Original Article

Influence of orthodontic adhesives and clean-up procedures on the stain susceptibility of enamel after debonding

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ABSTRACT

Objective: To determine the influence of the type of orthodontic adhesive system, such as conventional acid-etching (CE) and self-etching primers (SEPs), on the stain susceptibility of enamel surface after debonding. Effects of clean-up procedures on the enamel surface were also determined.

Materials and Methods: Two types and four brands of adhesive systems were investigated using 135 human premolars. Unbonded teeth were used as controls. Three-dimensional scanning of the enamel surface was performed before bracket bonding, after debonding, and after clean-up procedures. The color of each tooth was measured before bracket bonding and again after debonding and clean-up procedures. This was followed by methylene blue staining. The stain susceptibility of the enamel surface was measured after finishing only (F-condition) and after finishing/polishing (FP-condition).

Results: After debonding, the amount of residual adhesive resins in CE materials was greater than that in SEP materials. For the F-condition, staining color change in SEP materials was significantly higher than that in CE materials. For the FP-condition, staining color change in both CE and SEP materials was not different from those of the control.

Conclusions: The SEP system would show less stain susceptibility if the thin residual adhesive resin layer after debonding is removed by polishing. (*Angle Orthod.* 2011;81:334–340.)

KEY WORDS: Adhesive systems; Staining color change; Polishing

INTRODUCTION

Orthodontic treatment can lead to adverse effects on enamel surfaces.[1,2] These are manifested as enamel loss caused by etching,[1,2] enamel surface alterations leading to decalcification,[1] and microcracks and scratches induced during debonding and clean-up procedures.[2–5] Besides structural defects, the aforementioned changes can adversely affect color and esthetics of enamel.[3] Therefore, there have been needs to improve the bonding properties in which clinically sufficient bond strength is obtained, while minimizing enamel defect and discoloration.[5,6]

Self-etching primers (SEPs) have generated wide interest, leading to popular use[7–11] chiefly due to their simple application procedures. Besides saving chair time, fewer bonding steps would produce less error and reduce technique sensitivity. SEPs are also reported to show a more conservative etching pattern[3,5,6] but maintain adequate bond strength.[10]

Two main causes for enamel discoloration after debonding were reported—formation of white spots and irreversible penetration of resin tags into enamel structures.[12] Resin tags could absorb colorants[13] and corrosion products of the orthodontic appliance.[14] For the evaluation of stain susceptibility in restorative resin composite, a staining method was used,[15] and methylene blue is one of the dyes generally used.[16]

Although there have been studies on the bond strength of orthodontic adhesives and enamel loss after debonding,[9,10,17] limited information is available on the stain susceptibility of debonded and cleaned-up enamel surfaces associated with orthodontic adhesives and clean-up procedures such as finishing and polishing. The purposes of this study were to: (1)

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Table 1. Materials Investigated in This Study

Adhesive System[a]	Code	Brand Name	Batch Number	Manufacturer
CE	Transbond			
	Primer	Transbond XT	6CY	3M Unitek, Monrovia, Calif
	Adhesive	Transbond XT	7AX	
	Ortho Solo			
	Primer	Ortho Solo	2767643	Ormco Corp, Glendora, Calif
	Adhesive	Blugloo	2848183	
SEP	Transbond Plus			
	Primer	Transbond Plus Self Etching Primer	269689C	3M Unitek
	Adhesive	Transbond XT	7AX	
	Prompt L-Pop			
	Primer	Prompt L-Pop	307461	3M ESPE, Seefeld, Germany
	Adhesive	Transbond XT	7AX	3M Unitek

[a] CE indicates conventional etching; SEP, self-etching primer.

determine the influence of the orthodontic adhesive systems such as conventional acid-etching (CE) and SEP on the stain susceptibility of the enamel surface after debonding and clean-up (finishing only [F-condition] and finishing/polishing [FP-condition]) procedures and (2) determine the effects of clean-up procedures on the enamel surface morphology using a three-dimensional (3D) profilometer.

MATERIALS AND METHODS

Tooth Specimen Preparation

Two types and four brands of adhesive systems were studied (Table 1), and 135 human premolars extracted for orthodontic reasons were used as substrates. Teeth were selected based on visual evaluation for soundness of crown and stored in a 70% ethanol solution at room temperature before use.[18]

The root was removed with a disk and the crown portion was embedded in a resin block (20 × 20 × 20 mm acrylic resin block with a hole of 13 mm in diameter and 5 mm in depth at the center) with an acrylic resin to expose the labial surface. After cleaning with water, the exposed tooth surface was polished with nonfluoride pumice with a rubber cup for 10 seconds, rinsed with water, and dried with compressed air.[18]

Determination of Remaining Adhesive

The labial surface of the tooth was scanned with a 3D profilometer (MTS, St Paul, Minn) at each of three steps—before bracket bonding (baseline), after debonding, and after clean-up procedures for the area of 5 mm in the occlusogingival direction and 7 mm in the mesiodistal direction. The interval for each line scan (scan across mesiodistal direction) was 100 μm, and the number of points in each line scan was 70 at 10-μm intervals.[19]

Three scanned surfaces for each tooth were overlapped using a software (Ansur 3, Minnesota Dental Research Center for Biomaterials and Biomechanics, Minneapolis, Minn) to calculate the volume changes (mm³) between each pair of measurements[19]; therefore, quantifications of the remaining adhesive and enamel loss were possible.[20]

Color Measurement

The color of the natural enamel surface was measured before bracket bonding (baseline). Color was also measured after methylene blue staining following debonding and F-condition or debonding and FP-condition. In the control group, the same staining was performed, and the color was measured again. Measurement was made after blot drying[16] according to the CIELAB (Commission Internationale de l'Eclairage) scale relative to the CIE standard illuminant D65 on a reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan) with the specular component excluded geometry. Illuminating and viewing configuration was CIE diffuse/10° geometry, and the aperture size of the measuring port was 3 mm.[21] In this system, color is expressed in terms of CIE L^*, a^*, and b^* color coordinates. The L^* coordinate corresponds to the degree of lightness and darkness, and the a^* and b^* coordinates correspond to the chroma, where $+a^*$ is red, $-a^*$ is green, $+b^*$ is yellow, and $-b^*$ is blue. Chroma was calculated as $C^*_{ab} = (a^{*2} + b^{*2})^{1/2}$. Color difference ($\Delta E^*_{ab}$) between two colors was calculated as follows[22]:

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

Bracket Bonding and Debonding Procedures

Teeth specimens were randomly divided into 9 groups of 15 teeth (Table 2). A metal bracket for

Table 2. Groups Investigated in This Study[a]

Code	Finishing (F)	Polishing (P)
Control	No treatment	
Transbond-*F*	O	X
Transbond-*FP*	O	O
Ortho Solo-*F*	O	X
Ortho Solo-*FP*	O	O
Transbond Plus-*F*	O	X
Transbond Plus-*FP*	O	O
Prompt L-Pop-*F*	O	X
Prompt L-Pop-*FP*	O	O

[a] O indicates performed; X, not performed.

Table 3. Amount of Volume Changes (mm³) After Debonding and Clean-up Procedures[†]

Group	Volume of Remaining Adhesive Resin After Debonding (mm³) Mean (SD)	Enamel Loss After Clean-up (Finishing or/and Polishing) (mm³) Mean (SD)
Ortho Solo-*F*	2.296 (0.499)[a]	−0.143 (0.038)[c]
Ortho Solo-*FP*	2.399 (0.488)[a]	−0.183 (0.069)[c]
Prompt L-Pop-*F*	0.835 (0.397)[b]	0.004 (0.041)[d]
Prompt L-Pop-*FP*	1.052 (0.376)[b]	−0.047 (0.044)[d]

[†] Same letters demonstrate no statistically significant differences based on Scheffe multiple comparison test ($P > .05$).

maxillary premolars (Gemini, 0.018-inch twin, 3M Unitek, Monrovia, Calif) was bonded on the labial surface of the tooth following the protocols recommended by the manufacturers. For the control group, the bracket was not bonded.

For CE groups, the enamel surface was etched with 32% phosphoric acid (UNI-ETCH, Bisco, Schaumburg, Ill). For the Transbond-*F* and Transbond-*FP* groups, adhesive primer (Transbond XT) was applied on the etched enamel surface, and adhesive (Transbond XT) was applied on the bracket base. For the Ortho Solo-*F* and Ortho Solo-*FP* groups, adhesive primer (Ortho Solo) was applied on the etched enamel surface, and adhesive (Blugloo) was applied on the bracket base (Table 1).

For the Transbond Plus-*F* and Transbond Plus-*FP* groups, the enamel surfaces were pretreated with SEP (Transbond Plus), and adhesive (Transbond) was applied on the bracket base. For the Prompt L-Pop-*F* and Prompt L-Pop-*FP* groups, the enamel surfaces were pretreated with SEP (Prompt L-Pop), and adhesive (Transbond XT) was applied on the bracket base.

After that the bracket was pressed firmly on the enamel surface, and excess adhesive resin was removed with a probe. Light curing was performed for 10 seconds from mesial and distal sides, respectively, with a LED unit (Elipar FreeLight 2, 3M ESPE, St Paul, Minn) with an intensity setting of 600 mW/cm². Specimens with bonded bracket were stored in 37°C distilled water for 24 hours. Debonding was performed with a universal testing machine (Model 4465, Instron, Canton, Mass) with a cross head speed of 1 mm/min.[19]

Clean-up Procedures

For the F-condition, residual adhesive resin was removed with a 12-fluted tungsten carbide bur (Orthocare, Bradford, UK)[3,4] using a light unidirectional brush-stroke at low speed,[23] without water cooling. Removal of residual adhesive resins was considered complete when the enamel surface seemed smooth and free of

resin to the naked eye, under the light of the operatory lamp.[24]

The FP-condition included the same procedure as the F-condition followed by polishing with a rubber cup (Shinhung, Seoul, Korea) with pumice slurry (Dentsply, Milford, Del)[25] with water for 30 seconds.

Staining Procedure

For staining, the clean-up specimens were immersed in a 37°C, 0.5% methylene blue solution for 1 hour, and rinsed with distilled water for 10 seconds and blot dried.[21] Color measurement with the same protocols for the unbonded enamel surface was performed.

Statistical Analysis

Means and standard deviations were calculated. The amount of adhesive remnant, color coordinates before staining, and changes in color coordinates after staining were analyzed using one-way analysis of variance (ANOVA) by group, including the brand of adhesives and clean-up procedures. Scheffe multiple comparison test was performed at the .05 level of significance.

RESULTS

The amount of remnant on the enamel surface was significantly influenced by group (Table 3) ($P < .05$). CE material (Ortho Solo) left more adhesive resin on the enamel surface than SEP material (Prompt L-Pop) after debonding, and enamel loss in CE material was bigger than SEP material regardless of the clean-up procedures. Typical enamel surfaces before bonding, after debonding, and after finishing procedures are presented in Figure 1.

Ranges of the each color coordinate of enamel surface before bonding were as follows: CIE *L**, 45.0 to 50.3; CIE *a**, −1.0 to −1.5; CIE *b**, 1.4 to 3.4; and CIE C* *ab*, 2.6 to 3.9. There were no significant differences in these values among the groups (Table 4).
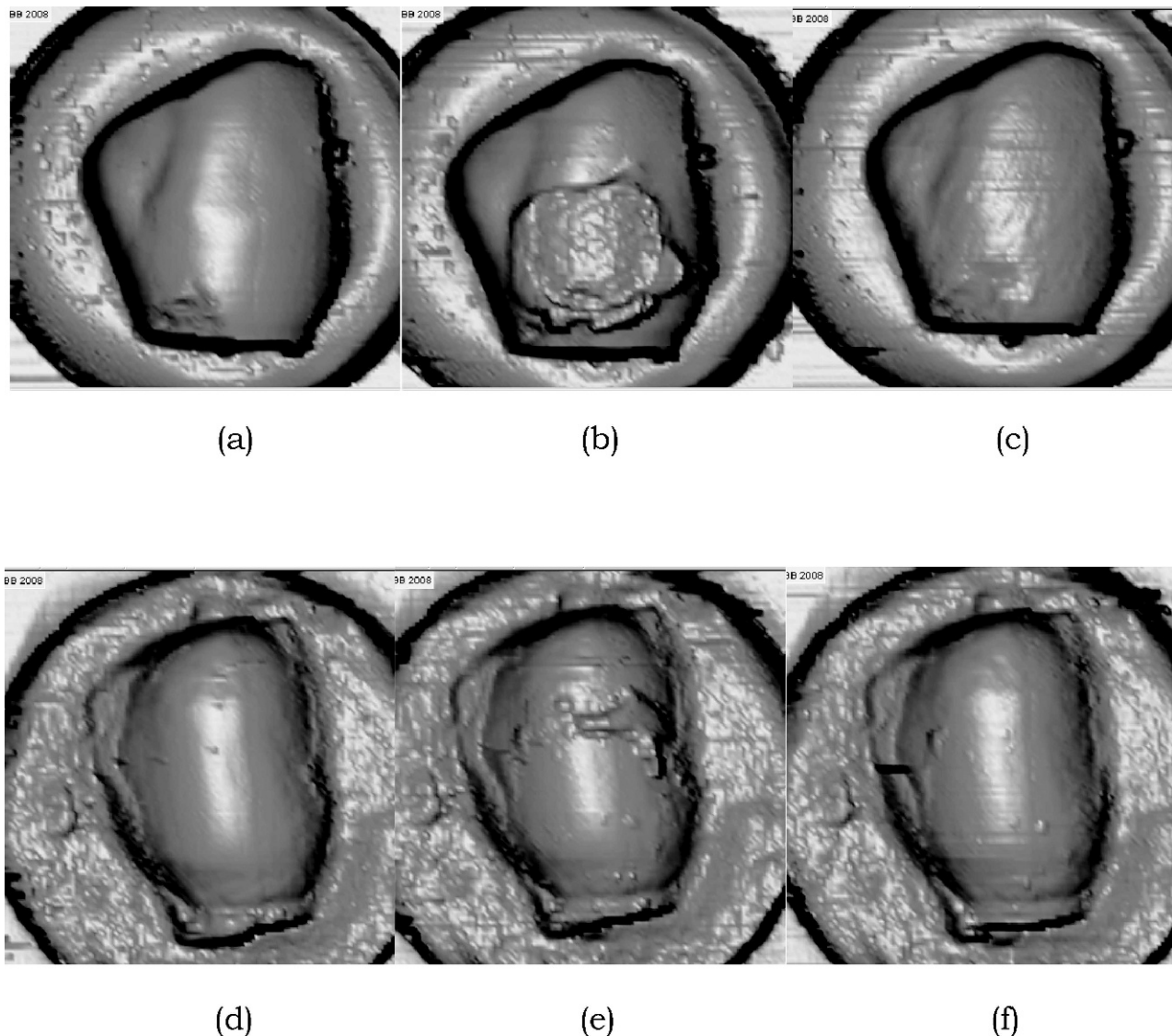
Figure 1. Tooth surface scanned with a 3D profilometer (MTS). (a,d) Before bracket bonding. (b) Ortho Solo, after debonding. (c) Ortho Solo, after finishing (Ortho Solo-*F*). (e) Prompt L-Pop, after debonding. (f) Prompt L-Pop, after finishing (Prompt L-Pop-*F*).

Changes in color and color coordinates after staining procedures are listed in Table 5. Comparing the F-condition groups, color changes (ΔE^*_{ab}) for SEP materials (15.8 and 17.6) were significantly higher than those of CE materials (10.0 and 11.2) ($P < .05$). Comparing the FP-condition groups, there was no significant difference in the color changes among the four experimental groups ($P > .05$).

The CIE *L** value decreased (shifted to dark direction) and the chroma value increased (became more chromatic) in all of the groups after staining ($P < .05$). The CIE *a** value decreased (shifted to green direction), and the CIE *b** value also decreased (shifted to blue direction) after staining in all of the groups ($P < .05$).

DISCUSSION

Previous studies have shown that SEP materials exhibited a more conservative etching pattern and thereby reduced enamel dissolution[3,5,6] and yielded shorter resin tags than CE materials.[6,10] Resin remaining on the enamel surface and increased roughness of the enamel surface after debonding could cause enamel discoloration.[5,27,28]

In the present study, SEP materials showed higher stain susceptibility than CE materials in the F-condition, but there was no significant difference between the two adhesive systems in the FP-condition. Higher stain susceptibility of SEP materials in the F-condition might have been caused by the greater

Table 4. Mean CIE *L*, a*, b** and Chroma of Natural Teeth Before Bonding[†]

Code	*L** Mean (SD)	*a** Mean (SD)	*b** Mean (SD)	*C*ab* Mean (SD)
Control	45.79 (4.32)	−0.95 (0.56)	2.90 (1.81)	3.38 (1.19)
Transbond-*F*	47.94 (3.97)	−1.14 (0.88)	2.22 (2.62)	3.39 (1.53)
Transbond-*FP*	45.16 (4.33)	−1.19 (0.47)	2.63 (1.62)	3.04 (1.39)
Ortho Solo-*F*	48.30 (3.01)	−0.95 (0.51)	1.74 (1.51)	2.59 (1.04)
Ortho Solo-*FP*	49.86 (3.29)	−1.42 (0.42)	1.37 (1.86)	2.92 (0.65)
Transbond Plus-*F*	44.97 (3.44)	−0.99 (0.68)	3.22 (2.40)	3.74 (1.86)
Transbond Plus-*FP*	46.79 (3.63)	−1.45 (0.32)	1.63 (2.01)	2.67 (1.42)
Prompt L-Pop-*F*	48.57 (4.18)	−0.97 (0.68)	3.41 (1.72)	3.93 (1.24)
Prompt L-Pop-*FP*	50.33 (3.08)	−1.25 (0.57)	1.81 (2.54)	3.04 (1.48)

[†] There were no significant differences between the groups in each of the color coordinates ($P > .05$).

amount of thin residual adhesive resin layers undetectable by the naked eye that remained after finishing. However, further studies such as element analysis should be performed to confirm this supposition.

In both adhesive systems, the F-condition showed significantly higher stain susceptibility than the FP-condition (Table 5). It was reported that the finishing and polishing treatment, compared with the finishing only treatment, showed lower surface irregularity.[29] In the present study, the amount of residual resin and volume changes of tooth surface were greater in CE than in SEP material ($P < .05$), and tooth surface in the FP-conditions showed greater enamel loss than that of the F-conditions (Table 3), though the changes were not significantly different.

Staining color changes ($\Delta E*_{ab}$) in the F-condition of CE materials were 11.2 (Transbond-*F*) and 10.0 (Ortho Solo-*F*), and those of SEP materials were 17.6 (Transbond Plus-*F*) and 15.8 (Prompt L-Pop-*F*) (Table 5). Therefore, SEP materials in the F-condition showed significantly higher staining than CE materials ($P < .05$). Generally, the F-condition enamel surface showed greater staining color changes than the control tooth. However, there was no significant difference between the control and the FP-condition of CE and SEP materials. These results imply that enamel

staining due to remaining adhesive resin in SEP materials was not observed in the FP condition, which indicates that the visually undetectable thin residual adhesive resin layer was almost completely removed during the polishing procedure.

Residual adhesive resins on enamel surfaces after debonding are cleaned up in a number of ways.[3,4,23,30] A spiral fluted tungsten carbide bur with a low-speed handpiece is reported to produce the finest scratch and the least enamel loss[3,4,23]; therefore, this method was used in the present study.

Polishing of the enamel surface after removal of residual adhesive resins was recommended to restore the enamel surface.[4,30] Polishing with a slurry of zirconium silicate on a rotating rubber cup[31] was reported to produce a morphologically acceptable enamel surface and minimize enamel loss. Therefore, in the present study polishing was done with a rubber cup and pumice. Polishing the enamel surface with pumice and a rubber cup removed about 10.7 µm of enamel surface.[32] Further research for the comparison of the amount of enamel loss after clean-up procedures between CE and SEP materials is recommended.

Color is important for the esthetics of the teeth, which results from volume scattering of light; illuminating light follows highly irregular light paths through

Table 5. Changes in Color, Color Coordinates, and Chroma After Staining Procedure[†]

Code	$\Delta E*_{ab}$ Mean (SD)	$\Delta L*$ Mean (SD)	$\Delta a*$ Mean (SD)	$\Delta b*$ Mean (SD)	$\Delta C*_{ab}$ Mean (SD)
Control	6.6 (1.6)[a]	−2.7 (1.0)[d]	−2.0 (0.5)[hi]	−5.6 (1.0)[j]	2.0 (1.5)[m]
Transbond-*F*	11.2 (1.6)[b]	−6.4 (1.9)[de]	−2.6 (0.8)[hi]	−8.8 (1.4)[jk]	5.2 (1.8)[m]
Transbond-*FP*	7.6 (1.3)[ab]	−5.6 (1.7)[de]	−1.3 (0.1)[i]	−5.1 (0.7)[j]	2.2 (1.0)[m]
Ortho Solo-*F*	10.0 (2.0)[ab]	−8.0 (2.7)[efg]	−2.4 (0.6)[hi]	−5.0 (2.0)[j]	2.6 (1.7)[m]
Ortho Solo-*FP*	7.2 (1.5)[ab]	−5.6 (1.7)[de]	−1.9 (0.6)[hi]	−3.8 (2.4)[j]	2.4 (1.4)[m]
Transbond Plus-*F*	17.6 (3.2)[c]	−12.0 (2.8)[g]	−2.5 (0.8)[hi]	−12.5 (3.1)[k]	6.1 (3.2)[m]
Transbond Plus-*FP*	8.8 (2.2)[ab]	−4.3 (2.1)[de]	−2.2 (0.5)[hi]	−7.7 (1.1)[jk]	5.9 (1.6)[m]
Prompt L-Pop-*F*	15.8 (3.6)[c]	−10.7 (3.7)[fg]	−3.5 (1.1)[h]	−11.0 (4.0)[k]	6.0 (3.3)[m]
Prompt L-Pop-*FP*	9.0 (1.9)[ab]	−6.9 (2.0)[def]	−1.8 (1.2)[i]	−3.7 (3.1)[j]	2.1 (1.6)[m]

[†] Same letters indicate no statistically significant differences based on Scheffe multiple comparison test ($P < .05$).

the tooth before it emerges at the surface of incidence and reaches the eye of the observer.[33] Light paths are determined by multiple lights scattering inside the tooth. After clean-up procedures, the refractive index of the enamel surface should have been changed, which might have influenced the diffusely reflected light. This phenomenon has influenced the color parameters of tooth because it was reported that tooth surface after debonding and clean-up was mainly composed of cut enamel prism infiltrated by resin tags, occupying the sites of enamel rods dissolved by acid-etching.[12] In the present study, although the influence of the changes in refractive indices after clean-up procedures was not determined, changes in color and color parameters after staining might include these changes.

Discoloration of tooth-colored, resin-based materials is caused by intrinsic and extrinsic factors. Extrinsic factors include staining by adsorption or absorption of colorants.[15] Enamel surface irregularities increased the opportunity for the retention of discolored integuments.[29] After clean-up procedures, the long-term presence of residual adhesive resin in the enamel surface that extends over the middle third rendered the instability of tooth color,[34] which occurred by direct absorption of exogenous colorants. Therefore, in the present study, methylene blue was used to determine stain susceptibility by resin tags in the enamel surface after clean-up procedures.

Since the sensitivity of the human eye in detecting small color differences is limited, and the interpretation of visual color comparisons is subjective, color measuring instruments are used to obtain reproducible results.[22] In the present study, color measurement was performed according to the CIELAB color scale relative to the CIE standard illuminant D65 on a reflection spectrophotometer. Generally, ΔE^{*}_{ab} values less than 1 unit are considered a color match since they cannot be identified by human observers.[35] Differences exceeding 2 ΔE^{*}_{ab} units may indicate color change,[26] but some studies set the proposed perceptible limit for color matching to 3.7 ΔE^{*}_{ab} units.[36] In the present study, enamel color changes in all groups were higher than the perceptible limit of color difference ($\Delta E^{*}_{ab} >$ 3.7, Table 3). However, it should be mentioned that these are experimental results, not clinical results.

Evaluation of the amount of remaining adhesive resin after debonding showed that SEP materials left less adhesive resin on the enamel surface than CE materials (Table 4). As to the enamel loss after clean-up procedures, SEP material showed less change than CE material regardless of the clean-up procedures. This fact would be another advantage for clinicians when removing the residual adhesive resin after debonding.[37]

CONCLUSIONS

- SEP material showed a smaller amount of residual adhesive resin after debonding than CE material.
- SEP material showed higher stain susceptibility than CE material when only the finishing procedure was performed, which might have resulted from a greater amount of residual adhesive resin not detectable by the naked eye in SEP material.
- Additional polishing resulted in similar staining susceptibility in SEP material compared with CE material.
- SEP would show less stain susceptibility if the residual adhesive resin layer were removed by a polishing step.

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