# Bond strength and micro-computed tomographic evaluation of pre-coated brackets

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*Objectives:* The aim of the present study was to assess and compare the shear bond strength (SBS) of metal pre-coated orthodontic brackets bonded to fluorotic and non-fluorotic teeth treated with three different etching techniques. A second aim was to determine the volume of adhesive remaining on the tooth at debond using micro-computed tomography ( $\mu$ CT). *Methods:* Ninety extracted premolars were selected to include 45 fluorotic (test group) and 45 non-fluorotic (control group) teeth. Each group was divided into three subgroups of 15 each, which were treated as follows: 1) micro-etched; 2) acid-etched; and 3) both micro-etched and acid-etched. A bonding agent was applied to the prepared surfaces; pre-coated and light-cured brackets were attached to all teeth. An Instron universal testing machine was used to record the debonding force. Specimens were then scanned using a microCT to evaluate the amount of adhesive remaining on the teeth. The significance of the statistical tests was pre-determined at p < 0.05.

*Results:* Two-way ANOVA showed that fluorosis of teeth had no influence on the SBS (p = 0.165) whereas the volume of adhesive remnants was significantly higher in the control group compared with the test group (p < 0.001).

*Conclusions:* Fluorosis had no influence on the SBS of brackets, whereas it had a negative influence on retaining adhesives onto the tooth surfaces.

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# Introduction

A consistent adhesive bond between orthodontic brackets and tooth enamel is a requisite for successful treatment,<sup>1</sup> and necessitates careful attention to tooth surface preparation, the design of bracket base and the bonding material.<sup>2</sup> Fluorosed enamel has been emphasised as the most challenging enamel surface to which to gain adherence.<sup>3</sup> Brackets bonded to fluorotic teeth may fail due to an inability to effectively etch the hypermineralised and acid-resistant enamel.<sup>4</sup> The bond strength between fluorosed enamel and composite materials has been previously examined<sup>5-8</sup> but the results indicate that inconsistencies exist. Ng'ang'a et al.<sup>9</sup> found no significant difference in the tensile bond strength of non-fluorotic teeth compared with mild and moderately fluorotic teeth following the use of 40% phosphoric acid etchant. However, Weerasinghe et al.<sup>8</sup> observed that the severity of fluorosis affected the micro-shear bond strength of a self-etching bonding system to fluorosed enamel. Similarly, Adanir et al.<sup>10</sup> reported a considerable difference in shear bond strength (SBS) of normal and moderately fluorosed teeth after etching using 37% phosphoric acid. Furthermore, several studies have evaluated and compared the SBS of orthodontic brackets bonded to enamel surfaces pre-treated using various techniques.<sup>11-13</sup>

The preferred site of bond failure during a debonding procedure is at the resin-bracket interface so that minimal adhesive remains on the tooth surface. Bonding failure at the resin-enamel interface is considered undesirable as the enamel surface may tear during the debonding process.<sup>2</sup> Apart from the Adhesive Remnant Index (ARI) system,14 additional measurement protocols may be used to monitor the adhesive remaining on enamel. These include three-dimensional (3D) laser scanning,15 3D optical scanning,<sup>16</sup> 3D profilometry,<sup>17</sup> scanning electron microscopy,<sup>18</sup> and stereomicroscopy,<sup>19</sup> planar surfometry<sup>20</sup> and optical coherence tomography.<sup>21</sup> Although the quality of the bond at the enamelbracket interface has been assessed using microcomputed tomography ( $\mu$ CT),<sup>22</sup> to date, the volume of the adhesive remnants after debonding has not been studied using microCT. Therefore, the aim of the present study was to assess and compare the SBS of pre-coated orthodontic metal brackets bonded to fluorotic and non-fluorotic teeth treated by three different etching techniques. An additional aim was to determine the volume of adhesive remaining on the tooth surface using microCT.

## Materials and methods

This study was registered at and approved by the College of Dentistry Research Center (Registration number: NF 2252), King Saud University, Riyadh, Saudi Arabia.

# Specimen collection and assessment of fluorosis

A total of 90 human maxillary first premolars extracted as part of an orthodontic treatment plan were obtained from the Department of Oral and Maxillofacial Surgery, College of Dentistry, King Saud University. The teeth were selected to include 45 with dental fluorosis (test group) and 45 without fluorosis (control group). Upon extraction, the teeth were debrided of soft tissue remnants by one investigator and were stored in sterilised normal saline at room temperature to prevent dehydration. Dental fluorosis was assessed by the same investigator according to the Thylstrup and Fejerskov Fluorosis Index (TFI)<sup>23</sup> and only teeth with moderate to severe fluorosis were included in the test group. Each sample was divided into three equal subgroups. Each 15-tooth subgroup was treated by either micro-etching, acid-etching, or both micro-etching and acid-etching.

# Different enamel surface preparation techniques

The teeth were mounted in an upright position on

plastic models using a metal and acrylic indicator. The method orientated the teeth so that force could be applied parallel to the buccal surface. Each tooth was embedded in self-curing acrylic resin and stored at room temperature in distilled water until required. The enamel surfaces of normal and fluorotic teeth within the first subgroup were micro-etched using 50 micron aluminum oxide particles (Aurum Ceramic Dental Laboratories, Saskatoon, Canada) for 5 seconds. Treatment utilised the Basic Professional Air Abrasion Gun (Micro Cab, Danville Engineering Inc., CA, USA) with a straight tip perpendicular to the buccal surface of the tooth. The teeth were finally rinsed with distilled water for 30 seconds and dried with oil-free compressed air for 10 seconds. Bonding agent (Transbond<sup>TM</sup> XT, 3M Unitek, CA, USA) was applied to the prepared surfaces and light cured using an LED curing unit (Ortholux, 3M Unitek, CA, USA). Victory Series<sup>™</sup> pre-coated premolar metal brackets (3M Unitek, CA, USA) were centred on the buccal surface of the teeth 4 mm from the occlusal surface using a bracket-positioning gauge (Ormco, CA, USA). The adhesive was cured for 5 seconds.

The enamel surfaces of normal and fluorotic teeth of the second subgroup were acid-etched using 37% phosphoric acid for 30 seconds (Total Etch, Ivoclar . Vivadent, Schaan, Liechtenstein). The etched surfaces were washed with water for 15 seconds and dried. Bonding material (Transbond<sup>TM</sup> XT, 3M Unitek, CA, USA) was applied to the prepared surfaces and Victory Series<sup>TM</sup> pre-coated premolar metal brackets (3M Unitek, CA, USA) were bonded. The enamel surfaces of normal and fluorotic teeth of the third subgroup were first micro-etched using 50 micron aluminum oxide particles (Aurum Ceramic Dental Laboratories, Saskatoon, Canada) for 5 seconds. The teeth were rinsed with distilled water for 30 seconds. dried with oil-free compressed air for 10 seconds and etched using 37% phosphoric acid for 30 seconds (Total Etch, Ivoclar Vivadent, Schaan, Liechtenstein). The etched surfaces were washed with water for 15 seconds and dried, bonding material applied and precoated premolar metal brackets bonded following the same protocol.

# Testing shear bond strength

The specimens were mounted in the jig of a universal testing machine (Instron Corp., High Wycombe, England) and adjusted to orientate the bracket base parallel to the direction of the applied force. This was expected to produce a shear force at the bracket-tooth interface. The Instron machine generated a 1 kN load cell at a crosshead speed of 0.5 mm/min during the test. The load at failure was recorded in newtons (N) and converted into megapascals (MPa) using the bracket base surface area of 10.23 mm<sup>2</sup>. The Instron machine produced a shear-peel force approximating the clinical situation.

### Micro-computed tomographic evaluation

The microCT evaluation of the adhesive residue after debonding was performed using a SkyScan 1172 (Micro-CT SkyScan, Kontich, Belgium). The X-ray generator of the microCT was operated at an accelerated potential voltage of 70 kV with current of 130 µA using an aluminum and copper filter with a resolution of 15 µm. Projection images were recorded in steps of 0.4 degrees from 0 to 360 degrees. A threedimensional reconstruction was performed using the scanner's 'N Recon' (1.6.5.0) software (Belgium) utilising a filtered back-projection algorithm. The reduction of the beam hardening effect was 40% and ring artifact correction was 12% to produce the precise image cross-section. The resulting data set of 15 µm resolution for each sample was analysed with 'CT An' (1.12.11.0+) software and post-scan adhesive remnants were measured in mm<sup>3</sup>. The mean percentage of adhesive was calculated and recorded for each sample. Visualisation in 3D was rendered using CT VOL software provided by SkyScan (Belgium). The software produced a 3D picture and movie projection in the axial, sagittal and transaxial dimensions to facilitate adhesive assessment.

## Statistical analysis

#### Statistical analysis was performed using SPSS 16.0

(SPSS Inc, Chicago, IL, USA). The analysis of variance (ANOVA) was used to assess differences in mean values between test and control group for SBS and remnant adhesive volume assessment. Posthoc multiple comparisons were applied to assess the variance in mean values for each group. The significance value was set at p < 0.05.

## Results

### Shear bond strength

The mean SBS was highest in the combined treatment (micro-etching followed by acid-etching) subgroup of the control group, and the lowest in the micro-etched subgroup of the test group. The mean values and standard deviations of SBS are presented in Table 1. Two-way ANOVA showed that fluorosis of the teeth had no influence on the SBS (p = 0.165). However, the differences produced by enamel treatment techniques were statistically significant (p < 0.001). Post-hoc analysis (Table II) revealed that, in the control group, combined treatment resulted in a significantly higher SBS compared with the acid-etched (p < 0.05) and micro-etched (p < 0.001) subgroups, whereas no significant difference in SBS was found between the acid-etched and micro-etched subgroups (p = 0.065). The combined treatment in the test group resulted in a significantly higher SBS compared with that of the micro-etched subgroup (p < 0.05). Furthermore, the acid-etched subgroup demonstrated a significantly higher SBS compared with the micro-etched subgroup (p < 0.05).

## Volume of adhesive remnants

The mean values and standard deviations of the volume of adhesive remaining (mm<sup>3</sup>) after debonding brackets from the test and control teeth are presented in

Table I. The mean shear bond strength (MPa) of brackets bonded to test and control teeth.

Group	Treatment	Mean	SD	Maximum load	Minimum load
Test	Micro-etched	3.71	1.48	7.34	2.21
	Acid-etched	7.50	4.61	14.66	2.29
	Micro- and acid- etched	8.84	4.43	16.65	2.78
Control	Micro-etched	5.09	1.66	8.26	2.62
	Acid-etched	7.36	3.37	13.97	2.56
	Micro- and acid- etched	10.76	3.69	17.29	3.41

SD: Standard Deviation

Table III. The mean volume of adhesive remnant was found to be highest on the acid-etched teeth of the control group and the lowest on the micro-etched teeth of the test group. Two-way ANOVA indicated that the volume of adhesive remaining was significantly higher in the control group compared with the test group (*p* < 0.001). In addition, the volume of adhesive remnant left after the surface treatment techniques showed a statistically significant difference (p < 0.001). Posthoc analysis (Table IV) revealed that, in the control group, a significantly higher adhesive remnant (Figure 1b) was found in the subgroup that underwent the combined treatment compared with the acid-etched subgroup (Figure 1a) (p < 0.05). Furthermore, the acid-etched subgroup showed a significantly higher volume of adhesive residue compared with the microetched subgroup (Figure 1c) (p < 0.05). The volume of adhesive remnants in the test group was significantly higher in the combined treatment subgroup (Figure 2b) compared with the micro-etched (Figure 2c) (p < 0.001) and acid-etched (Figure 2a) subgroups (p < 0.05). Moreover, the acid-etched subgroup had

significantly greater adhesive remaining compared with the micro-etched subgroup (p < 0.05).

Since using microCT for the measurement of the amount of adhesive residue is a relatively novel technique, a test for the reliability of this technique was performed. Thirty specimens were randomly selected and the scanning procedure was repeated. Pearson correlation coefficients showed that a significant correlation (p < 0.001) existed between readings obtained before and after the reliability test.

## Discussion

Although the bonding of brackets has revolutionised and improved orthodontic clinical practice, further improvements in the bonding procedure are essential to save time and to minimise enamel loss without compromising clinically useful bond strength. This is particularly related to the uncertainties in predicting the etching patterns<sup>24</sup> and the contradictory reports on the bond strength attained on fluorotic teeth.<sup>5-8</sup> The results of the present study showed that enamel



Figure 1. Micro-computed tomography images illustrating adhesive remnants subsequent to debonding brackets bonded to (a) acid-etched, (b) micro-etched followed by acid etched (combined treatment), and (c) micro-etched teeth, in the control group.

Figure 2. Micro-computed tomography images illustrating adhesive remnants subsequent to debonding brackets bonded to (a) acid-etched, (b) micro-etched followed by acid-etched (combined treatment), and (c) micro-etched teeth, in the test group.

Crewe	Transforment	Treatment J	Magn difference II		95% Confidence Interval		
Gloup	Irealment I		Mean allierence I-j	p value	Lower bound	Upper bound	
Test		A-E	-3.79	0.020*	-7.00	-0.58	
	/V1-E	M-A-E	-5.13	0.002**	-8.35	-1.92	
		M-E	3.79	0.020*	0.58	7.00	
	A-E	M-A-E	-1.34	0.706	-5.50	2.82	
	M-A-E	M-E	5.13	0.002**	1.92	8.35	
		A-E	1.34	0.706	-2.82	5.50	
Control	M-E	A-E	-2.34	0.065	-4.80	0.13	
		M-A-E	-5.74	0.00***	-8.40	-3.08	
	A-E	M-E	2.34	0.065	-0.13	4.80	
		M-A-E	-3.40	0.035*	-6.59	-0.20	
	M-A-E	M-E	5.74	0.00***	3.08	8.40	
		A-E	3.40	0.035*	0.20	6.59	

Table II. Multiple comparison of shear bond strength according to the three enamel treatment techniques.

\*Level of significance at 0.05 \*\*Level of significance at 0.001 \*\*\*Highly significant M-E: Micro-etched; A-E: Acid-etched; M-A-E: Micro- and acid-etched

Table III.	Volume of	adhesive	remnants	(mm3)	after	debonding	brackets	bonded t	o test	and o	control	teeth.
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Group	Treatment	Mean	SD
Test	Micro-etched	0.04	0.06
	Acid-etched	0.42	0.45
	Micro- and acid-etched	0.97	0.46
Control	Micro-etched	0.66	0.46
	Acid-etched	1.39	0.77
	Micro- and acid-etched	0.63	0.42

SD: Standard Deviation

Table IV. Multiple comparison of the volume of adhesive remnants according to the three enamel treatment techniques.

	Translation 1	Treatment J	Mean		95% Confidence Interval		
Gloup	Ireatment I		difference I-J	p value	Lower bound	Upper bound	
Test		A-E	-0.38	0.014*	-0.69	-0.08	
	/V-E	M-A-E	-0.93	0.00***	-1.25	-0.61	
	ΛE	M-E	0.38	0.014*	0.08	0.69	
	A-E	M-A-E	-0.54	0.009**	-0.96	-0.13	
	M-A-E	M-E	0.93	0.00***	0.61	1.25	
		A-E	0.54	0.009**	0.13	0.96	
Control		A-E	-0.74	0.012*	-1.32	-0.15	
	/v-L	M-A-E	0.03	0.987	-0.38	0.44	
	۸ F	M-E	0.74	0.012*	0.15	1.32	
	A-E	M-A-E	0.76	0.008**	0.19	1.33	
	M-A-E	M-E	-0.03	0.987	-0.44	0.38	
		A-E	-0.76	0.008**	-1.33	-0.19	

\*Level of significance at 0.05 \*\*Level of significance at 0.001 \*\*\*Highly significant M-E: Micro-etched; A-E: Acid-etched; M-A-E: Micro- and acid-etched

treatment techniques, rather than the type of teeth (fluorotic or non-fluorotic), had a more profound influence on SBS, whereas the amount of surface adhesive remaining was lower in the fluorotic compared with non-fluorotic teeth.

Several studies have compared the bond strength obtained on debonding orthodontic brackets bonded to normal (non-fluorotic) teeth after acid-etching, micro-etching and a combination of both.<sup>11-13</sup> It was concluded that micro-etching without acid-etching produced lower bond strength than combined microetching and acid-etching, which favourably compares with the results of the present study. Suma et al.<sup>25</sup> assessed the SBS of fluorotic enamel surfaces treated by micro-etching, acid-etching and a combination of both. It was revealed that, irrespective of the bonding material employed, micro-etching followed by acidetching provided significantly higher bond strength compared with acid-etching alone. In concordance with the results of the present study, etching time but not the severity of fluorosis was found to have a significant effect on the SBS of the composite material to fluorosed enamel.<sup>5</sup>

Clinicians ideally require high bracket bond strength and a low adhesive remnant index. After debonding, the resin material may be found adhering to the tooth surface and/or the bracket base. Adherence of bonding material to the bracket base suggests that the bond to the bracket is stronger than the bond to enamel.<sup>26</sup> The present study supports this view as it was found that the micro-etching only subgroup contained most of the adhesive remnants on the base of the bracket. A weaker bond between the adhesive and the enamel would make it easier for clinicians to remove resin from the enamel surface after debonding. However, the bond failure that occurs within the adhesive leaving remnants on both the tooth surface and the bracket base may be considered detrimental, as the removal of the remaining material from the tooth surface may damage enamel and increase chairside time.<sup>26</sup> The present study found that bond failure in the acidetching only subgroup and micro-etching followed by acid-etching subgroup occurred within the adhesive. Therefore, a balance between the bond strength and the volume of adhesive remnant is encouraged

The resin remnant left by different brands of orthodontic adhesive after debonding was determined quantitatively using a 3D profilometer by Lee and Lim.<sup>17</sup> The debonded enamel profile was quantified

using the 3D profilometer, which provided quantitative data at a micrometer scale. The height change at each measurement point was calculated by overlapping the surfaces before bonding and after debonding using software, which enabled remnant quantification. It was found that the amount of adhesive remaining after etching normal (non-fluorotic) teeth with 32% phosphoric acid (Uni-etch, Bisco, IL, USA) for 15 seconds and using Transbond XT (3M ESPE, MN, USA) was 1.4 mm<sup>3</sup> and the mean bond strength was  $4.7 \pm 2.0$  MPa. In the present study, the volume of adhesive remnant after etching normal teeth with 37% phosphoric acid for 30 seconds and using Transbond XT was 1.39 mm<sup>3</sup>, whereas the mean bond strength was 7.36 ± 3.37 MPa. An additional study<sup>16</sup> reported that the mean volume of composite remnant determined using a 3D laser scanner was 0.22 mm<sup>3</sup>  $\pm$  0.32 mm<sup>3</sup> and, in a recent study,<sup>15</sup> the volume of adhesive remnant measured using a 3D optical scanner was reported to range from 0.05 mm<sup>3</sup> to 4.16 mm<sup>3</sup> with a median of 0.98 mm<sup>3</sup>. Differences in experimental methodologies and the composite materials used may have led to the inconsistencies in the results of the studies.

The ARI system,<sup>14</sup> used to score adhesive remnants, provides rank scores and is a surface area assessment rather than a 3D volumetric measure produced by a microCT evaluation. A microCT scan therefore provides a true volumetric assessment of the adhesive remnant. Earlier studies used SEM and energy dispersive X-ray spectrometry to determine ARI and the calcium remnant index left on the bracket bases,<sup>18</sup> as well as an enamel detachment index.<sup>27</sup> However, sample preparation for SEM evaluation was necessary and quantification of the remaining adhesive was not possible. A later study used planar surfometry, which utilised two line scans to determine the differences in enamel height with that of an untreated reference plane during the bonding and debonding processes.<sup>20</sup> This method provided two-dimensional height changes, compared with the 3D quantitative measurement established by microCT.

## Conclusion

Within its limitations, the present study showed that fluorosis had no influence on the SBS of bonded brackets, whereas there was a negative influence on retaining adhesives to enamel surfaces. In addition, micro-etching followed by acid-etching provided higher SBS values compared with micro-etching or acid-etching alone. The use of microCT for the quantification of the adhesive remnant following debonding was found to be a feasible and reliable method.

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