



## Shear Bond Strength and Excess Adhesive Surface Topography of Different Bonding Systems after Thermocycling: A Comparative *In-vitro* Study

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### ABSTRACT

**Background:** To evaluate shear bond strength (SBS) and surface topography of excess adhesive of the recently introduced APCTM Flash-Free (FF) bracket-bonding system. **Material and Methods:** Sixty extracted human upper first premolars were randomly divided into five groups (12 per group), and used for this *in vitro* study: group 1, APCTM Flash-Free adhesive coated appliance system; group 2, APC<sup>TM</sup> Plus adhesive coated appliance system; group 3, APC<sup>TM</sup> II adhesive coated appliance system; group 4, manual application of adhesive bracket bonding system (Conven. 1) (with the use of Transbond<sup>TM</sup> XT Primer); group 5, manual application of adhesive bracket bonding system (Conven. 2) (with the use of Transbond<sup>TM</sup> Plus Self Etching Primer). Bond strength was measured using an Instron machine at a cross-head speed of 1 mm/min. The excess adhesive was evaluated with stereomicroscopic. **Results:** There were highly significant differences among all groups using ANOVA F-test; furthermore, there were no significant differences in (SBS) between APC<sup>TM</sup> Flash-Free group and the remaining groups, except with APC<sup>TM</sup> II group, and the mean shear bond strength were 12.633, 13.578, 15.080, 13.852, and 11.143 MPa respectively. The adhesive excess, which was metrically measured from the bracket edge, ranged from 206.245  $\mu$ m to 75.488  $\mu$ m APC<sup>TM</sup> Flash-Free group, 530.258  $\mu$ m to 155.750  $\mu$ m APC<sup>TM</sup> Plus group, 522.329  $\mu$ m to 188.681  $\mu$ m APC<sup>TM</sup> II group, 519.562  $\mu$ m to 157.151  $\mu$ m manual application Conven.1 group, and 508.449  $\mu$ m to 162.208  $\mu$ m manual application Conven.2 group, so there were high significant difference between APC<sup>TM</sup> Flash-Free group and the remaining groups. **Conclusions:** With the use of APC<sup>TM</sup> Flash-Free adhesive bracket system there was no need to clean up excess adhesive, which simplifies the bracket-positioning process and facilitate a smooth and sufficient marginal surface of the adhesive, which clinically might improve reduction of plaque accumulation, with an optimum shear bond strength value for clinical routine use.

**Keywords:** Shear bond strength, Excess adhesive, Surface topography, Bonding systems

### INTRODUCTION

For decades, various orthodontic adhesive and bonding techniques have been developed and have been subjected to multiple *in vivo* and *in vitro* studies. In all techniques, there is often flash that remains around the bracket-tooth interface that needs to be removed. While complete removal is desirable, clinicians often leave flash after bracket placement [1], which exposes a rough composite surface that becomes a critical site for plaque accumulation [2-4], and development of white spot lesions, which diminishes the final esthetic outcome [5,6].

The company 3M Unitek (Monrovia, Calif) had developed a new APC<sup>TM</sup> Flash-Free bracket-adhesive system as an attempt to eliminate the need for excess adhesive (flash) removal, each bracket of this type is individually packaged with an optimal amount of adhesive pre-pasted on the bracket base by the manufacturer, allowing the practitioner to place the bracket and cure the composite without the need for flash removal.

The purposes of this study were to examine the different bracket-bonding systems (APC<sup>TM</sup> Flash-Free, APC<sup>TM</sup> Plus, APC<sup>TM</sup> II, and regular (Conventional 1 and Conventional 2) light cure adhesive of the same bracket company (3M Unitek)) in regard to shear bond strength (SBS) and surface topography of excess adhesive.

## MATERIALS AND METHODS

### Teeth

Sixty extracted human upper first premolars, stored in a solution of 0.1% (weight/volume) thymol. The inclusion criteria for tooth selection were intact buccal enamel, not subject to any chemical agents (such as hydrogen peroxide), no cracks, no caries, and free of hypoplastic area [7]. Each tooth was embedded into a cold-cure acrylic resin prior to orthodontic bonding, with coding of the acrylic blocks to get randomization. The teeth were polished with pumice and rubber cup for 10 seconds, then randomly divided into five groups (12 teeth per group).

### Brackets

All brackets which were used are from 3M Unitek, Monrovia, USA. Stainless steel, victory series™ low profile brackets of  $0.022 \times 0.030$  MBT™ system, with the base area of all brackets was  $9.08 \text{ mm}^2$ . The total number was sixty maxillary first premolar brackets, that were divided into five groups (12 brackets/group): (APC™ Flash-Free (FF), APC™ Plus, APC™ II, Conventional bracket (Conven. 1) with the use of Transbond™ XT Primer, Conventional bracket (Conven.2) with the use of Transbond™ Plus Self Etching Prim).

### Bonding Procedure

The teeth of the 1<sup>st</sup>, 2<sup>nd</sup>, 3<sup>rd</sup>, and 4<sup>th</sup> groups were etched with 37% phosphoric acid for 30 seconds, then washed for 20 seconds, and drying for 10 seconds [8-11]. After etching, thin uniform coat of primer (Transbond™XT Primer; 3M Unitek) were applied. While the 5<sup>th</sup> group was bonded with the Transbond plus self-etch primer (3M Unitek).

The brackets were bonded according to the following procedures:

#### APC™ Flash-Free Group

In this group, 3M APC™ Flash-Free adhesive coated bracket applied to the tooth with a constant force at the ideal occluso-gingival and mesio-distal position [12,13].

#### APC™ PLUS Group

In this group, 3M APC™ Plus adhesive coated bracket applied to the tooth with a constant force at the ideal occluso-gingival and mesio-distal position. Excess adhesive resin was removed with an explorer [13].

#### APC™ II Group

In this group, 3M APC™ II adhesive coated bracket applied to the tooth with a constant force at the ideal occluso-gingival and mesio-distal position, excess adhesive resin was also removed with an explorer.

#### Manual Bonding Groups (Group 4 and 5)

For both groups (Transbond™ XT Primer and Transbond™ Plus Self Etching Primer respectively). Transbond™ XT light cure adhesive paste (3M Unitek) applied onto a metal bracket base with a plastic instrument, and the bracket applied to the tooth with a constant force at the ideal occluso-gingival and mesio-distal position. Excess adhesive resin was also removed with an explorer.

All adhesive resin was polymerized for a total of 20 seconds with a visible light-curing unit (Three H XLITE-II Shenghi Industry; China) at an intensity of  $600 \text{ mw/cm}^2$ . After bonding, the teeth were stored in distilled water at  $37^\circ\text{C}$  for 24 hours to allow complete polymerization of the bonding material, then thermocycling was performed between  $50^\circ\text{C}$  and  $550^\circ\text{C}$  for 500 cycles. The thermocycling in deionized water baths was done manually following the recommendation of the (ISO/TR 11405: 1994), the exposure to each bath was 30 seconds, and the transfer time between the two baths was 5-10 seconds [14,15].

#### Shear Bond Strength Test

Shear test was accomplished using a Tinius-Olsen Universal testing machine (H50KT, England) with a 5 KN load cell and a crosshead speed of 1 mm/minute [16,17], and a custom made chisel rod (that was fitted inside the upper arm of the testing machine) was used to apply an occluso-gingival load at the enamel bracket interface, while the specimen was secured in the lower jaw of the testing machine. The force required to debond the brackets was measured in Newton (N), and the shear bond strength in Megapascals ( $1 \text{ MPa}=1\text{N/mm}^2$ ) was then calculated by dividing the force values (in Newton) by the bracket base area ( $9.08 \text{ mm}^2$ ) (Figure 1).

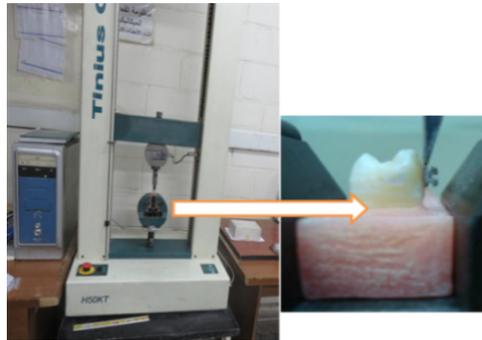


Figure 1 The sample fixed to the universal testing machine to test the shear bond strength

**Measurement of Surface Topography**

Conventional stereomicroscope (Hamilton, Italy) was used to evaluate the degree of excess adhesive (flash) in relation to bracket edge margin. Pictures were captured for every bracket under 10X magnification of the stereo-microscope. These pictures were used with special software (motic images advanced 3.2, Co., LTD, China) to measure the distance in (µm) between the bracket edge and the most/least leaked adhesive margin after measuring the magnification factor by scale bar considering for all the brackets and for every side of the brackets, multiple (minimum seepage adhesive/ maximum seepage adhesive) values were measured (Figure 2).

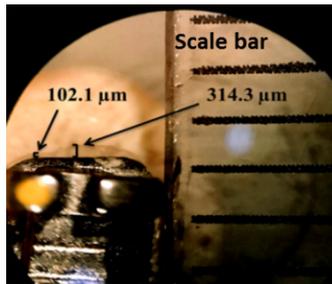


Figure 2 Measurement of surface topography

**Statistical Analysis**

All statistical tests and calculations were made using Statistical Package for Social Science software (SPSS for windows, 19.0, Chicago, USA). Maximum, minimum, mean values, and standard deviations were calculated as part of the descriptive analysis. Statistical significances were measured using a repeated-measure analysis of variance (ANOVA) to explore the differences between the five groups and Post hoc Tukey tests were performed to determine statistical significance between pairs of groups.

**RESULTS AND DISCUSSION**

Table 1 showed descriptive statistics and groups’ differences of the shear bond strength in different groups there were highly significant differences among all groups using ANOVA F-test. Also Post hoc Tukey’s HSD test was used to test the mean differences and showed that there were no significant differences in (SBS) between APCTM Flash-Free group and the remaining groups, except with APC™ II group; as shown in Table 2.

**Table 1 Descriptive statistics and groups’ differences of the shear bond strength (MPa) in different groups**

Groups	N	Descriptive statistics				Comparison (df=59)	
		Mean	S.D.	Min.	Max.	F-test	P-value
APC (FF)	12	12.633	2.185	9.04	15.3	6.933	0.00***
APC (plus)	12	13.578	1.776	10.82	16.31		
APC (II)	12	15.08	1.846	11.18	17.16		
Conven. 1	12	13.852	2.103	10.64	16.7		
Conven. 2	12	11.143	1.711	8.55	13.98		

\*\*\*Highly significant

**Table 2 Post hoc Tukey's HSD test of the shear bond strength between the different groups**

Groups		Mean Difference	p-value
APC (FF)	APC (plus)	-0.945	0.753 #
	APC (II)	-2.448	0.024 *
	Conven. 1	-1.219	0.539 #
	Conven. 2	1.489	0.336 #
APC (plus)	APC (II)	-1.503	0.328 #
	Conven. 1	-0.274	0.997 #
	Conven. 2	2.434	0.025 *
APC (II)	Conven. 1	1.228	0.531 #
	Conven. 2	3.937	0.000 ***
Conven. 1	Conven. 2	2.708	0.010 ***

\*Significant; \*\*\* Highly significant; # Non-significant

Table 3 showed descriptive statistics and groups' differences of the maximum excess adhesive measurements in different groups. Also, Post hoc Tukey's HSD test was used to test the mean differences and showed that there were high significant differences between APC Flash Free group and the remaining groups, while the differences between the other groups were not significant; as shown in Table 4.

**Table 3 Descriptive statistics and groups' differences of the maximum excess adhesive measurements ( $\mu\text{m}$ ) in different groups**

Groups	N	Mean	S.D.	Min.	Max.	F-test	P-value
APC (FF)	12	206.245	15.062	179.17	227.3375	431.239	0.00***
APC (plus)	12	530.258	32.99	489.3925	592.2		
APC (II)	12	522.329	16.405	489.2175	546.9675		
Trad. 1	12	519.562	26.229	486.765	558.7175		
Trad. 2	12	508.449	21.867	482.1325	543.9175		

\*\*\* Highly significant

**Table 4 Post hoc Tukey's HSD test of the maximum excess adhesive measurements between the different groups**

Groups		Mean Difference	p-value
APC (FF)	APC (plus)	-324.013	0.00
	APC (II)	-316.084	0.00
	Conven. 1	-313.317	0.00
	Conven. 2	-302.204	0.00
APC (plus)	APC (II)	7.929	0.921
	Conven. 1	10.696	0.797
	Conven. 2	21.809	0.168
APC (II)	Conven. 1	2.767	0.998
	Conven. 2	13.88	0.599
Conven. 1	Conven. 2	11.114	0.773

Table 5 showed descriptive statistics and groups' differences of the minimum excess adhesive measurements in different groups. Also, Post hoc Tukey's HSD test was used to test the mean differences, and showed that there were high significant differences between APC Flash Free group and the remaining groups, also the differences was significant between APC (plus) and APC (II), and APC (II) and Conven.1, while the differences between the other groups were not significant; as shown in Table 6.

**Table 5 Descriptive statistics and groups' differences of the minimum excess adhesive measurements ( $\mu\text{m}$ ) in different groups**

Groups	N	Mean	S.D.	Min.	Max.	F-test	P-value
APC (FF)	12	75.488	18.353	39.145	100.1825	40.744	0.00
APC (plus)	12	155.75	22.143	119.5525	186.025		
APC (II)	12	188.681	28.651	154.855	254.76		
Conven. 1	12	157.151	18.374	123.27	191		
Conven. 2	12	162.208	26.204	103.9825	207.685		

**Table 6 Post hoc Tukey's HSD test of the minimum excess adhesive between the different groups**

Group	Group	Mean Difference	p-value
APC (FF)	APC (plus)	-80.261	0.00
	APC (II)	-113.193	0.00
	Conven.1	-81.663	0.00
	Conven.2	-86.72	0.00
APC (plus)	APC (II)	-32.932	0.008
	Conven.1	-1.402	1.00
	Conven.2	-6.458	0.959
APC (II)	Conven.1	31.53	0.013
	Conven.2	26.473	0.052
Conven. 1	Conven.2	-5.057	0.983

Table 7 showed the relation between the average of maximum and minimum excess adhesive measurements and the mean shear bond strength. As shown in the table, there was a strong association between average excess adhesive measurements and mean shear bond strength in APC (FF) group, while in APC (II) group there was a moderate association, in regard to APC (plus), there was a weak association, furthermore there were no association in Conven.1 and Conven.2 groups.

**Table 7 Relation between the average of maximum and minimum flash measurements and mean shear bond strength for each group**

Groups	Relation	Average
APC (FF)	r	0.879
	p-value	0.000 ***
APC (plus)	r	-0.244
	p-value	0.445 #
APC (II)	r	-0.776
	p-value	0.003 ***
Conven. 1	r	-0.165
	p-value	0.609 #
Conven. 2	r	-0.082
	p-value	0.800 #

\*\*\* Highly significant; # Non-significant

### Shear Bond Strength

Generally, the high mean shear bond strength value does not necessarily refer to better clinical performance [17]. Consequently, the serious issue about the shear bond strength in clinical orthodontic practice is to attain sufficient bond strength value that permits safe detachment of fixed appliance components than to get the highest potential value [18]. In this study, the mean SBS in all groups was higher than the clinically adequate SBS (5.9 to 7.8 MPa) as proposed by Reynolds [19], which means that the adhesive systems that were used can resist shear stress to

adequate level. The SBS of the brackets in each group was less than the bond strength reported by Uysal, et al. [20], but comparable to the bond strength demonstrated by Bishara, et al. [8] and Reddy, et al. [21]. In the current study, APC (II) group had the highest value of the mean shear bond strength, followed by group Conven.1, which occupied a second place in SBS value, the difference between these two groups was not significant and similar to the results demonstrated by Samir, et al. [14], who compared between APC (II) and conventional adhesive brackets with the use of 37% phosphoric acid and Transbond XT primer (group 4), and found that there was no significant difference between them, and this results also agreed with Bishara, et al. [7]. However, the brackets of APC (II) group were coated with adhesive by the manufacturer, so there is less opportunity for bracket contamination during the bonding procedure; therefore, the adhesion between the adhesive and the bracket may be more uniform and effective than in the conventional procedure, in which the clinician has to apply the adhesive onto the bracket base. Furthermore, this new system contains hydrophilic monomers that improve tolerance to humidity in comparison with Transbond XT or other systems that contain mainly BisGMA or other similar hydrophobic monomers. APC (Plus) group was at the third place in SBS value, this may be attributed to the differences in the percentages of the various ingredients incorporated in the material. In addition, the coloring agents and the release of fluoride may affect SBS of this type of brackets, although fluoride releasing nature of this type of brackets aimed at preventing decalcification of the enamel adjacent to the brackets, the present finding correlates with the study done by Asension and Luis [22], whose found that a non-significant differences were observed in the shear bond strength for APC (Plus) and conventional brackets. Concerning APC (FF) group, which had the fourth place, but it was not significantly different from the other groups except with group APC (II), this may be due to the mate nature of the adhesive and the presence of optimum amount of adhesive (precoating) that was applied by the manufacturer and subsequently a little amount of excess adhesive around this type of brackets. Finally, Conven. 2 group had the lowest value of SBS. This may be due to the use of self-etch primer and this outcome is congruent with the findings noted by Cehreli and Altay [23], Cehreli, et al. [24], Bishara, et al. [25], Faltemeier, et al. [26], Paschos, et al. [27], Söderholm, et al. [28], and Selma, et al. [29]. Yet, the present study observations do not match with the results of other previous studies, these findings disagreed with Caccifesta, et al. [30], Cal-neto, [31], Attar, et al. [32], and David, et al. [33], who found that there was no significant difference between the self-etching primer and the conventional etch light cure adhesive system. The results of the current study were obtained after thermocycling in which the samples are subjected to thermal changes and additional water exposure, because of the differences in the coefficient of thermal expansion among the metal bracket, adhesive and tooth, repetitive contraction/ expansion stresses are generated, so these stresses may affect the adhesion of the resin to the bracket and the tooth, resulting in bond failure, this was confirmed with Gale, et al. [34], Anusavice and Brantley [35], Helvatjoglu, et al. [36], and Daub, et al. [37].

### Excess Adhesive Measurements

To our knowledge, only one study has analyzed the excess adhesive flash during the use of the flash-free adhesive system. While the areas surrounding the brackets and excess adhesive are critical places for plaque accumulation and enamel demineralization [4] and can act as a mechanical irritation to the gingivae and therefore potentially increase the incidence of white spot lesion [38-40]. It has been shown that the influence of material (ceramic or metal brackets) on microbial accumulation is not significant, and the common species are equally existent [41]. However, the shape and the surrounding surface of both bracket and adhesive are important factors for plaque accumulation [42]. According to the manufacturer, APC (FF) system is realized by a nonwoven, polypropylene fibber mat, which is directly positioned on the base of the bracket. Recent stereomicroscopic images revealed remarkable less excess adhesive flash in group APC (FF), with an average amount of visible resin between 0.20 and 0.07 mm (as a maximum and minimum excess adhesive) measured at the bracket periphery and this findings agree with that of Mortiz, et al. [12], who found high significant difference in mean of excess adhesive between APC (FF) and APC (plus). In the current study, APC (Plus), Conven.1, and Conven.2 groups had an average of maximum and minimum adhesive which are fairly close, and this result agrees with David, et al. [33], in that study, they did a comparison between APC (Plus) and Conven.1 and found that there were no statistically significant differences in excess adhesive flash, so we can conclude that the addition of a color change feature in the bonding agent do not reduce the amount of excess adhesive around orthodontic brackets. APC (II) group had the maximum amount of excess adhesive at the periphery of the brackets, but this increase is not significantly different from the other groups, except with APC (FF) group.

### The Relation Between the Average of Maximum and Minimum Excess Adhesive Measurements and the Mean Shear Bond Strength

The results of this study showed that there is a strong association between average excess adhesive measurements and mean shear bond strength in APC (FF) group, and this result can be used as an explanation for the decline of shear bond strength (as the excess adhesive flash decreases, the shear bond strength decreases too), this may be due to that such group had the lowest values of shear bond strength and excess adhesive flash, while APC (II) group had a moderate association between shear bond strength and excess adhesive flash (as the excess adhesive flash increases, shear bond strength increases too), due to the fact that this group had the highest values of shear bond strength and excess adhesive flash. The association between shear bond strength and excess adhesive flash in APC (plus) group was weak, and this can be due to the decrease of shear bond strength and increase of excess adhesive flash. While there were no association with Conven.1 and Conven.2, this may be due to the differences in the values of shear bond strength and excess adhesive flash.

### CONCLUSION

Within the limitation of this in vitro study, the following conclusions should be drawn:

1. With the use of APCTM (FF) there is no need to clean up excess adhesive (flash), which simplifies the bracket-positioning process. The resulting adhesive layer and resin-bracket margins facilitate a smooth and narrow surface. This appears to improve marginal integrity and might reduce plaque accumulation and subsequent demineralization around the bracket.
2. There is no reduction in the amount of excessive adhesive around orthodontic brackets with the addition of a color change feature in the bonding agent (as in APC plus bracket-adhesive system).
3. All five adhesive systems have shear bond strength values above the minimum for clinical routine use.
4. The self-etching primer (Transbond Plus) provided clinically acceptable bond strength values compared with the conventional method after 500 thermal cycles.

### DECLARATION

#### Conflict of Interest

The authors and planners have disclosed no potential conflicts of interest, financial or otherwise.

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