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Color stability of esthetic restorative materials: a spectrophotometric analysis

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ABSTRACT

Objective: The aim of this *in vitro* study was to evaluate the color stability of different restorative materials (one microfilled composite, one nanofilled composite, one nanohybrid composite and one Ormocer-based composite) after exposure to different staining solutions (coffee, coca-cola and red wine).

Material and methods: All materials were polymerized into silicon rings (2 mm ×6 mm ×8 mm) to obtain specimens identical in size. Thirty cylindrical specimens of each material were prepared. They were immersed in staining solutions over a 28-day test period. A colorimetric evaluation according to the CIE $L^*a^*b^*$ system was performed by a blind trained operator at 7, 14, 21, 28 days of the staining process. The Shapiro–Wilk test and Kruskal–Wallis ANOVA were applied to assess significant differences among restorative materials. The paired *t*-test was applied to test which CIE $L^*a^*b^*$ parameters significantly changed after immersion in staining solutions.

Results: All restorative materials showed clinically perceptible color differences after immersion in coffee. L^* and b^* values showed the highest variability. Coca cola and red wine did not influence the color stability for all restorative materials except for Filtek Supreme XTE.

Conclusions: Coffee caused a significant color change in all types of tested composite resins. Filtek Supreme XTE demonstrated alone a staining susceptibility to red wine; no other significant differences among the materials were demonstrated. Long-term exposure to some food dyes (coffee in particular) can significantly affect the color stability of modern esthetic restorative materials regardless of materials' different composition.

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Introduction

A crucial property of esthetic restorative materials is their long-term color stability and an unacceptable color match is a primary reason for the replacement of composite resin restoration.[1] As possessing good esthetic properties, resin composite materials are widely used in clinical practice. Any esthetic restorative material should duplicate the appearance of a natural tooth in color, and the success of an esthetic restoration depends first on the color match and then on the color stability of the material.[2] However, a major disadvantage is the discoloration of the restorative material after prolonged exposure to the oral environment.[3]

Three types of discolorations are generally described: external discoloration due to the accumulation of plaque and surface stains (extrinsic stain), surface or subsurface color alteration implying superficial degradation or slight penetration and reaction of staining agents within the superficial layer of composite resins (absorption) and body or intrinsic discoloration due to physicochemical reactions in the deeper portion of the restoration.[4] Discoloration of composite resin materials can be caused by intrinsic and/or extrinsic factors.[5] Extrinsic discoloration is mainly caused by colorants contained in beverages and foods. Numerous studies *in vitro* have demonstrated that common drinks and food ingredients could cause significant change in surface color.[6,7]

The structure of the resin matrix and characteristics of the filler particles directly affect the surface smoothness and susceptibility to extrinsic staining.[8,9] The staining susceptibility may be explained by the nature of the resin matrix, and also may be correlated with the dimension of the filler particles.[10,11] The resin plays a major role in the color stability of

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esthetic restorative materials. The affinity of a resin for stains is modulated by its conversion rate and its chemical characteristics, water sorption rate being particularly important.[12] According to some authors, the small dimension of the particles of nanofilled composite resin can permit low-staining susceptibility.[13]

Resin composites have been classified according to various characteristics, such as size, content and filler type, and the physical and mechanical properties of the materials.[14] Nanotechnology, known as molecular nanotechnology, is the production of functional materials and structures, at a range of 0.1–100 nm, by various physical and chemical methods.[15] A nanohybrid is a hybrid resin composite with nanofiller in a prepolymerized filler form, whereas nanofill is a composite resin that is composed of both nanomers and nanoclusters.[16]

The term Ormocer is an abbreviation for Organically Modified Ceramics.[13] Ormocers were initially used together with dimethacrylates, but a recent material formulated with a pure-ormocer-based resin matrix has been developed.[17] An ormocer is a hybrid molecular structure. This combines organic and inorganic components at nanoscopic scale through the sol-gel method, and the main characteristic of this type of material is the incorporation of organic groups linked to the inorganic backbone.[18] Ormocer materials contain inorganic–organic co-polymers in addition to the inorganic silanated filler particles. Ormocers are described as three dimensionally cross-linked copolymers. The ormocer matrix is a polymer even prior to light curing. It consists of ceramic polysiloxane, which has low shrinkage as against the organic dimethacrylate monomer matrix seen in composites.[19]

The aim of this *in vitro* study was to evaluate and compare the color stability of different esthetic restorative materials (one microfilled hybrid resin composite, one nanofilled composite, one nanoybrid composite and one Ormocer-based composite) after exposure to different staining solutions (coffee, coca-cola and red wine). The null hypothesis is that esthetic restorative materials do not change color clinically when staining agents are routinely applied.

Materials and methods

Specimens' preparation

One microfilled composite (Gradia Direct), one nanofilled composite (Filtek Supreme XTE), one nanoybrid composite (Ceram-X Duo) and one nanoybrid Ormocer-based composite (Admira Fusion) were evaluated in this study (Table 1). For each brand, the A3 Vita shade was selected. All materials were polymerized according to manufacturers' instructions into silicon rings (height 2 mm; internal diameter 6 mm; external diameter 8 mm) to obtain specimens identical in size. Gradia Direct, Filtek Supreme XTE and Admira Fusion were polymerized for 20 s, Ceram-X Duo for 30 s with the light curing unit Celalux II (Voco, Cuxhaven, Germany). Cavities of these rings were slightly overfilled with material, covered with

Table 1. Esthetic restorative materials tested in this study.

Material	Composition	Type	Filler content % (w/w) (v/v)	Lot #
GC Gradia Direct (GC Corporation, Tokyo, Japan)	Matrix: urethane dimethacrylate(UDMA), dimethacrylate camphorquinone Filler: fluoro-alumino-silicate glass silicapowder	microfilled composite	73 (w/w) 64 (v/v)	140127A
Filtek Supreme XTE (3M ESPE, St Paul, MN, USA)	Matrix:Bis-phenol Adiglycidylmethacrylate (Bis-GMA), triethylene glycoldimethacrylate (TEGDMA), UDMA, bisphenol A polyethyleneglycoldietherdimethacylate Filler: silicanano fillers (5–75 nm) zirconia/silica nanoclusters (0.6–1.4 μm)	nanofilled composite	78.5 (w/w) 59 (v/v)	N595296
Ceram-X Duo (Dentsply De Trey, Konstanz, Germany)	Matrix: methacrylate-modified polysiloxane, dimethacrylate resin, fluorescent pigment, UV stabilizer, stabilizer, camphorquinone, ethyl-4(dimethylamino) benzoate, iron oxide pigments, titanium oxide pigments, aluminum sulfosilicate pigments Filler: barium-aluminium-borosilicate glass (1.1–1.5 μm), methacrylate-functionalized silicon dioxide nano filler (10 nm)	Nanoybrid composite	76(w/w) 67 (v/v)	1407000927
Admira Fusion (Voco, Cuxhaven, Germany)	Matrix: resine Ormocer Filler: silicon oxide nano filler, glass ceramics filler (1μm)	Nanoybrid Ormocer-based composite	84 (w/w) 69 (v/v)	1508065

Mylar strip (Henry Schein, Melville, NY) and pressed between glass plates. One light polymerization mode was used for each material – standard: 1000 mW/cm^2. The intensity of the light was verified with a radiometer (SDS Kerr, Orange, CA). The light was placed perpendicular to the specimen surface, at distance of 1.5 mm. The upper surface of each specimen was then polished with fine and superfine polishing disks (Sof-Lex Pop On; 3M ESPE, St. Paul, MN) to simulate clinical conditions. Thirty cylindrical specimens of each material were prepared in this manner, for a total of 120 specimens. After polymerization and during the experimentation, the specimens were stored in distilled water at 37 °C.

Staining process

The staining solutions used were coffee (Nescafe Classic, Nestle, Vevey, Switzerland), Coca-Cola (Coca-Cola Company, Milan, Italy), red wine (Bonarda Tenuta Casa Re, Montecalvo Versiggia, Pavia, Italy) and physiological solution (solution 0.9% NaCl, negative control).

The specimens were immersed in staining solutions at room temperature over a 28-day test period. Solutions were changed daily and put in vials with cover that prevent evaporation of staining solutions. Spectrophotometric analysis was made after 7, 14, 21 and 28 days at the beginning of the experimentation. Before each measurement, the specimens were gently rinsed for 5 s with distilled water and air-dried.

Color testing

A colorimetric evaluation according to the CIE $L^*a^*b^*$ system was performed by a blind trained operator at five experimental periods: immediately after light-polymerization and at 7, 14, 21, 28 days of the staining process. The control samples have not been subjected to the staining process. Color of the specimens was measured with a spectrophotometer (SP820λ; Techkon Gmbh, Konig-Stein, Germany) against a black background in order to simulate the absence of light in the mouth against a white background. All specimens were chromatically measured out of the staining solution four times (two per surface) and the average values were calculated; then each color parameter for each specimens of the same shade was averaged. The CIE 1976 L^* a^* b^* color system is used for the determination of color differences. [20,21] The L^* value refers to 'lightness;' the higher is the L value, and it is the lightness (a value of 100 corresponds to perfect white and that of zero to black). CIE L^* a^* b^* values are called the 'chromaticity coordinates;' 'a^*' shows red color on positive values and green color on negative values; 'b^*' shows yellow color on positive values and blue color on negative values. [22] The total color differences (ΔEab^*) were calculated as follows:

$$\Delta Eab^* = [(\Delta L^*)2 + (\Delta a^*)2 + (\Delta b^*)2]1/2$$

where L^* is lightness, a^* is green-red component ($-a^*$ = green; $+a^*$ = red) and b^* is blue-yellow component ($-b^*$ = blue; $+b^*$ = yellow). A value of $\Delta Eab^* < 3.3$ was considered clinically acceptable in the present study.[1,22,23] Color measurements of the experimental groups were compared with those of the control group.

Statistical analysis

Differences in color change by the immersion protocols were calculated and a statistical analysis was performed using statistical software (Stata 12; College Station, TX). Descriptive statistics that included mean, standard deviation, median and minimum and maximum values were calculated for each CIE $L^*a^*b^*$ parameter. The Shapiro–Wilk test was applied to assess the normality of the distribution of each CIE $L^*a^*b^*$ parameter. A two-way nonparametric analysis of variance test (Kruskal–Wallis' ANOVA) was applied to determine whether significant differences existed among the groups. The Mann–Whitney test was used as *post-hoc*. A preset alpha level of (0).05 was used for all statistical analyses. Adjunctive analysis with paired *t*–*t* was applied to each CIE $L^*a^*b^*$ parameter when restorative materials were immersed in coffee.

Results

Results are summarized in Table 2. Shapiro–Wilk's test confirmed that the values were not normally

Table 2. Mean ± standard deviation of ΔE calculated from mean ΔL^*, Δa^* Δb^* values for each composite material referring to initial values. *Clinically perceptible color differences.

	Control	Coffee	Coca-Cola	Red wine
Gradia Direct	0.4289 ± 0.0992	4.0585 ± 0.1145*	0.6966 ± 0.0850	2.0035 ± 0.1046
Filtek Supreme XTE	1.4922 ± 0.0879	7.2975 ± 0.0941*	0.2777 ± 0.0276	7.1652 ± 0.1034*
Ceram-X Duo	0.6944 ± 0.0577	3.6030 ± 0.0538*	0.7453 ± 0.0671	2.1431 ± 0.0773
Admira Fusion	0.6343 ± 0.0834	4.8462 ± 0.0991*	0.7680 ± 0.0693	1.3566 ± 0.0667

Table 3. Means ± standard deviation of CIE $L^*a^*b^*$ values measured before and after immersion in coffee. L^* and b^* values are significantly different for all restorative materials when comparing before and after immersion in coffee.

	CIE $L^*a^*b^*$ values					
	Before			After		
	L^*	a^*	b^*	L^*	a^*	b^*
Gradia	52.57 ± 1.25	0.72 ± 0.11	9.23 ± 0.79	49.36 ± 1.54	0.97 ± 0.18	11.70 ± 0.85
Filtek	61.27 ± 0.68	0.18 ± 0.09	12.22 ± 0.61	58.54 ± 0.95	0.11 ± 0.14	19.34 ± 1.19
CeramX	49.16 ± 0.92	2.21 ± 0.15	13.89 ± 0.83	46.28 ± 0.90	2.44 ± 0.18	16.04 ± 0.59
Admira	52.76 ± 0.69	−0.48 ± 0.07	7.81 ± 0.57	50.15 ± 0.52	0.10 ± 0.23	12.58 ± 1.16

distributed. Kruskal–Wallis' ANOVA found significant differences among the various groups. The *post hoc* Mann–Whitney test showed that Ceram-X Duo and Admira Fusion maintained similar values in control group and after immersion in Coca-Cola ($p > 0.05$) while different staining was registered after immersion in coffee and red wine ($p < 0.05$). Admira Fusion showed higher values after immersion in coffee and lower values after immersion in red wine when compared with Ceram-X Duo ($p < 0.05$). Clinically perceptible color differences were registered for both the materials after immersion in coffee ($\Delta E > 3.3$). Filtek Supreme XTE showed highest ΔE values for all groups except after immersion in Coca-Cola and for Control group. Gradia Direct showed a lower staining in control group and after immersion in Coca-Cola group when compared with remnant restorative materials. Clinically perceptible color difference for Gradia Direct was registered only after immersion in coffee. In Table 3, we reported mean values for each parameter of the CIE $L^*a^*b^*$ formula for each composite material before and after 28 days of immersion in coffee. Paired *t*-test confirmed that for each material one or more parameters significantly changed after immersion in coffee ($p < 0.05$). L and b parameters significantly changed for all restorative materials ($p < 0.05$).

Discussion

The problem of surface discoloration of modern composites was investigated in this study considering their susceptibility to being stained by normal diet and food dyes. The color stability of a microfilled, a nanofilled, a nanohybrid and an ormocer composite was assessed after exposure to various drinks, which are commonly consumed by the general population. The null hypothesis of the study was partially rejected because coffee presented a significant staining capacity regardless of the composite resin. The results of the present investigation showed significant differences in color stability among the different groups.

Color change can be assessed both visually and by specific instruments.[24] The methodology used in the present study was in accordance with previous studies that used spectro-photometry and the CIE $L^*a^*b^*$ coordinate system, which is a recommended method for dental purposes.[25] The CIE $L^*a^*b^*$ coordinate system was chosen to evaluate the color variation (DE) because it is well suited for the determination of small color changes and has advantages such as repeatability, sensitivity, and objectivity.[25] Several authors have reported that ΔE values ranging from 1 to 3 are perceptible to the naked eye [26] and ΔE values greater than 3.3 are clinically unacceptable.[23,27]

Different *in vitro* studies have demonstrated that common food substances, such as coffee, cola or red wine,[28] but also tea, fruit juices, soy sauce, mustard and ketchup could cause significant change in surface color of composite resin materials.[29,30] In this study, control group (physiological solution) maintain the same color, with no discoloration. Contrariwise, all restorative materials showed clinically perceptible color differences after immersion in coffee. Coca-Cola and red wine did not have significant influence on the color stability of the restorative materials except for Filtek Supreme XTE exposed to red wine. Our results are in accordance with other studies which demonstrated that certain substances (e.g. coffee) may cause more severe staining than other (e.g. cola) though they have similar color parameters.[8,24] Khokhar et al. [31] also showed a strong staining of composites after exposure to coffee for 48 h. Coffee has shown a high capacity of staining anterior composite resins and natural teeth.[32] According to Guler et al. [28], the average time for consumption of 1 cup of coffee is 15 min, and among coffee drinkers, the average consumption is 3.2 cups per day. Therefore, 15 days of storage simulated consumption of the drink over 1 year. Cola drink does not appear to be strongly implicated in color change of composites, despite the presence of phosphoric acid.[33] Acids behave differently in promoting dissolution and hence in eroding the materials. In addition, the presence of phosphate ions in Coca-Cola may suppress the dissolution since these ions have been shown to reduce the dissolution rate of calcium phosphate from the tooth.[33]

The staining susceptibility of composite resins can explain the staining capacity of coffee. This capacity might be attributed to the degree of water sorption and the hydrophilicity of the matrix resin.[34] Composite resins can absorb water and are also able to absorb other fluids with pigments, which results in discoloration. It is assumed that water acts as a vehicle for stain penetration into the resin matrix.[35] Water sorption occurs mainly as direct absorption in the resin matrix. The glass filler particles do not absorb water. Therefore, greater amount of resin matrix results in greater water sorption. It has been reported that composite resins with a lower amount of inorganic fillers presented more color change because the greater resin matrix volume allows greater water sorption.[36] Further water sorption may decrease the durability of composite resins by expanding and plasticizing the organic matrix as well as by hydrolyzing the silane.[5]

It has been reported that water uptake in Bis-GMA-based composite resins increased from 3 to 6% as the proportion of TEGDMA increased from 0 to 1% 811). UDMA seems to be more stain-resistant than Bis-GMA.[37] Under normal curing conditions, UDMA-based composite resin presented lower water sorption and higher color stability than having other dimethacrylates in their resin matrix.[37] Higher monomer conversion indicates low amount of unreacted monomer, lower solubility and higher color stability.[38] Degree of conversion of composite resins light-cured under identical conditions ranges according to concentration of some monomers. Some monomers present lower degree of conversion than others in the following order: Bis-GMA < Bis-EMA < UDMA < TEGDMA.[38] In the present study, these differences were not clearly evident. However, all composites presented color alteration when immersed in coffee. No significant difference was noted between Gradia Direct, Ceram-X Duo and Admira Fusion; while Filtek Supreme XTE reported significant color change also after immersion in red wine. Compared to the other tested materials, Filtek Supreme XTE demonstrated the highest ΔE values except after immersion in Coca-Cola and for Control group. On the basis of the above considerations, this finding can be explained by the presence of Bis-GMA monomer and by the lower filler content (59% v/v) in Filtek Supreme XTE chemical composition.

Recently, manufacturers are producing composites with smaller filler particles in an attempt to produce materials with similar surface smoothness as that of dental enamel. The lower particle size and better distribution within the resin matrix produce smoother surfaces.[39] Although some studies have shown that the small dimension of the particles of nanofilled composite resin permits low-staining susceptibility [13]; other researchers reported that increased particle size resulted in less color change due to a decrease in the proportion of organic filler matrix.[40] This study seems to support the second thesis. In fact, the nanofilled composite Filtek Supreme XTE showed the highest ΔE values after immersion in coffee and wine, while the microfilled composite Gradia Direct showed lower values in control group and after immersion in Coca-Cola, when compared with remnant restorative materials. Clinically perceptible color difference for Gradia Direct was registered only after immersion in coffee. These observations are in accordance with a study by Villalta et al.,[1] which demonstrated that nanofilled composite resins absorb stains more easily than microfilled ones. Lee et al. [23] obtained similar results, concluding that the smoothest surfaces were not necessarily the most stain resistant, and staining ability was influenced by each composite monomer and filler composition.

Ormocer materials contain inorganic–organic copolymers in addition to the inorganic silanated filler particles. It is synthesized through a solution and gelation process (sol-gel process) from multifunctional urethane and thioether(meth)acrylate alkoxysilanes. [18] To the polysiloxane chains in ormocer, polymerizable side chains are added to react during curing and form the setting matrix. These inorganic molecules explain the material's lower volumetric shrinkage. Incorporation of filler particles decreases volumetric shrinkage from 2–8% when it has no fillers to 1–3% when fillers are incorporated. The filler particles are 1–1.5 μm in size.[18] Ormocers were formulated in an attempt to overcome the problems created by the polymerization shrinkage of conventional composites and also because ormocers include low shrinkage, high abrasion resistance, biocompatibility and protection against caries.[19] Recent studies compared the staining susceptibility of ormocers.[41] In our study, Admira Fusion ormocer composite showed ΔE values similar to Ceram-X nanohybrid composite and to Gradia Direct microfilled composite. However, in our study, in accordance to a research by Ayad,[42] Ormocer composite reported significantly higher ΔE values after exposure to coffee and lower color susceptibility if compared to nanofilled composites.

Conclusions

Under the conditions for this *in vitro* study, it can be concluded that the immersion of specimens in coffee

caused a significant color change in all types of tested composite resins. All restorative materials showed in fact clinically perceptible color differences after exposure to coffee. Coca-Cola did not influence the color stability for all restorative materials. Filtek Supreme XTE alone demonstrated a staining susceptibility to red wine; no other significant differences of staining among the different types of composite resins tested were demonstrated. Clinical choice of the composite material to be used for the patient could be related to habits and to general teeth discoloration.

Disclosure statement

The authors deny any conflicts of interest related to this study.

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