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Effect of cavity preparation method on microtensile bond strength of a self-etching primer vs phosphoric acid etchant to enamel

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Abstract This study evaluated the effect of cavity preparation using air abrasion or carbide bur on bond strength to enamel treated with a self-etching primer (*Tyrian SPE*) or a phosphoric acid etchant. Twenty-four molars were divided into three groups: high-speed; *standard* handpiece (ST air abrasion) or *supersonic* handpiece (SP air abrasion) of the same air-abrasive system. The enamel surfaces were treated with one of the two etchants and the same adhesive agent *One Step Plus*, and then composite buildups were done with Filtek Z250. After 24 h at 37 °C, beams (0.8 mm^2) were obtained and subjected to tensile stress in a universal testing machine (0.5 mm/min). The data were submitted to analysis of variance and Tukey's test ($P < 0.05$). For the conditioning agents, it was observed that the specimens conditioned with phosphoric acid presented superior results than the specimens that used *Tyrian SPE*. For the preparation techniques, it was verified that the SP air abrasion groups showed the highest bond strengths and carbide-bur groups presented the lowest bond strengths when the specimens were conditioned with *Tyrian SPE*. It can be concluded that the influence of the cavity preparation method was dependent on the conditioning system used, only when using carbide-bur preparation technique.

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Introduction

With advances in adhesive systems and composite resins, emphasis in restorative dentistry has been placed on conservative cavity design [1, 2]. Adhesive restorations require only the removal of tooth structure necessary to provide access, eliminate the carious lesion and produce a proper bonding surface [2, 3].

Conservative cavity preparation, which includes handpieces and burs, leads to undesirable removal of tooth structure [3]. Due to this excessive loss of sound tissue and the significant discomfort or fear that this method may cause to the patient, efforts have focused on new techniques such as air abrasion [2, 3].

Air abrasion in dentistry was first introduced by Black in 1945 [4], whose interest was focused on the possibility of cutting human teeth efficiently without creating heat, vibration, pressure and noise, offering improved patient comfort. In the 1950s, this technique had limited success because it did not allow suitable cavity design for amalgam and gold as the high-speed handpiece.

Modern air abrasion units employ a high-speed stream of purified aluminum oxide particles propelled by high-velocity air pressure [5]. The abrasive particles that strike the tooth remove small amounts of tooth structure producing cavity contours that are compatible with the needs of adhesive dentistry. This technique has been also proposed for removing carious lesions [6, 7] and existing restorations [8], and for alternative treatment of enamel and dentin surfaces instead of acid etching [9, 10]. The air abrasion provides a rough irregular surface and increases its wettability for the adhesive systems [11, 12].

This technique was used for surface treatment and restored employing a self-etching adhesive system, showing a better value of bond strength than to diamond-bur

group [13]. However, the bond strength of this system was not evaluated in dental cavities prepared using abrasive particles.

Both traditional burs and air abrasion device remove caries [7] and prepare the cavity surface [14] for the subsequent adhesive restoration. But, since the surface of the cavity preparation produced by these devices differ, it is important to know what effect the preparation device has on bonding to enamel and dentin. Considering the above-mentioned facts, the aim of the present study was to evaluate the microtensile bond strength of an acetone-based adhesive used as a total or self-etching bonding system to enamel after three different methods of cavity preparation: (1) conventional bur, (2) air abrasion with *standard* handpiece and (3) air abrasion with *supersonic* handpiece. The null hypothesis to be tested was: (1) there will not be influence of cavity preparation technique on bond strengths to enamel; (2) the bond strengths over air-abraded smear layer covered enamel will not be dependent on the acidity of surface conditioners and; (3) the ultimate strength of the adhesives will not be different among each other for the different cavity preparation techniques separately.

Materials and methods

This study was approved by the Ethics Research Committee on Human Beings of the School of Dentistry of Ribeirão Preto (University of São Paulo, São Paulo, Brazil). Fig. 1 illustrates the specimen preparation method used for microtensile bond strength testing.

Twenty-four human third molars extracted within a six-month period and stored in saline solution were selected and cleaned with water/pumice slurry in dental prophylactic cups. Roots were sectioned 2 mm below the cemento-enamel junction, and crowns were bisected longitudinally in a mesiodistal direction with a water-cooled diamond saw at low speed in a sectioning machine (Minitron, Struers A/S, Copenhagen, Denmark), yielding 48 enamel sections.

Cavity dimensions were standardized using a piece of insulating tape with a mesiodistal width and an occluso-gingival measurement of 4 mm performing an area of 16 mm^2. The depth of the cavity was approximately 1 mm and was calibrated by measuring with a periodontal probe. Three preparation methods were used, and for each tooth, the buccal and lingual surfaces were prepared by different techniques. The specimens were, then, randomly divided into six groups, each containing eight enamel specimens, according to the preparation technique and adhesive system used.

For groups I and II, the cavities were prepared using #58 tungsten carbide bur [12] (KG Sorensen Ltda, Barueri, SP, Brazil) at high-speed with air/water spray and finished with sharp hand instruments. New burs were used after every five preparations. For groups III and IV, cavities were

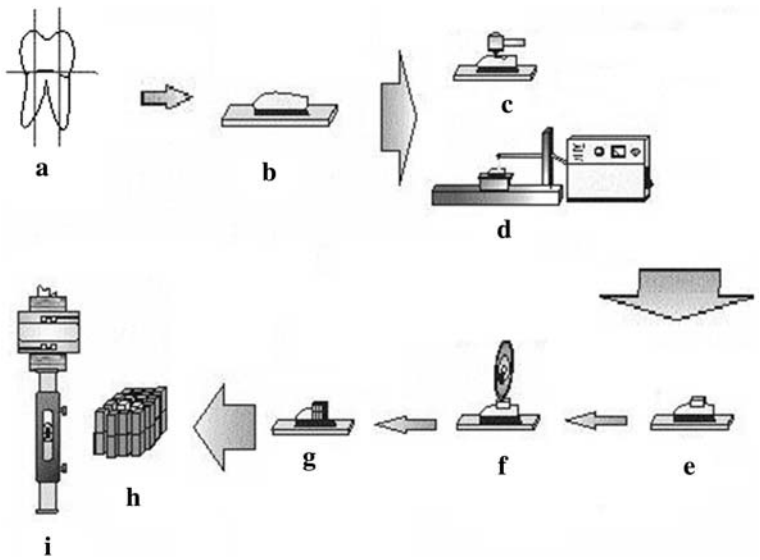

Fig. 1 Schematic showing the specimen preparation method used for microtensile bond strength testing: (**a**) crowns were bisected longitudinally in a mesiodistal direction; (**b**) cavity dimensions were standardized. (**c**) conventional cavity preparation using a #58 carbide bur; (**d**) cavity preparation with air abrasion system with the *standard* or *supersonic* handpiece; (**e**) specimen after conditioning, bonding and composite resin application; (**f**) each specimen was mounted on a low speed saw and serially sectioned perpendicular to the bonded surface; (**g**) each slab was sectioned to obtain up to three beams of approximately 0.8 mm^2; (**h**) four beams from each specimen were selected; (**i**) the specimens were attached to a special testing apparatus and subjected to tensile stress in a universal testing machine at a crosshead speed of 0.5 mm/min

prepared by the *standard* handpiece (ST air abrasion) of the air-abrasive system (Kreative Mach 4.1-New Image do Brazil Imp Exp Ltda, São Paulo SP, 04543-000, Brazil) with a 0.014-inch-nozzle opening using a 4 g/min stream of 27.5 μm aluminum oxide particles at 60 psi air pressure. The cavity preparation was accomplished at a distance of 2 mm at a 90° angle with the tooth surface. The operation of the air-abrasion system was controlled using an experimental apparatus that held both the specimen and the handpiece. The device regulated the distance of the handpiece from the specimens, which were fixed with wax in a the semi-adjustable base that was alternately moved in right-to-left and forward-to-back directions, thereby allowing the air-abrasion beam to provide an accurate preparation of the entire specimen surface.

For groups V and VI, cavities were prepared by the *supersonic* handpiece (SP air abrasion) of the same air-abrasive system, with a 0.012-inch-nozzle opening and all other parameters were equal the groups III and IV. After air abrasion, the specimens were thoroughly rinsed with a vigorous air/water spray for 1 min to remove residual aluminum particles from the surfaces.

After cavity preparation, the enamel surfaces were treated as follows: groups I, III and V were etched with a 37% phosphoric acid gel (*Tooth Conditioner Gel*, DENTSPLY Indústria e Comércio Ltda., Petrópolis, RJ, 25665-010, Brazil) for 15 s, rinsed for the same time and gently dried with absorbent paper to keep the tooth surface visibly moist; for groups II, IV and VI it was applied a self-etching primer *Tyrian SPE* (Bisco, Inc., Schaumburg, IL, USA). Equal amounts of Part A and Part B were mixed together getting a purple color, then, the mixture was applied with a pellet for 10 s and the excess was removed until the purple color had completely disappeared, but keeping the tooth surface visibly moist. After the conditioning step, the adhesive agent *One Step Plus* (Bisco, Inc., Schaumburg, IL, USA) was placed by applying two successive coats, brushed for 10 s, gently dried for 10 s to evaporate the solvent, and finally light cured for 10 seconds with a visible light curing unit with an output of 450 mW/cm^2 (XL-3000, 3 M ESPE, St Paul, MN, USA). Resin composite build-ups were performed using a light-cured hybrid composite (Filtek Z250, 3 M ESPE, St Paul, MN, USA) in two 1 mm increments that were separately light-activated for 40 s. Specimens were stored in distilled water at 37 °C for 24 h.

For the microtensile bond strength test, each specimen was first mounted on a low speed saw and serially sectioned perpendicularly to the bonded surface to produce a minimum of four enamel/composite slabs per specimen. Each slab (0.9 mm thick) was sectioned to obtain up to three beams of approximately 0.8 ± 0.2 mm^2. For each specimen, four beams from the center of the slabs were selected from this protocol and the beams of the periphery were discarded, in case the results could be influenced by either the excess or insufficient amount of adhesive system at the interface. The selected beams were attached to a microtensile testing apparatus with cyanocrylate glue (Super Bonder gel, Loctite, Henkel Ltda., Brazil) and subjected to tensile stress in a universal testing machine (Emic, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min. Microtensile bond strength values were calculated in Kgf and converted to MPa. If a spontaneous interfacial debonding occurred while the specimen was mounted, the incident was recorded as 0 MPa.

The data were submitted to two-way analysis of variance and Tukey's test was used to detect differences in means ($P < 0.05$).

After the microtensile bond strength test, the enamel sides of the fractured interfaces were examined under a stereo-light microscope (Nikon 88286, Japan) at 80× magnification to determine the fracture mode that occurred during the microtensile bond strength evaluation. From that analysis, three types of failures were defined: adhesive failure was considered to be that at the tooth/adhesive system interface; cohesive failure occurred in the material or tooth, with no damage to the interface, and mixed was defined as involving both the interface and the material.

Nine additional third molars were used for SEM observation of the adhesive interfaces created by the preparation with burs or air-abrasion system after application of the different surface conditioning agents and the adhesive agent *One Step Plus*. The specimens were prepared in exactly the same way that the microtensile was determined and were further processed for SEM as follows: the specimens were bisected longitudinally in a buccolingual direction with a water-cooled diamond saw at low speed in a sectioning machine (Minitron, Struers A/S, Copenhagen, Denmark), yielding three sections of 1 mm *per* specimen. The sections were carefully polished with #600- up to 4000-grit SiC papers and prepared according to the following protocol: first, the resin/tooth interface was etched with a 37% phosphoric acid solution for 5 s, rinsed, the samples were ultra-sonicated for 10 min, thoroughly washed with distilled water and immediately immersed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4, for 12 h at 4 °C. After fixation, the samples were rinsed with 0.1 M sodium cacodylate buffer several times, sequentially dehydrated in an ascending ethanol series (25–100%), and then immersed in 100% hexamethyldisizilane (HMDS) for 10 min. Specimens were mounted on stubs with their treated surfaces up-faced and sputter-coated with gold. The enamel surfaces and adhesive interfaces were examined with a JSM T330 scanning electron microscope (JEOL, Osaka, Japan) operating at 15 kv, regarding the surface morphology provided by different treatments, and

formation or not of a hybrid layer, focusing on its integrity, homogeneity and continuity along the interface, as well as on the arrangement, uniformity of size and characteristics of hybridization of resin tags. The visual analysis was performed by three examiners that did not have the knowledge of the specimen's group, in order to assess the reproducibility of the interface produced by each technique. Table 1.

Results

The mean microtensile bond strengths and standard deviations are summarized in Table 2.

In general, when analyzed the factor surface conditioning agents, ANOVA showed that the specimens that were conditioned with 37% phosphoric acid (30.74 MPa) presented statistically superior results ($P = 0.018$) when compared to specimens that were conditioned with *Tyrian SPE* (26.64 MPa), regardless the used cavity preparation device. On the other hand, for the factor cavity preparation techniques, it was verified that the SP air abrasion groups showed the highest values of bond strength (31.82 MPa) and were not statistically different from ST air abrasion groups (28.28 MPa). These last groups, however, were not statistically different from carbide-bur groups (25.98 MPa), which presented the lowest value of bond strength when the specimens were conditioned with *Tyrian SPE*. In interactions between the conditioning agent and tooth cavity preparation devices, it can be observed that the group Carbide Bur using *Tyrian SPE* showed inferior adhesion phosphoric acid and the groups of SP air abrasion (Table 2).

Failure analysis revealed the highest amount of adhesive failures for all the groups tested. Some specimens exhibited mixed failures, considering that it had mostly occurred in the group that associated SP air abrasion with *Tyrian SPE*. A few specimens showed cohesive failures in enamel, however, the amount of this type of failure was higher in the groups that associated even carbide bur or SP air abrasion with phosphoric acid.

Table 2 Microtensile bond strength means (MPa) and standard deviation

	37% Phosphoric acid	*Tyrian SPE*
ST air abrasion	29.05 (±5.43)[ab]	27.52 (±7.61)[ab]
SP air abrasion	32.64 (±7.53)[a]	31.00 (±5.34)[a]
Carbide bur	30.54 (±8.08)[a]	21.40 (±5.92)[b]

Data marked with same superscript are not significantly different from one another

The analysis of SEM micrographs revealed that, in general, the air-abraded cavity preparations presented irregular bonding interfaces, different from the ones prepared by rotary instruments. These irregularities of air-abraded interfaces allowed the formation of a non-homogeneous hybrid layer.

In addition, this study analyzed the interfaces of lateral and pulpal walls of cavity preparations (Fig. 2). It was observed that the interfaces of lateral walls were similar in both preparation techniques. However, when analyzing the interfaces of pulpal walls, were verified more irregularities than the air-abraded specimens. It was also observed the formation of a hybrid layer on lateral walls, but it was less evident, with fewer resin tags formation and tenuous adhesive layer thickness, regardless of the preparation device used. On the other hand, the pulpal wall presented a homogeneous thickness of the adhesive layer.

The analysis of adhesive/enamel interfaces (Fig. 3) produced by oxide aluminum particles followed by the application of phosphoric acid showed the formation of a hybrid layer and resin tags similar to the rotary preparations, but contrary to this last, they presented a tenuous adhesive layer thickness. The application of self-etching primer after air-abraded preparation allowed the formation of resin tags with lesser extent than the acid conditioned

Table 1 List of materials tested

Product name	Components	Batch #	Manufacturer
Tyrian SPE	Part A: Ethanol	0300014733	Bisco, Inc. 1100 W Irving Park Road, Schaumburg, IL 60193 USA
	Part B: 2-Acrylamido-2-methyl propanesuflonic acid		
	Bis (2-(methacryloyloxy)ethyl) phosphate	0300014734	
	Ethanol		
One-step plus	Biphenyl dimethacrylate	0300014199	
	Hydroxyethyl methacrylate		
	Acetone		
	Dental glass		
Tooth conditioner gel	37% Phosphoric acid	6656	DENTSPLY Indústria e Comércio Ltda., Petrópolis, RJ, 25665-010, Brazil
	Coloidal silica		

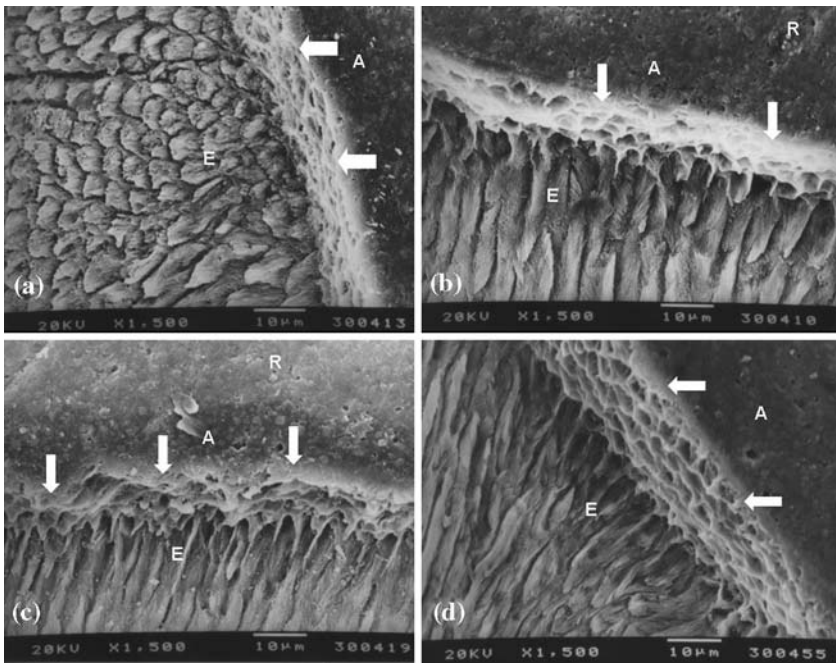

Fig. 2 Enamel/adhesive interfaces of pulpal and lateral walls of conventional and air-abraded cavity preparations. (**a**) bonding interface of lateral wall of cavity prepared by high-speed handpiece and treated with phosphoric acid. (**b**) bonding interface of pulpal wall of cavity prepared by high-speed handpiece and treated with phosphoric acid. (**c**) bonding interface of pulpal wall of cavity prepared by air abrasion with *standard* handpiece and treated with phosphoric acid. (**d**) bonding interface of lateral wall of cavity prepared by air abrasion with *standard* handpiece and treated with phosphoric acid. Hybrid layer (arrows), adhesive (A) and enamel (E)

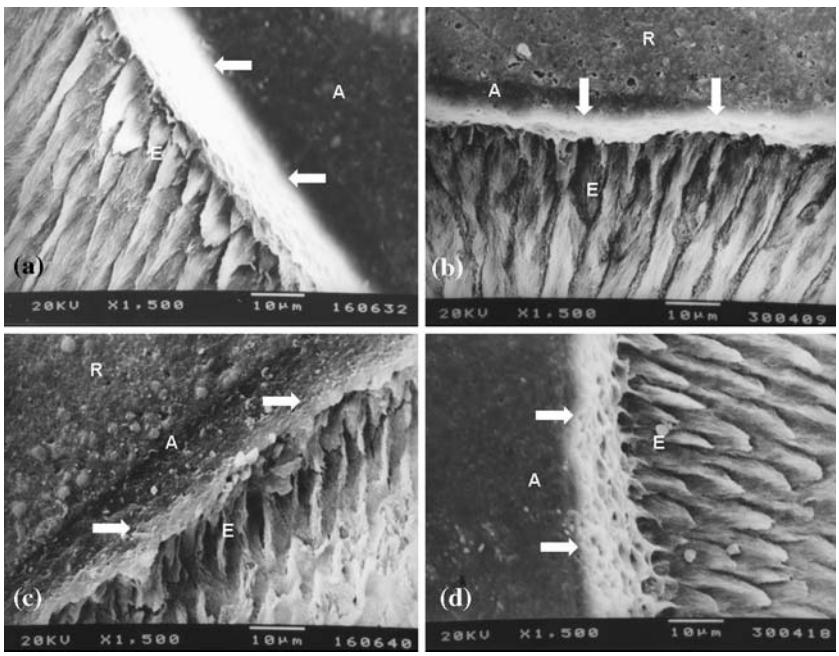

Fig. 3 Enamel/adhesive interfaces**.** (**a**) cavity prepared by air abrasion with *supersonic* handpiece and treated with phosphoric acid. (**b**) cavity prepared by air abrasion with *standard* handpiece and treated with self-etching primer. (**c**) cavity prepared by air abrasion with *supersonic* handpiece and treated with self-etching primer. (**d**) cavity prepared by high-speed handpiece and treated with self-etching primer. Enamel (E), hybrid layer (arrows), adhesive layer (A) and resin (R)

ones. In general, this preparation device maintained a less suitable interaction pattern than the presented by rotary instruments.

Discussion

The results obtained in the present study showed that the *One Step Plus* adhesive system when used as a total-etch system remains the more reliable bonding technique for enamel, as this system maintained a similar behavior for all cavity preparations techniques used. However, the results of bond tests using the self-etching primer employed in this study, when applied to SP air-abraded cavity preparations, were similar to the results of carbide bur cavity preparations treated with phosphoric acid and even superior to the results of conventional preparations that used the same self-etching primer. Such findings allow observing that the

phosphoric acid conditioning technique seems to suffer lesser influence on the type of device used for tooth preparation.

In a similar manner from the results obtained by the microtensile test, when analysed the morphological aspect of adhesive/enamel interfaces, it was observed that the enamel interface produced by the use of air-abrasive system prior to the application of phosphoric acid presented itself more micromechanical interlocking, formed moderately thick enamel hybridization, than the specimens treated with self-etching primer. In general, it promoted the formation of resin tags with greater extent and absence of gaps. However, the hybridization zones of this technique were inferior to that found with rotary instruments. This difference found in the bonding interfaces that received distinct forms of conditioning the enamel surface, may be due to the fact that the phosphoric acid employed has a greater ability to demineralize this substrate than the acid in the self-etching primer [16].

The self-etching primers' acidic components demineralize through the smear layer and diffuse a short distance into the underlying tooth surface, resulting in the creation of a thin hybrid layer with strong bonds mainly to dentin [17, 18]. However, the self-etching primers do not etch as well as a 35 or 37% phosphoric acid etchant because of their relatively high pH [19], due to the patterns etching is dependent upon aggressiveness of the acids used and/or etching time [20]. Therefore, it is believed that bond strengths of self-etching primer bonding systems to enamel could be affected by differences in the quantity of residual smear layer left on the surface due to the weak acidity of these etchants. The inferior bond strength in this group reflected the mild etching effect of this system on enamel. In this context, it was verified that the phosphoric acid, when applied to air-abraded enamel, allowed the creation of a more suitable surface to bonding and promoted the formation of a more uniform interface, the new structure that is part enamel and part resin [20], than the presented by the self-etching primer.

Some studies of literature [21–24] evaluated the influence of tooth surface preparation method on the bond strength of total and self-etching adhesive systems to enamel, and most of them reported that the total-etch systems are rather insensitive to the tooth surface preparation mode, while the self-etching systems suffer this influence. It seems to be the problem found with the self-etching primer used in this study, which has 1.7 pH with moderate etching capacity in comparison the total-etch, and the adhesion should be proportional to the strength of the adhesive infiltrated in demineralized tissue [20].

Another factor that must also be considered is that high-speed burs may induce increase in thermal and mechanical stress, which could affect the underlying enamel and promote a decrease in adhesion to this substrate. On the other hand, when using the air-abrasion system for cavity preparation, many studies have reported that the creation of heat, vibration or pressure does not occur [25–27], avoiding the problems associated to conventional preparations. Besides, air-abrasion technique promotes the formation of an irregular surface, because this technique alters the tooth surface morphology, increasing its surface area [9, 11, 28–30]. In this context, it can be observed in the present study that the superficial irregularities promoted by the use of air-abrasion device, regardless the used handpiece, were helpful to the adhesion of *Tyrian SPE/One Step Plus* system to enamel, allowing this to be similar to the adhesion obtained with the conventional acid etchant applied to carbide-bur cavity preparations.

In the same way, Van Meerbeeck et al. [13] showed that the self-etching system *Clearfil SE* presented lower bond strengths to bur-cut enamel than the total-etch system *OptiBond FL*. However, for air-abraded enamel, the difference was nearly significant ($P = 0.0562$, $\alpha = 0.01$).

When comparing the different tip designs employed with air-abrasion system, it was not observed any difference on morphological aspect. This fact may probably be explained by the little difference between the two inner diameter tips of the handpieces used. Also, the abrasive particles scattering that occurred when they were projected against the dental substrate allowed that the amount of particles that crashes into the tooth was similar. Earlier studies verified that tips with bigger inner diameters produced larger and deeper cuts in primary teeth [31] while in permanent teeth there was no difference on the cutting pattern [32]. Nevertheless, these studies did not evaluate the morphology of the resulting substrate promoted by the use of those different tip designs.

In the analysis of the lateral and pulpal walls of the cavity preparations, it was observed that the lateral wall interfaces of air-abraded preparations were similar to the ones obtained through the use of rotary instruments, while the pulpal wall interfaces presented more irregular interfaces. This cutting pattern may be resulted by the 90° nozzle angle of air-abrasion application, where the spray is parallel to the lateral walls, allowing a uniform cutting of these walls. At the same time, this spray concentrates the majority of the abrasive particles to impact the dental substrate on the pulpal wall, promoting more superficial irregularities. The cutting action of the peripheral spray is less efficient because of its low speed and reduced particle concentration [5], on the contrary of what occurs in the center of the tip, where the abrasive particles have maximum cutting force [33]. It was also observed that on the lateral walls there was the formation of hybrid layer, but it was less evident, with fewer resin tags formation and tenuous adhesive layer thickness, regardless the preparation

device used. On the other hand, the pulpal wall presented a homogeneous thickness of adhesive layer. In this context, it may have occurred because of the influence of material's viscosity and the gravity force on the adhesive system applied to the lateral walls. This fact promoted a tenuous thickness of adhesive layer, compromising the great pattern of bonding interface.

Analysing the results obtained in this study, it can be speculated that with the employment of air-abrasion system for tooth cavity preparation it may be possible to increase the freedom of choice for an adhesive system, without creating any type of damage to the adhesion to enamel. However, an accurate comparative analysis becomes difficult since the literature is scarce about studies that evaluate the influence that different tooth preparation techniques have on bond strength to the restorative materials tested.

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