ORIGINAL ARTICLE

Assessment of the effect of different surface treatments on the bond strength of brackets bonded to acrylic resin

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Abstract

Objective: To evaluate the influence of the surface treatment of acrylic resins on the shear bond strength of brackets bonded with composite resin. **Material and Methods:** Were fabricated 140 discs with autopolymerizing acrylic resin (Duralay®) and divided them into 14 groups (n = 10). In each group, the specimens received a different type of surface treatment. Group 1- untreated surface (control), Group 2- silane, Group 3- aluminum oxide blasting (AOB), Group 4- AOB + silane, Group 5- diamond bur, Group 6- diamond bur + silane, Group 7- hydrofluoric acid, Group 8- hydrofluoric acid + silane, Group 9- phosphoric acid, Group 10- phosphoric acid + silane, Group 11-methylmethacrylate monomer (MMA), Group 12- MMA + silane, Group 13- plastic conditioner (Reliance®); Group 14- plastic conditioner (Reliance®) + silane. After surface treatment the specimens were analyzed using a surface roughness tester. Subsequently, standard edgewise central incisor brackets (Morelli®) were bonded using Transbond XT® light-cure adhesive system, according to the manufacturer's instructions. **Results:** The silane-based wetting agent had no statistically significant effect on bond strength values. Treatments with AOB and bur generated the highest topographical changes on the surface of acrylic resin as well as the highest roughness values. A nonlinear correlation was found between bond strength and surface roughness. Monomer + AOB treatment yielded the highest bond strength values. **Conclusions:** Silane failed to increase the bond strength between brackets and acrylic resin. We encourage further studies on this subject since the bond strength achieved in our study was extremely low.

Keywords: Orthodontics. Acrylic resin. Composite resin. Brackets.

INTRODUCTION

Given the increasing number of adults that seek orthodontic treatment some procedures used in the office have had to be modified to fit this new patient profile. This age shift means that orthodontics is no longer just sought as an end in itself but also as a means towards achieving other goals. In other words, orthodontic treatment is now multidisciplinary and, as such, the treatment plan, previously developed by the orthodontist alone, must be carried out in conjunction with periodontists, endodontists, surgeons, GPs and implant dentists.[2,11] As a result, orthodontic treatment may serve as a pre-prosthetic activity as it is confronted not only with natural teeth but also extensive composite resin restorations, implants, metal crowns, ceramics and provisional restorations.[6,7] In short, given the fact that archwires and brackets are still needed to achieve orthodontic movements, a wide range of surfaces exists where brackets need to be bonded. Moreover, effective bracket bonding still plays a key role in the success of orthodontic treatment.

Bonding techniques comprising etching, primer and adhesive have been widely accepted and documented for bonding brackets to the surface of natural teeth.[1,3,9,12] They afford more than the necessary bond strength to enable orthodontic treatment.[10] Many studies have been conducted in attempts to accomplish a similar performance when bonding orthodontic attachments to amalgam, porcelain, composite resin and metal surfaces.[2,13,14] In fact, until recently, it was considered inconceivable that any clinically acceptable bond strength could be achieved in areas other than the enamel, particularly in the lower posterior region. However, advances in orthodontic materials and the development of new techniques have shown that the bonding of orthodontic attachments to surfaces other than enamel is also possible.[8,13-17]

However, the literature is still rather sparse when it comes to the bonding of orthodontic attachments to acrylic resin provisional restorations. This dearth of scientific studies leads practitioners to resort to empirical protocols, which ultimately cause brackets to debond frequently, thereby hindering the orthodontic treatment.

This study was designed to perform an in vitro analysis to measure and compare the roughness of acrylic resin surfaces after applying different surface treatments. It also aimed to characterize these surfaces by scanning electron microscopy (SEM) and assess the bond strength of metal brackets bonded to pretreated acrylic resin surfaces using composite resin. Furthermore, we assessed the effects of applying a silane-based wetting agent to the acrylic resin in terms of bond strength between bracket and acrylic resin.

MATERIAL AND METHODS

Table 1 lists the materials used in this study and their composition. All materials were used as received without adding any further formulation and/or treatment.

One hundred and forty acrylic resin (Duralay®) specimens were fabricated for bonding 40 central incisor standard Edgewise brackets (Morelli®, slot 0.022x0.028-in). The specimens were fabricated using as matrix an acrylic disc with 25 mm diameter and 5 mm in height with a central hole of 8 mm diameter and 5 mm in height, where the heat-polymerized acrylic resin (Duralay®) was inserted (Fig 1). In keeping with the usual procedure of sample preparation, sanding was performed to homogenize the surface of the specimens. We utilized silicon carbide abrasive paper—in decreasing order of abrasiveness (400 and 600)—under refrigeration/lubrication with abundant water. The sanded specimens were cleaned in an ultrasound device with distilled water for five minutes and then dried with hot air.

TABLE 1 - Materials used for surface treatment and bonding of brackets.

Material (Manufacturer)	Composition
Phosphoric acid 37% (DENTSPLY® Indústria e Com. Ltda.) L156380	Phosphoric acid (in aqueous solution, 37 wt% in gel form), silicon dioxide and pigments
Porcelain Conditioner® (DENTSPLY® Indústria e Com. Ltda.) L244578	Hydrofluoric acid (aqueous solution 9.6% in gel form)
Transbond XT Primer (3M- Unitek, Monrovia) 712-035	Triethyleneglycol-dimethac-rylate Bis-GMA
Transbond XT Paste (3M- Unitek, Monrovia) 712-035	Silica Bis-GMA Silane N-dimethyl benzocaine hexa-fluoride-phosphate
Duralay® Powder and Liquid (Reliance, Dental Mfg. Co. Worth) 60482	Powder - copolymer of methyl methacrylate color 81 Liquid - methyl methacrylate
Aluminum oxide blasting (BIO-ART® Ind. de Eq. Odont. Ltda.)	Aluminum oxide 50 µm / 220 mcsh
Plastic Appliance Conditioner® (Reliance, Ortho Prod. Inc) 0473	Composition was not informed
Ceramic Primer® (3M- ESPE, St. Paul) 551441000	Metacriloxipropil-trimetoxisi-lano (MPTS) 1 wt% Ethanol-water solution with acetic acid - pH = 4 - 99 wt%
Diamond bur # 3145 (KG- Sorensen) 1076/1103	Diamond and Stainless Steel

The surfaces were then treated in preparation for bonding the brackets. Fourteen different surface conditioning methods were used, which are summarized in Table 2. These conditioning methods are detailed in Table 3, by group. Ten samples were tested using 14 experimental conditions. The brackets were bonded to the acrylic resin using Transbond XT primer and adhesive (composite resin).

Before bonding the brackets, three specimens from each group were analyzed with a roughness tester to determine the effect of the various treatments on the surface topography of the samples. This procedure was adopted because the roughness of the substrate is a parameter that significantly influences how the adhesive spreads.[17]
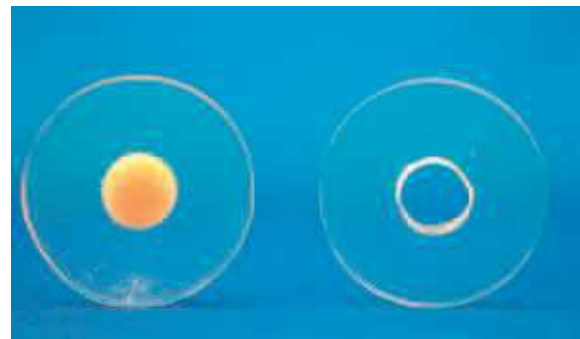

FIGURE 1 - Acrylic matrix with 25 mm diameter and 5 mm in height, containing a central 8 mm hole used as a basis for preparing the specimens.

TABLE 2 - Experimental conditions for bonding of brackets.

Groups	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Primer	x	x	x	x	x	x	x	x	x	x	x	x	x	x
Silane		x		x		x		x		x		x		x
Aluminum oxide blasting			x	x										
Diamond bur					x	x								
Hydrofluoric acid							x	x						
Phosphoric acid									x	x				
Monomer											x	x		
Plastic appl. conditioner													x	x

TABLE 3 - Description of surface treatments performed before bonding.

Group	Surface Preparation
1	(Control) - no surface preparation other than sanding
2	(Control) - with silane layer, allowed to act for 60 seconds. Subsequently, the specimens were dried with compressed air free of moisture and oil
3	Surface was abraded with aluminum oxide blasting (50 μm) for 10 seconds, using the Blast Microetcher®, Bio-art operating at a pressure of 80 pounds and at a distance of 1 cm from the surface with the tip of the device forming a right angle relative to specimen surface
4	Same as group 3 + application of silane as in group 2
5	Surface was abraded with diamond bur No. 3145 KG Sorensen positioned parallel to the surface of the specimen, with a rotation of 4000 rpm under water spray. Brushing bur movements were performed on the specimens
6	Same as group 5 + application of silane as in group 2
7	Surface was etched with hydrofluoric acid at 9.6% for 15 seconds. It was then washed with water jets for 60 seconds and dried with compressed air free of moisture and oil
8	Same as group 8 + application of silane as in group 2
9	Surface was etched with hydrofluoric acid at 37% for 30 seconds. It was then washed with water jets for 15 seconds and dried with compressed air free of moisture and oil
10	Same as group 9 + application of silane as in group 2
11	Application of Plastic Appliance Conditioner (plastic surface conditioner). After application there was a 60-second wait, as directed by the manufacturer
12	Same as group 11 + application of silane as in group 2
13	Methyl methacrylate monomer was applied for 60 seconds and dried with compressed air to remove any excess
14	Same as group 12 + application of silane as in group 2

Measurements were made in an automated Surface Roughness Tester, Mitutoyo (Illinois, USA).

In view of the fact that roughness can be characterized by several different parameters, in this investigation we used the arithmetic mean of roughness profiles, called Ra, which gives the arithmetic average of all profile departures from a given baseline (valley depth and peak height). Five roughness profiles of surface texture were measured in each sample in order to evaluate the uniformity of surface treatment of substrates, both in terms of cleaning and the treatment itself.

Topographic analysis of the treated surfaces was also performed using scanning electron microscopy (SEM). Analyses were performed on surfaces coated with a conductive metal layer using an electron acceleration voltage of 10-15 kV, 80 μA current, and secondary and/or backscattered electrons.

After completing surface treatment, we bonded the brackets using Transbond XT adhesive system, according to the manufacturer's instructions. The specimens were then attached to a device especially fabricated for bracket bonding.[18] This metal device was used to help standardize the distance and parallelism between bracket and specimen while providing bond strength and homogeneous polymerization (Fig 2).

Small amounts of Transbond XT adhesive were placed on the bracket bases with the aid of a No. 5 explorer and then pressed against the specimens. The device ensured that each specimen axis remained in an orthogonal position relative to the bracket (Fig 2). The entire assembly was light-activated for 60 seconds through a slit in the device, using an Optilux 400® unit with a light intensity of 500 ± 20 mw/cm². Five minutes after bonding,

the specimens were stored in zip lock bags and kept at room temperature. Twenty-four hours prior to shear bond strength testing, the specimens were stored in water at 37±2° C in conformity with ISO/TS 11405, of 2003.

Shear bond strength tests were performed on a Universal Testing Machine set to apply the load onto the specimen at a speed of 0.5 mm/min.

RESULTS

In examining Table 4, we noted that it was only in the Phosphoric Acid + Silane and Plastic + Silane treatment groups that the combination with silane yielded increased bond strength. In all other groups, the addition of silane caused a decrease in both the mean and the median (Table 5).

For statistical evaluation, two-by-two sample comparisons were carried out in order to check whether or not the silane had any bearing on the bond strength of each surface treatment. The comparison was performed by the Mann-Whitney test (Table 5, p value). The choice of this test was due to the fact that the samples did not display a normal pattern.

Overall, silane application did not stabilize bond strength measurements, causing instead increases and decreases in measurement variability, as shown in Table 4.

DISCUSSION

The effect of various treatments on the surface roughness of acrylic resin is shown in Table 4. It is noted that the groups undergoing mechanical bur treatment yielded much higher roughness values than all other groups, including those whose surface was treated with aluminum oxide blasting (AOB).

FIGURE 2 - Bonding device used to ensure standardization. Illustration of how the brackets were bonded to the acrylic resin discs.
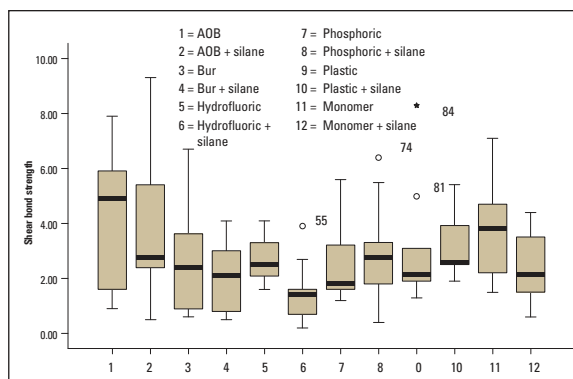

FIGURE 3 - Box plot chart showing variation in bond strength within and between groups.

TABLE 4 - Roughness evaluation (µm).

Groups	Surface Treatment	Mean	Standard Deviation	Cv
G1	Control	0.35	0.02	0.057
G3	AOB	0.85	0.23	0.270
G5	Bur	7.77	1.48	0.190
G7	Hydrofluoric	0.38	0.04	0.105
G9	Phosphoric	0.47	0.06	0.127
G11	Monomer	0.55	0.14	0.254
G13	Plastic	0.45	0.03	0.066

TABLE 5 - Shear bond test experimental results and descriptive statistics of the influence of silane on bond strength (MPa).

Treatment	Observations	Mean	SD	Median	Cv	P-value
AOB	10	4.23	2.32	4.90	0.55	0.545
AOB+silane	10	3.65	2.48	2.75	0.68	
Bur	10	2.69	1.88	2.40	0.70	0.384
Bur+silane	10	2.07	1.24	2.10	0.60	
Hydrofluoric	10	2.75	0.86	2.50	0.31	0.006*
Hydrofluoric+silane	10	1.47	1.13	1.40	0.77	
Phosphoric	10	2.39	1.34	1.80	0.56	0.405
Phosphoric+silane	10	2.94	1.88	2.75	0.64	
Plastic	10	2.97	2.16	2.15	0.73	0.210
Plastic+silane	10	3.13	1.07	2.60	0.34	
Monomer	10	3.66	1.74	3.80	0.47	0.880
Monomer+silane	10	2.44	1.21	2.15	0.50	

It is noteworthy that although the roughness of bur-treated surfaces was far superior to the others, their uniformity was adequate since their coefficient of variation was less than 30%, similar to that achieved with other treatments. It was also noted that treatment using phosphoric and hydrofluoric acid etching was unable to significantly alter acrylic resin topography as the surface roughness of these groups was similar to the control group. Treatments using conditioner and monomer also produced little change in acrylic resin roughness in comparison with the control group.

Experimental shear bond strength test results are shown in Figure 3. We were unable to determine the shear bond strength of the control groups (groups 1 and 2) because the brackets came off as we were handling the samples. This means that these control samples with no surface treatment had very limited bond strength. As shown in Table 4, standard deviation was high for all groups although the bonding procedure was performed as reproducibly as possible. A similar behavior has been observed by other authors.[19]

Table 5 shows the minimum and maximum values for each group, the first and third quartile and the median (represented by the solid line), as well as possible outliers. This chart shows shear bond strength variability within and between groups and reveals that the data do not follow a normal distribution and must therefore be analyzed by a nonparametric test.

By examining the data shown in Table 5 and in Figure 4 the influence of silane on the bond strength of the different surface treatments can be assessed. It becomes obvious that only the treatments using phosphoric acid and plastic conditioner yielded an increase in bond strength when the surface treatment was associated with silane. In the other groups, including the AOB treatment group, the use of silane caused a reduction both in mean and median bond strength. A statistical evaluation of these results was made by comparing the samples subjected to the same surface treatment, with and without the application of silane (e.g., groups 3 and 4).

This comparison was performed using the Mann-Whitney test to determine whether the use of silane was able to significantly alter bond strength. The Mann-Whitney test is indicated when samples do not show a normal distribution pattern, as was the case here. The test result is expressed by the p value in Table 5.

The hydrofluoric acid group (p = 0.006) was the only group that showed significant variation. However, the silane caused a decrease in bond strength, and not an increase, as is the case when bonding to porcelain.[14,15,16]

Figure 4 shows the correlation between roughness and shear bond strength. The data indicate that initially a small increase in roughness results in a large increase in bond strength. However, as soon as a 'critical' roughness value is reached the bond strength remains stable.

Figure 5 shows the microstructure of the various surfaces investigated in this study. Figure 5A (A1, A2 and A3) represents the control group, i.e., the surfaces have been sanded and are now ready to receive surface treatment. A number of grooves can be seen which result from the initial sanding process. They are homogeneously distributed in a mostly unidirectional arrangement. Acrylic resin debris is also visible. Figure 5B (B1, B2 and B3) shows the

untreated areas where silane has been applied. As can be observed, the silane does not alter the acrylic resin topography since the surface features that can be viewed here are basically the same as in Figure 5A.
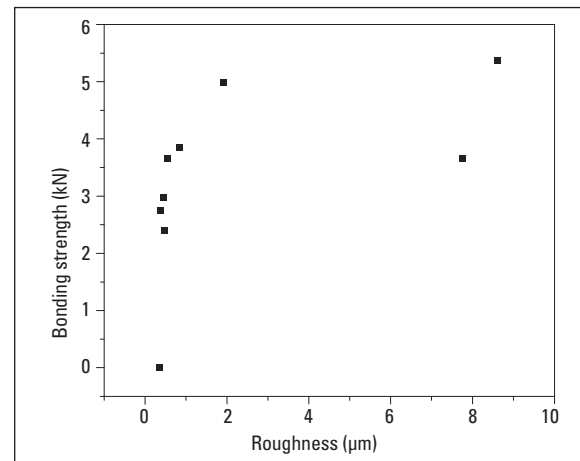

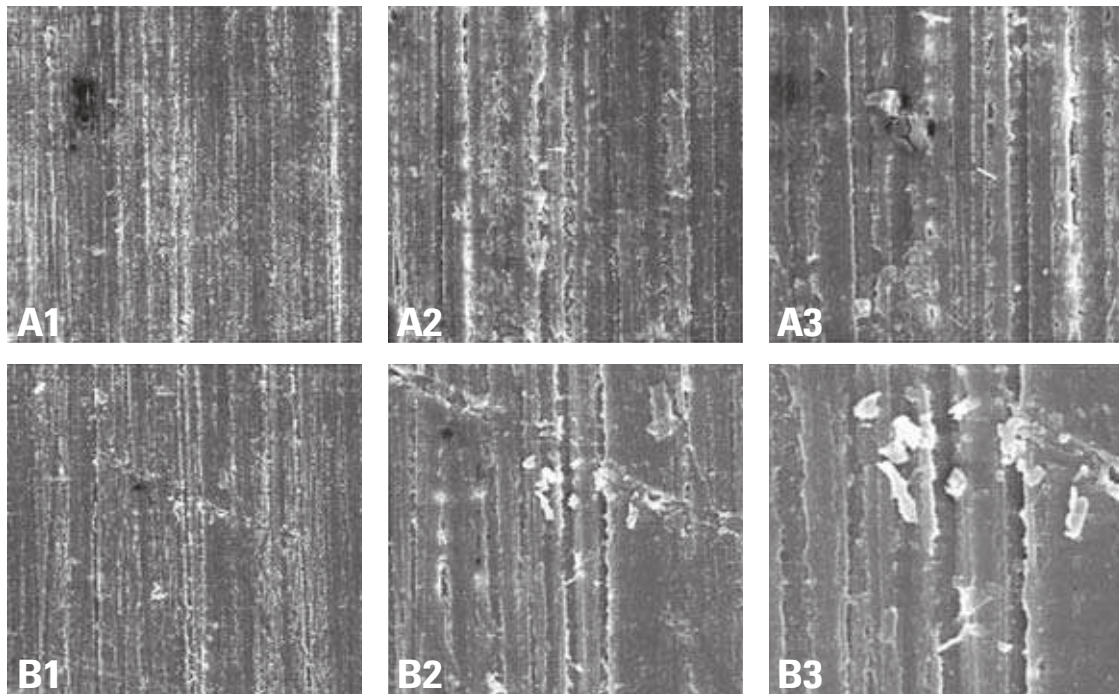FIGURE 4 - Variation in bond strength / roughness.

FIGURE 5 - Control groups surface topography: **A)** Group 1 (control), **B)** Control + Silane (Image magnification for each group: 1 = 200 times, 2 = 500 times and 3 = 1000 times).

When comparing the control group (Fig 6A) with the AOB group (Fig 6C) and bur-treated group (Fig 6D) we can see that these treatments were able to completely alter the topography of acrylic resin. In the AOB group, the surfaces were evenly blasted (C1), generating a uniform topography with a nearly equiaxed relief composed of valleys and peaks. The bur-treated surface (D) exhibits rounded lumps and a pronounced relief variation, which was confirmed by measurements taken with the roughness tester (7.77 Ra). As shown in Figure 4, increases in roughness also increase shear bond strength. This finding is in agreement with results achieved using the mechanical inter-locking bonding mechanism.[20]

Acid etching (hydrofluoric and phosphoric) resulted only in an apparent removal of impurities generated by the original sanding process, as can be shown in Figure 7. This seems to be the great advantage of using acid on the acrylic resin as the presence of debris on the surface has been known to affect bond strength.
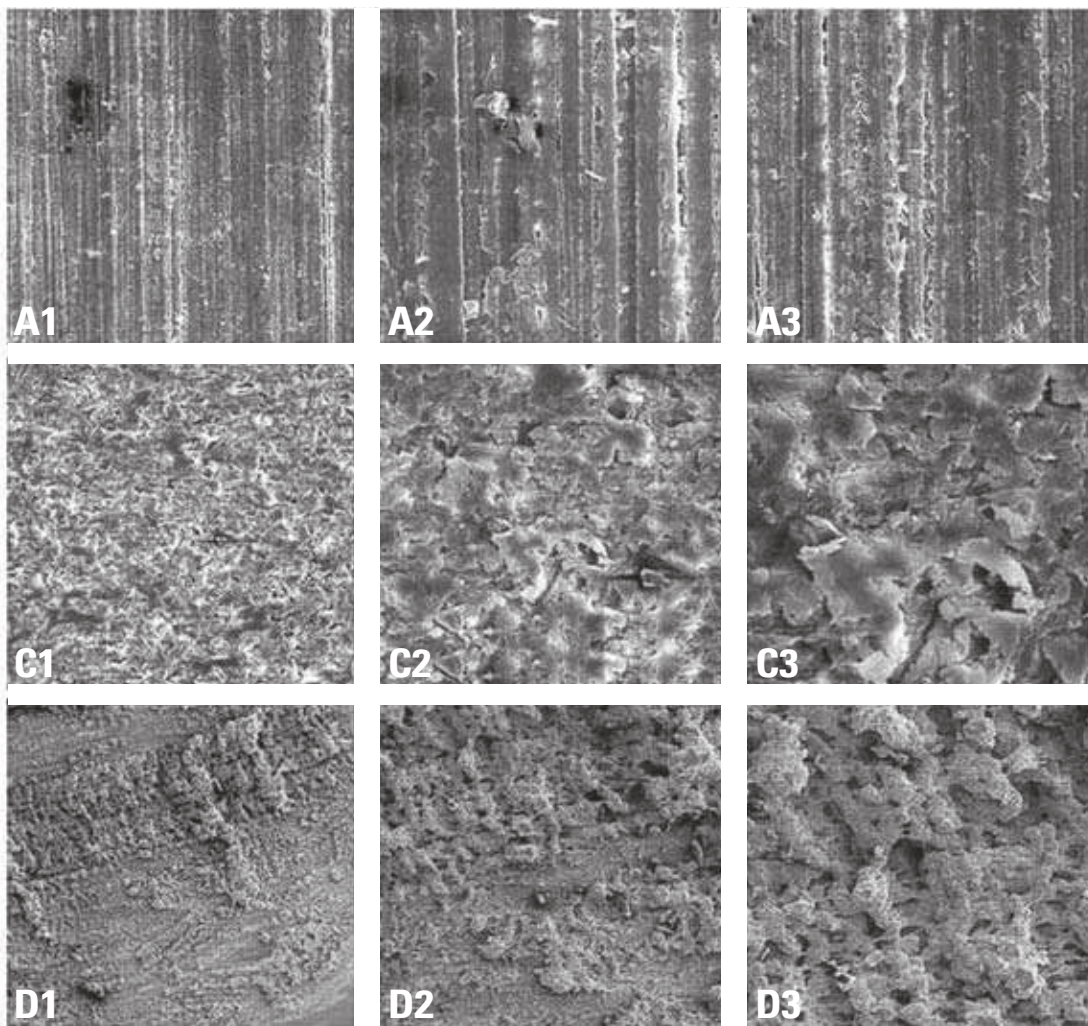
Figure 8 shows the effect of monomer and

FIGURE 6 - Acrylic resin surface topography. **A** = Group 1 (Control), **C** = Group 3 (Aluminum Oxide Blasting), **D** = Group 5 (Bur).

plastic conditioner treatments on the topography of acrylic resin. A comparison between the control group (A) and the monomer coated group (G) shows a total change in the topography of acrylic resin with the formation of randomly distributed micro-cracks. This modification in the substrate increased the bond strength of the composite resin (3.66 MPa) and led to an increase in surface roughness compared with the control group (from 0.35 Ra to 0.55 Ra). Although not within the scope of this investigation, studies should be conducted to find out whether these micro-crack substructures were generated in the acrylic resin or whether the application of the monomer covered the acrylic resin surface with a layer which, possibly by contraction, generated the system of micro-cracks. The area treated with surface plastic conditioner (group H) maintained the topographical structure (control A) originally caused by sanding. However, surface conditioning seems to produce a coating that acts as a glaze over the acrylic resin and negatively affects the bonding process.
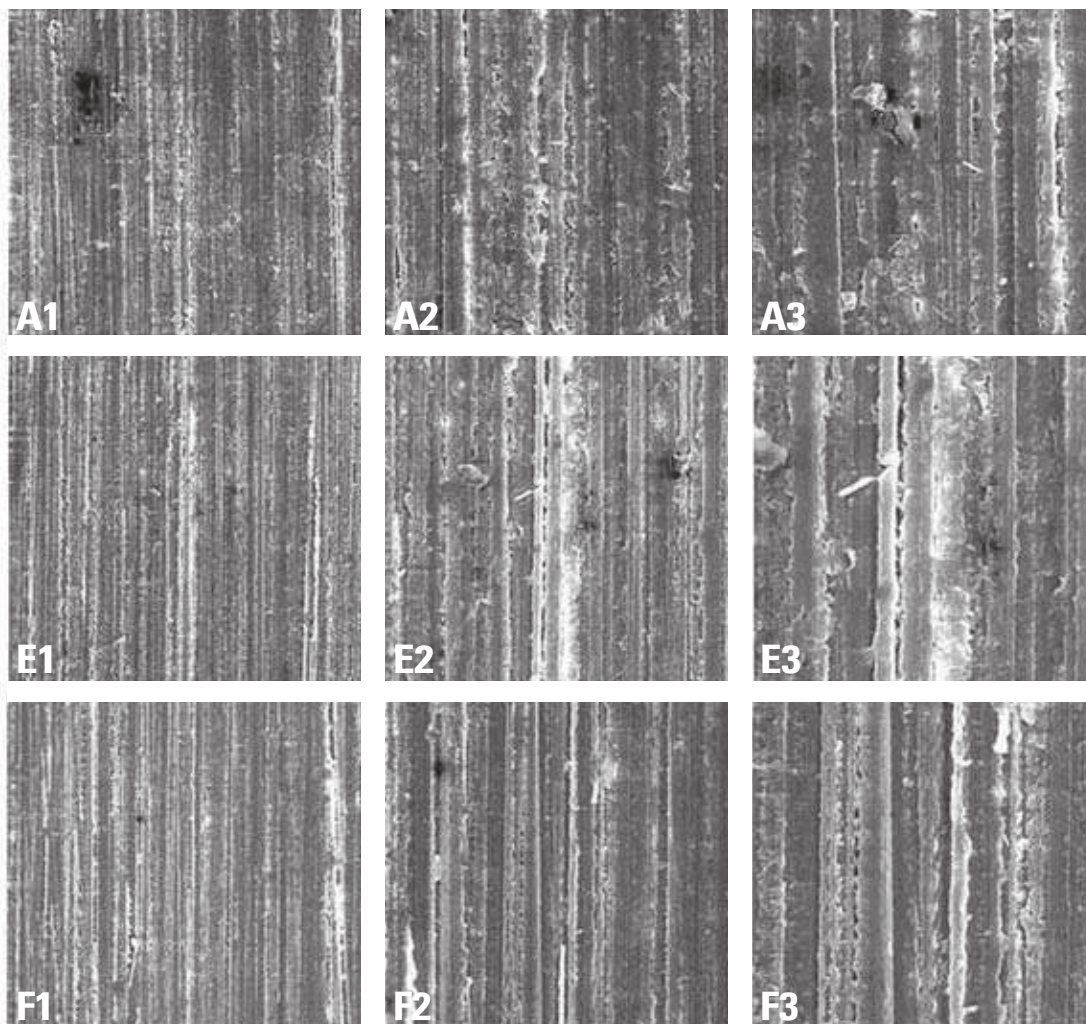

FIGURE 7 - Acrylic resin surface topography. **A** = Group 1 (Control), **E** = Group 7 (Hydrofluoric acid), **F** = Group 9 (Phosphoric acid).
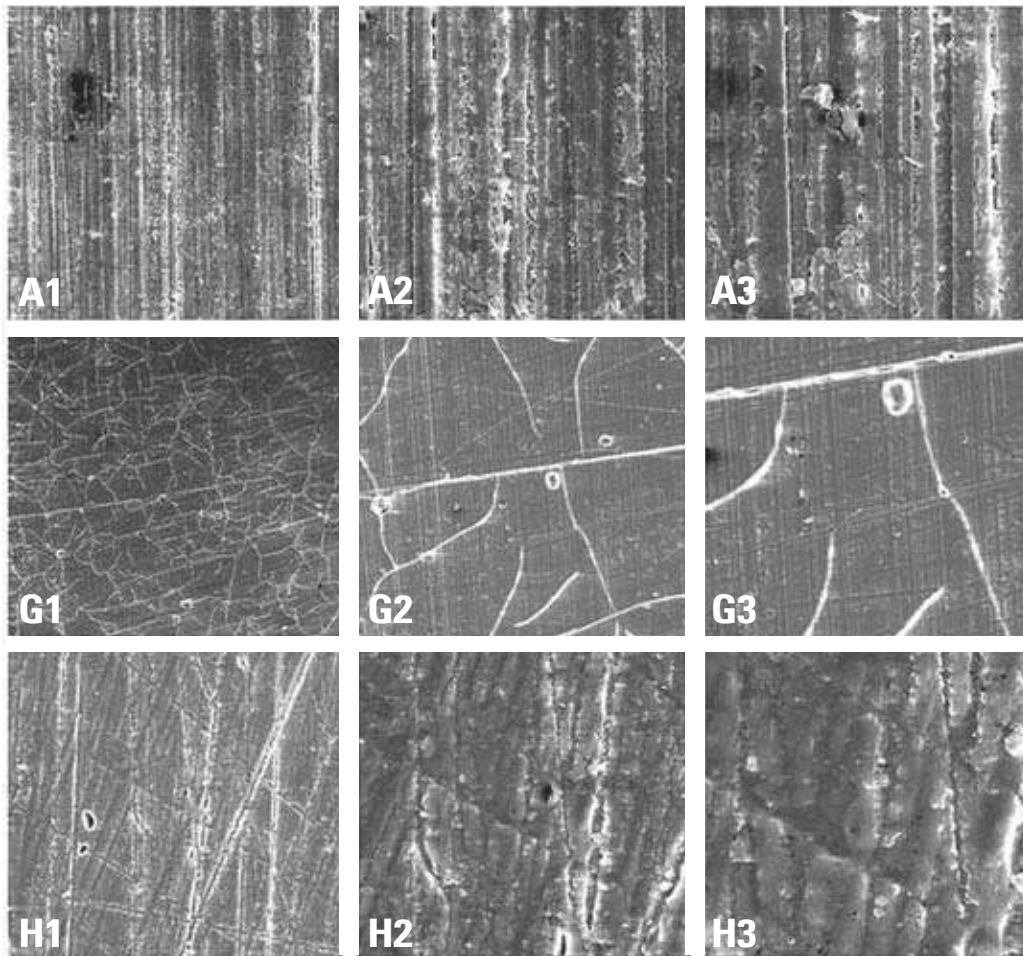
FIGURE 8 - Acrylic resin surface topography. **A** = Group 1 (Control), **G** = Group 11 (Monomer), **H** = Group 13 (Plastic Surface Conditioner).

CONCLUSIONS

Based on the experimental findings achieved through this study we have concluded that:

I) Silane application does not contribute to enhance bond strength values, leading only to non-significant variations. Thus, the use of silane after treating the acrylic surface, within the parameters of this study, does not improve the bond strength of orthodontic brackets.

II) No linear correlation was found between bond strength and surface roughness of acrylic resin. A critical roughness value was found which must be reached if maximum bond strength is to be achieved. Within the experimental parameters used in this research, such critical roughness value is approximately 2 μm.

III) Treatments involving aluminum oxide blasting (AOB) and burs produced the greatest changes in acrylic resin surface topography and produced greater roughness, which contributes to increase the bond strength between resin and bracket.

IV) Acid etching did not alter the original topography significantly, acting primarily as a

means of cleaning debris generated by the process of preparing the original acrylic resin.

V) Treatment with monomer caused a series of micro-cracks on the acrylic resin surface. These micro-cracks actually increased surface roughness and, consequently, enhanced bond strength. The plastic conditioner seems to produce a covering layer over the surface that hinders the contact between acrylic resin and bracket, thereby compromising bond strength.

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