UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

MILAGROS FALCON AGUILAR

# EFEITO DA DURAÇÃO DO JATEAMENTO COM ÓXIDO DE ALUMÍNIO E O TIPO DE CONDICIONAMENTO ÁCIDO NA RESISTÊNCIA DE UNIÃO DE UM ADESIVO UNIVERSAL EM DENTINA

# EFFECT OF AIRBORNE PARTICLE ABRASION DURATION WITH ALUMINUM OXIDE AND THE TYPE OF ACID ETCHING ON BOND STRENGTH OF A UNIVERSAL ADHESIVE IN DENTIN

Piracicaba, SP
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# EFFECT OF AIRBORNE PARTICLE ABRASION DURATION WITH ALUMINUM OXIDE AND THE TYPE OF ACID ETCHING ON BOND STRENGTH OF A UNIVERSAL ADHESIVE SYSTEM IN DENTIN

Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestra em Clínica Odontológica, na Área de Dentística.

Dissertation presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Dental Clinic, in Operative Dentistry Area.

Orientador: Prof. Dr. Flávio Henrique Baggio Aguiar

ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DA DISSERTAÇÃO DEFENDIDA PELA ALUNA MILAGROS FALCON AGUILAR E ORIENTADA PELO PROF. DR. FLÁVIO HENRIQUE BAGGIO AGUIAR

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## RESUMO

Existem estratégias que podem melhorar o desempenho dos adesivos atuais, especialmente os adesivos universais. Supõe-se que o pré-tratamento dentinário com jateamento com partículas de óxido de alumínio poderia aumentar a resistência de união de um sistema adesivo universal especialmente quando combinado com diferentes estratégias de condicionamento ácido. Este trabalho avaliou a resistência de união à dentina (μTBS) de um sistema adesivo universal usando diferentes tempos (0, 5 e 10 s) de abrasão por partículas de óxido de alumínio (APA) a 0,5 cm de distância da superfície com um ângulo de 90º e dois tipos de condicionamento ácido (ácido fosfórico a 35% - PhoA ou ácido fítico a 1% - PhyA) e sem condicionamento ácido (sem ácido- NA), através do teste de microtração (n=8). Após o teste, a análise de padrão de fratura foi feita. A topografia de superfície foi analisada com a finalidade de ilustrar a influência da duração do pré-tratamento com APA e do tratamento de condicionamento com ácido fosfórico e fítico na dentina (n=2). Os dados obtidos foram analisados estatisticamente por ANOVA e o teste de Bonferroni. O grupo que recebeu um pré-tratamento de APA de 5 segundos e PhoA (5APA+PhoA) apresentou os maiores valores de μTBS entre todos os grupos, sendo estatisticamente diferente quando comparado aos grupos PhoA, 10APA+PhoA e 5APA+PhyA. PhyA não afetou significativamente a resistência de união à microtração dos grupos que receberam APA. A falha adesiva foi considerada predominante para todos os grupos. A topografia de superfície mostrou que nas amostras tratadas apenas com APA, a dentina exibiu fissuras na superfície e túbulos dentinários ocluídos. No tratamento com APA e a posterior aplicação de ácido fosfórico (PhoA) ou fítico (PhyA), PhoA conseguiu desocluir melhor os túbulos dentinários. O pré-tratamento da dentina com APA usando óxido de alumínio quando aplicado por 5 segundos e associado ao PhoA foi eficaz para aumentar a resistência da união em um sistema adesivo universal.



**Palavras chaves:** Jateamento, adesivo universal, ácido fítico, ácido fosfórico, dentina, adesão.

## ABSTRACT

Certain strategies can improve the performance of current adhesives, especially universal adhesive systems. Supposedly, a dentin pretreatment called airborne- particle abrasion with aluminum oxide particles could increase the bond strength of a universal adhesive system especially when combined with different acid etching strategies. This work evaluated the dentin bond strength (μTBS) of a universal adhesive system using different times (0, 5 and 10 s) of abrasion by aluminum oxide particles (APA) 0.5 cm distance from the surface at a 90° angle and two types of acid conditioning (35% phosphoric acid - PhoA; 1% phytic acid - PhyA) and no acid conditioning (no acid- NA) by microtensile bond strength test (n=8). After the test, fracture pattern analysis was performed. The surface topography was analyzed in order to illustrate the influence of the duration of APA pretreatment and phosphoric and phytic acid etching treatment on sealed dentin (n=2). The data obtained were statistically analyzed by ANOVA and Bonferroni test. The group that received a 5-second APA and PhoA etching (5APA+PhoA) showed the highest μTBS values among all groups, and was statistically different when compared to PhoA, 10APA+PhoA and 5APA+PhyA groups. PhyA did not significantly affect the microtensile bond strength of the groups that received APA. Adhesive failure was predominant for all groups. The surface topography showed that in the samples treated with APA alone, the dentin exhibited surface cracks and occluded dentinal tubules. In APA treatment and the subsequent application of phosphoric acid (PhoA) or phytic acid (PhyA), PhoA was able to better disocclude the dentinal tubules. Pretreatment of dentin with APA using aluminum oxide when applied for 5 seconds and associated with PhoA was effective in increasing bond strength in a universal adhesive system.



**Keywords:** Airborne particle abrasion, universal adhesive, phytic acid, phosphoric acid, dentin, bonding.

# SUMÁRIO

# 1. INTRODUÇÃO

A introdução da técnica de condicionamento ácido do esmalte em 1955 transformou completamente a odontologia operatória, aumentando a demanda por tratamentos dentários estéticos restauradores (Buonocore MG, 1955; Perdigão J *et al.,* 2000; Van Meerbeek *et al.,* 2020). Os adesivos dentais podem ser classificados de acordo com a estratégia de adesão aos tecidos dentais como adesivos convencionais, o que significa que requerem um condicionamento prévio das superfícies com ácido fosfórico à 37%, ou como adesivos autocondicionantes, que não requerem esta etapa prévia e aderem à dentina por meio de monômeros funcionais (Cardoso MV *et al.,* 2011, 2019).

A adesão é influenciada pelas características morfológicas da estrutura do dente. A adesão ao esmalte é uma técnica confiável, enquanto a adesão à dentina representa um desafio maior, sendo um tecido orgânico intrinsecamente úmido penetrado por um labirinto tubular contendo o processo odontoblástico. Essa natureza inerente da dentina como substrato influencia na adesão e no selamento marginal, sendo relacionado ao insucesso clínico nas resinas compostas (Perdigão *et al.,* 2000).

A tecnologia de adesivos dentais tem evoluído nas últimas décadas para formulações complexas com procedimentos clínicos simplificados. Existe uma tendência entre os fabricantes de continuar simplificando a tecnologia de frasco único para atender à demanda dos clínicos por procedimentos adesivos mais rápidos, menos sensíveis à técnica e com menor incidência de sensibilidade pós-operatória (De Munck *et al.,* 2005; Chen C *et al.,* 2015).

Os adesivos universais ou multimodo combinam o condicionamento ácido, o primer e o adesivo, contendo assim monômeros funcionais ácidos, monômeros hidrófilos e hidrófobos, água e solventes orgânicos em uma única solução (Giannini *et al.,* 2015). Os adesivos universais são desenhados para aderir às estruturas dentárias através do modo convencional ou autocondicionante em esmalte e dentina. Ele também pode ser utilizado na técnica de condicionamento seletivo do esmalte (Erhardt *et al.,* 2008; Jacker-Ghur *et al.,* 2019; Cardoso *et al.,* 2019). Um adesivo que pode ser aplicado nos dois modos permite ao profissional

decidir sobre o protocolo adesivo específico mais adequado para a cavidade que está sendo preparada (Rosa *et al.,* 2015).

No entanto, a literatura demonstra que o condicionamento com ácido fosfórico antes da aplicação de adesivos universais em esmalte proporcionou de maneira significativa, um aumento da resistência de união do esmalte e durabilidade de união, em comparação com aqueles preparados sem pré-condicionamento como nos casos de adesivos autocondicionantes de dois passos e de um passo. Estudos clínicos mostraram que os defeitos marginais do esmalte e descolorações marginais foram observados com mais frequência em restaurações de adesivos autocondicionantes de dois passos sem condicionamento seletivo e que o condicionamento do esmalte com ácido fosfórico a 37% tem os melhores resultados para a durabilidade da união. Conseqüentemente, mesmo ao usar adesivos autocondicionantes, o condicionamento seletivo é frequentemente recomendado (Heintze *et al.,* 2015; Suzuki *et al.,* 2021).

O hexafosfato de inositol (IP6), conhecido como ácido fítico, é um ácido cíclico saturado e uma molécula essencial para diferentes funções biológicas. Contém uma alta densidade de cargas negativas devido a seus seis grupos de fosfato que conferem ao IP6 uma grande capacidade quelante e propriedades antioxidantes. O ácido fítico é abundante nas plantas e tem um importante papel nutricional como principal forma de armazenamento de fósforo em muitos tecidos vegetais, especialmente farelos e sementes. O IP6 é considerado um produto químico abundante, não tóxico e barato que pode ser facilmente obtido de diferentes fontes vegetais por procedimentos relativamente simples (Nassar *et al*., 2021)[14]. Em 2013, o ácido fítico foi avaliado como um agente de ataque dentinário aumentando a resistência da união resina - dentina. Exibe melhor biocompatibilidade com células pulpares e osteoblastos quando comparado ao ácido fosfórico ou EDTA e consegue estabilizar a morfologia da rede de colágeno, possivelmente via interação de reticulação (Nassar *et al.,* 2013; 2015; Kong *et al.,* 2015). O mecanismo exato pelo qual o ácido fítico aumenta a resistência de união ainda não é totalmente compreendido, mas acredita-se que ele reticule a rede de colágeno exposta (Kong *et al.,* 2015, 2016).

De acordo com alguns autores, os pré-tratamentos dentários que aumentam a rugosidade do dente podem afetar a resistência de união, melhorando o contato interfacial

entre a dentina e a superfície adesiva. A jateamento (APA) usando óxido de alumínio ($Al_2O_3$) é definida como "o processo de alteração da superfície de um material através do uso de partículas abrasivas propelidas por ar comprimido ou outros gases". Além disso, APA com óxido de alumínio é freqüentemente usada para preparar superfícies com o fim de melhorar a retenção micromecânica de materiais restauradores, como vitrocerâmicas e resinas compostas. Em resinas compostas, a APA tem sido usada como um pré-tratamento para melhorar a rugosidade do esmalte e da dentina e remover a "*smear layer*", melhorando a infiltração dos sistemas adesivos na dentina desmineralizada, o que pode resultar em resistências de união significativamente maiores (França *et al.,* 2007; Sutil *et al.,* 2017; Lima *et al.,* 2021). Segundo a revisão sistemática realizada por Lima *et al*.em 2021, a melhoria da resistência de união com APA em dentina é altamente dependente de fatores como tamanho de partícula e pressão de ar utilizada. Outros fatores, como a duração do APA, são descritos como não particularmente importantes para a resistência de união à dentina, no entanto, novas pesquisas devem ser feitas a esse respeito. (Lima *et al.,* 2021).

Desta forma, o objetivo geral no presente estudo foi avaliar o efeito da duração do pré-tratamento dentinário de abrasão por partículas aerotransportadas com óxido de alumínio nas propriedades adesivas de um sistema adesivo universal com dois tipos diferentes de condicionamento.

Os objetivos específicos deste estudo in-vitro foram avaliar a resistência de união de um adesivo universal, associado ou não ao jateamento com óxido de alumínio; do adesivo após condicionamento ácido com ácido fítico; após condicionamento ácido com ácido fosfórico, associado ou não a jateamento com óxido de alumínio e avaliar a superfície da dentina após o pré-tratamento com jateamento por partículas de ar usando óxido de alumínio.

# 2. ARTIGO

## 2.1 Artigo: Effect of airborne-particle abrasion duration with aluminum oxide and the type of acid etching on bond strength of a universal adhesive system.

**Milagros Falcon Aguilar, Rodrigo Barros Esteves Lins, Marcela Alvarez Ferretti, Jardel dos Santos Silva, Giselle Maria Marchi, Débora Alves Nunes Leite Lima, Flávio Henrique Baggio Aguiar.**

## ABSTRACT

**Objective:** This work aims to evaluate the bond strength to dentin (μTBS) of a universal adhesive system using different time periods (0, 5 and 10 s) of airborne particle abrasion by aluminum oxide particles (APA) and two types of acid etching (phosphoric acid - PhoA or phytic acid - PhyA) and without acid etching (acid-free - NA). **Methods:** Seventy two (72) dentine surfaces from human third molars were used, divided into 9 groups (n=8) according to the dentin pretreatment protocol and to the conditioning acid used. The occlusal surface of the specimens was removed using a silicon carbide (SiC) sandpaper. Groups that were pretreated with APA received air abrasion 0.5 cm away and 60 psi air pressure, and conditioned with PhoA or PhyA, for 5 and 10 s according to the group. Two coats of Single Bond universal adhesive were applied. All specimens were restored with 4 mm of resin in height by the incremental technique. All increments were light activated for 20 s with the 3rd generation LED light-curing device. After the restoration, sticks were obtained from each specimen for the microtensile bond strength (μTBS) test. Subsequently, the surface topography of each dentinal pretreatment and the fracture pattern were evaluated using SEM. The data obtained was statistically analyzed. **Results:** The group that received a pretreatment of 5 seconds APA and PhoA presented the higher values of μTBS among all groups, being statistically different when compared with groups PhoA, 10APA+PhoA and 5APA+PhyA. PhyA did not significantly affect the microtensile bond strength of the air abraded groups. Finally, adhesive failure was considered the predominant failure for all groups. **Conclusions:** Pretreatment of dentin with 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.

**Clinical significance:** When associated with phosphoric acid, the use of airborne particle abrasion for 5 seconds in a universal adhesive system successfully influenced bond strength. These results could be promising in reducing the exposure to aluminum oxide and working chair time.

**Keywords:** Airborne particle abrasion, universal adhesive, phytic acid, dentin, bonding.

# INTRODUCTION

Universal or multi-mode adhesives combine the etching, priming and bonding, thus containing acidic functional monomers, hydrophilic and hydrophobic monomers, water and organic solvents into a single solution. These adhesives are designed to bond to tooth structures via an etch and rinse, self-etch or selective enamel etch mode. Thus, an adhesive that can be applied in both ways allows the practitioner to decide on a specific adhesive protocol best suited for the cavity being prepared.[8, 9, 10, 11, 5]

Phosphoric acid (PhoA) etching remains the most efficacious technique for enamel bonding, however etching of dentin is a bigger challenge. The technique sensitivity of etch-and-rinse adhesive systems is the difficulty of controlling the degree of moisture of etched dentin, necessary to prevent collagen collapse that would interfere with the infiltration of resin monomers. Application of PhoA results in exposure of collagen fibrils totally deprived of hydroxyapatite. These fragile collagen networks are prone to collapse, hampering an optimal infiltration of resin, resulting in compromised bonding to dentin. Furthermore, these collagen fibrils are susceptible to degradation by matrix metalloproteinase (MMPs) jeopardizing the longevity of resin–dentin bonds. In attempts to overcome these problems, different etching agents have been evaluated as possible replaces to PhoA, such as maleic acid, citric acid, lactic acid or EDTA.[22, 23, 24, 18, 14]

Inositol hexaphosphate (PhyA), known as phytic acid, is a saturated cyclic acid and an essential molecule for different biological functions. It has a high density of negative charges due to its six phosphate groups that gives PhyA a great chelating ability and antioxidant properties. Phytic acid is abundant in plants and has an important nutritional role as the principal storage form of phosphorus in many plant tissues, especially bran and seeds. PhyA is considered an abundant, non-toxic and inexpensive chemical that can be easily obtained from different plant sources by relatively simple procedures.[14] In 2013, phytic acid was evaluated as a dentin etching agent, reporting to increase resin-dentin bond strength. It showed better biocompatibility with pulp cells and osteoblasts when compared to phosphoric acid or EDTA and stabilized collagen network morphology, possibly via cross-linking interaction. [15, 16, 17] The exact mechanism by which phytic acid increases bond strength is not yet fully understood, but it is believed to cross-link the exposed collagen network.[17, 18]

According to some authors, dental pretreatments that enhance tooth roughness may affect bond strength by improving interfacial contact between dentin and the adhesive surface. Airborne particle air abrasion (APA) using aluminum oxide ($Al_2O_3$) is defined as "the process of altering the surface of a material through the use of abrasive particles propelled by compressed air or other gasses". In restorative dentistry, APA with aluminum oxide is used as a pretreatment to enhance enamel and dentin roughness and remove the smear layer, improving the infiltration of adhesive systems into demineralized dentin, which may result in significantly higher bond strengths. Even though, studies suggest that APA must always be used with acid etching.[19, 20, 21] According to some studies, the improvement of bond strength with APA in dentin is highly dependent on factors like particle size and pressure of the air stream used. Factors like APA duration have been considered for some authors as not particularly important for the bond strength to dentin, though more studies should be conducted.[21]

In this way, the general objective of the present study was to evaluate the effect of the duration of dentinal pretreatment airborne particle abrasion using aluminum oxide on the adhesive properties of a universal adhesive system with two different types of conditioning. The first null hypotheses were: (1) there would be no differences in bond strength of a universal adhesive regardless the time duration of airborne particle abrasion; (2) there would be no differences in bond strength of a universal adhesive among the two types of acid conditioning regardless of the airborne particle abrasion.

# METHODOLOGY

## 1. Tooth preparation:

Seventy two human third molars were obtained, extracted and stored in distilled water solution with the approval of the Local Ethics Committee. Debris was initially removed with the help of 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 pre-selected, so that only teeth that have completely healthy crowns, without cracks or cavities, were used in the research. After this procedure, the cleaned and selected teeth were stored in 0.1% thymol solution (Dinâmica, Piracicaba, São Paulo, Brazil).

## 2. Adhesive and restorative procedure:

All teeth were then fixed in acrylic plates with the aid of thermoplastic glue, the coronal portions were separated from the root portion 2 mm below the cementoenamel limit, and then sectioned 3 mm above the height of the cemento-enamel junction limit through perpendicular sectioning of the tooth element in relation to its long axis. A precision metallographic cutter (Isomet 1000, Buehler, Lake Buff, IL, USA), in which a high concentration diamond disc (Isomet Diamond Wafering Blades, Buehler) was adapted and, rotating at low speed, under constant irrigation with distilled water, the necessary cuts were made to obtain the samples.

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

All samples were stored in distilled water and randomly distributed in the nine groups (n=8) according to the air abrasion duration (0, 5 and 10 seconds) and to the etching protocol (no acid - NA, 35% phosphoric acid - PhoA; or 1% phytic acid - PhyA):

(1) NA with 0s APA;
(2) 15s of PhoA with 0s APA;
(3) 30s of PhyA with 0s APA;
(4) NA with 5s APA;
(5) 15s of PhoA with 5s APA;
(6) 30s of PhyA with 5s APA;
(7) NA with 10s APA;
(8) 15s of PhoA with 10s APA;
(9) 30s of PhyA with 10s APA.

Next, for the groups that were abraded, the exposed flat dentin surfaces of the specimens were air abraded with 50 μm aluminum oxide ($Al_2O_3$) (Bioart, São Carlos, SP, Brazil) according to the group; for 5 seconds 0.5 cm away from surface with an angle of approximately 90º, using a Microetcher intraoral device (Bioart, São Carlos, SP, Brazil) at 60 psi air pressure, then rinsed with water and air for 10 seconds; or air abraded for 10 seconds and rinsed with water and air for 20 seconds.

After this step, the exposed flat dentin surfaces of the specimens were etched with 35% phosphoric acid (Ultra-Etch - Ultradent, South Jordan, UT, USA) for 15 seconds or with 1% phytic acid (Sigma-Aldrich, St Louis, MO, USA) for 30 seconds, and in both cases, rinsed with water for 30 seconds, according to the manufacturer's recommendations, then dried with a hydrophilic coffee filter squares (Melitta, Alcobendas, MAD, ES), leaving a visibly moist dentin surface.

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

The restorative procedure was performed by inserting the composite A2 Filtek Z250 XT (3M ESPE, Saint Paul, MN, USA) through the incremental technique, with 2 mm increments on the dentin surface of the dental element, using a Goldstein spatula Flexi-thin2 (Hu Friedy, Chicago, IL, USA), until obtaining a restoration of 4mm in height in relation to the prepared buccal surface. Each increment of composite was then light-activated with the 3rd generation LED light-curing device (Valo– Ultradent, South Jordan, UT, USA). In this way, the 72 restored specimens of this study were obtained.

## 3. Performed analysis

### 3.1. Microtensile test (μTBS)

For the microtensile test, the restored specimens were fixed on acrylic plates with sticky wax (Asfer Indústria Química Ltda., SP, Brazil). Then fixed on the precision metallographic cutter (Isomet 1000, Buehler, Lake Buff, IL, USA), in which a high concentration diamond disc (Extec Corp., Enfield, CT, USA) was adapted and rotating at low speed and under constant water irrigation, performed serial cuts in the mesiodistal direction, to obtain slices with 1 mm of thickness. Afterwards, the tooth was repositioned and cuts were made in the buccolingual direction, obtaining sticks of approximately 1 x 1 mm.

Next, the sticks were attached to the microtensile device (Geraldeli) with a cyanoacrylate-based adhesive (Three Bond Super Gel, ThreeBond Ltda., Diadema, SP, Brazil) by their ends, in order to position them parallel to the traction loading. Subsequently, they were taken to the universal testing machine (EZ Test L Shimadzu, Japan) and the test was conducted with a 500 kgf load cell, at a speed of 0.5 mm/min, until rupture. The force required to cause the specimens to rupture, in kilogram-force (kgf), was noted and the dimensions of the adhesive interface of the specimens were measured with a digital caliper (Mitutoyo Corporation, Tokyo, Japan).

Fracture strength in MegaPascal (MPa) was calculated by dividing the maximum force recorded during the test (in kgf) by the bond area (in $mm^2$) and expressed in MPa.

### 3.2. Analysis of specimens in Scanning Electron Microscope (SEM)

#### 3.2.1. Fracture pattern analysis:

After the μTBS test, the debonded specimens were recovered and mounted in aluminum stubs, covered with gold (Baltec Sputter Coater - SCD – 050), in order to be evaluated in the SEM with magnification of 80 x. The failure mode was classified as: A) Adhesive fracture mode, Cr) Cohesive in resin fracture mode, Cd) Cohesive in dentin fracture mode, M) Mixed fracture. The patterns were then quantified and converted to percentage.

#### 3.2.2. Surface topography:

For the surface topography test and in pursuance of illustrate the influence of the duration of pretreatment with air abrasion and the phosphoric and phytic acid conditioning treatment on sealed dentin better, two extra samples from each of the following groups: 5APA; 10APA; 5APA+PhoA; 5APA+PhyA; 10APA+PhoA; 10APA+PhyA after the dentin surface treatment, were separated. The samples were fixed on aluminum acrylic holders and coated with gold by vacuum metallization (Baltec Sputter Coater - SCD – 050). Then, they were evaluated in the SEM. For each sample, two sequences of images were recorded with magnifications of 1.000 and 3.000 x. The surface topography was descriptively analyzed.

## STATISTICAL ANALYSIS

The remaining data were tested for normality and equity variances using the Shapiro-Wilk and Levene tests, respectively ($p > 0.05$). Two-way ANOVA was performed with Bonferroni *post-hoc* test for the bond strength data. Analyses were statistically analyzed by Software SPSS 21.0 (SPSS Inc. Chicago, IL, USA) with a significance level set at 5%.

# RESULTS

## 1. Microtensile strength (μTBS)

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

ANOVA test showed double interaction among the factors (p = 0,018). Bonferroni *post-hoc* test showed that the air abraded group using oxide aluminum for 5 seconds and conditioned with phosphoric acid (5APA+PhoA) presented the highest value of bond strength, being statistically different from the other groups treated with phosphoric acid (PhoA and 10APA+PhoA) and also, from the group air abraded for 5 seconds 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 and it statistically differed from the 10APA+PhoA and 10APA+PhyA, also differed from the NA+0APA and 5APA groups. The 0 APA groups did not differ from themselves (PhoA, PhyA, NA).

Table 1. Mean (standard deviation) bond strength values in MPa

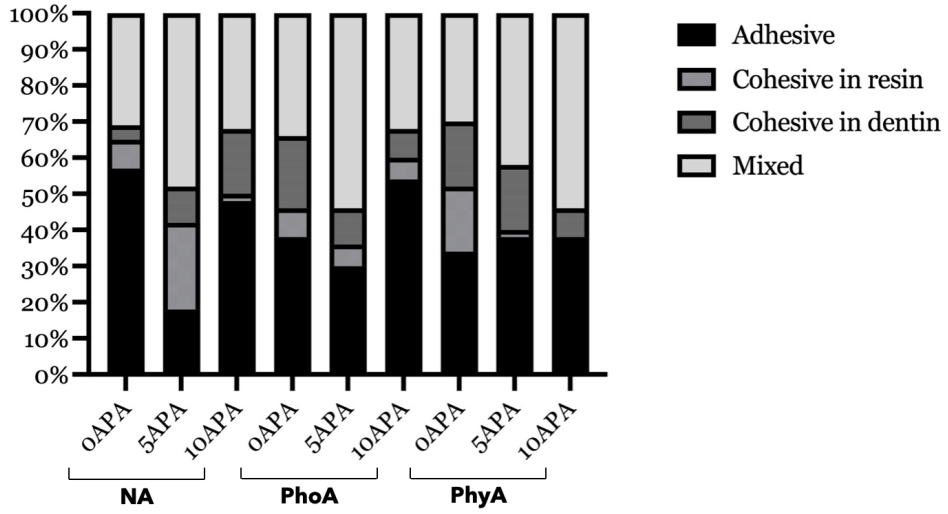
| | 0 APA | 5 APA | 10 APA |
|---|---|---|---|
| PhoA | 52.07 (±9.87) aB | 73.25 (±12.98) aA | 56.39 (±11.27) aB |
| PhyA | 57.72 (±13.58) aA | 60. 01 (±7.52) bA | 59.85 (±11.12) aA |
| NA | 61.85 (±10.93) aA | 61.34 (±14.53) abA | 43.08 (±13.46) bB |

*Abbreviations- NA: No acid applied, PhoA: Phosphoric acid, PhyA: Phytic acid, 0APA: No airborne particle abrasion was performed, 5APA: Airborne particle abrasion for 5 seconds, 10APA: Airborne particle abrasion for 10 seconds.

Different lowercase letters represent statistical differences in relation to type of acid, in each duration of APA. Different capital letters represent statistical differences in relation to times, in each type of acid.

## 2. Fracture pattern analysis

Figure1. Fracture pattern (%) of all groups




The observed failure patterns are shown in Figure 1. The predominant failure types were Adhesive followed by Mixed failures. In general, the groups that received a pretreatment with air abrasion presented mainly Mixed failures. Overall, the rate of Cohesive resin and Cohesive dentin failures were low for all tested groups.

### 3. Surface topography in the Scanning Electron Microscope (SEM)

Figures 2, 3 and 4 show representative SEM images of all groups treated with airborne particle abrasion (APA). On samples treated only with APA using aluminum oxide for 5 seconds (Figure 2A and 2C) and 10 seconds (Figure 2B and 2D), the dentin exhibited fissures on the surface and occluded tubules. The treatment with APA and subsequent phosphoric (PhoA) or phytic acid (PhyA) application produced superficial fissures and surface irregularities, exposing tubular and intertubular dentin (Figure 3 and Figure 4). In comparison to conditioning with PhoA after APA, PhyA presented more occluded tubules, as pointed out by the red arrows.

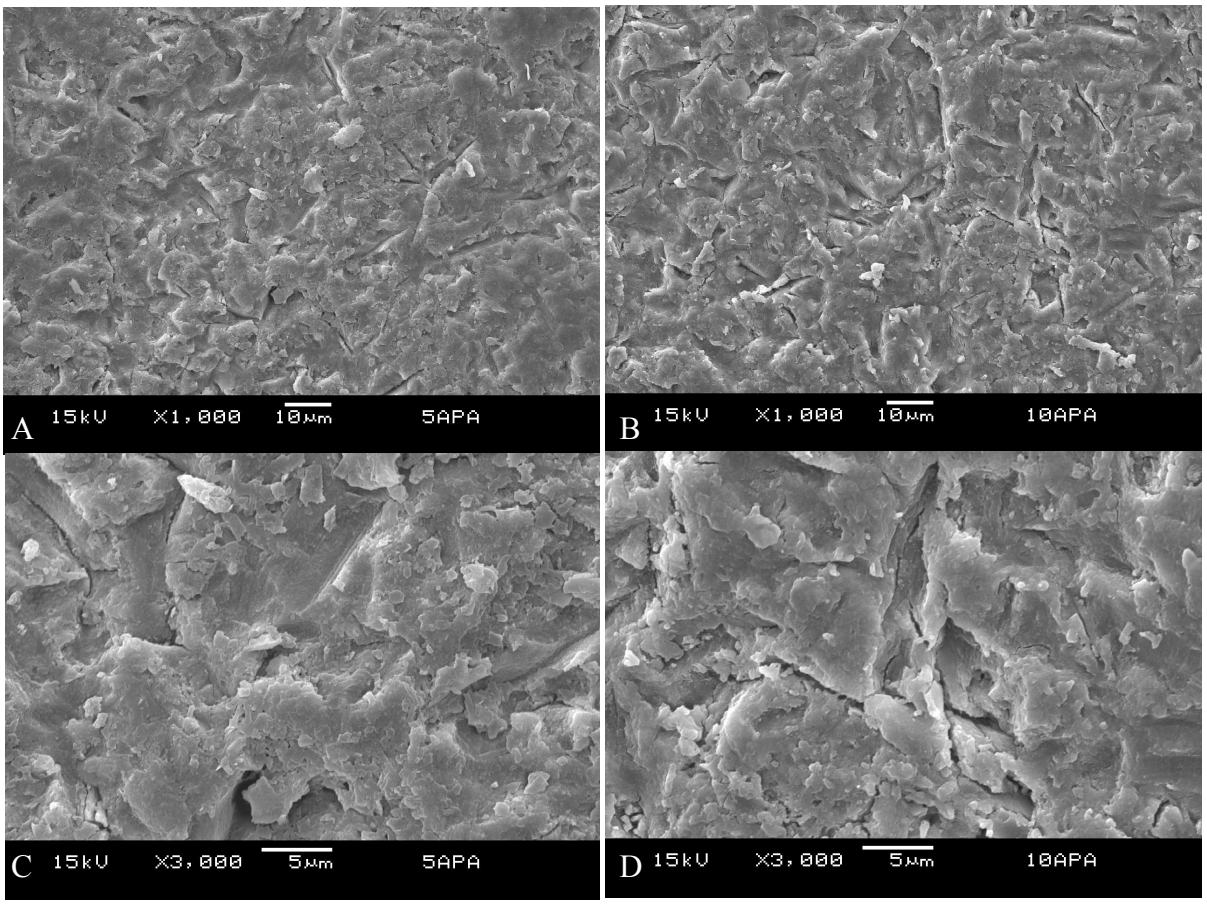



Figure 2. Representative images of air abraded samples for 5 seconds with APA at 1000x and 3000x magnification (A and C), respectively. Air abraded samples for 10 seconds with APA at 1000x and 3000x magnification (B and D), respectively.

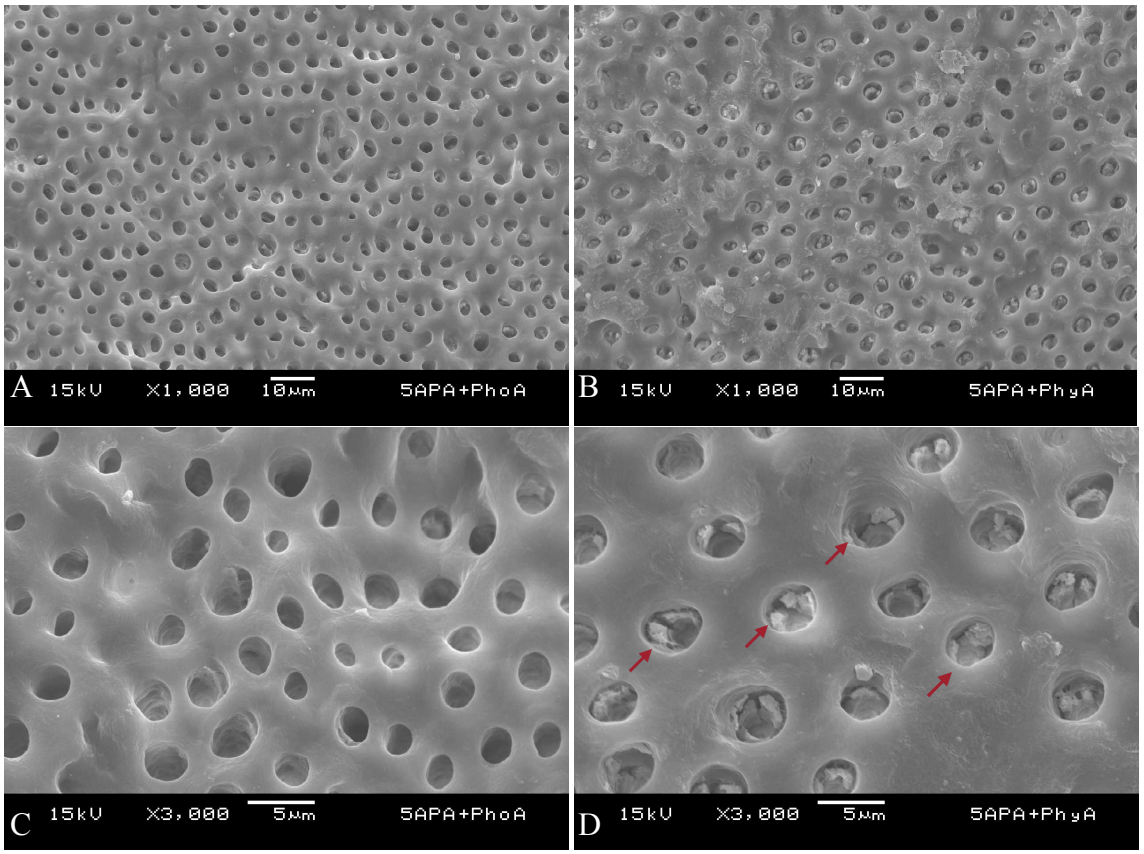



Figure 3. Representation of samples air abraded for 5 seconds conditioned with PhoA at 1000x and 3000x (A and C) and with PhyA at 1000x and 3000x (B and D) magnification, respectively. The arrows point occluded dentinal tubules.

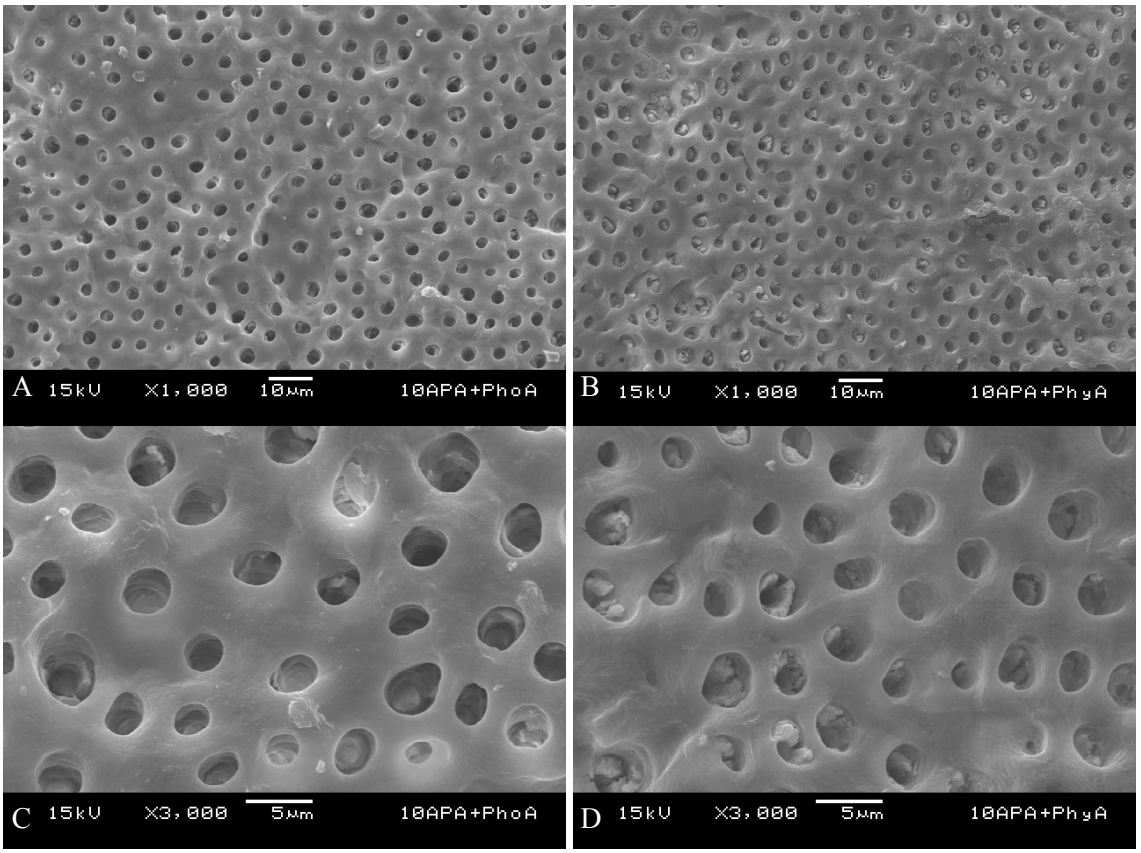



Figure 4. Representation of samples air abraded for 10 seconds and conditioned with PhoA at 1000x and 3000x (A and C) and with PhyA at 1000x and 3000x (B and D), respectively.

## DISCUSSION

Universal or multi-mode adhesives accomplish to simplify the adhesive procedures. In order to improve its adhesive properties to dentin, different approaches could be investigated. This particular study found that airborne particle abrasion with aluminum oxide applied for 5 seconds in association with phosphoric acid, successfully improved bond strength in a universal adhesive system. Thereby, our first null hypothesis was rejected.

To the best of our knowledge, this may be the first article comparing APA duration and 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 results could mean that air abrading for half of the time could also be successful in creating the mechanical factors to increase the bond strength in composites. This outcome may be supported by one systematic review that establishes APA duration as not specifically important for the bond strength to dentin.[16] Yet more importantly, a clinically shorter time operating APA with aluminum oxide ($Al_2O_3$) could mean a decreased risk of exposure for the patient and clinician to a hazard particle that can cause nose, throat and lungs irritation.

Airborne particle abrasion is believed to improve the bond strength when compared to groups not abraded. Results showed that using 5 seconds of APA and conditioning with PhoA did increase the bond strength when compared with other groups conditioned with the same acid. Since a pretreatment of dentin with aluminum oxide air abrasion could improve the dentin bond strength due to the superficial removal of the smear layer by air abrasion increasing perhaps the infiltration of the resin monomers. Additionally, a roughness increment in the irregular surface, presumably increasing the surface area and contact between the dentin and the adhesive and consequently, enhancing dentin adhesion.[14, 15, 17, 18, 19] Nevertheless, these beneficial effects may only be achieved when the dentin surface is conditioned. The group only air abraded for 10 seconds (10APA) also statistically differed when compared to the 10APA groups conditioned with PhoA and PhyA. The improvement in bond strength of both conditioned groups could be explained because of the air abrasion pretreatment but also in regard to the Etch-and-rinse strategy. Specifically, the acid conditioning removing the smear layer and aluminum oxide particles left on the dentin surface, exposing the dentinal tubules and improving a tag formation and enhancing the hybrid layer by enhancing the infiltration of the adhesive, as seen in Figures 3 and 4 when

compared with Figure 2.[14,15, 18] This last affirmation is supported by many articles that conclude that oxide air abrasion must always be used with acid etching .[19, 20, 21]

Etching with phytic acid has been reported to enhance the bond strength in dentin. In this study, the 5APA group displayed significant differences when conditioned with PhoA (5APA+PhoA) in comparison to PhyA (5APA+PhyA). These findings contradict previous results, Nassar *et al.* (2013)[11] found that the bond strength of resin to dentin was increased when compared to PhoA, upon etching with PhyA. Also consistent with these results, Attia *et al.,* (2022)[22], reported that PhyA enhanced the microtensile bond strength of a universal adhesive in comparison to PhoA etching or using the universal adhesive in a self-etch mode. The discrepancy in bond strengths, meaning PhoA having a higher bond strength than PhyA, may be due PhoA accomplished not only to remove the smear layer and aluminum oxide particles left on the dentin surface after the dentinal pretreatment, but to better expose the dentinal tubules in comparison to the dentin treated with PhyA, in which the dentinal tubules were in majority partially blocked, as observed in Figures 3B and 3D.[14,15,18]

Moreover, and even though in this case PhyA did not enhance the dentine bond strength when APA was used, it should be taken into consideration that the obtained results with PhyA show that it did act effectively when compared with the actual *gold standard,* PhoA. This while having less adverse effects on pulpal cells and at a much lower concentration than PhoA, an important factor knowing that etching of dentin with PhoA is now considered too aggressive. Unlike controlled concentrations of PhyA which seem to be not aggressive to dentin and create a stable collagen network which might be clinically translated to better longevity to resin-based restorations.[6, 23]

No statistical difference was shown when the universal adhesive was used in a Self-Etch (SE) and an Etch-and-Rinse (ER) mode in groups where APA was not used. These results can be supported with several studies using Scotchbond Universal adhesive (3M) indicating that the bonding strategy, meaning the ER or SE approach, does not have a significant effect on the bond strength of the adhesive in dentin. This can be explained considering that SE adhesives contain acidic monomers that “condition” and “prime” the dental substrates, not requiring a prior phosphoric acid etching step. Literature has established that prior acid etching does not influence the dentin bond strength for universal adhesives with mild acidity, being the Scotchbond Universal adhesive an adhesive of mild acidity with

a pH=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 a pretreatment with air abrasion using aluminum oxide presented Mixed failures. Literature claims that in the presence of a strength bond, the fracture path begins in the composite and propagates through the bonding interface reaching the dentin substrate. For that reason, mixed failures suggest that the elements involved in dentin adhesion acted as a single body rather than separate layers, which translates into adequate bond strength results.[24, 25]

Within the limitations of an in-vitro study, our findings suggest that certain dentin pretreatments could benefit the subsequent adhesive procedures, one of them being airborne particle abrasion (APA) with aluminum oxide and that factors like duration of APA and use of certain type of acid conditioning could be important factors in order to improve the performance of Single Bond Universal adhesive system. Yet one of the main limitations in this study that can not be disregarded would be that we tested only immediate bond strength (after 24 hours), thus, more studies are needed to evaluate the influence of these approaches on the adhesive effectiveness of universal adhesive systems in the long-term.

## CONCLUSION

The use of airborne particle abrasion with aluminum oxide when applied for 5 seconds and associated with phosphoric acid was effective in enhancing bond strength in a universal adhesive system. When used in association with phytic acid, abrasion was not effective. Airborne particle abrasion in a Self-Etch mode was not effective and even detrimental when used for 10 seconds.

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# ANEXO 1- Certificado do Comitê de Ética em Pesquisa

FACULDADE DE ODONTOLOGIA DE PIRACICABA DA UNIVERSIDADE DE CAMPINAS - FOP/UNICAMP

## PARECER CONSUBSTANCIADO DO CEP

**DADOS DO PROJETO DE PESQUISA**

**Título da Pesquisa:** EFEITO DO TEMPO DE JATEAMENTO COM ÓXIDO DE ALUMÍNIO E DO TIPO DE CONDICIONAMENTO ÁCIDO NA RESISTÊNCIA DE UNIÃO EM DENTINA DE DENTES RESTAURADOS COM RESINA COMPOSTA

**Pesquisador:** Milagros Falcon Aguilar

**Área Temática:**

**Versão:** 3

**CAAE:** 66646123.7.0000.5418

**Instituição Proponente:** Faculdade de Odontologia de Piracicaba - Unicamp

**Patrocinador Principal:** Financiamento Próprio

**DADOS DO PARECER**

**Número do Parecer:** 5.882.981

**Apresentação do Projeto:**

O parecer inicial é elaborado com base na transcrição editada do conteúdo do registro do protocolo na Plataforma Brasil e dos arquivos anexados à Plataforma Brasil. Os pareceres de retorno, emendas e notificações são elaborados a partir do último parecer e dos dados e arquivos da última versão apresentada.

Pendência 1 (atendida em 07/02/23). A EQUIPE DE PESQUISA citada na capa do projeto de pesquisa inclui MILAGROS FALCON AGUILAR (Cirurgiã Dentista, Mestranda no PPG em Clínica Odontológica, área de Dentística, da FOP-UNICAMP, Pesquisadora responsável, Orientanda), FLÁVIO HENRIQUE BAGGIO AGUIAR (Cirurgião Dentista, Docente da área de Dentística da FOP-UNICAMP, Orientador), o que é confirmado na declaração dos pesquisadores e na PB.

DELINEAMENTO DA PESQUISA: Trata-se de estudo laboratorial, comparativo, que envolverá 72 dentes terceiros molares humanos, obtidos de pacientes adultos, sem distinção de sexo, da área de Cirurgia da FOP-UNICAMP. Este trabalho tem como objetivo avaliar a resistência de união à dentina de adesivos autocondicionantes com jateamento de óxido de alumínio utilizando dois

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PIRACICABA, 08 de Fevereiro de 2023

**Assinado por:**
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**(Coordenador(a))**

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## Anexo 2. Certificado de submissão do artigo ao periódico “Clinical, Cosmetic and Investigational Dentistry”

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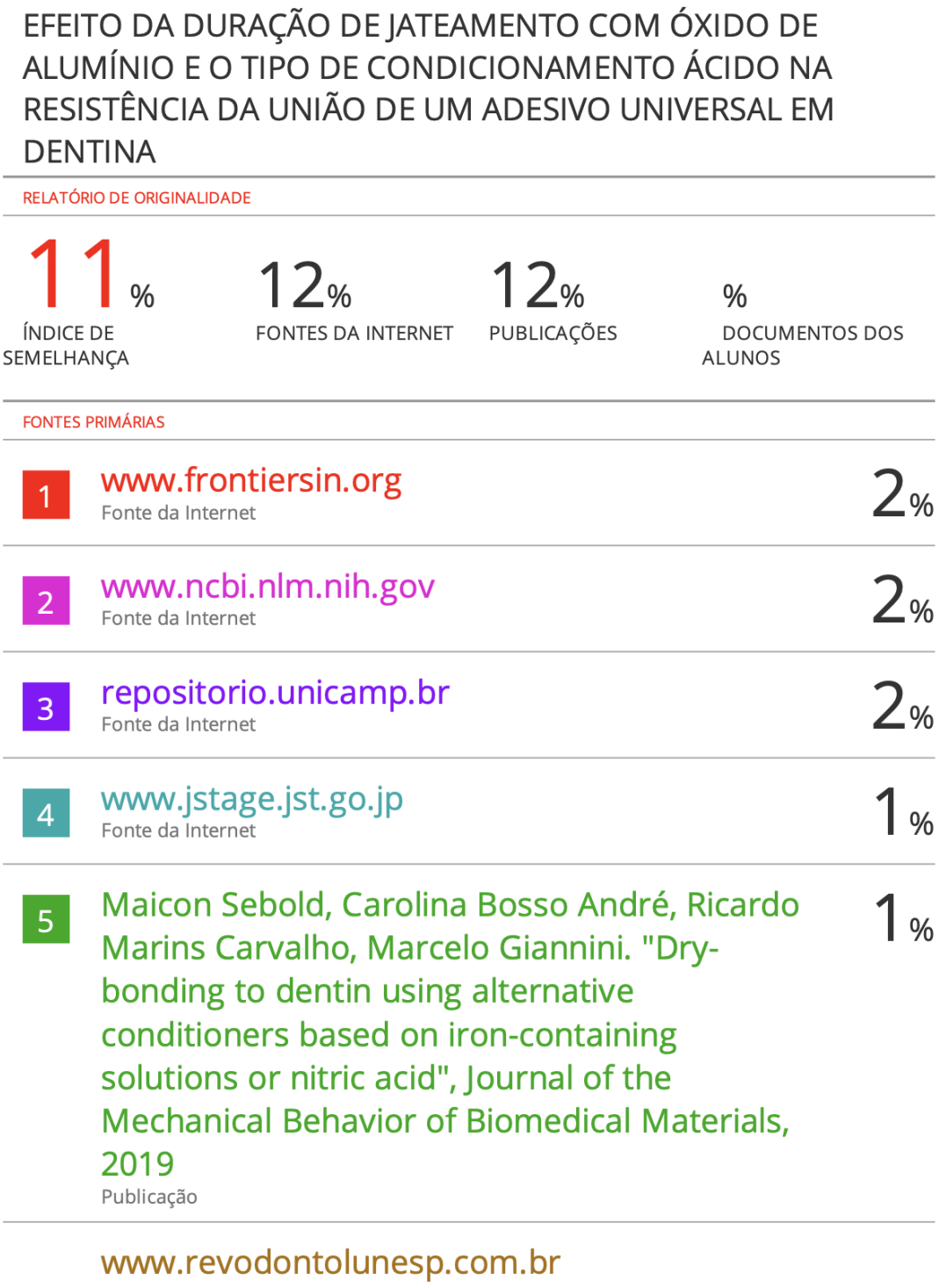

EFEITO DA DURAÇÃO DE JATEAMENTO COM ÓXIDO DE ALUMÍNIO E O TIPO DE CONDICIONAMENTO ÁCIDO NA RESISTÊNCIA DA UNIÃO DE UM ADESIVO UNIVERSAL EM DENTINA

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