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ORIGINAL ARTICLE

Color stability of siloranes versus methacrylate-based composites after immersion in staining solutions

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Abstract

Objectives The purpose of this study was to determine, by using a spectrophotometer device, the color stability of silorane in comparison with four methacrylate-based composites after being immersed in different staining solutions such as coffee, black tea, red wine, orange juice, and coke, and distilled water as control group.

Methods Four restorative methacrylate-based composites (Filtek Z250, TetricEvoCeram, Venus Diamond, and Grandio) and one silorane (FiltekSilorane) of shade A2 were selected to measure their color stability (180 disk samples) after 4 weeks of immersion in six staining solutions: black tea, coffee, red wine, orange juice, coke, and distilled water. The specimen's color was measured each week by means of a spectrophotometer (CIE L*a*b* system). Statistical analysis was carried out performing an ANOVA and LSD Test in order to statistically analyze differences in L*a*b*and ΔE values.

Results All materials showed significant discoloration ($p < 0.05$) when compared to the control group (immersed in distilled water). The Highest ΔE observed was with red wine, whereas coke led to the lowest one. Silorane showed the highest color stability compared with methacrylate-based composites.

Conclusions Methacrylate-based materials immersed in staining solutions showed lower color stability when compared with silorane. Great differences in ΔE were found among the methacrylate-based materials tested.

Clinical relevance Although color stability of methacrylate-based composites immersed in staining solutions has been widely investigated, this has not been done for long immersion periods with silorane-based composites.

Keywords Resin composites · Staining · Color stability · Silorane

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Introduction

During the last decades, resin-based composites have undergone many modifications. The use of these materials for the restoration of anterior teeth has begun to increase [1–3] due to the improvements of not only its physicomechanical but also esthetic properties. The esthetic restoration of the anterior dentition presents one of the greatest challenges in daily practice. Defining esthetics as “the art of the imperceptible” [4], esthetic restorative materials should mimic the appearance of natural teeth. The latter is directly related to the material's degree of opacity and translucency, opalescent and iridescent effects, and fluorescence. In the long term, also other factors such as color stability, surface roughness, and surface gloss can determine success or failure of restorations [1, 4, 5]. Unacceptable color match is one of the main reasons for replacement of resin-based composite restorations [6].

When exposed to the oral environment [1, 6, 7], resin-based composites may present color instability due to intrinsic discoloration or extrinsic staining [4, 5, 8–10]. As described in many studies, extrinsic staining may be caused by insufficient degree of polymerization, heat, UV irradiation, water sorption, or adsorption of food colorants such as red wine, coffee, coke, tea [1, 6–14]. The degree of discoloration varies according to the oral hygiene, eating–drinking, and smoking habits of the patient [5, 7, 10, 13]. These external color changes may be eliminated by subsequently scaling and polishing the surface, but if deeper layers are involved, the discoloration is mostly irreversible [1, 8]. Intrinsic discoloration on the other hand, involves the staining of the resin material itself [1, 5, 6, 9–11]. This may be related to (1) the type of resin matrix, e.g., urethane dimethacrylate seems more stain resistant than bis-GMA because of its low viscosity and low water absorption [10, 11]; and (2) the fillers, e.g., an unfilled resin specimen generally exhibited less color change than did resin-based composite specimens [5, 10]; the filler's particle size and distribution seem to be directly correlated to optical properties; a smaller filler size might contribute to decrease staining and enhance esthetic appearance [1].

In order to avoid these problems related to the intrinsic structure of methacrylate-based composites, a newly developed material has been recently introduced onto the market. This innovative monomer system known as "silorane", is obtained from the reaction of oxirane and siloxane molecules [15–17]. Siloranes have been suggested as alternatives to methacrylates as matrix resin components for resin-based composites because of their hydrophobicity and lower polymerization shrinkage [15, 18, 19]. Various studies have confirmed a decreased water sorption, solubility, and associated diffusion coefficient of silorane compared with conventional methacrylate-based composites [16, 19, 20]. In light of these favorable properties, this new monomer system may be a promising solution to overcome the negative effects of oral fluids on the mechanical properties of resin-based composites [21]. Although color stability of methacrylate-based composites immersed in staining solutions has been widely investigated [1, 4–6, 8–11], there are no in vitro studies that have evaluated the color stability of silorane immersed in staining solutions.

The purpose of this study was, therefore, to determine the color stability of silorane in comparison with four methacrylate-based composites after immersion in different staining solutions such as coffee, black tea, red wine, orange juice, and coke, using reflection spectrophotometry based on the CIE L*a*b* color system. There were three null hypotheses:

1. The type of material does not influence the color stability of the material.
2. The type of staining solution does not influence the staining of the material.
3. The duration of storage in the staining solution does not influence the color stability of the material.

Material and methods

One hundred eighty disk samples were prepared from five different resin-based composite materials (Table 1) of shade A2 (n=30) by condensing the material into a standardized metal mold. The mold with the composite resin was held between two glass slides, each one covered with a transparent polyester strip (Mylar, DuPont, Wilmington, Del., USA). The slides were then gently pressed together to remove excess material. All specimens were polymerized by a LED light-curing lamp Bluephase (IvoclarVivadent AG, Schaan, Liechtenstein) with light intensity of 1,200 mW/cm^2 for 20 s of exposure on top and bottom surfaces, respectively. Irradiance was tested by a radiometer Demetron LED (Kerr Corp, Orange, CA, USA). The distance between the light source and the specimen was standardized by the use of a 1-mm glass slide. Specimens' dimensions were 10 mm in diameter and 2 mm thick. After polymerization, the specimens were stored in distilled water at 37 °C

Table 1 Composites used in the investigation

Composite	Composition[a]	Manufacturer	Color	Lot
Filtek silorane (A)	Bis-3,4-Epoxycyclohexylethyl-Phenyl-Methylsilane 3,4-Epoxycyclohexylcyclopolymethylsiloxane	3M/ESPE, St. Paul, MN, USA	A2	9BY/9CC
Filtek Z250 (B)	Bis-GMA, Bis-EMA, UDMA, TEGDMA	3M/ESPE, St. Paul, MN, USA	A2	8HU/9JA
Tetric evoceram(C)	Bis-GMA, UDMA, DDDMA	IvoclarVivadent AG, Schaan, Principality of Liechtenstein	A2	M08007
Venus diamond (D)	TCD-DI-HEA, UDMA	HeraeusKulzer, Hanau, Germany	A2	010026
Grandio (E)	Bis-GMA, UDMA, dimethacrylate, TEGMA	Voco, Cuxhaven, Germany	A2	918270

Bis-GMA Bisphenol-A diglycidylether methacrylate, *Bis-EMA* bisphenolApolyethyleneglycoldietherdimethacrylate, *UDMA* Urethane dimethacrylate, *TEGDMA* Triethyelene glycol dimethacrylate, *DDDMA* DecandiolDimetacrylate, *TCD-DI-HEA* 2-Propenoic acid, (octahydro-4,7-methano-1H-indene-5-diyl) bis(methyleneiminocarbonyloxy-2,1 ethanediyl) ester

[a] Composition as given by manufacturers

for 24 h for rehydration and completion of the polymerization, following the methodology of previous studies [4, 6, 8, 10, 12]. Before the immersion in staining solutions, specimens of each composite were randomly divided into six groups, corresponding to six samples per staining solution (coffee, black tea, red wine, orange juice, and coke, and distilled water as control group) (Table 2).

The specimen was rinsed for 10 s with distilled water and wiped dry with gauze before being immersed in staining solutions. At this moment, baseline color measurements (T0) were made. These measurements were performed using a reflectance spectrophotometer (SpectroShade, Handy Dental Type, MHT, Arbizzano, Italy) using the CIE L*a*b* system. The SpectroShade consisted of a D_{65} light source (6,500 K). This light was split so that the specimen could be illuminated simultaneously from a 45-degree angle using an intraoral camera. Spectrophotometric measurements were made using a white background, as in other studies [22]. Before each measurement session, the spectrophotometer was calibrated according to manufacturer recommendations using the supplied calibration standards. Color measurements were then made according to the same procedure at a time interval of 1 week (T1), 2 weeks (T2), 3 weeks (T3), and one month (T4). Staining solutions were renewed every 2 days to avoid bacteria or yeast contamination [23].

Using the SpectroShade software, differences in color (ΔE) and color coordinates (ΔL^*, Δa^*, Δb^*) between baseline (T0) and T1, T2, T3, and T4 measurements were calculated for each resin-based composite material and staining solution, where ΔL^* is the change in luminosity, Δa^* the change in red–green parameter and Δb^* the change in yellow–blue parameter. Color stability was determined evaluating the color difference (ΔE). The latter was calculated from the mean ΔL^* Δa^* and Δb^* according to the formula of Pythagoras [1, 4, 8, 10–14, 24–26]:

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

Table 2 Different staining solutions used in this investigation

Solution	Abbreviation	Manufacturer
Black tea	BT	Label Tea, Lipton Tea®, Unilever, France
Coffee	CO	Nescafé Classic®, Nestlé, Spain.
Red wine	RW	Enate®, Tempranillo-Cavernet Sauvignon 2004, Spain.
Orange juice	OJ	Granini Orange®, Spain.
Coke	CK	Coca-Cola®, The Coca-Cola Company, Spain.
Distilled water	CG	Lasda S.A, Spain

As data were normally distributed (Kolmogorov–Smirnov, Shapiro–Wilk), statistical analysis was performed using multifactorial ANOVA and LSD test in order to evaluate differences in ΔE, ΔL, Δa, and Δb between groups. The data analysis was carried out using the Statgraphics software, (Statpoint Technologies, Warrenton, Virginia, USA) with a significance set at $p=0.05$.

Results

The mean values of color change of the evaluated materials after the first week immersion in the staining solutions are represented in Table 3. Regarding the stainability of the materials, TetricEvoceram immersed in red wine showed the lowest color stability overall (ΔE=30.9) in the first week, while the highest color stability was Filtek Z250 immersed in coke (ΔE=1.1). Red wine had the highest staining potential overall, followed by coffee, black tea, orange juice, and coke. No statistically significant differences were found in ΔE between orange juice and coke ($p>0.05$). For the second week, the behavior of the materials and colorants is represented in Table 4. TetricEvoceram immersed in red wine showed the lowest color stability overall (ΔE=27.3), while the highest color stability was Filtek Z250 immersed in coke (ΔE=1.4). Regarding the staining potential, red wine showed the highest potential overall, followed by coffee, black tea, orange juice, and coke. Coffee and black tea showed no statistically significant differences in ΔE between these materials ($p>0.05$); no statistically significant differences in ΔE were also found between orange juice and coke ($p>0.05$). The behavior of the materials and colorants for the third week are represented in Table 5. Regarding the stainability of the materials, Grandio immersed in coffee showed the lowest color stability overall (ΔE=28.0), while the highest color stability was Filtek Z250 immersed in coke (ΔE=1.6). Red wine had the highest staining potential overall, followed by black tea, coffee, orange juice, and coke. Red wine, black tea, and coffee showed no statistically significant differences in ΔE between these materials ($p>0.05$); no statistically significant differences in ΔE also were found between orange juice and coke ($p>0.05$). For the fourth week, the behavior of the materials is represented in Table 6. Filtek Z250 immersed in red wine showed the lowest color stability overall (ΔE=30.2), while the highest color stability was once again Filtek Z250 immersed in coke (ΔE=1.4).

Regarding the staining potential, red wine showed the highest potential overall, followed by coffee, black tea, orange juice, coke, and distilled water. Coffee and red wine showed no statistically significant differences in ΔE between these materials ($p>0.05$), and coffee and black tea

Table 3 Mean (SD) of color changes (ΔE) for each composite and colorant (week 1)

Composite	Color change (ΔE)						
	Black Tea	Coffee	Red wine	Orange Juice	Coke	Distilled water	Homogeneous Groups
Silorane	5.4 (0.93)	6.1 (3.6)	4.2 (1.2)	2.2 (1.0)	2.0 (0.9)	1.1 (0.2)	A
Filtek Z250	16.2 (2.7)	13.4 (5.7)	24.7 (3.2)	3.1 (0.9)	1.1 (0.3)	0.5 (0.2)	C
TetricEvoceram	15.7 (5.9)	15.9 (3.7)	30.9 (1.3)	1.6 (0.6)	1.7 (0.3)	1.3 (0.7)	C
Venus Diamond	9.1 (2.6)	12.4 (2.9)	16.1 (4.9)	1.6 (0.7)	2.1 (0.5)	0.8 (0.3)	B
Grandio	10.0 (0.8)	26.3 (10.4)	19.5 (2.8)	2.1 (0.6)	1.7 (0.5)	0.5 (0.1)	C

Groups not connected with the same letter are significantly different

showed no statistically significant differences in ΔE between these materials ($p>0.05$). Also, no statistically significant differences in ΔE were found between orange juice, coke, and distilled water ($p>0.05$). Control group showed the lowest staining potential overall, after 4 weeks silorane showed the highest color stability ($\Delta E=1.9$) according to Table 6.

Discussion

Restorative dental materials are continuously exposed to saliva, beverages, and food stains in the oral environment [1, 6, 21]. It is important to determine their both intrinsic color stability and staining resistance, as this will influence the restorations' imperceptibility [1]. Color stability has been previously studied in vitro and in vivo studies [27–33] for a variety of esthetic restorative materials [1, 4, 7, 10–14]. In this study, a severe clinical situation was simulated. Therefore, samples were not polished but obtained by pressing the resin between two glass plates. This is an attempt to simulate the most severe clinical situation, where the restorative material is polymerized against a Mylar strip, thus richer in matrix resin, as can occur especially in the proximal region [23].

The color stability of a resin composite is related to the resin matrix, dimensions of filler particles, depth of polymerization, and coloring agents. Satou et al. [34] remarked that the chemical differences among resin components such as purity of the oligomers and monomers, concentration or type of activators, initiators and inhibitors, and oxidation of the unreacted carbon–carbon double bonds may also affect the color stability. Microcracks and microvoids located at the interface between the filler and the matrix are the most likely penetration pathways for stain. The roughness of the surface caused by wear and chemical degradation may also affect gloss and consequently increase the extrinsic staining. The staining susceptibility of resin-based composite materials is directly related to their degree of water sorption, related to the hydrophilic/hydrophobic nature of the resin matrix. If a composite resin can absorb water, it is also more likely to absorb water-soluble pigments, resulting in composite discoloration [1, 11, 35, 36].

The staining solutions used in this study were black tea, coffee, red wine, orange juice, and coke. These are common in our daily diet, and some are known to have potential to stain restorative materials [1, 8, 27, 28]. Storage in distilled water was used as control group. For this group, color differences were clinically acceptable ($\Delta E<3.0$); these results confirm that water absorption by itself did not alter the color of composites to a considerable extent [6, 37]. An immersion period of 4 weeks was chosen, as according to Ertas et al. [27], which should be equivalent to about 2.5 years of clinical aging (24 h of staining in vitro corresponds to about 1 month in vivo).

Table 4 Mean (SD) of color changes (ΔE) for each composite and colorant (week 2)

Composite	Color change (ΔE)						
	Black Tea	Coffee	Red wine	Orange Juice	Coke	Distilled water	Homogeneous Groups
Silorane	8.2 (0.7)	7.4 (3.7)	7.4 (1.3)	2.4 (0.9)	1.9 (0.5)	1.2 (0.4)	A
Filtek Z250	22.7 (2.7)	17.5 (2.4)	26.8 (7.6)	4.4 (1.1)	1.4 (0.5)	1.0 (0.5)	C
TetricEvoceram	23.6 (4.2)	18.2 (4.0)	27.3 (10.8)	2.4 (0.5)	2.2 (1.4)	2.0 (0.2)	C
Venus Diamond	8.9 (3.0)	15.1 (2.7)	18.6 (5.5)	2.9 (0.3)	2.7 (0.8)	1.5 (0.1)	B
Grandio	14.2 (2.4)	25.0 (7.3)	20.7 (4.0)	2.7 (0.6)	1.6 (0.1)	1.1 (0.2)	C

Groups not connected with the same letter are significantly different

Table 5 Mean (SD) of color changes (Δ*E*) for each composite and colorant (week 3)

Composite	Color change (Δ*E*)						
	Black Tea	Coffee	Red wine	Orange Juice	Coke	Distilled water	Homogeneous Groups
Silorane	9.1 (1.9)	7.5 (3.8)	6.9 (1.4)	2.6 (1.6)	2.7 (0.7)	1.8 (0.3)	A
Filtek Z250	27.5 (8.6)	18.4 (1.5)	26.4 (8.5)	6.3 (1.5)	1.6 (0.4)	1.5 (0.5)	C
TetricEvoceram	26.4 (2.9)	19.8 (2.9)	26.1 (8.9)	3.5 (0.6)	3.1 (0.7)	2.6 (0.3)	C
Venus Diamond	11.7 (2.0)	16.1 (2.6)	18.0 (4.3)	3.1 (0.8)	2.3 (0.4)	2.1 (0.3)	B
Grandio	18.6 (5.0)	28.0 (9.0)	22.5 (7.1)	3.7 (1.1)	2.4 (0.5)	1.9 (0.3)	C

Groups not connected with the same letter are significantly different

To avoid bias due to individual subjective evaluation of color, a spectrophotometric device was used in this study, allowing a quantitative color assessment [10, 11, 13, 22] The CIE L*a*b* system was chosen to measure color of the samples, as it is well suited for the determination of small color differences and has been widely used in previous studies [14, 23]. It has been actually claimed that a color difference of Δ*E*<1.0 is imperceptible for the human eye, while a Δ*E*>3.3 is clinically unacceptable [11, 35]. A white background was used as according to Dietschi et al. [22], it is clinically the most relevant background in small Class III restorations.

Regarding staining potential, the solutions were ranked in the following order: red wine >coffee >black tea >orange juice >cola. Surprisingly, cola showed Δ*E* values similar to the control group. This is probably due to the low staining potential of the components. As according to Um and Ruyter [35], even if cola has an acidic pH that might deteriorate the surface of the material, it contains few yellow stains with low polarity. Red wine, on the other hand, performed the highest staining potential, followed by coffee and tea. Several studies have reported that alcohol facilitates staining by softening the resin matrix [5, 10, 28]. However, it was not explained whether staining by red wine was due to the alcohol or the presence of pigments in wine [5]. The staining ability of tea may be attributed to the presence of tannic acid and stains. Regarding coffee, it contains yellow stain molecule that seem to be responsible for the staining because of their affinity to the polymer network [23].

Regarding the stainability of the materials, FiltekSilorane showed the highest color stability (Δ*E*=8.1), according to Table 6, as described by various studies [15, 16, 18, 37, 38, 39]. This might be due to the reduction in water sorption and solubility, which can be attributed to the hydrophobic backbone of the silorane molecule formulated from the incorporation of siloxane groups. Venus diamond (Δ*E*=16.4), was the second composite with the highest color stability, followed by Grandio (Δ*E*=23.8), Tetric (Δ*E*=26.3), and Filtek Z250 (Δ*E*=30.2), according to Table 6. Venus diamond contains a modified urethane dimethacrylate resin matrix and is bis-GMA-free. This matrix consists of resin-containing aliphatic chains that are less hydrophilic, which results in a lower water sorption and water solubility when compared to conventional Bis-GMA resin matrix having hydrophilic hydroxide groups, as present in Z250, Tetric, and Grandio [36]. Statistically significant differences in Δ*E* were not found between Grandio, Z250, and Tetric. Visually imperceptible differences in Δ*E* values (Δ*E*<1.0) between Z250, Tetric, and Grandio were observed. These may be attributed to small differences in chemical composition of the materials, quality of filler-resin silanization, or different affinity of the colorants to specific resin matrix components.

It is important to emphasize the impossibility of establishing the exact correlation between in vitro and in vivo tests, since the oral environment cannot be reproduced in the laboratory, and restorative materials are never subjected to staining medias for such a long consecutive period of time. The drinking habits of the patients must be considered when choosing restorative composite materials, especially on the

Table 6 Mean (SD) of color changes (Δ*E*) for each composite and colorant (week 4)

Composite	Color change (Δ*E*)						
	Black Tea	Coffee	Red wine	Orange Juice	Coke	Distilled water	Homogeneous Groups
Silorane	12.9 (3.6)	7.6 (3.4)	8.1 (1.5)	3.4 (1.2)	2.9 (0.7)	1.9 (0.5)	A
Filtek Z250	16.9 (5.9)	20.1 (2.3)	30.2 (7.7)	7.3 (1.8)	1.4 (0.2)	2.1 (0.6)	C
TetricEvoceram	29.1 (3.5)	23.7 (2.3)	26.3 (8.5)	3.3 (1.0)	3.6 (0.6)	3.0 (0.6)	D
Venus Diamond	12.7 (2.3)	16.4 (1.5)	16.4 (4.7)	3.8 (0.6)	2.9 (0.5)	2.6 (0.2)	B
Grandio	19.0 (6.1)	29.9 (9.1)	23.8 (4.8)	3.9 (0.8)	2.6 (0.5)	2.8 (0.2)	CD

Groups not connected with the same letter are significantly different esthetic zone. The restorative material's composition is also important, as well as the polymerization degree, an adequate surface texture, and a competent way of using it in order to obtain its best properties, thus guaranteeing longevity and success [10].

Conclusion

Within the limitations of the present study, all three hypotheses were rejected, as ΔE values depended on the material and the staining solution in which the material was immersed. Further, from T0 to T4, ΔE increased gradually for every material, for which the stainability is also time dependent. Silorane composite showed the highest color stability overall.

It can be concluded that the drinking habits of the patients must be considered when choosing restorative composite materials, as the staining potential of a solution is material dependent. It can be supposed that color of esthetic restorations can be maintained over a longer period of time in the oral environment either by introducing some restrictions to a patient's dietary habits or carefully choosing the type of material best compatible with the dietary lifestyle.

Conflict of interest The authors declare that they have no conflict of interest.

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