Influence of surface treatment on shear bond strength of orthodontic brackets

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Introduction: The shear bond strength of orthodontic brackets bonded to micro-hybrid and micro-particulate resins under different surface treatment methods was assessed.

Methods: Two hundred and eighty test samples were divided into 28 groups (n = 10), where 140 specimens were filled with Durafill micro-particulate resin and 140 with Charisma composite. In 140 samples, a coupling agent (silane) was applied. The surface treatment methods were: Phosphoric and hydrofluoric acid etching, sodium bicarbonate and aluminum oxide blasting, stone and burs. A Universal Instron Machine was used to apply an occlusal shear force directly to the resin composite bracket surface at a speed of 0.5 mm/min. The means were compared using analysis of variance and multivariate regression to assess the interaction between composites and surface treatment methods.

Results: Means and standard deviations for the groups were: Sodium bicarbonate jet 11.27 ± 2.78 ; burs 9.26 ± 3.01 ; stone 7.95 ± 3.67 ; aluminum oxide blasting 7.04 ± 3.21 ; phosphoric acid 5.82 ± 1.90 ; hydrofluoric acid 4.54 ± 2.87 , and without treatment 2.75 ± 1.49 . An increase of 1.94 MPa in shear bond strength was seen in Charisma groups. Silane agent application reduced the Charisma shear bond strength by 0.68 Mpa, but increased Durafill means for bicarbonate blasting (0.83), burs (0.98) and stone drilling (0.46).

Conclusion: The sodium bicarbonate blasting, burs and stone drilling methods produced adequate shear bond strength and may be suitable for clinical use. The Charisma micro hybrid resin composite showed higher shear bond means than Durafill micro particle composite.

Keywords: Shear bond strength. Dental materials. Orthodontic brackets. Orthodontics.

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INTRODUCTION

Adult patients undergoing orthodontic treatment often have teeth restored with resin composites commonly used for esthetic purposes and for restorations.^{1,2} This type of material is also widely found in the mouths of young patients due to a high incidence of trauma during permanent dentition (30%).³

The use of dental composites for bonding orthodontic brackets to enamel is convenient as it allows careful positioning of the orthodontic accessories while promoting bond strength capable of withstanding tooth movement during application of different types of forces. The literature is rich in methods and properties used in the direct bonding of orthodontic metal accessories to ceramic surfaces.^{4,5,6} However, few studies have been conducted to evaluate this procedure on composite restorations. Therefore, there is great clinical demand for an effective technique capable of allowing teeth restored with resin composite to be subjected to different types of force application.

In order to develop an efficient method for bonding brackets onto resin composite surfaces it is necessary to learn about the factors that directly influence the success of the procedure, such as type of resin composite (hybrid or micro-particulate) and bond strength. Research carried out by Schwartz, Tyas and West⁷ and Lai, Woods and Tyas,⁸ demonstrated that metallic brackets bonded to hybrid and micro-particulate restorative composites could withstand 7.8 MPa of shear force, which according to Lopez⁹ would be sufficient to ensure a successful bond in clinical practice.

Several methods based on the repairing technique of resin composites have been reported as capable of improving bond strength. These include acid etching,^{10,11} air abrasion^{12,13} and the use of solvents and silanes.^{14,15} However, no consensus has yet been reached regarding the results obtained with different procedures hence, this study. The aim of this study, therefore, was to assess the bond strength resulting from the direct bonding of orthodontic brackets to surfaces composed of micro-particulate and micro-hybrid restorative materials subjected to different chemical and physical surface treatment methods.

MATERIAL AND METHODS

Two hundred and eighty cylinders were fabricated from acrylic (specimens) with 25 mm diameter and 5 mm in height, with an 8 mm diameter and 1.5 mm depth hole, with grooves on the bottom for increased retention of the resin composite. Of these, 240 were used for surface treatment testing and the remaining for the control group. In order to prevent the Transbond XT adhesive (3M/Unitek, Monrovia, CA, USA) from flowing and thereby affecting the yield strength values on shearing, the bonding area was bounded with tape prior to surface preparation.

Two types of resin composites were tested: (a) Durafill resin composite (HeraeusKulzer, Hanau, Germany), and (b) Charisma micro hybrid resin composite (HeraeusKulzer, Hanau, Germany). These materials were all placed in one go to reduce the incorporation of air bubbles, and light cured for 40 seconds, 20 at the top and 20 at the bottom. A glass slide was used for compression and homogenization of the material (Fig 1). Ortholux XT Visible Light Curing Unit (3M Unitek, Monrovia, CA, USA) was used, and had its light intensity measured prior to curing at each stage, in all groups (Optilux Radiometer Model 100, SDS Care, Danbury, CT, USA). The intensity found was $450 \pm 10 \text{ mW/cm}^2$. Additionally, the bonding procedures were all performed in the same environment and with a 1kVA voltage stabilizer connected to the curing light. The specimens were stored in plastic containers with distilled water, covered with an opaque lid and kept in an oven at $37 \pm 1^{\circ}$ C for 7 days for aging.

Sample group composition

The specimens were divided into 28 groups of 10 elements, separated according to the type of surface treatment, type of resin composite and the use / non-use of silane (Table 1). The resinous surfaces were treated by the following agents: 37% phosphoric acid (Dental G Conditioner, Dentsply, Petrópolis, RJ) for



Figure 1 - Light curing of specimens filled with resin composite.

Table 1 - Presentation of groups, types of chemical surface treatment	nt, use or non-use of silane, and type of resin composite used.
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Groups	Surface treatments	Silane application	Resin
1	Phosphoric acid no Durafill	No	Durafill
2	Phosphoric acid with Durafill	Yes	Durafill
3	Hydrofluoric acid no Durafill	No	Durafill
4	Hydrofluoric acid with Durafill	Yes	Durafill
5	Aluminum oxide blasting no Durafill	No	Durafill
6	Aluminum oxide blasting with Durafill	Yes	Durafill
7	Sodium bicarbonate no Durafill	No	Durafill
8	Sodium bicarbonate blasting with Durafill	Yes	Durafill
9	Mounted stone no Durafill	No	Durafill
10	Mounted stone with Durafill	Yes	Durafill
11	Diamond bur no Durafill	No	Durafill
12	Diamond bur with Durafill	Yes	Durafill
13	No treatment no Durafill	No	Durafill
14	No treatment with Durafill	Yes	Durafill
15	Phosphoric acid no Charisma	No	Charisma
16	Phosphoric acid with Charisma	Yes	Charisma
17	Phosphoric acid no Charisma	No	Charisma
18	Hydrofluoric acid with Charisma	Yes	Charisma
19	Aluminum oxide blasting no Charisma	No	Charisma
20	Aluminum oxide blasting with Charisma	Yes	Charisma
21	Sodium bicarbonate blasting no Charisma	No	Charisma
22	Sodium bicarbonate blasting with Charisma	Yes	Charisma
23	Mounted stone no Charisma	No	Charisma
24	Mounted stone with Charisma	Yes	Charisma
25	Diamond bur no Charisma	No	Charisma
26	Diamond bur with Charisma	Yes	Charisma
27	No treatment no Charisma	No	Charisma
28	No treatment with Charisma	Yes	Charisma

30 seconds (Fig 2A); 10% hydrofluoric acid (Porcelain Conditioner, Dentsply, Petrópolis, RJ) for 2 minutes (Fig 2B); aluminum oxide blasting (blaster manufactured by Bio Art dental equipment Ltd, São Carlos, São Paulo, Brazil, 50-micron particles) (Fig 2C) and bicarbonate blasting (Profi II Ceramic, Dabi Atlante, Ribeirão Preto, São Paulo, Brazil, 60 pounds) (Fig 2D); mounted cylindrical stone (Jon, São Paulo, SP, Brazil) (Fig 2E) and high speed cylindrical diamond bur (3101, KG Sorensen, Barueri, SP, Brazil) (Fig 2F). In the two latter groups unidirectional grooves were observed by stereomicroscopy. The cleaning and drying procedures were similar in all other groups, and consisted in air/water sprays for 30 seconds followed by drying with air for another 30 seconds.

Thereafter, maxillary central incisor metallic brackets (standard edgewise) (Dental Morelli Ltda., Sorocaba, SP, Brazil) with 3.8×3.6 mm base and 0.022×0.030 -in slot were bonded. The brackets were bonded to the composite restorative system of the specimens using Transbond XT orthodontic adhesive system according to manufacturer's specifications (Fig 3).

In groups where silane (Angelus, Londrina, PR) was applied, a microbrush (KG Sorensen Indústria and Comércio Ltda., Barueri, SP, Brazil) was used, and after a 1 minute interval, was homogenized by air jet for 3 seconds as well as the primer (Transbond XT Primer, 3M Unitek, Monrovia, CA, USA), which was applied with a microbrush and also homogenized by air jet.

Transbond XT adhesive system was applied to the base of the bracket and positioned on the surface of the pretreated resin composite. The brackets were pressed lightly avoiding any movement, with the purpose of causing the adhesive paste to flow and allow for the removal of adhesive flash with



Figure 2 - Surface treatment using: A) 37% phosphoric acid, B) hydrofluoric acid, C) aluminum oxide blasting, D) sodium bicarbonate blasting, E) mounted stone, and F) diamond bur.



Figure 3 - Orthodontic Brackets bonded with Transbond XT light-cured resin onto previously treated surfaces.

an explorer #5 (DUFLEX, Petrópolis, RJ, Brazil). Light curing was performed on the whole set for 20 seconds (10 seconds on the mesial and 10 seconds on the distal surface). After bracket bonding, the same storage protocol described above was performed. The specimens were then once again stored in distilled water at 37° C for 7 days for aging.

Shear test

For the mechanical shear strength test a universal testing machine (EMIC DL 10000 MF cell Trd 21) owned by the Laboratory of Biomaterials at the Military Engineering Institute was used. The specimens are fastened to a device so that the bracket base was kept parallel to the tip of the chisel (Fig 4). Tests were performed with a load cell of 10 kg at a speed of 0.5 mm/min,¹⁶ and data recorded by a computer program (TESC version 3.04, Pinhais, PR, Brazil)

Adhesive remnant index (ARI)

After shearing the specimens, the bases of the brackets and the resin surfaces were examined in a stereomicroscope with 20x magnification. Adhesive remnant index (ARI) was categorized as follows: Score 0 (no adhesive present on the bracket bases); 1 (less than half); 2 (more than half) and 3 (the entire adhesive remained on the bracket base).¹⁷ A descriptive analysis of the data with means, medians and standard deviations was carried out. Comparisons between shear force means were made using analysis of variance (ANOVA) with Scheffe correction for multiple comparisons. Kruskall-Wallis test was used for multiple comparisons between the scores, complemented by Mann-Whitney test.

RESULTS

The mean shear force values ranged between 2.30 MPa and 12.02 MPa, whereas the groups treated with acid etch-



Figure 4 - Bracket shearing in an Emic DL 10000 MF universal testing machine. Note the metallic support for fixation of specimens.

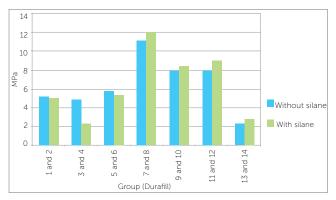


Figure 5 - Diagram showing shearing forces of specimens filled with Durafill resin arranged according to the use or non-use of silane.

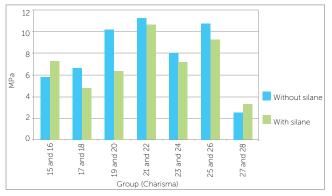


Figure 6 - Diagram showing shearing forces of specimens filled with Charisma resin arranged according to the use or non-use of silane.

ing attained lower values than those observed in the groups treated by blasting and bur/stone. In specimens filled with Durafill resin, silane application improved the means after treatment with bicarbonate blasting, bur and stone, but higher standard deviations were observed (Fig 5). Regarding Charisma, the highest mean was obtained with bicarbonate blasting without silane, although the application of silane worsened the mean yield strength values, with the exception of phosphoric acid etching (Fig 6). The stone without silane group showed the largest standard deviation followed by etching with hydrofluoric acid without silane. The brackets in the control groups of both resin composites had lower shear strength scores (Table 2).

Bivariate analyses showed that there are differences between the means of the groups, i.e., Durafill and Charisma, in terms of surface preparation and silane application. The Charisma group obtained higher means than the Durafill group. The resins tested exhibited their best performance with bicarbonate blasting, and the latter group with diamond burs. The effects produced — regardless of the type of resin composite (Durafill or Charisma) — types of surface preparation and use of silane were assessed by multiple linear regression. Analysis of the model showed significant interaction between type of composite and type of preparation (Table 2).

In trivariate analysis, the Charisma group treated with hydrofluoric acid (Fl) without silane (5.85 MPa) was used as a basis for comparison. Compared to this group, all other surface preparations were significantly different in terms of specimen shear values, except for the use of phosphoric acid (p = 0.23). Charisma tests showed that the best surface preparation was bicarbonate blasting (5.42 MPa) followed by the use of burs (4.52 MPa), aluminum oxide (3.01 MPa), and stone (2.23 MPa), compared to hydrofluoric acid. In the control group, the non-use of silane decreased shear strength by 1.94 MPa compared to the use of hydrofluoric acid under the same conditions (Table 2).

In Table 3, the multivariate linear regression model highlights the interaction between the composite type and type of surface preparation variables. Charisma with hydrofluoric acid was used as a basis for comparison, where bonding performed with silane application reduced bracket adhesion, i.e., 0.68 MPa (p = 0.05). It was found that the use of Durafill with the same surface preparation, hydrofluoric acid resin without silane, compared to Charisma, caused the shear force to worsen Table 2 - Description of mean and standard deviation values of shear forces comprising 4 characteristics. Analysis of Variance (ANOVA) with Scheffe correction.

	Mean <u>+</u> SD	Size Sample	p value	
Composite in restoration				
Charisma	7.45 <u>+</u> 3.91	130	0.028	
Durafill	6.44 <u>+</u> 3.72	130		
Restoration composite preparation type				
Hydrofluoric acid	$4.54^{a.b.c.d} \pm 2.87$	40		
Phosphoric acid	5.82 ^{e.f.g} ± 1.90	40		
Bur	9.26 ^{a.e.h} ± 3.01	40	< 0.00	
Bicarbonate blasting	11.27 ^{b.d.f.ijk} ± 2.78	40		
Aluminum oxide	7.04 ^{c.il} ± 3.21	40		
Stone	7.95 ^{d,j,m} ± 3.67	40		
None	2.75 ^{e.g.h.k.Lm} ± 1.49	20		
Presence of silane				
No	7.99 ^{a.b} ± 3.81	120	< 0.00	
Yes	6.70 ^{b.c} ± 3.61	120		
Total	6.95 <u>+</u> 3.84	260	-	

Pairs of means with same letters show statistically significant differences at 5% level after correction using Scheffe's multiple comparisons test.

by up to 1.94 Mpa (Table 3). Although Durafill resulted in smaller magnitudes than Charisma, the combined use of Durafill with bicarbonate blasting resulted in an additional increase in shear force (2.63 MPa) compared with the observed data (means), when the use of bicarbonate blasting and Charisma were combined. The stone group also had a significant increase of 2.37 MPa (p = 0.05) relative to the comparison group, Charisma and hydrofluoric acid without silane (Table 3).

Statistical differences between specimen ARI scores in the various groups are depicted in Table 4. Analysis of specimen ARI scores after debracketing revealed a tendency of Charisma group specimens with surfaces treated by hydrofluoric acid, aluminum oxide, mounted stone (with application of silane) and diamond burs (without silane) to exhibit failures at the adhesive/ bracket interface. In Durafill groups, the same interface failure was observed in specimens conditioned with hydrofluoric acid and aluminum oxide (without silane application). Charisma groups treated by phosphoric acid, bicarbonate blasting and use of stone (without silane application) and burs (with silane) tended to show failures at the adhesive/specimen interface. The same behavior was observed in the Durafill groups conditioned by phosphoric acid, bicarbonate blasting and after use of stone and diamond burs (Table 4).

The use of silane did not produce ARI differences in many groups, except for 3 specimen groups filled with

Charisma, and surfaces treated by hydrofluoric acid, mounted stone and diamond burs.

DISCUSSION

The shear force of both types of resin composites tested were considered different, and were influenced by the vast majority of surface preparations analyzed. According to Swift, Valley and Boyer¹⁰ the shear force of hybrid resin bonding was higher than that of microparticulate resin, although their study did not test adhesion using orthodontic brackets.

Most studies reviewed tested the bond strength of different types of resin in procedures to repair esthetic restorations. In these cases, several authors^{18,19,20,21} reported the need to etch the composite in order to enhance its adhesiveness. In this study it was found that the effect of surface preparation on both resin composite types were similar to procedures to repair restorations.

Hydrofluoric acid treatment produced a significant decrease in shear force compared to blasting procedures (aluminum oxide and bicarbonate) or grooved surface (bur and stone) (Tables 2 and 3). This finding corroborates works reported in the literature.^{16,22,23}

Swift, Valley and Boyer¹⁰ stated that the effect of hydrofluoric acid is related to its percentage, size and type of inorganic composition. Therefore, depending on the type of resin composite, the effect of hydrofluoric acid may alter the effect of cohesion between

		Force Cl 95%		n volen	Variable combination description			
		(MPa)	Lower	Upper	p-valor	Composite	Adhesive	Preparation
Group used for comparison		5.85	4.62	7.08	-	Hydrofluoric acid	Without silane	Charisma
Effect of adhesive	With silane	-0.68	-1.37	0.00	0.05	Hydrofluoric acid	With silane	Charisma
Composite effect	Durafill	-1.94	-3.61	-0.27	0.02	Hydrofluoric acid	Without silane	Durafill
	Phosphoric acid	1.02	-0.65	2.69	0.23	Phosphoric acid	Without silane	Charisma
	Bur	4.51	2.84	6.19	0.00	Bur	Without silane	Charisma
Preparation effect	Bicarbonate blasting	5.42	3.74	7.09	0.00	Bicarbonate blasting	Without silane	Charisma
	Aluminum oxide	3.01	1.34	4.68	0.00	Aluminum oxide	Without silane	Charisma
	Stone	2.23	0.55	3.90	0.01	Stone	Without silane	Charisma
	None	-1.94	-3.84	-0.05	0.05	None	Without silane	Charisma
	Phosphoric acid	0.52	-1.84	2.89	0.67	Phosphoric acid	Without silane	Durafill
	Bur	0.42	-1.94	2.79	0.73	Bur	Without silane	Durafill
Interaction Durafill <i>versus</i>	Bicarbonate blasting	2.63	0.26	4.99	0.03	Bicarbonate blasting	Without silane	Durafill
	Aluminum oxide	-1.01	-3.38	1.35	0.40	Aluminum oxide	Without silane	Durafill
	Stone	2.37	0.01	4.74	0.05	Stone	Without silane	Durafill
	None	1.61	-0.76	3.97	0.18	None	Without silane	Durafill

Table 3 - Coefficients of multivariate regression with the shear force interaction terms between composite and preparation.

Table 4 - Differences between group scores (ARI).

Group	Differed from groups
1	4, 19, 20, 24, 26
2	4, 19, 20, 24, 26
3	-
4	8, 9, 12, 13, 14, 18, 23, 27, 28
5	8, 9, 12, 13, 14, 23, 27
6	-
7	20
8	19, 20, 24, 26
9	19, 20, 24, 26
10	-
11	20
12	19, 20, 24, 26
13	19, 20, 24, 26
14	19, 20, 24, 26
15	-
16	-
17	18*
18	19, 20
19	23, 27, 28
20	23, 27, 28
21	-
22	-
23	24*, 26
24	27
25	26*
26	27
27	-
28	-

the resins, reducing yield strength. Hydrofluoric acid acts by dissolving glass particles, leading to increased surface porosity, which results in increased retention caused by the mechanical bonding agent. An application of this acid at 9.6% for 2 minutes may be regarded as aggressive since short duration conditioning can dissolve the filling and compromise resin softness. The risks involved in handling hydrofluoric acid and the poor results it yielded may render it less indicated as a surface conditioning agent. This preparation procedure behaved similarly in this study.

Air abrasion showed better results in this study, as well as abrasive grooves. This finding is in agreement with several studies conducted previously.^{10,23} Depending on the type of resin composite treated, differences were observed between preparation methods with abrasive blasting. Durafill's shear force performed better with silane application than without it, unlike Charisma, whose higher values were obtained when silane was not applied. These data corroborate the findings of Martin and Lopez¹⁶ but contrast with those of DeSchepper, Tate and Power, who reported improved bond strength with the use of silane after abrasive procedures.²⁴ Silane application seems to improve the bond strength of the composite after acid etching, blasting and preparation with burs and stones, when it comes to porcelain repairs.²⁵ Silane application and the use of adhesives seems to slightly increase bond strength compared to using

adhesive only. The same results were obtained after subjecting both Durafill and Charisma to sodium bicarbonate blasting and grooves with burs and stone, i.e., an increase in shear strength due to silane in areas etched with phosphoric acid (Figs 5 and 6).²⁶ Although Söderholm and Roberts²⁷ did not find statistically significant differences in repairing composite surfaces after using silane, this study found an increase in shear strength of surfaces prepared with sodium bicarbonate blasting and grooves with burs and stone in specimens filled with Durafill. Silane may be responsible for the increase in shear forces observed on micro-hybrid resin surfaces.

The action of coupling agents is justified to the extent that it facilitates infiltration of resin composite in microscopic grooves produced in a prior surface preparation.¹⁶ The process of groove creation enables the detachment of inorganic particles, leaving exposed a deeper region in the composite's resin matrix. Given that there is a structural similarity between orthodontic bonding materials and the resin composites evaluated in this study, shear strength values would be expected which allow the clinical longevity of brackets to be bonded. This increase in shear strength after silane application, however, was not observed in Charisma, which might indicate changes in the exposure of deeper portions of the inorganic matrix material when compared to micro-hybrid composites (Fig 6). Additional studies are needed to substantiate this laboratory finding. ARI differences were observed which may be related to the unique features of the two types of composites and the efficiency shown by different types of surface treatments (Table 4). ARI differences were not found between the use or non-use of silane in many groups, a finding that agrees with some studies.²⁷

In terms of the mean yield strength values of the brackets relative to the specimens, it should be noted that according to Lopez,⁹ such yield strength should withstand at least 7.8 MPa of shear force, an amount sufficient to ensure successful bonding in clinical practice.

In this case, only the specimens filled with Durafill after preparation with bicarbonate blasting, bur and stone, reached means that were above the means suggested by Lopez.⁹ As for Charisma, the surface preparation that obtained values greater than 7.8 MPa were the groups that had previously been treated with aluminum oxide blasting, sodium bicarbonate blasting, bur and stone without silane (Table 2). Thus, in Durafill micro-particulate restorations of anterior teeth, orthodontic brackets would be less prone to bond failures if the surfaces were first prepared with bicarbonate blasting, bur or stone, without silane. However, in the case of brackets bonded to esthetic restorations in posterior teeth restored with Charisma micro-hybrid composite, the best surface preparation according to the results would be aluminum oxide and bicarbonate blasting, bur and stone without silane.

There seems to have been operator influence in surface preparation since standard deviation values relative to the mean values were not low, especially in groups prepared with bicarbonate blasting, bur and stone. This suggests that choosing a method for bonding orthodontic brackets onto resin composites can be influenced by the individual orthodontist performing the procedure (Table 2).

CONCLUSIONS

- 1. Charisma, the micro-hybrid resin composite, displayed higher yield strength during shear testing of the brackets than Durafill, the micro-particulate resin composite.
- 2. Silane application improved surface preparation of Durafill when performed in conjunction with bicarbonate blasting and grooves with bur and stone.
- 3. When performed on Charisma, silane application worsened the means obtained for the yield strength of orthodontic brackets in all preparation surfaces, with the exception of phosphoric acid.
- 4. Surface preparation with bicarbonate blasting, bur and stone experienced greater operator influence than others.
- 5. Surface treatment with sodium bicarbonate blasting, bur and stone in both types of resin composites tested showed a mean yield strength greater than 7.8 MPa, a value normally recommended and sufficient to ensure successful bonding in clinical practice.

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