

Effect of Phytic Acid Etching and Airborne-Particle Abrasion Treatment on the Resin Bond Strength

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Objective: This study aimed to evaluate the bond strength of a universal adhesive to dentin (μ TBS) using different time periods of airborne particle abrasion (APA) and two types of acid etching.

Methods: Seventy-two human third molars were divided into 9 groups ($n=8$) according to dentin pretreatment: APA duration (0, 5, or 10s) and acid etching (no acid - NA, 37% phosphoric acid - PhoA, or 1% phytic acid - PhyA). APA was performed at a 0.5 cm distance and air pressure of 60 psi using 50 μ m aluminum oxide particles. Afterwards, two coats of Single Bond Universal adhesive (3M) were applied to the dentin surface. Composite blocks were built using the incremental technique, sectioned into 1×1 mm slices and subjected to microtensile bond strength (μ TBS) testing. Fracture patterns and surface topography of each dentinal pretreatment were evaluated using a Scanning electron microscope (SEM). Bond strength data were analyzed using two-way ANOVA and Bonferroni *post-hoc* tests.

Results: The group that received pretreatment with 5s APA and PhoA presented higher μ TBS values among all groups, which was statistically different when compared with the PhoA, 10APA+PhoA, and 5APA+PhyA groups. PhyA did not significantly influence the bond strength of the air-abraded groups. Finally, adhesive failure was considered the predominant failure in all groups.

Conclusion: Dentin pretreated by airborne particle abrasion using aluminum oxide demonstrated an increase in bond strength when abraded for 5 seconds and conditioned with phosphoric acid in a universal adhesive system.

Keywords: dental air abrasion, dental bonding, universal adhesive, microtensile bond strength, phytic acid, phosphoric acid

Introduction

Universal or multimode adhesives are capable of etching, priming, and bonding, and thus, their content of functional, hydrophilic, and hydrophobic monomers in a single solution. This type of adhesive offers three diverse application methods: self-etching, etch and rinse, and selective enamel etching. Allowing clinicians to decide on a specific adhesive protocol for each clinical situation.^{1–5}

Phosphoric acid (PhoA) etching of enamel is a simple and reliable technique. Nevertheless, dentin etching is considered a complicated substrate for dental adhesion mainly because of its hydrophilicity. PhoA etching efficiently eliminates the formed smear layer and increases the surface roughness, thereby improving adhesion to this particular surface. However, it is well established that the challenge of using PhoA in dentin relies on the difficult control of dentin moisture, which may lead to a collapse of the collagen fibrils, interfering with the infiltration of adhesive monomers, and finally resulting in compromised adhesion to dentin. Furthermore, PhoA etching in dentin has also been associated with the activation of collagen fibril degradation by the matrix metalloproteinase (MMPs) jeopardizing the long-term durability of restorations on the dentin surface. Different etching agents have been examined as possible substitutes for PhoA, such as phytic acid (PhyA).^{6–10}

PhyA, also known as Inositol hexaphosphate, is an important molecule that has various biological functions. The six phosphate groups provide PhyA with high negative charge, antioxidant properties, and chelating ability. This saturated cyclic acid is abundant in plants and is the principal form of phosphorus storage in seeds and bran. PhyA is an abundant, biocompatible, and inexpensive chemical that can be obtained from several plant sources.^{6,11}

In restorative dentistry, the use of PhyA as a dentin etchant has been shown to increase the resin-dentin bond strength. Nassar et al found that PhyA effectively eliminates the smear layer, promoting enhanced resin monomer infiltration through a mild etching mechanism and minimal depth of demineralization, thereby preserving healthy tooth tissue integrity. PhyA also seems to stabilize collagen network morphology by cross-linking with the exposed collagen network. Although, the exact mechanism by which it could enhance the bond strength in dentin remains elusive.^{7,11–14}

Aluminum oxide (Al_2O_3) airborne particle abrasion (APA) is used in restorative dentistry as a pretreatment to enhance enamel and dentin roughness and the interface between the dental and adhesive surfaces. Specifically, in dentin, it is believed to aid in increasing the adhesive system infiltration into the demineralized dentin tubules, thereby improving tensile strength.^{15–20} This bond strength improvement in dentin is highly dependent on factors such as particle size and pressure of the air stream used. Interestingly, some authors have regarded factors such as the duration of APA as not important, often employing a standardized duration of 10 seconds, thus resulting in a lack of data concerning the influence of APA duration and its effects on the dentin surface and bond strength.¹⁵

Thus, this study aimed to evaluate the effect of dentinal pretreatment using aluminum oxide airborne duration on the adhesive properties of a universal adhesive system with two different types of conditioning. The null hypothesis was that there would be no significant difference in dentin bond strength according to the duration of airborne particle abrasion regardless of the tested etchants.

Materials and Methods

Tooth Preparation

Seventy-two intact human third molars were extracted and selected according to the protocol approved by the Ethics Committee in Research of the Piracicaba Dental School, University of Campinas (CAAE 66646123.7.0000.5418). The teeth were obtained from patients undergoing extraction procedures within the department of Oral and Maxillofacial Surgery of the Piracicaba Dental School, after obtaining their written informed consent. Debris was initially eliminated using a scalpel blade and polished using a rubber cup with pumice stone (SS White LTDA; Rio de Janeiro, RJ, Brazil) and water. The teeth were preselected so that only teeth with completely healthy crowns, without cracks or cavities, were used in the study. After this procedure, the cleaned and selected teeth were stored in 0.1% thymol solution (Dinâmica, Piracicaba, São Paulo, Brazil) at 4 °C for no longer than four months.

Adhesive and Restorative Procedure

All teeth were then fixed in acrylic plates with the aid of thermoplastic glue, and the coronal portions were divided from the root portion 2 mm below the cemento-enamel limit and then sectioned 3 mm above the cemento-enamel junction limit through perpendicular sectioning of the tooth element in relation to its long axis. A high-precision metallographic cutter (Isomet 1000, Buehler, Lake Buff, IL, USA) equipped with a high-concentration diamond disc (Isomet Diamond Wafering Blades, Buehler) was used to create the required cuts for the sample preparation. The cutter was rotated at low speed and constantly irrigated with distilled water.

The surface was polished after removing the occlusal enamel and exposing the dentin to the cervical third. The samples were glued to plastic covers to standardize the preparation of the occlusal faces. Each sample had a dentin surface polished with silicon carbide (SiC) sandpaper #600 under constant irrigation of water, using a rotary polisher (AropolE, Arotec, Cotia, SP, Brazil) to flatten and polish the surface for 30s, standardize the smear layer of the dentin surface, and simulate the clinical situation of the use of diamond tips during cavity preparation.

All samples were randomly distributed across nine groups ($n=8$) in compliance with the air abrasion duration (0, 5, or 10s) and etching protocol (no acid-NA, 37% phosphoric acid-PhoA, or 1% phytic acid-PhyA).

The groups subjected to APA had exposed, flat dentin surfaces treated with air abrasion using 50 μm aluminum oxide (Al_2O_3) (Bioart, São Carlos, SP, Brazil) for 5s at a distance of 0.5 cm from the surface, at an approximately 90° angle, utilizing a Microetcher intraoral device (Bioart, São Carlos, SP, Brazil) at 60 psi air pressure, followed by a 10-seconds water rinse and subsequent air drying, or 10s of air abrasion with a subsequent 20-seconds water rinse and air dry.

After this step, the exposed flat dentin surfaces of the specimens were etched or not (NA; control) with 37% phosphoric acid (Ultra-Etch - Ultradent, South Jordan, UT, USA) for 15s or with 1% phytic acid (Sigma-Aldrich, St Louis, MO, USA) for 30s. For both scenarios, a 30-second water rinse was conducted according to the manufacturer's instructions and then dried with hydrophilic coffee filter squares (Melitta, Alcobendas, MAD, Spain), leaving a visibly moist dentin surface.

The samples were then coated with two layers of Single Bond Universal adhesive (3M ESPE, St Paul, MN, USA) using a disposable brush (Microbrush - KG Sorensen, Cotia, SP, Brazil) in light friction movements for 20s each. Then, it was air dried with a light air jet for 10s, excess was removed with a piece of coffee filter, and finally light-cured for 20s using a 3rd generation LED light curing unit (Valo - Ultradent, South Jordan, UT, USA) in standard mode: 1000 mW/cm² for 20s (20 J/cm²).

The restorative procedure involved building a 4 mm in height composite block with Filtek Z250 XT (3M ESPE, Saint Paul, MN, USA) using the incremental technique and a Goldstein spatula Flexi-thin2 (Hu Friedy, Chicago, IL, USA). Each increment of resin composite was light-activated with the 3rd generation LED light-curing device (Valo - Ultradent, South Jordan, UT, USA). Restored samples were stored in distilled water at 37°C for 24 hours.

Performed Analysis

Dentin Microtensile Bond Strength Test (μTBS)

For the microtensile test, the restored specimens were fixed on acrylic plates with sticky wax (Asfer Indústria Química Ltd., SP, Brazil). The tooth was then positioned on a high-precision metallographic cutter (Isomet 1000, Buehler, Lake Buff, IL, USA), equipped with a high-concentration diamond disc (Extex Corp., Enfield, CT, USA), and underwent sequential cuts at low speed with continuous water irrigation. These cuts were made in the mesiodistal direction to create slices. Afterwards, the tooth was repositioned, and cuts were made in the buccolingual direction, resulting in sticks of approximately 1×1 mm in size.

Next, the slices were affixed to the microtensile device (Geraldeli) at their ends using a cyanoacrylate-based adhesive (Three Bond Super Gel, ThreeBond Ltda., Diadema, SP, Brazil), in order to position them parallel to the traction loading.

The samples were then tested on a universal testing machine (EZ Test L Shimadzu, Japan) equipped with a 500 kgf load cell operating at a speed of 0.5 mm/min until rupture occurred. The force needed for specimen rupture, measured in kilograms (kgf), was recorded. The dimensions of the adhesive interface were assessed using digital calipers (Mitutoyo Corporation, Tokyo, Japan).

The fracture strength (MPa) was determined by dividing the maximum recorded force during the test (N) by the bond area (mm²), expressing the result in MPa.

Failure Mode Analysis Under Scanning Electron Microscope (SEM)

Following the μTBS test, the debonded surfaces of each specimen, derived from the dentin microtensile bond strength, were retrieved. They were then affixed onto aluminum stubs, positioning the fractured surface facing upwards, and subsequently coated with a layer of gold (SDC-050 Sputtercoater, Baltec, Balzers, Liechtenstein), in order to be evaluated in the SEM at a magnification of 80 X. Failure modes were classified as: A) Adhesive fracture mode (fracture in the interface between dentin and resin), Cr) Cohesive in resin fracture mode (fracture between the body of the resin), Cd) Cohesive in dentin fracture mode (fracture between the body of the dentin), M) Mixed fracture (cohesive fractured mixed with adhesive fracture). Patterns were quantified and converted to percentages.

Surface Topography Under SEM

For the surface topography and to illustrate the influence of the duration of air abrasion and phosphoric and phytic acid etching treatment on sealed dentin, two extra samples from each of the following groups were separated: 5APA, 10APA,

5APA+PhoA, 5APA+PhyA, 10APA+PhoA, and 10APA+PhyA. The samples were fixed on aluminum acrylic holders and coated with gold via vacuum metallization (SDC-050 Sputtercoater; Baltec, Balzers, Liechtenstein). They were evaluated using SEM. For each sample, two sequences of images were recorded at magnifications of $1000\times$ and $3000\times$. The surface topography was descriptively analyzed.

Statistical Analysis

The collected data were subjected to the Shapiro–Wilk normality test and Levene’s test to check for equal variances. Dentin microtensile bond strength was then assessed using two-way ANOVA (factors: APA duration, etchant), followed by Bonferroni’s test ($\alpha=0.05$). All analyses were conducted using SPSS 21.0 (SPSS Inc., Chicago, IL, USA), with a significance level set at 5%.

Results

Dentin Microtensile Bond Strength (μ TBS)

The results of microtensile bond strength are shown in Table 1.

The ANOVA test showed double interactions among the factors ($p = 0.018$). The Bonferroni *post-hoc* test showed that the air-abraded group using aluminum oxide for 5s and conditioned with phosphoric acid (5APA+PhoA) presented the highest value of bond strength, which was statistically different from the other groups treated with phosphoric acid (PhoA and 10APA+PhoA) and from the group air abraded for 5s and conditioned with PhyA (5APA+PhyA). The PhyA groups did not differ from themselves (0APA+PhyA, 5APA+PhyA, 10APA+PhyA). The 10APA group presented the lowest bond strength value, which was statistically different from that of the 10APA+PhoA and 10APA+PhyA, also differed from the NA+0APA and 5APA groups. The 0 APA groups did not differ significantly from one another (PhoA, PhyA, and NA).

Failure Mode Analysis

The observed failure patterns are shown in Figure 1. The predominant failure type was adhesive failure, followed by mixed failure. In general, the groups that received pretreatment with air abrasion presented mainly mixed failures. In general, the occurrence of cohesive resin and cohesive dentin failures was low across all the examined groups.

Surface Topography

Figures 2–4 show the representative SEM images of all groups treated with airborne particle abrasion (APA). In samples treated only with APA using aluminum oxide for 5s (Figure 2A and B) and 10s (Figure 2C and D), dentin exhibited fissures on the surface and occluded tubules. Treatment with APA and subsequent phosphoric (PhoA) or phytic acid (PhyA) application produced superficial fissures and surface irregularities, exposing the tubular and intertubular dentin

Table 1 Mean (Standard Deviation) Bond Strength Values in MPa

	0 APA	5 APA	10 APA
PhoA	52.07 (± 9.87) aB	73.25 (± 12.98) aA	56.39 (± 11.27) aB
PhyA	57.72 (± 13.58) aA	60.01 (± 7.52) bA	59.85 (± 11.12) aA
NA	61.85 (± 10.93) aA	61.34 (± 14.53) abA	43.08 (± 13.46) bB

Notes: Different lowercase letters represent statistical differences in relation to the type of acid used for each APA duration. Different capital letters represent statistically significant differences in APA duration for each type of acid.

Abbreviations: NA, no acid applied; PhoA: Phosphoric acid; PhyA: Phytic acid; 0APA, no airborne particle abrasion; 5APA, airborne particle abrasion for 5s; 10APA, airborne particle abrasion for 10s.

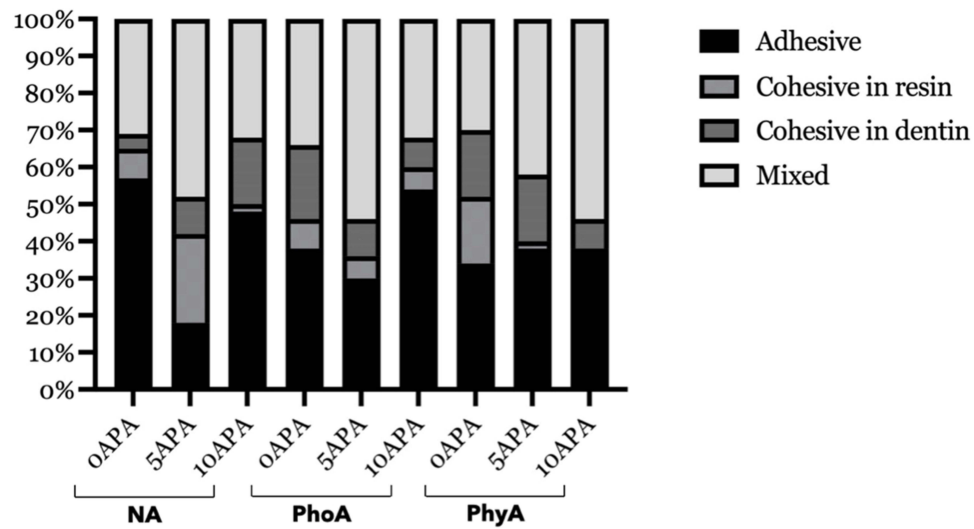


Figure 1 Fracture pattern (%) in each group.

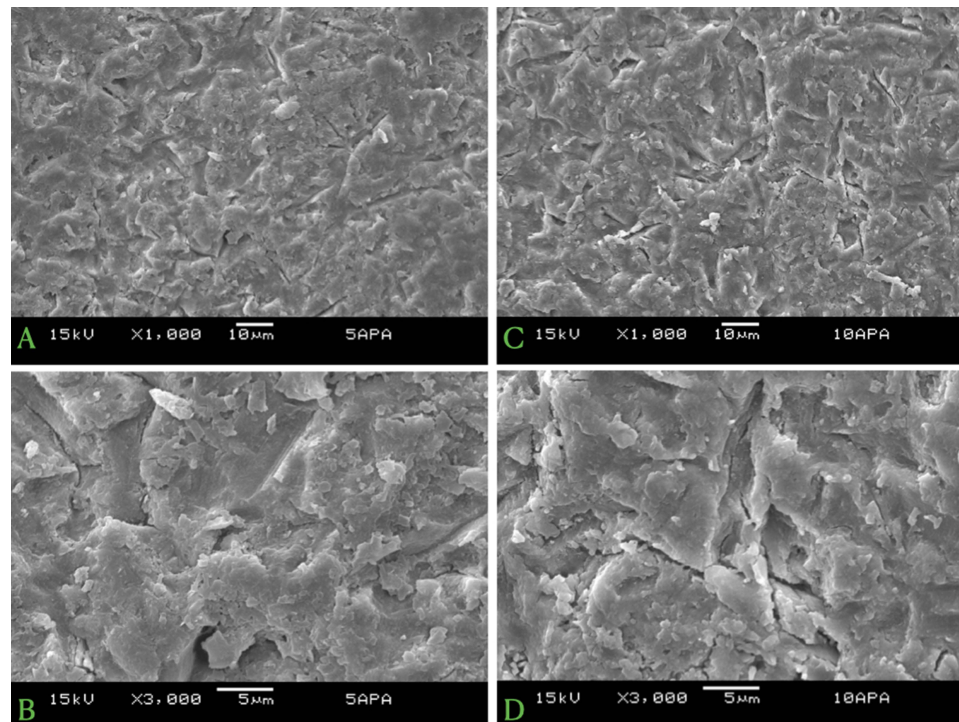


Figure 2 Representative images of air abraded samples for 5 seconds with APA at 1000x and 3000x magnification (**A** and **B**), respectively. Air abraded samples for 10 seconds with APA at 1000x and 3000x magnification (**C** and **D**), respectively.

(Figures 3 and 4). Compared to conditioning with PhoA after APA, PhyA presented more occluded tubules, as indicated by red arrows.

Discussion

Universal or multi-mode adhesives simplify adhesive procedures. Various approaches have been investigated to improve its adhesive properties to dentin. This study found that using aluminum oxide airborne particle abrasion associated with

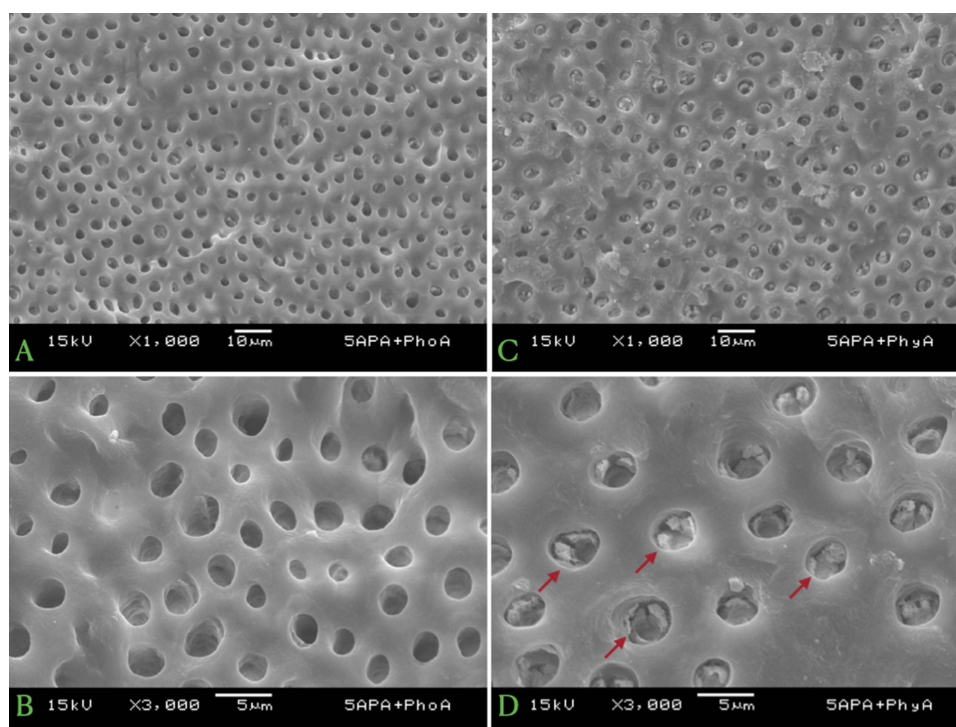


Figure 3 Representative images of samples air abraded for 5 seconds conditioned with PhoA at 1000x and 3000x (**A** and **B**) and with PhyA at 1000x and 3000x (**C** and **D**) magnification, respectively. The arrows point occluded dentinal tubules.

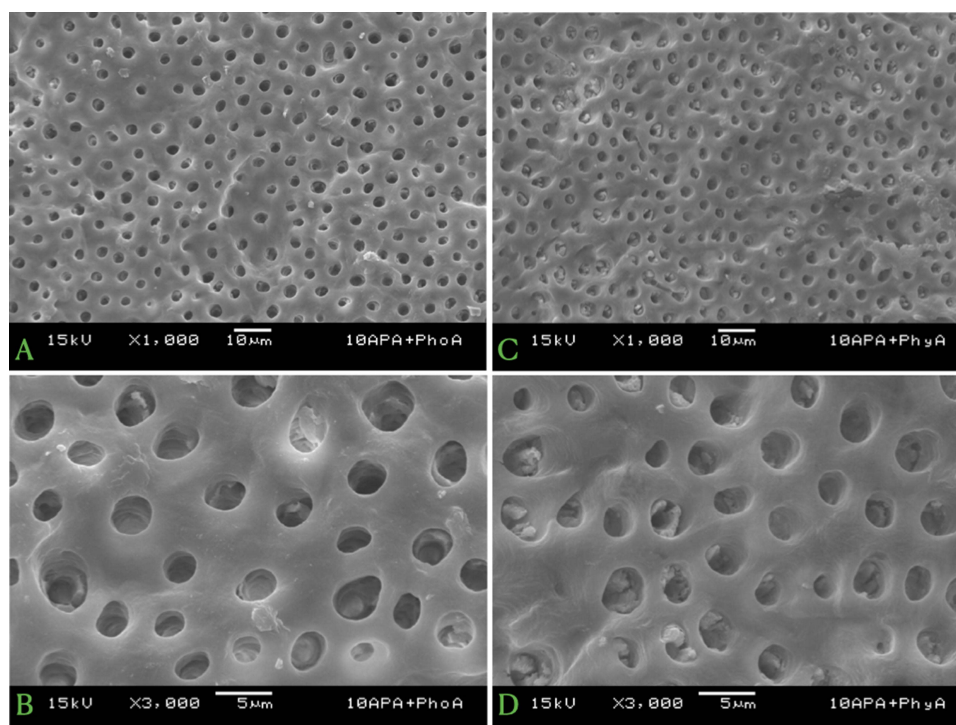


Figure 4 Representative images of samples air abraded for 10 seconds and conditioned with PhoA at 1000x and 3000x (**A** and **B**) and with PhyA at 1000x and 3000x (**C** and **D**), respectively.

phosphoric acid for 5 seconds effectively enhanced the bond strength in a universal adhesive system. Therefore, the first null hypothesis was rejected.

To the best of our knowledge, this is the first study to compare APA duration using different types of acids. Based on the results presented with an APA duration of 5 seconds, and although most of the published studies use a 10 seconds APA, our findings suggest that air abrasion for half of the time could also be successful in creating mechanical factors to increase the bond strength in dentin. This outcome contradicts some authors' affirmations that APA duration is not an important factor for bond strength to dentin.¹⁵ Yet more importantly, a clinically shorter time operating APA with aluminum oxide (Al_2O_3) could decrease the risk of exposure for the patient and clinician to hazardous particles that can cause nose, throat, and lung irritation.

Airborne particle abrasion is believed to improve the tensile strength compared with non-abraded groups. The results showed that using 5s of APA and conditioning with PhoA increased the resin-dentin bond strength when compared with other groups conditioned with the same acid. Dentin pretreatment using aluminum oxide air abrasion can potentially enhance dentin tensile strength by incrementing the roughness of the dentin surface presumably increases the surface area and contact between the adhesive and dentin, consequently enhancing dentin adhesion.^{15–20} Nevertheless, these beneficial effects may only be achieved when the dentin surface is conditioned. The group only air abraded for 10s (10APA) statistically differed from the 10APA groups conditioned with PhoA and PhyA. The improvement in the bond strength of both conditioned groups could be explained by the air abrasion pretreatment as well as the etch-and-rinse strategy. Specifically, acid conditioning could remove the smear layer and residual aluminum oxide particles on the dentin, thus exposing the dentinal tubules and enhancing tag formation and the hybrid layer by increasing the infiltration of the adhesive, as shown in Figures 3 and 4, when compared with Figure 2.^{16,17,19} This last affirmation is supported by many studies that concluded that oxide air abrasion must always be used with acid etching.^{16,21,22}

Studies have shown that phytic acid etching can improve the tensile strength in dentin. In this study, the 5APA group displayed significant differences when conditioned with PhoA (5APA+PhoA) compared to PhyA (5APA+PhyA). These findings contradict previous results; Nassar et al (2013)¹² found that the bond strength to dentin increased when compared to PhoA upon etching with PhyA. Consistent with these results, Attia et al (2022)²³ reported that PhyA enhanced the tensile bond strength of a universal adhesive compared with PhoA etching or the self-etch mode. This discrepancy could be explained by the fact that some studies had a higher application time (60s) of PhyA than that used in this study, implying that a longer acid application time contributes to a higher dissolution of the smear layer and allows better resin monomer diffusion on the dentin surface. Consistent with this explanation, the surface topography images showed that PhoA removed both the smear layer and residual aluminum oxide particles on the dentin surface following pretreatment and exposed the dentinal tubules better than the dentin surface treated with PhyA, in which the dentinal tubules were partially blocked, as observed in Figures 3C, 3D, 4C, and 4D.^{12–14,21}

Moreover, although in this case PhyA did not enhance the dentine bond strength when APA was used, it should be taken into consideration that the results obtained with PhyA showed that it effectively demineralized dentin and obtained clinically acceptable bond strength results when compared with the actual *gold standard*, PhoA. This, whilst having fewer adverse effects on pulpal cells and a much lower concentration than PhoA, an important factor knowing that etching of dentin with PhoA is now considered too aggressive. Additionally, controlled concentrations of PhyA have been demonstrated to be non-aggressive to dentin and capable of creating stable collagen networks, which could be clinically interpreted as better longevity in composite restorations.^{6,22}

No statistical difference was observed between the use of the universal adhesive in Self-Etch (SE) and Etch-and-Rinse (ER) modes in the groups in which APA was not used. These results are supported by several studies using Scotchbond Universal adhesive (3M), indicating that the bonding strategy, that is, the ER or SE approach, does not have a significant effect on the bond strength of the adhesive in dentin. This can be explained by the fact that SE adhesives contain acidic monomers capable of both “conditioning” and “priming” dental surfaces without requiring previous acid etching. Preliminary studies have established that acid etching of dentin does not influence the tensile strength of universal adhesives featuring mild acidity, such as the Scotchbond Universal adhesive with a pH of 2.7.^{1,4}

Considering the predominant failures in each group, and despite the most common type of failure mode being adhesive failure, the majority of the groups that had pretreatment with air abrasion using aluminum oxide presented

mixed failures. It can be assumed that, with a strong bond, the fracture initiates within the composite and extends through the bonding interface before reaching the dentin substrate. Therefore, mixed failures suggest that the components involved in dentin adhesion function as a unified entity rather than distinct layers, resulting in satisfactory bond strength results.^{23,24}

Given the inherent limitations of an in vitro study, our findings suggest that certain dentin pretreatments could benefit subsequent adhesive procedures, such as airborne particle abrasion (APA) with aluminum oxide, and that factors such as duration of APA and the use of certain types of acid conditioning could be important in improving the performance of the Single Bond Universal adhesive system. However, one of the main limitations of this study is that it only tested the immediate bond strength (after 24 h); hence, further research is necessary to assess the influence of the approaches used on the long-term bonding effectiveness of universal adhesive systems.

Conclusion

The use of airborne particle abrasion with aluminum oxide when applied for 5 seconds in combination with phosphoric acid was effective in enhancing the bond strength in a universal adhesive system. The abrasion was not effective when used in combination with phytic acid. A Self-Etch strategy in association with airborne particle abrasion for 10 seconds is ineffective and even detrimental when using a universal adhesive.

Funding

Funding for this research was provided by the Coordination for the Improvement of Higher Education Personnel (CAPES) under Code 001.

Disclosure

The authors declare no conflict of interest with respect to the authorship and/or publication of this article.

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