The objective of the study was to evaluate the color stability of 8 recently developed resin composites when exposed to various staining agents. Six disc-shaped specimens made out of 8 resin composite materials were immersed in artificial saliva, coffee, coke, tea, orange juice and red wine. The initial color (T0) of the 288 specimens was assessed by a calibrated reflectance spectrophotometer (SpectroShade, MHT) over a black as well as a white background. All specimens were kept in an incubator (INP-500, Memmert GmbH) at 37 °C for 4 weeks. Colorant solutions were changed every 7 days to avoid bacteria or yeast contamination. After 4 weeks of storage in the colorants (T1), spectrophotometric measurements were repeated and ΔE00 was calculated. When analyzed over a white background, the mean ΔE00 values varied from 1.5 (Venus Diamond) to 52.4 (Miris 2). When analyzed over a black background, the mean ΔE00 values varied from 0.8 (Venus Diamond) to 40.0 (ELS). Significant differences were detected when comparing values at T0 and T1 (ΔE00) for both backgrounds (Anova and Fisher’s LSD post hoc test). Resin composite [...]
Color stability of recent composite resins

S. Ardu1 · O. Duc2 · E. Di Bella3 · I. Krejci2

Abstract The objective of the study was to evaluate the color stability of 8 recently developed resin composites when exposed to various staining agents. Six disc-shaped specimens made out of 8 resin composite materials were immersed in artificial saliva, coffee, coke, tea, orange juice and red wine. The initial color (T0) of the 288 specimens was assessed by a calibrated reflectance spectrophotometer (SpectroShade, MHT) over a black as well as a white background. All specimens were kept in an incubator (INP-500, Memmert GmbH) at 37 °C for 4 weeks. Colorant solutions were changed every 7 days to avoid bacteria or yeast contamination. After 4 weeks of storage in the colorants (T1), spectrophotometric measurements were repeated and \( \Delta E_00 \) was calculated. When analyzed over a white background, the mean \( \Delta E_00 \) values varied from 1.5 (Venus Diamond) to 52.4 (Miris 2). When analyzed over a black background, the mean \( \Delta E_00 \) values varied from 0.8 (Venus Diamond) to 40.0 (ELS). Significant differences were detected when comparing values at T0 and T1 (\( \Delta E_00 \)) for both backgrounds (Anova and Fisher’s LSD post hoc test). Resin composite materials do behave in a significantly different way (p value < 0.01) when exposed to various staining solutions.

Keywords Composite · \( L^* \) a* b* · Staining · Spectrophotometer

Introduction

Resin composites today have the potential to reproduce the beauty of the natural tooth’s appearance, allowing a very conservative approach. These are the main reasons for increasing the use of composites for restorations of anterior and posterior teeth [1–3] as a valid alternative to ceramic veneers and ceramic or PFM crowns.

Nevertheless even the most recent composite resin products, due to their resin matrix’s nature, still absorb more moisture than ceramics and are thus more prone to the penetration of various staining agents [4, 5]. Unfortunately, only information furnished by manufacturers is available on the discoloration potential of composite resin materials so far and there is a lack of independent scientific data regarding their possible behavior in the clinical environment. To test their staining predisposition, an “in vitro” study can allow all tested materials to be submitted to equal staining stress. The results can be analyzed to understand the staining susceptibility and—even if limited—“in vivo” predictions become possible.

Within the last 2 years, several authors [6–15] have investigated the staining susceptibility of newly developed resin composite materials because of the patients’ desire to receive not only functional, but esthetically pleasing restorations for a reasonable life span.

The aim of this in vitro study was to evaluate the staining susceptibility of recent composite resins when submitted to different staining solution which are common in daily diet.
The first hypothesis tested was that the materials included into the study do not change their color after 4 weeks immersion in staining agents. The second goal was to rank their behavior per staining solution tested and also to pool the results in all staining solutions together.

**Materials and methods**

Two hundred and eighty-eight disc-shaped specimens measuring 10 mm in diameter were made out of eight composite materials tested (Tables 1, 2) by gently pressing the material between two glass slides to the thickness of 1 mm (Fig. 1). The composite resins were light cured for 20 s from a distance of 1 mm using an LED curing device (Valo, Ultradent, South Jordan, USA) used in “standard mode” with a power density of $>1000 \text{ mW/cm}^2$ (checked by LED Demetron radiometer 910726, Kerr Corporation Middleton, USA). After 24 h dry storage in an incubator (INP-500, Memmert), the initial color of each specimen was assessed by a calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, Serial No. HDL0090, MHT, Arbizzano di Negar, Verona, Italy). These measurements were performed with a white as well as a black background.

Specimens were then randomly divided into 6 groups and immersed in 5 staining solutions represented by red wine, juice, coke, tea and coffee or artificial saliva which constituted the control group (Table 3).

![Fig. 1 Image of the composite sample “in fieri”, just before polymerization, obtained by gently pressing the material between two glass slides to the thickness of 1 mm. The polymerization lamp was then inserted perpendicular and in contact with the upper glass slide.](image)

**Table 1** Tested materials, % charge by weight and by volume

<table>
<thead>
<tr>
<th>Composite</th>
<th>Manufacturer</th>
<th>% Charge by weight</th>
<th>% Charge by volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>Estelite posterior</td>
<td>Tokuyama dental</td>
<td>84</td>
<td>70</td>
</tr>
<tr>
<td>ELS</td>
<td>Saremco</td>
<td>74</td>
<td>49</td>
</tr>
<tr>
<td>Saremco microhybrid</td>
<td>Saremco</td>
<td>76</td>
<td>52</td>
</tr>
<tr>
<td>Filtek supreme</td>
<td>3M Espe</td>
<td>72</td>
<td>56</td>
</tr>
<tr>
<td>Inspiro SN</td>
<td>Edelweiss</td>
<td>82</td>
<td>65</td>
</tr>
<tr>
<td>Venus diamond</td>
<td>Heraeus Kulzer</td>
<td>81</td>
<td>64</td>
</tr>
<tr>
<td>Miris 2 NR</td>
<td>Coltene-Whaledent</td>
<td>80</td>
<td>65</td>
</tr>
<tr>
<td>Filtek silorane</td>
<td>3M Espe</td>
<td>76</td>
<td>59</td>
</tr>
</tbody>
</table>

**Table 2** Composition of the materials tested

<table>
<thead>
<tr>
<th>Composite</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Estelite posterior</td>
<td>Silica zirconia filler (mean particle size 2 μm, range 1–10 μm). Bis-GMA, TEG-DMA, Bis-MPEPP</td>
</tr>
<tr>
<td>ELS</td>
<td>Inorganic fillers (size 50–3000 nm). Bis-GMA, Bis-EMA</td>
</tr>
<tr>
<td>Saremco microhybrid</td>
<td>Inorganic fillers (size 4–3000 nm). Bis-GMA, Bis-EMA, TEG-DMA</td>
</tr>
<tr>
<td>Filtek supreme</td>
<td>Silica filler and aggregated zirconia fillers (size 4–20 nm) clusters 0.6–10 μm. Bis-GMA, UDMA, TEG-DMA, PEG-DMA, Bis-EMA</td>
</tr>
<tr>
<td>Inspiro SN</td>
<td>Barium alumino fluoride glass (size 0.02–2 μm). Bis-GMA</td>
</tr>
<tr>
<td>Venus diamond</td>
<td>Barium alumino fluoride glass TCD-DI-HEA, UDMA</td>
</tr>
<tr>
<td>Miris 2 NR</td>
<td>Barium alumino fluoride glass Bis-GMA, TEG-DMA, UDMA</td>
</tr>
<tr>
<td>Filtek silorane</td>
<td>Quartz fillers (size 0.1–2 μm) silorane matrix</td>
</tr>
</tbody>
</table>
All specimens were kept in an incubator at 37 °C in the dark for 28 days. Staining solutions were changed every week to avoid bacteria or yeast contamination.

After 28 days of storage, samples were removed from staining solutions, rinsed for 60 s with a high-pressure hot water airbrush (0.4 MPa, 135 °C, Minivapor 93, Effegi Brega s.r.l., 29010 Sarmato, PC-Italy) and, afterward, air dried. Spectrophotometric measurements were repeated for each sample to determine the color changes according to the classical CIEDE 2000 (ΔE00) formula (Fig. 2).

### Table 3 Composition of the staining solutions

<table>
<thead>
<tr>
<th>Staining agent</th>
<th>Producer</th>
</tr>
</thead>
<tbody>
<tr>
<td>RED wine</td>
<td>Côtes du Rhône (DOC), Les Arènes, Vacqueyras</td>
</tr>
<tr>
<td>Orange juice</td>
<td>Hohes C, Eckes-Granini, Switzerland</td>
</tr>
<tr>
<td>Coke</td>
<td>Coca-Cola; Coca-Cola beverages AG, CH-8306 Brüttisellen, Switzerland</td>
</tr>
<tr>
<td>Tea</td>
<td>Twinings Earl Gray tea, London, England</td>
</tr>
<tr>
<td>Coffee</td>
<td>Arpeggio, Nespresso, Nestle, Switzerland</td>
</tr>
</tbody>
</table>

![Fig. 2 CIEDE 2000 (ΔE00) formula](image-url)
More specificities regarding the methodology used in this study are explained in detail in a precedent publication which already tested the behavior of resin-based composite materials in an in vitro long term-simulated staining test [16].

Statistical analysis was performed by means of ANOVA on the log-transformed $\Delta E_{00}$ values to guarantee the required normality assumptions as tested by the Kolmogorov–Smirnov test to test the effect of the staining solutions (first goal of the paper). Furthermore, all staining values were pooled together per composite and the $\Delta E_{00}$ values were submitted to Fisher’s LSD post hoc test (second goal of the paper).

**Results**

All tested materials showed significant ($p < 0.01$) color changes after 28 days of staining immersion.

When analyzed over a white background, the mean $\Delta E_{00}$ values varied from 1.5 (Venus Diamond) to 52.4 (Miris 2).

When analyzed over a black background, the mean $\Delta E_{00}$ values varied from 0.8 (Venus Diamond) to 40.0 (ELS).

When all staining solutions’ means were pooled together per composite material and analyzed over a white background, the mean $\Delta E_{00}$ values varied from 10.6 (Estelite Posterior) to 19.2 (Filtek Supreme).

When all staining solutions’ means were pooled together and analyzed over a black background, the mean $\Delta E_{00}$ values varied from 11.4 (Filtek Silorane) to 16.6 (Filtek Supreme).

When immersed in red wine, Estelite Posterior performed best, while Saremco Microhybrid and Filtek Supreme showed the worst results independently of the background.

When immersed in coffee, Filtek Silorane performed best, while Venus Diamond, ELS and Filtek Supreme showed the worst results independently of the background.

When immersed in tea, Filtek Silorane performed best, while Venus Diamond and Filtek Supreme showed the worst results independently of the background.

When immersed in coke and in orange juice, Estelite Posterior performed best, while Inspiro SN showed the worst results independently of the background.

These results are summarized in Table 4a, b.

**Discussion**

Resin composite restorations are continuously exposed to staining agents due to food and beverage pigments which are present in common diet. According to previous studies, the degree of composite staining can be affected by several factors such as incomplete polymerization, [17, 18] water sorption, [19, 20] chemical reactivity [21, 22], diet, [23–

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**Table 4 a, b** Average $\Delta E_{00}$ differences before and after the staining process per group analyzed over a black and white background and corresponding groupings ($A =$ best, $F =$ worst) defined on the basis of the Fisher’s LSD test (significant differences of group mean for $p$ value $< 0.01$)
with Ertas et al. [45], who considered 28 days to be highly clinically relevant [44]. This is even in accordance with simulated consumption of the drink over 2 years, which is three cups per day. Therefore, 28 days of storage is three days. Moreover, 28 days of storage is three days. As materials’ degree of conversion may be a crucial point, all composites tested were of enamel translucency A2 shade. In case A2 was not available, the manufacturers were asked to provide an A2-equivalent shade. Enamel translucency was chosen due to the fact that it is the composite mass which is usually used as the most external mass and thus in contact with staining agents, and A2 is one of the most used enamel shades.

The decision of not polishing samples was taken to mimic clinically relevant conditions such as the part of a class IV restoration, which is polymerized against a Mylar strip in the front teeth or against the sectional matrix at the contact point in the posterior area. Under this clinical condition, actually, the composite is richer in matrix resin [38] and thus could be more prone to discoloration. In contrast with this hypothesis, Garoushi et al. [39] tested the influence of polishing resin composite materials with similar chemical base as the ones we tested (except for silorane), clearly showing no difference between the polished and unpolished samples. Anyway, and even if literature is not univocal concerning the influence of polishing on staining [39–41], we prefer to consider the obtained results as clinically relevant only for the above-mentioned clinical situation. A further study with the same protocol and materials and methods but polished samples will be carried out to elucidate definitively this topic.

Twenty eight days immersion time was chosen in accordance with two previous massive reviews of the literature of the last 10 years showing that the maximum immersion time used was 30 days [42, 43]. The average time for consumption of one cup of coffee or tea is 15 min and, among coffee or tea drinkers, the average consumption is three cups per day. Therefore, 28 days of storage simulated consumption of the drink over 2 years, which is highly clinically relevant [44]. This is even in accordance with Ertas et al. [45], who considered 28 days to be equivalent to about 2.5 years of clinical aging (24 h in vitro staining corresponds to 1 month in vivo).

The use of a spectrophotometer was made to avoid all bias due to human eye evaluation, in accordance with precedent studies [6, 46–48] and to allow a double evaluation over a white as well as a black background. This double evaluation can simulate two different clinical situations [48]; the black background mimics class IV composite restorations, where no tooth structure exists in the back. White background can mimic the situation, where one of the walls is still present, i.e., class I, II, III and veneers.

Considering the staining solution tested, red wine had the most staining capacity, followed by coffee and tea. The highest values of ΔE00 reached by red wine could be explained by its relative acidity (pH 4.5) and its content of tannins.

Coffee was tested through Arpeggio (Nespresso) capsule. Despite its strength 9 (on a scale up to 13), the fact of having been brewed in the “lungo mode” could have lowered the staining potential due to dilution of colorants.

Tea, (Twinnings Earl Gray tea, London, England) is a light and aromatic blend of fine black tea, with a strength of 2 on a scale up to 5. Its staining potential may be considered medium to low.

The use of other staining solution or other solution brands may lead to different results according to different staining potentials of the applied products.

Considering the results obtained in this study, Filtek Silorane showed the best performance. Its excellent behavior can be explained by the hydrophobicity of the resin matrix which minimizes water sorption and consequently prevents staining. Its matrix is made out of oxirane and siloxane which are highly hydrophobic. Specifically, colorants of coffee and tea, as well as the ones present in orange juice, were not able to attack the resin matrix of the silorane composite, resulting in an excellent behavior. Its underperformance in the coke and saliva groups could be due to the postpolymerisation behavior rather than due to a specific acid attack. ΔE00 values are, in fact, quite low and similar to each other (coke ΔE00 = 3.6, saliva ΔE00 = 3.3).

Estelite Posterior also performed well. This material, even if made out of common Bis-GMA resin, is highly charged, which means that a lower proportion of resin can be exposed to staining solutions. Furthermore, the presence of Bis-MPEPP could have enhanced the hydrophobicity of the matrix. In addition, it is speculated that the conversion rate of this material might be quite elevated.

Venus Diamond which has substituted the classical Bis-GMA by a patented TCD-DI-HEA monomer did not show any advantage with respect to staining behavior. Specifically, the high polarity colorants of tea and the low polarity
colorants of coffee seem to have quite a great affinity with this resin matrix.

The substitution of TEG-DMA (Saremco Microhybrid) by Bis-EMA (ELS) did not show any improvement in terms of staining prevention in comparison to the standard composite matrix composition. The general trend of both materials, with respect to colorants’ affinity, seems very similar independently of the background.

Inspiro SN seemed to be easily attacked by acidic beverages such as orange juice and coke. This could be due to the evenly distributed charge, which could allow a larger exposition of the fillers and a consequent etching of their surface. The presence of some micro-porosities within the composite could have further promoted pigment apposition. The batch used in this study was one of the first to be produced. More studies may be needed to confirm or reject our results.

Filtek Supreme, accordingly to other studies [16, 42], seems to be the most prone to staining, probably due to a sub-ideal integration of the nano-aggregated particles. These structures, even if not hydrophilic “per se”, may lead to discoloration when their interface is not perfectly silanized and integrated into the resin matrix, thus facilitating water and colorant infiltration.

Furthermore, a higher level of TEGDMA, which is a hydrophilic monomer, could have contributed to a less than ideal in vitro behavior of the material. This resin composite material performed the worst when merged with high polarity (tea), low polarity (coffee) colorants or tannins, independently of the background.

The slightly different ranking obtained with the two different backgrounds could be due to different opacity of the resin composites which might modify the color perception on different backgrounds. Anyway, the general behavior trend of the tested materials is confirmed.

Some limitations have to be considered due to the nature of the study: it is an in vitro experiment, the tested materials were continuously exposed to staining solutions, the surfaces of the samples were not polished and no brushing effect was used during the staining process. Future studies which investigate all these variables are warranted for more precise evaluations as well as in vivo studies to confirm the obtained results.

Conclusions

Resin composite materials do behave in a significantly different way (p value < 0.05) when soaked in various staining solutions.

The first hypothesis was thus rejected.

The second goal of the present work was to rank resin composite materials’ behaviors per staining solution and pooling all staining solution results together per composite material which resulted in significantly (p value < 0.01) better performance of Estelite Posterior and Filtek Silorane when considered over a white background and Filtek Silorane when considered over a black background.

Compliance with ethical standards

Conflict of interest All the authors declare that they have no conflict of interest.

References


