staining of esthetic restorations. *Odontology.* 2019;107(4):507-512.
- De Goes MF, Murillo-Gómez F, Lima RBW, Sartori CG. Effect of air polishing procedure with glycine and bicarbonate powders on glass ceramic and composite resin surfaces. *Int J Dent Oral Health.* 2016;2(5):1-6.
- Santak V, Zaplotnik R, Milosevic S, Klaric E, Tarle Z. Atmospheric Pressure Plasma Jet as an Accelerator of Tooth Bleaching. *Acta Stomatol Croat.* 2014;48(4):268-78.
- Diklić D, Sever EK, Galić N, Spajić J, Prskalo K. Attitudes of Students of Different Schools of University of Zagreb on Tooth Bleaching. *Acta Stomatol Croat.* 2016;50(4):301-309.
- Ning K, Bronkhorst E, Bremers A, Bronkhorst H, van der Meer W, Yang F, Leeuwenburgh S, Loomans B. Wear behavior of a micro-hybrid composite vs. a nanocomposite in the treatment of severe tooth wear patients: A 5-year clinical study. *Dent Mater.* 2021;37(12):1819-1827.
- Omanović-Miklićanin E, Badnjević A, Kazlagić A, Hajlovac M. Nanocomposites: a brief review. *Health Technol.* 2020;10(1):51-59.
- Ertas E, Güler AU, Yücel AC, Köprülü H, Güler E. Color Stability of Resin Composites after Immersion in Different Drinks. *Dent Mater J.* 2006;25(2):371-376.
- Villalta P, Lu H, Okte Z, Garcia-Godoy F, Powers JM. Effects of staining and bleaching on color change of dental composite resins. *J Prosthet Dent.* 2006;95(2):137-142.
- Ceci M, Viola M, Rattalino D, Beltrami R, Colombo M, Poggio C. Discoloration of different esthetic restorative materials: A spectrophotometric evaluation. *Eur J Dent.* 2017;11(2):149-156.

11. Alkhadim YK, Hulbah MJ, Nassar HM. Color Shift, Color Stability, and Post-Polishing Surface Roughness of Esthetic Resin Composites. *Materials (Basel)*. 2020;13(6):1376.
12. Al-Nahedh HN, Awliya WY. The effectiveness of four methods for stain removal from direct resin-based composite restorative materials. *Saudi Dent J*. 2013;25(2):61-67.
13. Ghinea R, Pérez MM, Herrera LJ, Rivas MJ, Yebra A, Paravina RD. Color difference thresholds in dental ceramics. *J Dent*. 2010;38 Suppl 2:e57-64.
14. S. Madhyastha P, G. Naik D, Kotian R, Srikant N, M. R. Bhat K. Effect of Staining Solutions on Color Stability of Silorane and Methacrylate Restorative Material. *Int J Biomed Sci*. 2015;11(1):29-34.
15. Ozera EH, Pascon FM, Correr AB, Puppin-Rontani RM, Castilho AR, Correr-Sobrinho L, Paula AB. Color Stability and Gloss of Esthetic Restorative Materials after Chemical Challenges. *Braz Dent J*. 2019;30(1):52-57.
16. Reddy PS, Tejaswi KL, Shetty S, Annapoorna BM, Pujari SC, Thipswamy HM. Effects of commonly consumed beverages on surface roughness and color stability of the nano, microhybrid and hybrid composite resins: an in vitro study. *J Contemp Dent Pract*. 2013;14(4):718-723.
17. Manojlovic D, Lenhardt L, Milićević B, Antonov M, Miletic, V, Dramićanin M. D. Evaluation of Staining-Dependent Colour Changes in Resin Composites Using Principal Component Analysis. *Sci Rep*. 2015;5:14638.
18. Duc O, Di Bella E, Krejci I, Betrisey E, Abdelaziz M, Ardu S. Staining susceptibility of resin composite materials. *Am J Dent*. 2019;32(1):39-42.
19. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. *Dental Materials*. 2006;22(3):211-22.
20. Schulze KA, Marshall SJ, Gansky SA, Marshall GW. Color stability and hardness in dental composites after accelerated aging. *Dent Mater*. 2003;19(7):612-619.
21. Malekipour MR, Sharafi A, Kazemi S, Khazaei S, Shirani F. Comparison of color stability of a composite resin in different color media. *Dent Res J (Isfahan)*. 2012;9(4):441-446.
22. Ardu S, Duc O, Di Bella E, Krejci I, Daher R. Color stability of different composite resins after polishing. *Odontology*. 2018;106(3):328-333.
23. Sideridou ID, Achilias SD. Elution Study of Unreacted Bis-GMA, TEGDMA, UDMA, and Bis-EMA from Light-Cured Dental Resins and Resin Composites Using HPLC. *J Biomed Mater Res Part B: Appl Biomater*. 2005;74(1):617-626.
24. Alberton Da Silva V, Alberton Da Silva S, Pecho OE, Bacchi A. Influence of composite type and light irradiance on color stability after immersion in different beverages. *J Esthet Restor Dent*. 2018;30(5):390-396.
25. Sartori N, Knezevic A, Peruchi LD, Phark JH, Duarte S Jr.. Effects of Light Attenuation through Dental Tissues on Cure Depth of Composite Resins. *Acta Stomatol Croat*. 2019;53(2):95-105.
26. Maran BM, de Geus JL, Gutiérrez MF, Heintze S, Tardem C, Barceiro MO, Reis A, Loguercio AD. Nanofilled/nanohybrid and hybrid resin-based composite in patients with direct restorations in posterior teeth: A systematic review and meta-analysis. *J Dent*. 2020;99:103407.
27. Kwon SR, Wertz PW. Review of the Mechanism of Tooth Whitening. *J Esthet Restor Dent*. 2015;27(5):240-257.