

# Usefulness of orthodontic adhesive-containing fluorescent dye

Yasuhiro Namura\*, Takashi Tsuruoka\*, Chiyako Ryu\*, Masahiro Kaketani\*\* and Noriyoshi Shimizu\*

Departments of \*Orthodontics and \*\*Dental Materials, Dental Research Center, Nihon University School of Dentistry, Tokyo, Japan

Correspondence to: Dr Yasuhiro Namura, Department of Orthodontics, Nihon University School of Dentistry, 1-8-13 Kandasurugadai, Chiyoda ward, Tokyo 101-8310, Japan. E-mail: namura-y@dent.nihon-u.ac.jp

**SUMMARY** Orthodontic adhesive is often left on the tooth surface when a multibracket appliance is debonded, and it is difficult to remove because its colour is similar to that of the tooth. If the adhesive changed colour during debonding, residual adhesive could be more easily removed. This *in vitro* study evaluated the usefulness of adhesive mixed with a small amount of fluorescent dye for clinical orthodontics.

Sixty-four metal brackets were bonded to flattened bovine enamel surfaces using adhesives with three concentrations (0.001, 0.002, and 0.003 per cent) of fluorescent dye, and the shear bond strength (SBS) and adhesive remnant index (ARI) scores for each adhesive were determined. Colour penetrating through the transparent bracket was measured using a colour analyser. SBS and fluorescence intensity were examined to determine the stability of the adhesives after they were subjected to a thermal cycle test (1000 cycles). For data that were normally distributed, one-way analysis of variance followed by the Student–Newman–Keuls test was used to identify significant differences among the groups. If the data were not normally distributed, the Kruskal–Wallis *H*-test followed by the Mann–Whitney *U*-test with Bonferroni correction was used. Differences in ARI were determined with the chi-square test.

The SBS of the adhesive with 0.003 per cent fluorescent dye was significantly lower than that of the control (Transbond). In ARI tests, significantly more of the adhesive with 0.003 per cent dye was left on the tooth surface after 24 hours compared with the other adhesives. With regard to colour penetration, the adhesive with 0.003 per cent dye was five times more visible than to others. SBS and fluorescence intensity of the adhesives were not affected by thermal cycling. Therefore, an adhesive containing less than 0.002 per cent fluorescent dye provides both sufficient bond strength for orthodontic brackets and sufficient fluorescent colour for easy visualization without aesthetic impairment.

## Introduction

The adhesives used in orthodontics and clinical dentistry have been improving since Buonocore (1955) introduced the principle of enamel etching. In modern orthodontic treatment, in almost all cases, light-cured adhesive is used for bracket bonding (Krishnaswamy and Sunitha, 2007). Although brackets adhere strongly because of the improved bonding adhesives used, removal of the brackets and the cured adhesive takes considerable time.

Some resin adhesive is often left on the tooth surface when the brackets are removed because the resin colour is similar to that of the tooth (Fields, 1982). Residual resin results in unaesthetic staining as well as caries at the boundary between the tooth and the residual resin (Zachrisson and Büyükyilmaz, 2005). To facilitate more complete removal of adhesive during bracket debonding, the focus of the present study was to find an adhesive that could be distinguished by a fluorescent colour inducible by visible light such as that from a dental curing unit. Although fluorescent dye has been applied to a visible bonding agent for crown restoration (Tay *et al.*, 2002), application of fluorescent dye to an orthodontic bracket adhesive has not

been reported. The aim of this study was to investigate the effect of incorporating fluorescent dye into a bracket adhesive that would make it possible to visualize the adhesive left on the tooth during bracket debonding without compromising the aesthetics of a clear bracket or decreasing shear bond strength (SBS).

## Materials and method

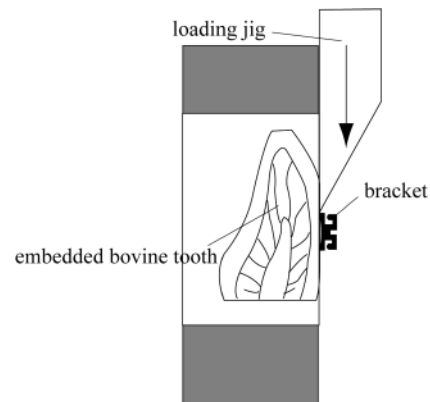
Adhesive paste (composed of dimethacrylates, photoinitiators, and fillers) containing three different concentrations (F1, 0.001 per cent; F2, 0.002 per cent; and F3, 0.003 per cent) of a coumarin-derived fluorescent dye (Nippon Kankoh Shikiso Kenkyusho, Okayama, Japan), which is excited by light with a wavelength of 380–540 nm, and self-etching primer (SP-2; Tokuyama dental, Tokyo, Japan) were used (Table 1). Because Transbond Plus Self Etching primer and XT paste (3M Unitek, Monrovia, California, USA) has been used in many studies and is a clear paste that has appropriate aesthetic qualities and possesses sufficient bond strength, it was selected as the control adhesive.

**Table 1** Composition and application protocol of the materials used in the present experiment.

Group	Concentration of fluorescent dye (%)	Components	Pretreatment of tooth surface	Application protocol
F1	0.001	Base resins (Bisphenol A glycidyl ether dimethacrylate, Triethylene glycol dimethacrylate, etc.)	SP-2 (prototype self-etching primer, Lot No. 070522; Tokuyama dental, Tokyo, Japan)	The tooth surface was treated with primer for 3–5 seconds, dried with moisture-free air and adhesive paste was applied to the bracket base. The bracket was pressed onto the tooth surface, and then the adhesive was light cured from each interproximal side of the bracket for 10 seconds (total curing time, 20 seconds)
F2	0.002	Photo initiators (Camphor quinone, etc.)		
F3	0.003	Coumarin derivatives fluorescent dye		
Control (Transbond XT paste, Lot No. 6WY; 3M Unitek, Monrovia, California, USA)	—	Silica filler Bisphenol A glycidyl ether dimethacrylate Bisphenol A bis (2-hydroxyethyl ether) dimethacrylate Silane-treated quartz Silane-treated silica	Transbond Plus Self-etching primer (Lot No. 228883A; 3M Unitek)	
			35 wt% 65 wt% 10–20 wt% 5–10 wt% 70–80 wt% Less than 2%	

### SBS testing

The specimens for SBS testing were prepared according to the method of Yamamoto *et al.* (2006) using bovine enamel. Briefly, the separated crowns of the bovine teeth were embedded in acrylic resin, and their enamel surfaces were treated with wet 600-grit silicon carbide paper to form a flat bonding surface. The bovine teeth ( $n = 64$ ) used in bond testing were randomly divided into four groups (F1, F2, F3, and control) of 16 teeth each, and each group was subsequently divided into two groups (to be tested immediately or 24 hours after curing) of eight teeth each. Self-etching primer was applied to the prepared flat enamel surface for 3–5 seconds using a disposable applicator and was then gently evaporated in air. The adhesive paste was applied to the base of a metal bracket (bracket base area, 15.26 mm<sup>2</sup>; New DynaLock; 3M Unitek) for the maxillary central incisors, and the bracket was pressed firmly onto the enamel surface. Excess paste was removed from around the base of the bracket, and the adhesive was cured with a light-emitting diode (light intensity, 400 mW/cm<sup>2</sup>; Ortholux; 3M Unitek) for 10 seconds on each interproximal side (total curing time, 20 seconds). After bonding, the specimens were measured immediately or were stored in distilled water at 37°C for 24 hours and then measured. Clinically, a wire is placed immediately after the brackets are bonded. However, the orthodontic adhesive exhibited increased bonding strength over a short time (e.g. 24 hours after bonding) due to maturation of the material (Yamamoto *et al.*, 2006). Each specimen was tested in shear mode (static load cell:  $\pm 1$  kg N) using a universal testing machine (5567; Instron, Norwood, Massachusetts, USA) at a crosshead speed of 1 mm/minute (Figure 1). SBS values were calculated based on the peak load at failure divided by the bracket area (15.26 mm<sup>2</sup>), measured with a sliding calliper (NTD12P-15C; Mitutoyo, Kawasaki, Japan). All tests were conducted at  $23 \pm 1^\circ\text{C}$  and  $50 \pm 5$  per cent relative humidity. The SBS of the control group was measured under the same conditions.



**Figure 1** Schematic of the shear bond test.

### Adhesive remnant index

After determination of the SBS, each specimen was examined under an optical microscope (SZH-131; Olympus, Tokyo, Japan) at  $\times 16$  magnification to identify the location of the bond failure. The residual adhesive on each tooth was assessed using the adhesive remnant index (ARI; Årtun and Bergland, 1984). The ARI of the control group was examined under the same conditions.

### Colour penetration test

To ensure maintenance of the aesthetic bracket colour, which might be impaired if the fluorescent dye colour was perceptible through a clear bracket, a colour penetration test was carried out.

Medium-sized clear brackets for maxillary central incisors made of monocrystalline alumina (Inspire; Ormco, Orange, California, USA) were used because these brackets provide greater optical clarity compared with polycrystalline brackets (Liu *et al.*, 2005). Eighteen brackets were divided into three groups (F1, F2, and F3) of six brackets each and bonded onto white acrylic plates using the respective adhesives. The brackets were then embedded in transparent epoxy resin, which was adjusted to be level with the height of the bracket to closely touch the aperture of the colour analyser. The final finish was accomplished by grinding the top surface of the epoxy resin with 2000 grit silicon carbide paper (Figure 2). For each specimen, the colour value according to the Commission International de l'Eclairage (1978),  $L^*a^*b^*$  colour system was measured using a colour analyser (TC-1800MKII; Tokyo Denshoku, Tokyo, Japan) without visible light irradiation. The  $L^*$  value (from 0 to 100) represents lightness and  $a^*$  (from -100 to 100) and  $b^*$  (from -100 to 100) are the chromatic coordinates of the green-red and blue-yellow axes, respectively. The colour difference  $E_{ab}^*$  was calculated to compare the difference in colour penetration among the specimens using the following equation (Arikawa

*et al.*, 2007):  $\Delta E_{ab}^* = [(L^*)^2 + (a^*)^2 + (b^*)^2]^{1/2}$ , where  $L^*$ ,  $a^*$ , and  $b^*$  are the differences in value compared with the control group (Transbond).

### Thermal cycle testing

To confirm the stability of SBS and the intensity of fluorescence, thermal cycling was performed. Adhesive paste (diameter, 3 mm; thickness, 0.15 mm) was applied to bovine teeth and light cured. The brackets were bonded to specimens using the same procedure as for the SBS test. These specimens were subjected to continuous thermal cycling for 1000 cycles between 4 and 60°C in a water bath with a 30-second dwell time in each bath. After thermal cycling, SBS tests were performed, and fluorescence intensity was measured.

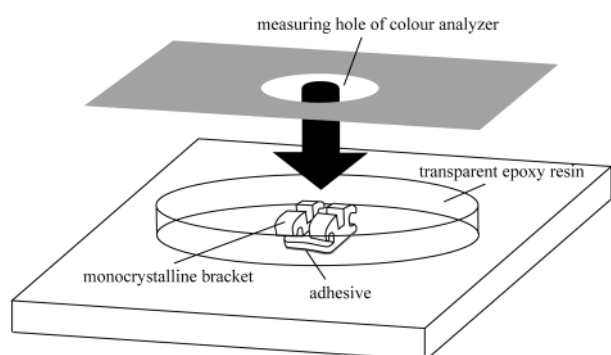
### Statistical analysis

Descriptive statistics, including the mean, standard deviation, and minimum and maximum values, were calculated for the bond strength and colour value. The Kolmogorov-Smirnov normality test was applied to the bond strength data and the colour difference  $E_{ab}^*$  data. If the data showed normal distribution, one-way analysis of variance was used followed by the Student-Newman-Keuls test to identify significant differences among the groups, and if the data did not have a normal distribution, the Kruskal-Wallis  $H$ -test followed by the Mann-Whitney  $U$ -test with Bonferroni correction was used. The chi-square test was used to analyse differences in the distribution of the ARI scores. The significance level for all statistical tests was  $\alpha = 0.05$ .

## Results

The SBS of the adhesive for each dye concentration is shown in Table 2. The bond strength of F3 was significantly less ( $P < 0.05$ ) than that of the control, but no significant difference in bond strength was observed between F1, F2, and the control. After 24 hours, the ARI distribution of the residual F3 adhesive differed significantly from that of the control and the F1 and F2 adhesives (Table 3). The results of SBS testing showed that bond strengths after thermal cycle testing were  $7.0 \pm 1.2$  MPa for F1 and  $6.3 \pm 2.4$  MPa for F2, and the bond strengths of the F1 and F2 adhesives were not significantly different from that of the control adhesive.

Regarding colour penetration through the brackets, a yellow-green colour was clearly visible with the F3 adhesive, as evidenced by the low value of  $a^*$  (-5.19) and the high value of  $b^*$  (12.66), but this colour was not visible in the F1 or F2 adhesive (Figure 3). The  $E_{ab}^*$  of F3 was significantly greater ( $P < 0.05$ ) than that of the other two concentrations (Table 4 and Figure 4). The fluorescence intensity was



**Figure 2** Brackets were bonded on a white acrylic plate using each adhesive and were embedded in transparent epoxy resin. The epoxy resin was adjusted to be level with the height of the bracket to closely touch the aperture of the colour analyser. The final finish was accomplished using 2000 grit silicon carbide paper to grind the top surface of the epoxy resin.

**Table 2** Shear bond strength at each concentration of fluorescent dye.

Group (MPa)	Sample size	Immediate measurement					After 24 h					After thermal cycle testing			
		Mean	Standard deviation	Minimum	Maximum	95% Confidence interval (lower)	95% Confidence interval (upper)	Mean	Standard deviation	Minimum	Maximum	95% Confidence interval (lower)	95% Confidence interval (upper)	Mean	Standard deviation
F1	8	7.2	1.2	4.8	8.6	6.2	8.2	17.0	3.6	12.6	21.1	14.0	20.0	7.0	1.2
F2	8	6.6	1.0	4.8	8.0	5.8	7.5	16.2	3.2	11.5	22.3	13.6	18.9	6.3	2.4
F3	8	5.2*	0.6	4.6	6.3	4.7	5.7	9.3*	2.2	6.3	12.3	7.5	11.1	—	—
Control	8	7.3	1.2	6.1	9.3	6.3	8.3	18.6	4.4	13.9	25.9	15.0	22.3	—	—

Adhesive containing 0.001% F1, 0.002% F2, and 0.003% F3 fluorescent dye.

\*The value of F3 is significantly ( $P < 0.05$ ) less than that of the other concentrations and the control.

**Table 3** Adhesive remnant index (ARI) at each measurement time ( $n = 8$ ).

Group	Immediate measurement				After 24 h			
	ARI scores				ARI scores			
	0	1	2	3	0	1	2	3
F1	6	2	0	0	8	0	0	0
F2	7	1	0	0	8	0	0	0
F3	1	7	0	0	1	3	3	1
Control	4	3	1	0	8	0	0	0
$\chi^2$ value	14.0513 ( $P = 0.1211$ )				26.88 ( $*P = 0.0015$ )			

ARI scores: 0, no adhesive left on tooth surface; 1, less than 50 per cent of adhesive left on tooth surface; 2, more than 50 per cent of adhesive left on tooth surface; and 3, all adhesive left on the tooth surface. Adhesive containing 0.001% F1, 0.002% F2, and 0.003% F3 fluorescent dye.

\*For ARI measured at after 24 h there is significant difference (F3 adhesive). There is no significant difference in immediate measurement.

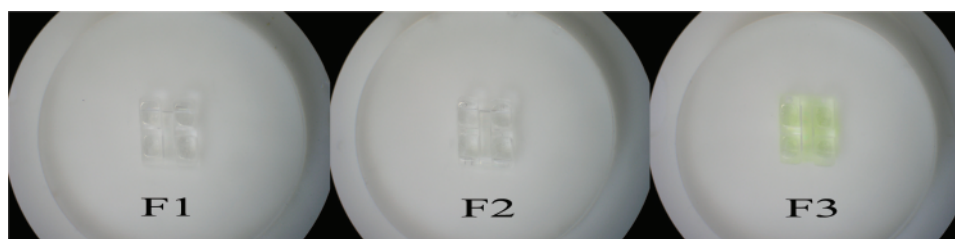
maintained after 1000 cycles in the thermal cycle test (Figure 5).

## Discussion

In this study, the bond strength of the adhesive with 0.003 per cent fluorescent dye (F3) was significantly less than that of the Transbond adhesive, which has been widely used as a control (Gibb and Katona, 2006; Bishara *et al.*, 2007, 2008; Chalgren *et al.*, 2007; Vicente and Bravo, 2007). With regard to SBS, Yamamoto *et al.* (2006) reported that Transbond adhesive with self-etching primer had a SBS of 6.5 MPa 5 minutes after bonding and 10.4 MPa 24 hours after bonding. In the study of Fjeld and Øgaard (2006), the SBS of Transbond paste with self-etching primer was 8.8 MPa 5 minutes after bonding and 11.0 MPa 15 minutes after bonding. Kitayama *et al.* (2007) reported an SBS of 17.6 MPa 24 hours after bonding. In present study, the Transbond control had an SBS of 7.3 MPa immediately after bonding and 18.6 MPa 24 hours after bonding, similar to the results of previous studies. Because the F3 adhesive had a SBS as of 5.2 MPa and 9.3 MPa immediately after and 24 hours after bonding, respectively, it is doubtful that bond strength would be sufficient for clinical application.

Reynolds (1979) suggested that a minimum bond strength of 6–8 MPa is adequate for most clinical orthodontic needs, as this strength is sufficient to withstand masticatory and orthodontic forces. However, those values cannot be compared with the bond strengths in the present study because the method of testing bond strength differed. Furthermore, this study used bovine teeth, which are easily obtained, and the bond strength would be likely to differ from that measured using human enamel, which has been reported to be 21–44 per cent stronger (Oesterle *et al.*, 1998). Thus, caution must be exercised regarding the





**Figure 3** Colour penetration through a bracket. Fluorescent dye concentration of adhesives: F1, 0.001; F2, 0.002; and F3, 0.003 per cent.

**Table 4** Value of colour penetration through a bracket.

Group	Sample size		Median	Mean	Standard deviation	Minimum	Maximum	95% Confidence interval (lower)	95% Confidence interval (upper)
F1	6	$L^*$	63.88	63.44	1.42	60.73	64.83	61.94	64.93
		$a^*$	-1.04	-1.03	0.04	-1.06	-0.95	-1.07	-0.98
		$b^*$	2.08	2.07	0.17	1.87	2.33	1.89	2.25
		$\Delta E_{ab}^*$	1.64	1.88	0.53	1.46	2.73	1.33	2.43
F2	6	$L^*$	65.06	65.01	0.57	64.07	65.77	64.41	65.60
		$a^*$	-1.09	-1.13	0.22	-1.56	-0.92	-1.37	-0.90
		$b^*$	1.73	1.82	0.44	1.40	2.66	1.36	2.28
		$\Delta E_{ab}^*$	2.33	2.34	0.27	1.97	2.80	2.06	2.63
F3	6	$L^*$	65.61	65.42	1.12	63.46	66.60	64.25	66.59
		$a^*$	-5.31	-5.26	0.35	-5.63	-4.62	-5.63	-4.89
		$b^*$	12.90	12.76	0.99	11.23	13.76	11.72	13.79
		$\Delta E_{ab}^*$	13.22	13.11	0.92	11.73	14.19	12.14	14.08
Control	5	$L^*$	63.48	63.15	0.57	62.32	63.58	62.44	63.85
		$a^*$	-0.24	-0.24	0.04	-0.30	-0.19	-0.290	-0.18
		$b^*$	0.98	0.92	0.15	0.69	1.04	0.73	1.10

Adhesive containing 0.001% F1, 0.002% F2, and 0.003% F3 fluorescent dye.  $L^*$  indicates lightness and  $a^*$  and  $b^*$  are chromatic coordinates of the green-red and blue-yellow axes. All  $\Delta E_{ab}^*$  values were calculated with the following formula, after the mean values of the control ( $L^* = 63.15$ ,  $a^* = -0.24$ , and  $b^* = 0.92$ ) were subtracted from those of each adhesive:  $\Delta E_{ab}^* = [(L^*)^2 + (a^*)^2 + (b^*)^2]^{1/2}$

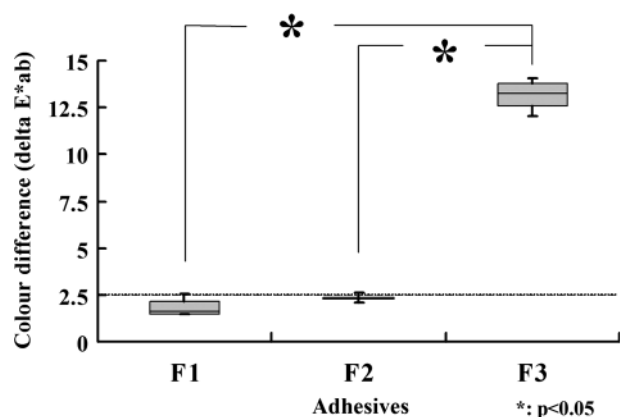
clinical relevance of the results of this *in vitro* study, even though a commercial orthodontic adhesive (Transbond) was used as the control.

The ARI scores demonstrated that a significant amount of the F3 adhesive remained on the tooth surface 24 hours after bonding compared with the residual amounts of F1, F2, and the control. Thus, it is likely that the F3 adhesive differed in some respects from the others. With metal brackets, the ARI scores of the F1 and F2 adhesives were 0 at 24 hours after bonding. However, as Ozcan *et al.* (2008) demonstrated, polycarbonate or ceramic brackets may leave more adhesive on the tooth surface when they are debonded, and the remaining adhesive is not readily removed.

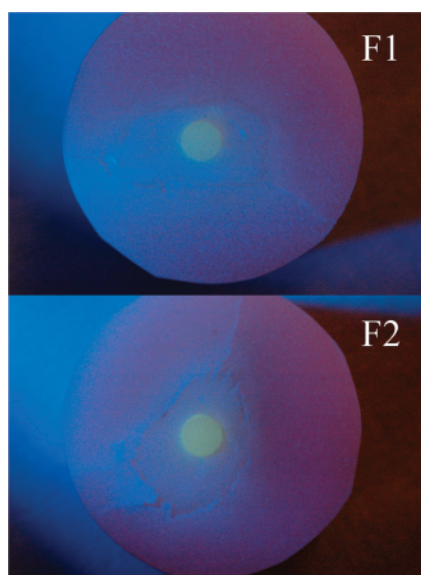
To evaluate colour penetration through the bracket, the colour difference method was used (Figure 3 and Table 4). The  $L^*$  value represents lightness and  $a^*$  and  $b^*$  are chromatic coordinates of the red-green and yellow-blue axes, respectively (Eliades *et al.*, 2004; Hosoya *et al.*, 2006). In the present study, the  $a^*$  value of F3 was less than that of F1 or F2, while the  $b^*$  value was greater. The yellow-green colour of F3 showed strongly through the brackets. These results are supported by the finding that the  $\Delta E_{ab}^*$  of the F3

adhesive, but not of the F1 and F2 adhesives, was greater than 2.5 (or 3.7) of the  $\Delta E_{ab}^*$  minimum acceptance limit. Generally,  $\Delta E_{ab}^*$  values in the range of 1 unit are considered an exact colour match because they cannot be perceived by independent observers, and most studies set the proposed acceptance limit for colour matching to 2.5 (3.7 units), beyond which the differences are clinically visible (Eliades *et al.*, 2004; Hosoya *et al.*, 2006). Thus, the F1 and F2 adhesives, as seen through clear brackets, would match the colour of the tooth surface and would not create an aesthetic problem.

This study focused on a fluorescent dye that has already been used as a bonding agent in general dentistry (Tay *et al.*, 2002) and has been shown to be safe. The remaining F1 and F2 adhesives were not detected on the tooth surface without irradiation by visible light; however, these adhesives were easily seen during irradiation with visible light (Figure 6). Fluorescence occurs when a molecule or quantum dot relaxes to its ground state after being electronically excited by electromagnetic radiation (light). According to the manufacturer, the fluorescent dye used in this experiment is excited by light with wavelengths between 380 and 540 nm.



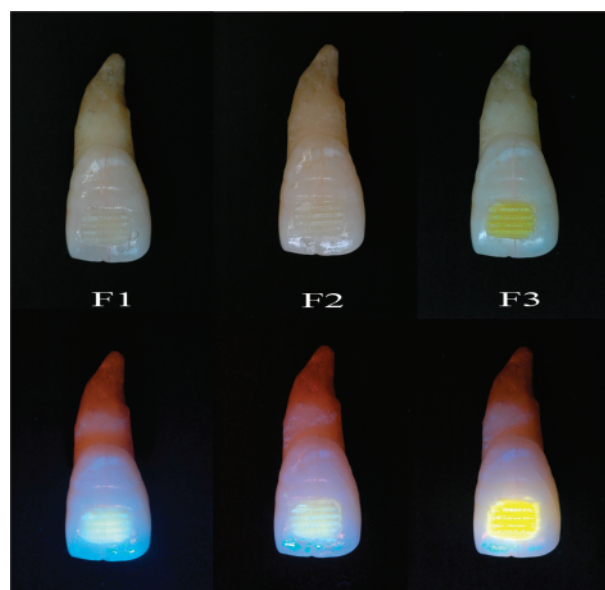
**Figure 4** Colour difference ( $\Delta E^*_{ab}$ ). Values of all ( $\Delta E^*_{ab}$ ) were calculated using the formula ( $\Delta E^*_{ab} = [(L^*)^2 + (a^*)^2 + (b^*)^2]^{1/2}$ ) after subtracting the mean values for the control ( $L^* = 63.15$ ,  $a^* = -0.24$ , and  $b^* = 0.92$ ) from those of each fluorescent adhesive. The  $\Delta E^*_{ab}$  of the F3 adhesive is greater than 2.5 (broken line), which is the limit above which independent observers can perceive mismatch of colours, and is significantly greater ( $P < 0.05$ ) than that of the other adhesives with other concentrations of dye.



**Figure 5** The fluorescence of the adhesives during light irradiation after thermal cycle testing (1000 cycles). The adhesives were selected based on the results of the shear bond strength testing, adhesive remnant index, and colour penetration tests.

This excitation wavelength range corresponds well with that typically emitted from a dental light-curing unit (~390–530 nm; *Staudt et al., 2005*). Thus, an adhesive containing this dye could be readily distinguished by irradiation with a dental light-curing unit without the need for a special apparatus, making this dye an appropriate choice for clinical use in orthodontic adhesives.

Active orthodontic treatment takes 2 years, and therefore, the SBS and intensity of fluorescence of an



**Figure 6** Typical images of remaining adhesive-containing fluorescent dye. Upper: without visible light irradiation, the F1 and F2 adhesives were not detected on the tooth surface. Lower: remaining adhesive irradiated with visible light. All the adhesives were excited by irradiation and were clearly distinguishable from the tooth surface.

orthodontic-adhesive-containing fluorescent dye should be maintained after 1000 cycles of thermal cycle test, as they were in F1 and F2 (Table 2 and Figure 5). Although the F1 and F2 adhesives were not evaluated, they would be quite likely to maintain sufficient bond strength and intensity of fluorescence during active orthodontic treatment.

## Conclusions

Based on the bond strength and colour penetration through clear brackets of the adhesives containing 0.001–0.003 per cent fluorescent dyes, it can be concluded that less than 0.002 per cent of fluorescent dye mixed with adhesive results in sufficient bond strength for orthodontic brackets, while providing sufficient fluorescent colour for easy visualization without aesthetic impairment.

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