Evaluation of the staining potential of a caries infiltrant in comparison to other products

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In this study, we evaluated in vitro the staining susceptibility of an infiltration resin (Icon, DMG, Hamburg, Germany) and compared it with several marketed bonding systems. Fifty 1-mm-thick disk-shaped specimens were prepared for Icon and for each bonding material. Initial specimen color was assessed by a spectrophotometer. Specimens in each group were then randomly divided into five sub-groups and stored in an incubator at 37°C in the dark for 60 days. Groups 4 and 5 were used as negative controls by being stored dry and in tap water respectively. Test groups were stored in (1) coffee, (2) tea, or (3) red wine. After 60 days of storage, new spectrophotometric measurements were performed and \( dE \) (color difference) was calculated to determine color change. Icon showed higher staining susceptibility. The clinician should be aware of the staining potential of infiltration resins over time.

*Keywords: Caries Infiltration, Spectrophotometer, Staining susceptibility*

**INTRODUCTION**

The technique of infiltrating interproximal and vestibular carious lesions with low-viscosity resins seems to be an interesting complement to the widely used treatment approaches of remineralization by fluorides and restorations\(^1\). Hinging on the modern philosophy of minimal invasive dentistry, the infiltration technique has gained a lot of interest in the past few years. It is more time consuming than the application of preventive varnishes, but less invasive than microabrasion and restorative treatments\(^2\). The infiltration technique is based on the fact that low-viscosity, hydrophilic, light-cured resins can literally penetrate initial caries lesion, thus inhibiting the diffusion pathway to cariogenic bacteria and their by-products and preventing further lesion progression under demineralizing conditions\(^3\). Another positive effect of infiltration resins is that they also change the refractive index (RI) of an initial caries lesion.

The whitish appearance of an initial caries lesion is due to the difference in RI between sound enamel and caries lesions, resulting in highly unaesthetic appearance—especially in the anterior zone. Infiltration resin can fill the pores of an initial caries lesion, thus eliminating the RI mismatch between sound and carious enamel\(^4\). This effect is particularly important for patients presenting white spot lesions resulting from orthodontic treatments. The presence of plaque accumulation around orthodontic brackets is often associated with white spot lesions, representing a common undesirable effect of orthodontic treatment with a highly unaesthetic side effect\(^5,6\). Studies have shown that the infiltration technique is highly recommended for patients with white spot lesions, because highly esthetic results can be achieved instantly. Infiltration technique is less invasive than microabrasion or restorative treatments with composite resins\(^7\), but the infiltration resin is capable of penetrating deeply into the lesions, much more than the remineralization effect of fluorides.

An infiltration resin showing good penetration characteristics (Icon, DMG, Hamburg, Germany) was developed and marketed recently. This low-viscosity light-curing material is used for the infiltration technique, and has revealed good results in masking white spot lesions. In fact, several studies showed that Icon exhibited a very positive masking effect on white spot lesions\(^2,4,5,7,8\). Icon is an unfilled, low-viscosity hydrophilic resin more or less similar to a dentin primer, thus susceptible to water sorption and degradation. The aim of infiltrating white spot lesions with Icon is to have highly esthetic results, not just immediately after treatment but for long-term effect. A study showed that artificial lesions infiltrated with Icon were not sensitive to discoloration by sunlight\(^9\). However, no studies have shown how Icon reacts to natural, everyday staining agents found in food and beverages, such as coffee, red wine, or tea. If the infiltrated carious lesions become stained over time, the esthetic outcome of the treatment would be compromised.

Therefore, the aim of this study was to evaluate in vitro the staining susceptibility of Icon after long-term exposure to various staining agents and compare it with several marketed bonding agents. The null hypothesis was that the color of Icon would remain stable even when in contact with natural staining agents, and that there would be no significant differences when compared with other bonding agents.

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MATERIALS AND METHODS

Fifty 1-mm-thick disk-shaped specimens were prepared for the infiltration resin (Icon) and for each of the four bonding materials. A total of 250 resin specimens were gently pressed between two glass slides. To ensure complete polymerization, the resins were light-cured for 30 s on each side of the disk, with the light tip placed 1 mm above the specimen, using a high-power halogen curing device, Swiss Master Light (EMS SA), at a light intensity of 1,000 mW/cm². Ten specimens of each material were randomly assigned to one of the following five groups:

- Group A: Clearfil SE Bond (Kuraray Noritake Dental, Tokyo, Japan; Lot No. 01474A; Expiry date: 2012-09)
- Group B: Heliobond (Ivoclar Vivadent, Schaan, Liechtenstein; Lot No M50564; Expiry date: 2014-07)
- Group C: OptiBond FL (Kerr, West Collins, USA; Lot No. 4367177; Expiry date: 2013-05)
- Group D: Scotchbond Universal Adhesive (3M ESPE, St. Paul, USA; Lot No. 457855; Expiry date: 2013-11)
- Group E: Icon (DMG, Hamburg, Germany; Lot No. 621424; Expiry date: 2010-10)

Initial specimen color was assessed using a quantitative numerical measurement approach with a calibrated reflectance spectrophotometer (SpectroShade, Handy Dental Type 713000, MHT, Zürich, Switzerland). CIE $L^*a^*b^*$ measurements of each specimen were performed using a white background. Measurements were performed under a D65 light source (6,500 K). This light source was split to have each sample illuminated simultaneously from both sides at a 45-degree angle. Reflected light was directed at 0 degrees on the two system detectors (each having a 18×13-mm surface). One detector was a color charge-coupled device (CCD) chip that generated the color video image; the other was a black-and-white CCD detector which recorded the spectrophotometric data. Polarization filters were used to eliminate surface gloss. All measurements were captured in a proprietary image file format used to create detailed CIE $L^*a^*b^*$ data.

After recording their initial $L^*a^*b^*$ data, specimens in each group were randomly divided into five subgroups (10 specimens per bonding material were used in each staining solution) and stored in an incubator (at 37˚C) for 60 days during the testing phase. Groups 4 and 5 were used as negative controls by being stored dry and in tap water respectively. Test groups were stored in the following solutions:

- Sub-Group 1: 1.5 mL of coffee solution (Ristretto, Nespresso)
- Sub-Group 2: 1.5 mL of tea solution (Twinings Classics Prince of Wales tea, Twinings)
- Sub-Group 3: 1.5 mL of red wine (Château Etang des Colombes 2010, Henri Gualco, France)
- Sub-Group 4: Negative control (dry)
- Sub-Group 5: Negative control (water)

Test solutions were changed every 7 days to avoid bacteria or yeast growth. After 60 days of storage, specimens were removed from the staining solutions, rinsed for 60 s with a high-pressure hot-water airbrush (0.4 MPa, 135˚C, Minivapor 93, Effegi Brega) and air-dried. New spectrophotometric measurements were performed and $L^*a^*b^*$ data were recorded. Color change (staining susceptibility) was determined by comparing these results with the initial data according to the following formula:

$$dE = \sqrt{\left(L^*_{\text{final}} - L^*_{\text{initial}}\right)^2 + \left(a^*_{\text{final}} - a^*_{\text{initial}}\right)^2 + \left(b^*_{\text{final}} - b^*_{\text{initial}}\right)^2}$$

This methodology of color assessment was based on a study performed by Ardu et al.10.

Statistical analysis was performed with SPPS 16.0 for Windows. ANOVA and Duncan’s post hoc tests were used to specifically identify differences between groups. Confidence level was set at 95%.

RESULTS

Table 1 presents the mean color change values for each adhesive material group in each staining agent. Table 2 presents the mean color change values of each adhesive material group amongst three staining agents. Figure 1 presents the different colors of the specimens before and after staining.

Regarding the staining potential of colorants,
Table 2  
$dE$ values (Mean (SD)) of each adhesive material in three colorants (coffee, red wine, tea). Groups with different letters are significantly different ($p<0.05$)

<table>
<thead>
<tr>
<th>Adhesive Material</th>
<th>$dE$</th>
<th>Letter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil SE Bond</td>
<td>13.6 (4.2)</td>
<td>A</td>
</tr>
<tr>
<td>Scotchbond Universal Adhesive</td>
<td>25.6 (8.6)</td>
<td>B</td>
</tr>
<tr>
<td>Heliobond</td>
<td>28.4 (5.4)</td>
<td>BC</td>
</tr>
<tr>
<td>OptiBond FL</td>
<td>31.8 (4.7)</td>
<td>C</td>
</tr>
<tr>
<td>Icon</td>
<td>44.1 (20.2)</td>
<td>D</td>
</tr>
</tbody>
</table>

Fig. 1  
Discoloration of adhesive material by coffee, tea, red wine (from left to right) and the initial color: (a) Icon (b) OptiBond FL; (c) Heliobond; (d) Clearfil SE Bond; (e) Scotchbond Universal Adhesive.
red wine had the highest staining potential (mean $dE=34.76$), followed by coffee (mean $dE=33.64$) and tea (mean $dE=17.76$) with the latter exhibiting the lowest staining potential. The difference in staining potential among the three colorants is clearly visible in Fig. 1.

Regarding the color change of adhesive materials, Icon had the highest color change (mean $dE=44.1$, Fig. 1(a)), followed by OptiBond FL (mean $dE=31.8$, Fig. 1(b)), Heliobond (mean $dE=28.4$, Fig. 1(c)), Scotchbond Universal Adhesive (mean $dE=25.6$, Fig. 1(e)), and Clearfil SE Bond (mean $dE=13.6$, Fig. 1(d)) (Table 2).

Finally, regarding the staining potential of colorants in every group of adhesive material, Icon had the highest color change with coffee and red wine (mean $dE$ values were 60.3 and 55.2 respectively) and OptiBond FL had the highest color change with tea (mean $dE=26.4$). Clearfil SE Bond had the lowest color change with red wine, coffee, and tea (mean $dE$ values were 17.2, 13.9 and 9.5 respectively).

Compared with staining by colorants, the control groups showed significantly different color changes. Nevertheless, dry storage and water storage groups showed a mean $dE$ values ranging from 0.7 (Heliobond, water storage) to 5.9 (Clearfil SE Bond, water storage). Some $dE$ values were higher than 3.3.

Figure 1 presents the different colors of the specimens before and after staining. It could be clearly seen that red wine and coffee exhibited the highest staining potential.

**DISCUSSION**

Although the efficiency of caries infiltration resins, especially Icon, has been proved in many studies$^{1,3,6,11,12}$, the staining ability of Icon compared with other products has not been investigated yet. When a practitioner wants to treat white spot lesions associated with orthodontic brackets, his aim is to improve the esthetic appearance of the teeth. The infiltration technique of treating white spot lesions with Icon has been proved efficient by several studies$^{2,4,5,7,8}$. However, there is also a need for this treatment to remain color-stable with time despite the fact that the teeth are in contact with staining agents every day.

In this study, we simulated a long-term exposure with specimens stored for 2 months in different staining solutions and in an incubator at 37°C. According to the estimation of Ertas et al.$^{13}$, this duration should simulate around 5 years of clinical exposure to the staining agents (24 h in vitro corresponds to about 1 month in vivo), which is considered sufficient for long-term staining ability evaluation.

Specimens were rinsed with a high-pressure hot-water airbrush (0.4 MPa, 135°C, Minivapor 93, Effegi Brega) for 60 s so that only pigmentation that adhered irreversibly to the inner surface of the resin was evaluated. In a previous study, Ardu et al. showed that this rinsing method achieved an effect similar to polishing with an 80-µm prophylactic paste for 30 s$^{10}$, but which did not remove any material from the surface.

Measurements before and after the immersion period were done on white and black backgrounds. In some studies which compared the discoloration of different classes of composites, measurements were also done on white and black backgrounds$^{10,14}$. Adhesive materials are more or less transparent depending on their chemical compositions, but generally more transparent than composite resins. Therefore, measurements on a black background are often biased because of the transparency of the bonding resins. This was the reason why we decided not to consider measurements on a black background, but to keep only measurements done on a white background. Moreover, white spot lesions are situated in most cases on the vestibular surface of the tooth and not at the angle (class IV restoration). This means that the white background is more fitting to simulate a clinical situation than the black one.

When dealing with spectrophotometry, we set a level to distinguish between statistical differences and eye-visible color variations. $dE$ values higher than 1.1 were considered eye-visible and $dE$ values higher than 3.3 were esthetically disturbing and non-acceptable$^{15,16}$.

Adhesive resins are composed of different components which may influence their staining ability. The foremost common chemical components are the resin monomers, similar to those in composite restorative materials, which promote good covalent bonding to the composites. Table 3 presents the composition of the five adhesive materials tested in this study. According to Van Landuyt et al., both acrylates and methacrylates are vulnerable to water degradation (hydrolysis) of their ester groups. The spacer of the monomer does not have such an important function, except for keeping both functional and polymerizable groups well separated. However, it has an important influence on monomer properties, because its hydrophilicity may cause water uptake, which leads to higher hydrolysis susceptibility of the monomers as well as discoloration of the cured resin$^{17}$.

According to Sideridou et al., TEGDMA has the highest water sorption capability, followed by BisGMA and by UDMA$^{18}$. TEGDMA is the main component of the infiltration resin Icon because it has the best ability to infiltrate deep into the lesion. Paris et al. showed that a resin consisting mainly of TEGDMA seemed to be the preferred choice because a higher penetration coefficient into the lesion$^{12}$. Nevertheless, TEGDMA has the highest water sorption rate, which causes resin discoloration. Therefore, Icon (composed mainly of TEGDMA) became more discolored after storage in staining solutions as compared with other resins in this study. Dietschi et al. showed that staining may be linked to water sorption rate, with water being the carrier for pigments to penetrate deep into the resin matrix. Therefore, staining susceptibility tended to correspond with water sorption rate$^{19}$.

Scotchbond Universal Adhesive contains a polyalkenoic copolymer (Vitrebond Copolymer). The reason for using this polymer is to provide better
Table 3  Compositions of the adhesive materials tested

<table>
<thead>
<tr>
<th>Adhesive Manufacturer</th>
<th>Composition</th>
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<tbody>
<tr>
<td>OptiBond FL, Kerr, West Collins, USA</td>
<td>Bis-GMA, HEMA, GDMA, CQ, ODMAB, filler (fumed SiO$_2$, barium aluminoborosilicate, barium aluminosilicate, Na$_2$SiF$_6$), coupling factor A174</td>
</tr>
<tr>
<td>Clearfil SE Bond, Kuraray Noritake Dental, Tokyo, Japan</td>
<td>MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, photo-initiators, silanated colloidal silica</td>
</tr>
<tr>
<td>Heliobond, Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td>Bis-GMA, TEGDMA, catalysts, stabilizers</td>
</tr>
<tr>
<td>Scotchbond Universal Adhesive, 3M ESPE, St. Paul, USA</td>
<td>MDP Phosphate monomer, Dimethacrylate resins, HEMA, Vitrebond Copolymer, Filler, Ethanol, Water, Initiators, Silane</td>
</tr>
<tr>
<td>Icon, DMG, Hamburg, Germany</td>
<td>TEGDMA based resin, initiators, additives</td>
</tr>
</tbody>
</table>

moisture stability$^{20,21}$. Scotchbond Universal Adhesive and Clearfil SE Bond also contain MDP. Structurally, the long carboxyl chain renders this monomer quite hydrophobic. Therefore, 10-MDP provides hydrolysis stability as it keeps water at bay$^{17}$. This characteristic of Scotchbond Universal Adhesive and Clearfil SE Bond explained their good behavior when stored in different staining solutions. Moreover, Malacarne showed that Clearfil SE Bond had the lowest water sorption rate among four different adhesive systems. They claimed that water sorption, solubility and water diffusion coefficient of methacrylate-based resins were dependent on an adhesive’s composition and hydrophilicity$^{22}$. Among the three staining solutions, red wine had the highest mean $dE$ value (34.76), followed by coffee (mean $dE$=33.64) and tea (mean $dE$= 17.76). These data were in accordance with the findings of Ardu et al.$^{10}$ and Ertas et al.$^{13}$. Red wine is rich in tannins, which thus possesses the highest potential for discoloration. Red wine also contains alcohol, and the sorption of alcohol molecules into the resin matrix could result in softening of the adhesive resin surface and contributed to staining, which thus explained the results obtained for red wine solution$^{23}$.

Both tea and coffee contain yellow colorants. According to Um and Ruyter$^{16}$, the discoloration of materials by tea was mainly due to surface adsorption of the colorants. Discoloration by coffee was due to both adsorption and absorption of colorants. The absorption and penetration of colorants into the organic phase of materials were probably due to compatibility of the polymer phase with the yellow colorants of coffee$^{16}$.

Conversion rate after polymerization is also very important for the physical characteristics of resins after polymerization. As stated by different studies, low conversion rate may result in higher permeability$^{24}$, more water sorption$^{25}$, and thus higher staining potential. To avoid this problem, the materials in this study were polymerized with a calibrated, high-power halogen curing device, Swiss Master Light (EMS SA), at a light intensity of 1,000 mW/cm$^2$ for 30 s on both sides of the disk-shaped specimens.

All resin specimens in the control groups (water and dry storage) showed statistically significant differences in color change when compared with the other stained specimens. Nevertheless, both dry- and water-stored specimens exhibited slight change in color, with $dE$ (mean (SD)) values being even higher than 3.3 in some cases. In the case of dry storage, $dE$ values of Scotchbond Universal Adhesive and OptiBond FL were 3.7 (0.4) and 5.4 (0.9) respectively. In water storage, the $dE$ values of Scotchbond Universal Adhesive and Clearfil SE Bond were 5.7 (0.8) and 5.9 (1.1) respectively. This was probably due to post-polymerization of the adhesive materials in the case of storage in dry conditions, coupled with water sorption after storage in water$^{26}$.

CONCLUSION

The null hypothesis that the color of Icon would remain stable even when in contact with natural staining agents and that there would be no significant differences when compared with other bonding agents is rejected.

Results obtained from the present study could be of clinical relevance. Clinicians are provided with information on the staining potential of infiltration resins over time when teeth are exposed to natural staining agents. It is speculated that while Icon can fix the initial esthetic problem associated with white spot lesions, the resin may become more discolored than other adhesive materials over time — especially when the patient habitually consumes teeth-staining food and beverages. By taking this factor into consideration, patients should be advised to avoid consuming teeth-staining food and beverages.

REFERENCES

1) Kielbassa AM, Muller J, Gernhardt CR. Closing the gap between oral hygiene and minimally invasive dentistry:


