Effects of staining solutions on the discoloration of orthodontic adhesives: An in-vitro study

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Introduction: Our aim was to evaluate the effects of staining solutions on the discoloration of orthodontic adhesives. Methods: Six orthodontic adhesives were investigated (Transbond XT [3M Unitek, Monrovia, Calif, USA], Heliosit Orthodontic [Ivoclar Vivadent, Liechtenstein], Light Bond [Reliance Orthodontic Products, Itasca, IL, USA], Bisco Ortho [Bisco, Schamburg, IL, USA], Quick Cure [Reliance Orthodontic Products, Itasca, IL, USA], and Filtek Supreme XT [3M ESPE, St Paul, Minn, USA]), and 5 beverages (tea, cola, coffee, red wine, and yogurt). Sixty specimens were prepared. Five specimens from each group were stored in each of the 5 staining solutions. The 5 remaining specimens from each group served as the controls and were stored in distilled water. The specimens were immersed in staining solutions and water at 37°C ± 1°C for 5 days. The test period was 25 days. Before and after the test period, color measurements were carried out with a spectrophotometer, and color changes (ΔE*) were calculated. Statistical differences were evaluated by using analysis of variance (ANOVA) and the Tukey HSD tests. Results: Adhesive materials, staining agents, and their interactions were found to play statistically significant roles (P <0.001) in color changes. Among the adhesive materials, the Light Bond water control group consistently showed the lowest ΔE* value for all materials, and the Filtek Supreme XT group showed the highest ΔE* value for all materials. After the in-vitro experimental process for staining solutions and water, unsatisfactory color stability was observed for the conventional adhesive systems except for Light Bond, Transbond XT, and Bisco Ortho water control group (ΔE* >3.7), respectively. Conclusions: In esthetically critical areas, discoloration of adhesive materials for fixed orthodontics can cause patient dissatisfaction. Orthodontic composites will discolor from staining beverages during their lifespan. (Am J Orthod Dentofacial Orthop 2010;138:741-6)

For over 60 years, resin-based adhesive composites have been used for direct or indirect bonding of orthodontic attachments to teeth for orthodontic purposes. Most orthodontic adhesives are variations on adhesive and direct-restorative formulations produced for use in restorative dentistry.¹ ²

Direct bonding of orthodontic brackets to teeth has been an important subject in orthodontic research, because of the significance of a stable bond between a tooth etched with phosphoric acid and its bracket.³ Most studies in the orthodontic literature have focused on evaluation of the physical and mechanical properties of adhesives resins. However, discolorations of resin-based adhesive composites have been investigated in only a few studies.² ⁴ ⁵ A study showed that orthodontic bracket bonding and debonding can cause changes in the appearance of tooth enamel.⁶ In addition to the formation of structural and surface defects, some variables—eg, enamel loss caused by etching—can affect the enamel color, inducing various alterations on the enamel surface, including white spots.⁶ Although there are many findings about enamel white spot formation associated with orthodontic treatment, the incidence of enamel color changes caused by orthodontic bonding and debonding protocols has interested only a few investigators.² ⁴ ⁵ There is some evidence that adhesive resin tags could reach a depth of 50 μm in the enamel.⁴ As a result of these parameters, debonding and cleaning protocols cannot reverse adhesive resin impregnation into the enamel.⁴ Enamel discolorations can occur by direct absorption of food colorants and products from the corrosion of orthodontic appliances.⁷ The long-term presence of these resin residues in the enamel tags that extend over the middle third of the buccal surface makes the color stability of these materials critical for tooth color.⁵
The color stability of resin-based adhesive composites might be affected by external and internal factors. External influences are colored mouth rinses and staining foods (such as coffee and red wine), matrix (hydrophobic or hydrophilic), and type of filler particles (organic, inorganic, silica gel, silane coupling agent, and size). The material’s superficial roughness has an important role in the color changes of adhesive composites influenced by the different materials. A number of factors influence the extent of discoloration of photo-curing materials, such as the adsorption of dyes or plaques, water sorption, incomplete polymerization, photo-initiator components (eg, camphorquinone), resin matrix composition, light-curing devices, and irradiation times. The proof for internal color change of adhesive materials can be found in the aging under various physical and chemical conditions, such as humidity, ultraviolet irradiation, and thermal changes. The system of photo-initiators used in adhesive composite as well as on the applied form and the time span of curing have a certain effect on the discoloration of the material. They are induced by chemical changes in the materials’ matrices and hence affect all layers of the material; they cause irreversible discolorations in the polymer. In esthetically critical areas, resin-based adhesive materials must maintain not only the attachment of orthodontic appliance but also an esthetic appearance over the period of service. Discoloration of adhesive materials for fixed orthodontics can cause patient dissatisfaction; this is especially problematic when orthodontic adhesives are subjected to prolonged exposure to staining materials during lengthy treatment. Hence, the color change might be an important criterion in the selection of a particular orthodontic adhesive material for use in an esthetically critical area. Therefore, the aim of the study was to evaluate the combined effect of 5 staining beverages on the discoloration of orthodontic adhesives.

**MATERIAL AND METHODS**

The resin-based adhesive composites investigated were Bisco Ortho (Bisco, Schaumburg, Ill), Heliosit Orthodontic (Ivoclar Vivadent, Liechtenstein), Light Bond (Reliance Orthodontic Products, Itasca, Ill), Quick Cure (Reliance Orthodontic Products), Transbond XT (3M Unitek, Monrovia, Calif), and Filtek Supreme (3M ESPE, St Paul, Minn) to test for color changes (Table I). An Elipar FreeLight 2 (3M ESPE) was the light-curing device for preparing the light-polymerizing specimens. The specimens were stored in common colored beverages; for the control group, distilled water was used. Sixty cylindrical specimens (10 for each adhesive group) were manufactured in polytetrafluoroethylene molds at room temperature. Each disk had a diameter of 6 mm and a depth of 1 mm. The manufacturers’ instructions were used for preparing, handling, and polymerizing the materials. The mold was filled with material, and the upper and lower surfaces of the mold were covered by 2 glass slides (3 mm). These slides were gently pressed together for removing excess test material. The specimens were stored in common colored beverages; for the control group, distilled water was used. The drinks were black tea, black coffee, yogurt, red wine, and a soft drink based on cola (Table II).

Sixty cylindrical specimens (10 for each adhesive group) were manufactured in polytetrafluoroethylene molds at room temperature. Each disk had a diameter of 6 mm and a depth of 1 mm. The manufacturers’ instructions were used for preparing, handling, and polymerizing the materials. The mold was filled with material, and the upper and lower surfaces of the mold were covered by 2 glass slides (3 mm). These slides were gently pressed together for removing excess test material. The specimens were cured with the light-emitting diode device having a tip of 8 mm with light intensity of 1200 mW per square centimeter. Standardization of the distance between the light source and the specimen was provided by the thickness of glass slide, which also provided a smooth surface for testing. The specimens were then immersed in distilled water at 37°C ± 1°C for 24 hours for complete polymerization.

Sixty specimens of each adhesive composite resin were divided into 6 groups (n = 10) for staining.

<p>| Table I. Adhesive resins in this investigation |</p>
<table>
<thead>
<tr>
<th>Product</th>
<th>Material type</th>
<th>Manufacturer</th>
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<tbody>
<tr>
<td>Bisco Ortho</td>
<td>Bis-GMA (15%-40%), TEG-DMA (5%-15%), fused silica (30%-60%)</td>
<td>Bisco, Schaumburg, Ill Lot no: 0600004508</td>
</tr>
<tr>
<td>Heliosit Orthodontic</td>
<td>UDMA, Bis-GMA and decandiol-DMA (85%), dispersed silicon dioxide (14%), catalysts and stabilizers (1%)</td>
<td>Ivoclar Vivadent, Liechtenstein Lot no: G21064</td>
</tr>
<tr>
<td>Light Bond</td>
<td>UDMA, TEG-DMA (22%), fused silica, sodium fluoride (78%)</td>
<td>Reliance Orthodontic Products, Itasca, Ill Lot no: 0609234</td>
</tr>
<tr>
<td>Quick Cure</td>
<td>Silica-crystalline, silica-fused (50%-90%), Bis-GMA (1%-10%), TEG-DMA (5%-10%), sodium fluoride (0.1%-2.0%)</td>
<td>Reliance Orthodontic Products, Itasca, Ill Lot no: 0604936</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>Bis-GMA (5%-10%), Bis-EMA (10%-20%), TEG-DMA (5%-10%), silane-treated quartz (70%-80%), silane-treated silica (2%)</td>
<td>3M Unitek, Monrovia, Calif Lot no: 6CX6WM0088</td>
</tr>
<tr>
<td>Filtek Supreme</td>
<td>Bis-GMA, TEG-DMA, and Bis-EMA (35%), zirconia-silica (65%)</td>
<td>3M ESPE, St Paul, Minn Lot no: 6BY3910A1E</td>
</tr>
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</table>
Five randomly selected specimens from each group were stored in each of the 5 staining solutions. The 5 remaining specimens from each group were the controls and were stored in distilled water. The specimens were immersed in the staining solutions and water at 37°C ± 1°C for 5 days. The test period was 25 days. The solutions and water were changed daily. The black tea and coffee were prepared with boiled distilled water according to the manufacturer’s suggested concentrations. The yogurt, red wine, and cola came from the market ready to drink. The solutions (50 mL for each) and the specimens were placed in small receptacles. After 1 day in the solutions, the specimens were gently rinsed with distilled water for 5 minutes and dried with tissue paper.

The baseline color of all specimens was measured with a colorimeter (Vita Easyshade, Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany) against a white background according to the system of the Commission Internationale de l’Eclairage (CIE) — L*, a*, b* — after 24 hours of storage in distilled water.17 All measurements were repeated 3 times, and averages for the values of L*, a*, and b* were evaluated. After all discoloration processes, final color measurements was carried out.

The CIE system uses the 3-dimensional colorimetric measurements: L* values correspond to the brightness of a color, a* values to the red-green content, and b* values to the yellow-blue content. The color changes (ΔE*) were calculated from the L*, a*, and b* values for each specimen according to the following formula, which determines the 3-dimensional color space:

$$\Delta E^* = \left[ (L_1^* - L_2^*)^2 + (a_1^* - a_2^*)^2 + (b_1^* - b_2^*)^2 \right]^{1/2}$$

A perceptible color change that is ΔE* > 1.0 will be referred to as acceptable up to the value ΔE* = 3.7 in subjective visual determinations made in vitro under optimal lighting conditions.

### Statistical analysis

The descriptive analysis was determined for the control and treatment groups. Statistical differences were evaluated by using 2-way analysis of variance (ANOVA) and the Tukey HSD tests for adhesive materials with statistical software (SPSS for Windows, version 13.0, SPSS, Chicago, Ill). The significance level was set at α = 0.05.

### RESULTS

According to the ANOVA, the adhesive materials, staining beverages, and their interactions were statistically significant (P = 0.000) in the color changes (Table III). Mean values and standard deviations of ΔE* values for intervals of baseline and staining, and group differences of orthodontics adhesive materials are listed in Tables IV and V. After day 25 of the staining period, all adhesive materials had perceptibly significant discolorations (ΔE* > 3.7) in the treatment (staining solutions) groups. Filtek Supreme was the most affected of the adhesive materials, and Quick Cure was the least affected (Table IV). Overall, the control group (distilled water) had less discoloration than the adhesive materials, but 3 tested composites (Filtek Supreme, Heliosit Orthodontic, Quick Cure) had perceptibly significant discolorations (ΔE* > 3.7) (Table V). Light Bond’s water control group consistently showed the lowest ΔE* value of all adhesive materials. On the other hand, the Filtek Supreme group showed the highest ΔE* value of all the adhesive materials. After the in-vitro experimental immersion in staining solutions and water, unsatisfactory color stability was observed for the conventional adhesive systems except for Light Bond, Transbond XT, and Bisco Ortho water control groups (ΔE* ≤ 3.7).

### DISCUSSION

In the literature, there are many studies about discoloration of adhesive resins by staining beverages such as tea, coffee, red wine, yogurt, cherry juice, and cola. But in these studies generally, colorant beverages were investigated one by one; in other words, adhesive resin samples were prepared for discoloration, and the first group was immersed in coffee, and the second group was immersed in tea, and so on. Thus, the effects of
the staining solutions were compared separately, and the last word was that the first solution changed the color of the composite more than the second one. One study reported that coffee and tea caused clinically unacceptable color changes, and coffee had more staining capacity than tea. Another study came to the same conclusion. A study about discoloration reported that mouth rinse, tea, coffee, and red wine caused changes in color, and red wine caused the greatest changes below or above the value of 3.7 (ΔE*). Thus, color changes can be determined with various procedures, including spectrophotometric and visual. Visual investigation is not useful or even possible for the quantitative evaluation of minimal color changes and differences. The results of visual color comparisons are too subjective because of the investigator’s personal opinion, and, therefore, the reproducibility of these investigations is low. By using standardized color-quantifying devices such as the spectrophotometer, Vita Easyshade, reproducible, metric, objective, and statistically usable results of color determinations can be obtained. This testing design was based on that of Yannikakis et al and combined with other designs for using artificial treatment to examine color changes of adhesive materials. The discoloration protocol used this investigation might not dependably simulate the micro-environmental conditions of the oral cavity.

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Time is a major factor for changing the optical properties of adhesive resins. Also, the quality of polymerization type, light activation conditions, curing time, filler particles of the matrix of adhesive resins, actual colors of the surface, type of staining agent, and immersion procedures also affect and alter the optical properties. The color changes of polymeric materials might be caused by external or internal influences.
influences for color changes include staining by superficial adsorption or absorption of pigments from food dyes, tea, nicotine, colored beverages, and so on. Internal factors for color changes are directly related to changes of chemical structures of the adhesive materials; these are irreversible. Oxidation of the polymer matrix or oxidation of the double bonds of the matrix in the adhesive resin are related to chemical discolorations. Problems involved with insufficient polymerization include poor physico-chemical properties, increased solubility in the oral environment, increased microleakage, and consequent color changes. Also, the inorganic filler content of the adhesive resin, including the type and the amount, affects color stability. An adhesive resin with fewer inorganic filler might absorb a staining substance such as coffee or cola more easily than higher filled polymers. But in our study, although the orthodontic composites Quick Cure, Transbond XT, and Bisco Ortho have the same amount of filler content, their discolorations were different. According to these results, discoloration of orthodontic composites does not depend only on the amount of filler content but also on the type of filler and monomer, the connection capacity of monomer to filler, the degree of conversion, and so on.

Water sorption characteristics of the adhesive resins are affected by hydroxyl, carboxyl, and phosphate groups in monomers and their resultant polymers, and thus they acquire more hydrophilic behavior. The adhesive resins’ color could be affected irreversibly from their immersion in water for a long time, because of high levels of water absorption. The action of water facilitates the penetration of the beverage component into the adhesive resin because of the hydrophilic behavior of the polymeric matrix; this results in discoloration of the adhesive resin.

From our study, 3 brands of adhesive resins from the water control groups showed clinically acceptable and imperceptible color changes after treatment. The other 3 showed greater discolorations. Quick Cure from the water control group had the greatest color change. The ΔE* values were 5.11, 5.92, and 10.86 for the discolored composites Filtek Supreme, Heliosit Orthodontic, and Quick Cure, respectively. For the higher water discolorations of the control groups, there might be several explanations in the literature. Yannikakis et al. stated that a composite-based resin demonstrated clinically unacceptable color changes with water for 7 and 30 days of treatments. There might be a number of reasons for the excessive color changes: unreacted double bonds, prolonged periods in water, hydophilic behavior of the polymeric matrix, and the inorganic filler composition of the composites.

The color of all adhesives in the stained groups changed dramatically after treatment. Discoloration rates were high. Filtek Supreme had a high rate of discoloration. Although Quick Cure was the most discolored adhesive resin in the water control groups, it was the lowest one in the stained groups after treatment. As mentioned before, discoloration of composite resins with staining solutions depended on the material. The resin matrix or filler content of a composite resin might affect its staining capacity. According to the manufacturer’s declaration, Filtek Supreme is a nanocomposite with a primary 20-nm silica filler and loosely bonded cluster zirconia-silica particle size ranging from 0.6 to 1.4 mm. For example, Villalta et al. investigated Filtek Supreme for discoloration and concluded that, because of its composition, Filtek Supreme demonstrated high discoloration rates, similar to other authors’ findings. Filtek Supreme is a filling composite material for enamel in conservative dentistry and was used in this study as a control and for comparison with the fluid bonding resins of orthodontics.

CONCLUSIONS

Within the limitations this study, the following conclusions can be drawn.

1. Orthodontic composites will discolor from staining beverages during their lifespan.

2. Discoloration of orthodontic composites is complex, affected by the inorganic filler content of composite, monomer type, polymerization degree, and many other factors.

3. In esthetically critical areas, discoloration of adhesive materials for fixed orthodontics might cause patient dissatisfaction. The patient’s oral hygiene is an important factor for preventing color changes of the orthodontic composite.

In future clinical studies, orthodontic composites should be investigated for the level of discoloration they cause to the esthetics of tooth enamel.

REFERENCES


