Color stability of a resin composite: Effect of the immersion method and surface treatments

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ABSTRACT

Background: *In vitro* staining methods expose the entire specimen to staining solutions. In a real clinical situation, this is not observed, since one should consider that the bonded surface is not exposed to the oral environment. Theoretically, the clinical condition would be the best simulated if the specimens were exposed to staining solutions by partial immersion.

Aims: To evaluate if different immersion methods and surface treatments influence the color stability of resin-based specimens.

Methodology: A stainless steel matrix was used to prepare 30 disc-shaped specimens that were randomly allocated in three groups: Without polishing, polishing with abrasive discs, and surface sealant. Half of the specimens were isolated to maintain only the upper surface exposed to staining (partial immersion) and the other half was totally immersed in coffee solution for 48 h (total immersion). The coordinates $\Delta E^*$, $\Delta L^*$, $\Delta a^*$, $\Delta b^*$ were assessed by spectrophotometer.

Statistical Analysis: Two-way ANOVA and Tukey’s post hoc tests ($\alpha = 0.05$).

Results: Specimens submitted to partial immersion showed lower values of $\Delta E^*$, $\Delta L^*$, $\Delta a^*$, $\Delta b^*$, in comparison to total immersion ($P = 0.000$). Specimens covered by a surface sealant presented lower $\Delta E^*$ values regardless of the immersion method.

Conclusions: Specimens totally immersed in staining solutions could in somehow overestimate the color change, once that in most clinical conditions not all of the restoration surfaces are exposed to the oral environment. Moreover, as the surface sealant application produces color change values that are clinically acceptable, it might be used in esthetic restorations as an adjunct treatment.

Key words: Colorimetry, dental sealants, resin composite, spectrophotometer, staining

Improvements in the properties of resin composites are constantly conducted since resin-based materials are commonly employed as the first choice in restorative procedures. Despite the increased improvements, discoloration of resin composites remains one of the major reasons for the replacement due to esthetic dissatisfaction.\(^{[1,2]}\)

There are mainly two types of discoloration: Intrinsic discolorations-physical-chemical reactions within the resin matrix and extrinsic discolorations-biofilm and absorption/adsorption of dyes. While some mechanical properties can only be improved by manufacturers, some studies concerning surface treatments have attempted to enhance the color stability of resin composites.\(^{[3-5]}\)

Most of the studies aforementioned evaluated the color stability of resin-based materials by total immersion of disc-shaped specimens in staining solutions. Although the surface treatment is performed only upon the upper surface of the specimens, all surfaces are exposed to staining, once...
the specimens are totally immersed in beverages such as coffee, tea, or water.\textsuperscript{[6-8]} However, this staining pattern does not simulate clinical conditions, since at least one surface will be bonded to the remaining cavity walls.

Colorimeters and spectrophotometers are the most common methods used for color evaluation in \textit{in vitro} and \textit{in vivo} studies since visual observations can be affected by subjective interpretations inherent of the researcher and environmental conditions.\textsuperscript{[9,10]} Usually, the threshold of 3.3 in color change is considered clinically unacceptable, which leads to restoration replacement due to esthetics.\textsuperscript{[11]} Total immersion method exposes all surfaces to staining agents and may overestimate the real value of the color change. Values above 3.3 using the current method of staining should be interpreted carefully. Therefore, to produce a more reliable method for evaluation of external discoloration, the exposition of only one surface to the staining solution was proposed in this study.

The aim of this study was to evaluate if different immersion methods and surface treatments influence the color stability of resin-based specimens. The null hypotheses tested were (1) that the immersion methods of staining would not produce different color change values and (2) that different surface treatments would result in similar patterns of staining.

**METHODOLOGY**

**Specimens preparation**

The tested materials are shown in Table 1. A stainless steel matrix with 2 mm thickness and 8 mm diameter was used to prepare 30 disc-shaped specimens that were randomly assigned in three experimental groups according to the surface treatment.

Half of the specimens of each group were submitted to partial immersion in coffee solution and the other half were totally immersed in coffee (\(n = 5\)). The groups and immersion methods are shown in Figure 1. The matrix was filled in with resin composite in one increment and covered by a Mylar strip. Then, a glass slide was positioned over the matrix and pressured for 10 s to produce a smooth and standardized surface. The specimens were light-cured for 20 s with a light-emitting diode LED light-curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) at irradiance 1200 mW/cm\(^2\), monitored by a radiometer. After removal of the glass slide, the specimen was light cured for 20 s in both sides. Thickness of the specimens was checked with a digital caliper (Carbografite, Equipamentos Industriais Ltda., RJ, Brazil). Specimens were stored in distilled water at 37°C for 24 h. A single trained operator made all specimens.

Twenty specimens were sequentially polished with abrasive discs (Sof-Lex\textsuperscript{TM} XT Pop on, 3M ESPE, St. Paul, MN, USA), dark orange to yellow, in a mounted low speed handpiece under light pressure for 15 s on each grit on the top surface, which will be exposed to staining and rinsed with running water after polishing steps. The abrasive discs were discarded every four specimens. Then, a surface sealant (Biscover LV, Bisco, Schaumburg, IL, USA) was applied on 10 specimens, in accordance with manufacturer’s instructions.

**Color assessment**

Immediately after storage, baseline color measurement was performed according to the CIE L’a*b* color scale (Commission Internationale de l’Eclairage) relative to the standard illuminant D65 over a white background on a reflection spectrophotometer (SP60-EX-Rite/Grand Rapid, MI, USA). The CIE L’a*b* color system is a three-dimensional color measurement: L* refers to the lightness coordinate and its value ranges from 0 for perfect black to 100 for perfect white, a* and b* are chromaticity coordinates on the green-red (−a* = green; +a* = red) and blue-yellow (−b* = blue; +b* = yellow) axes. Three consecutive readings were performed upon the top surface of the specimen, and the average value of L*, a* and b* was calculated. The spectrophotometer was calibrated according to the manufacturer’s instructions before color measurements. All measures were performed at the same environmental conditions by a single operator.

Half of the specimens were totally immersed in coffee solution (3.6 g of coffee powder was dissolved in 300 ml of boiling water as per the manufacturer’s recommendation), and the other half was partially immersed. A sealing tape involving side and bottom of the specimens was placed, leaving only the upper surface uncovered [Figure 2a]. Five holes were made with a rubber dam punch, and each specimen was placed within the perforations [Figure 2b].

| Material       | Color | Particle size/composition | Manufacturer                      | Batch number       |
|----------------|-------|---------------------------|-----------------------------------|--------------------|
| Z250 (microhybrid) | A1    | 0.01-3.5 µm Zirconia, Silica, Bis-GMA, UDMA, Bis-EMA\textsuperscript{*} | 3M ESPE (St. Paul, Minissota, USA) | 1332300388         |
| BisCover LV     | -     | Dipentaerythritol Pentaacrylate (20%), ethanol | Bisco (Schaumburg, Illinois, USA) | 1300007021         |

\textsuperscript{*}Bis-GMA=Bisphenylglycidyl dimethacrylate, UDMA=Urethane dimethacrylate, Bis-EMA=Ethoxylated bisphenol-A dimethacrylate

**Table 1: Materials, composition, manufacturers’ information and batch number**
Cyanoacrylate adhesive was applied on the covered surfaces of the specimens, to ensure that no coffee would infiltrate through the isolation. Then, the rubber dam was attached to the strainers with elastics floating in coffee with only the uncovered specimen surfaces exposed to stain.

After 48 h, specimens were rinsed with running water and dried with absorbent paper before each color measurement. In the partial immersion groups, specimens were removed from the rubber dam isolation along with the sealing tape and washing was performed before color change evaluation. The final color of the specimens was measured once again upon the top surface using the spectrophotometer, as described earlier. Color change (\(\Delta E\)) was calculated as follows: 

\[
\Delta E^* = \left( (L_1^* - L_0^*)^2 + (a_1^* - a_0^*)^2 + (b_1^* - b_0^*)^2 \right)^{1/2}
\]

**Statistics**

Based on a pilot study, the sample size was obtained considering the power of 80% and significance level of 5%.

The normal distribution of the data was confirmed using Kolmogorov–Smirnov test. The mean \(\Delta E^*, \Delta L^*, \Delta a^*,\) and \(\Delta b^*,\) values were analyzed by two-way ANOVA and Tukey’s post hoc tests (\(\alpha = 0.05\)). Statistical analysis was performed using the Minitab software (Minitab Inc., State College, PA, USA).

**RESULTS**

The color change (\(\Delta E\)) mean and standard deviations for all experimental groups are shown in Table 2. Considering that the interaction among factors was not significant (\(P = 0.197\)), direct comparison among all experimental groups could not be performed.

Tables 3 and 4 present the mean and standard deviations of \(\Delta E^*, \Delta a^*, \Delta b^*, \Delta L^*\) according to the factors immersion and surface treatment, respectively. The groups submitted to partial immersion presented statistically significant lower values of all variables compared with groups where total immersion was performed (\(P = 0.000\)). When the surface treatment was evaluated, the color change (\(\Delta E\)) of the surface sealant group was statistically lower than polished groups that were similar to nontreated groups. Despite that the color change values (\(\Delta E\)) were not different among groups with or without polishing, statistically significant differences was observed (\(P = 0.000\)) for values of lightness (\(\Delta L\)), red-green parameter (\(\Delta a\)), and yellow-blue parameter (\(\Delta b\)).

**DISCUSSION**

At the moment, satisfactory in vitro and in vivo correlations may not yet exist, since laboratory studies often use “static tests” to evaluate the dental material properties. The materials evaluated in clinical studies are part of an assembly at the mouth surrounded by its biological environment daily submitted to many changes that in vitro studies cannot properly simulate. Many characteristics encountered clinically are difficult to achieve at the laboratory, e.g., saliva, pH challenges, abrasion by oral hygiene, plaque accumulation, and some of them can either...
have protective or deleterious effects on the material being tested. Color change is one of the characteristics that have a poor correlation between laboratory and clinical data.[14]

It should be stated that most studies regarding color stability evaluation perform aging techniques on the entire surface of the specimens.[6,7,15‑17] This pattern is not observed in most clinical restorations since at least one surface remains bonded to the remaining cavity walls. In an attempt to approximate the laboratory and clinical environments, this study aimed to create an immersion method to stain one surface of the specimen for color stability evaluation, and, therefore, compared the new immersion method to the conventional, in specimens with different surface treatments.

The color change can be assessed by different methodologies. Besides the reproduction of more reliable values, instrumental methods can be easily used to perform comparison of the color change in different resin composites.[9] Most of these instruments use the CIE *L*′,*a*′,*b*′ color system. The analysis is based on the coordinates (*L*′, *a*′, *b*′) in regard to its final and initial measures of each specimen. The current study used the ∆E value of 3.3 as a clinically acceptable threshold. This is in accordance with Ruyter et al., which considered that changes in one unit of ∆E are visually perceptible, but with no need for restoration replacement.[12]

Total immersion groups can be considered an extreme condition of staining. In this case, all surfaces are exposed to the dye solution that produce clinically unacceptable results,[18,19] as can be observed also in this study [Table 2] which groups exposed to total immersion achieved ∆E values higher than 3.3. Therefore, to predict a similar color change achieved in clinical conditions, a partial isolation of the specimens by a rubber dam was performed to ensure that only one surface was exposed to the staining agent. When total immersion was performed, higher values of all variables (∆E, ∆a, ∆b, and ∆L) were observed regardless of the surface treatment (*P* < 0.05). It is noteworthy that a shift of two units (∆E = 4.58 to ∆E = 2.91) was observed when the surface sealant group were submitted to partial immersion. Nevertheless, despite the interaction among factors was not significant, and then direct comparison between values cannot be performed, the color change was lower than 3.3. When totally immersed groups were compared, the values were not only clinically unacceptable, but also overestimated.

Surface treatments have been described as an effective way to enhance color stability of resin composite restorations.[18,19] Different polishing techniques may diminish the roughness of resin composite restorations performed with spatula, which may increase their color stability. However, some of the polishing steps may leave irregularities on the surfaces that may lead to the impregnation of dyes. Mylar strips are considered the “gold standard” in surface roughness, but their use are restricted to proximal surfaces on restorations. Furthermore, it is known that the composition of the resin matrix can influence the color stability. Bis-GMA monomers are more susceptible to staining in comparison to urethane dimethacrylate,[4] and due to the hydrophilicity, triethylene glycol dimethacrylate monomers have a higher staining potential.[20]

In the literature, it is still not well established if surface roughness alone can be directly associated with the color stability over time.[21] In this study, three types of surface treatment were evaluated. Once that nontreated groups and polished groups did not exhibit significant differences (*P* > 0.05) regarding color change (∆E) and both were made with the same resin composite, it is likely that the irregularities made by the polishing steps were not enough to produce a different ∆E between these groups, since the same pattern of staining was observed in both types of immersion. Noteworthy, the surface sealant applied does not have in its composition the monomers and fillers found in the matrix of the resin composite. Since polished specimens had similar ∆E values of nonpolished, and the sealant was applied upon the same resin composite in both immersion methods, other factors related to the material itself might have a significant impact on the color stability as previously stated.[21] More studies correlating surface roughness and color stability are desired to confirm this statement.

The use of surface sealant produced statistically lower values of ∆E, irrespective of the immersion method. This is in

### Table 2: Mean (standard deviation) values of color change (∆E) for all groups

| Groups          | Partial immersion | Total immersion |
|-----------------|-------------------|-----------------|
| Without polishing | 4.68 (0.47)       | 6.89 (0.31)     |
| With polishing  | 4.74 (0.37)       | 6.97 (0.39)     |
| With sealant    | 2.91 (0.35)       | 4.58 (0.40)     |

### Table 3: Mean (standard deviation) values of ∆E, ∆a, ∆b, ∆L considering immersion methods

| Groups          | ∆E     | ∆a     | ∆b     | ∆L     |
|-----------------|--------|--------|--------|--------|
| Total immersion | 6.15 (1.20) | 1.36 (0.45) | 4.87 (0.90) | −3.43 (1.00) |
| Partial immersion | 4.11 (0.95) | 0.25 (0.31) | 3.80 (0.84) | −1.51 (0.54) |

Within each column different superscripts indicate statistically significant differences (*P* < 0.05)

### Table 4: Mean (standard deviation) values of ∆E, ∆a, ∆b, ∆L for surface treatment

| Groups          | ∆E     | ∆a     | ∆b     | ∆L     |
|-----------------|--------|--------|--------|--------|
| Without polishing | 5.79 (1.22) | 0.73 (0.58) | 5.10 (0.87) | −2.52 (1.03) |
| With polishing  | 5.86 (1.23) | 1.26 (0.68) | 4.60 (0.48) | −3.26 (1.34) |
| With sealant    | 3.75 (0.95) | 0.43 (0.55) | 3.29 (0.62) | −1.62 (0.83) |

Within each column different superscripts indicate statistically significant differences (*P* < 0.05)
accordance with previous studies that showed better color stability when surface sealants were applied.\textsuperscript{18,19} The sealed group submitted to partial immersion showed clinically acceptable discoloration after staining, suggesting that the BisCover LV could be applied in esthetic restorations as an alternative procedure to prevent staining over time.

According to the results of this study, the null hypotheses must be rejected. Specimens, where the surface sealant was applied upon the resin composite, presented statistically lower ΔE values. Moreover, all variables tested (ΔE, Δa, Δb, ΔL) exhibited lower values when only the methods were considered, showing that the partial immersion method produces lower values of color change.

\section*{CONCLUSION}

The new method of staining seems to be more reliable in comparison with the conventional. All specimens submitted to partial immersion showed significantly lower color change values when immersed in coffee and the surface sealant group had the lowest value (<3.3), which is clinically acceptable. Therefore, surface sealant might be used in aesthetic restorations as an adjunct treatment to protected resin composites from external discoloration. Future studies for color change evaluation should consider the new immersion method of staining rather than total immersion methods since the latter could overestimate the staining of specimens.

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\section*{Conflicts of interest}

There are no conflicts of interest.

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