

Effectiveness of Etching by Three Acids on the Morphological and Chemical Features of Dentin Tissue

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ABSTRACT

Aim: The purpose of this study was to evaluate the microscopic and chemical effects of phosphoric acid gel, phosphoric acid liquid, and polyacrylic acid application for 15 seconds (s) on coronal dentin.

Materials and methods: Twelve extracted teeth were selected. Three etching acids were used to prepare the dentin surfaces. Scanning electron microscopy (SEM) and X-ray energy dispersive spectroscopy (EDX) were used to analyze the chemical and morphological changes of the dentinal surfaces, including the depth of demineralization. Collected data were statistically analyzed by the one-way analysis of variance test.

Results: Dentin etched with phosphoric acid gel or liquid showed greater peritubular dentin dissolution, including complete removal of the smear layer. In addition, there were many silica particles on the dentin etched by phosphoric acid gel 37%. The dentin that was etched with 25% polyacrylic acid for 15 seconds showed no smear layer removal. Chemical analysis (EDX) showed that dental surfaces etched with phosphoric acid liquid 37% for 15 seconds showed the strongest mineral dissolution at the calcium surface, with a calcium content of 5.25%. On the other hand, EDX analysis of the dental surface etched with 25% polyacrylic acid showed more surface enrichment in calcium (17.19%).

Conclusion: Although phosphoric acid (gel or liquid) 37% cleans the dental surface, phosphoric acid gel precipitates silica particles on the etched dentin surface. These particles cannot be removed by rinsing of this acid. The application of polyacrylic acid for 15 seconds does not noticeably demineralized dentin, nor remove the smear layer.

Clinical significance: The clinician should use phosphoric acid (gel or liquid) to clean dental surfaces to prepare them for the bonding process. The low demineralizing effects of the polyacrylic acid permits its use near the pulp.

Keywords: Dentin demineralization, Phosphoric acid, Polyacrylic acid.

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INTRODUCTION

Dentin is the substance located under the enamel, surrounding the pulp chamber and root canals. The dentin microstructure is composed of dentinal tubules that outwardly emerge into the dentin structure from the pulp to the outer cementum or the enamel boundary. Peritubular dentin and intertubular dentin are enriched with collagen fibers. There are variations between the size, quantity, and wall thickness of dentin tubules from the outside to the inside.¹ Dentinal tubules diameter ranges from 2.5 µm (near the pulp) to less than 1 µm (near the enamel), enamel adjacent surfaces bounded by a highly mineralized cuff of peritubular dentin. The intertubular dentin is composed of collagen fibers directed mainly perpendicular to dentin tubules, loaded with apatite crystallites.²

Important clinical considerations have been considered when etching the dentin, based on its composition. Dentin composition is roughly 70% mineral, 20% organic, and 10% water.³

The recent improvements in materials have made bonding to dentin more effective. Metal alloys, ceramics, and composites require special surface processing for effective bonding. A smear layer (an adherent layer of debris) usually covers tooth surfaces requiring bonding, hence requiring sufficient cleaning in the preparation process. This smear layer can form on dentin during procedural preparation with burs and hand instruments. It contains crushed hydroxyapatite and fractured collagen. Bacteria and saliva may also contaminate this layer.⁴

The dentin smear layer could be described as a disturbed film of organic and hydroxyapatite particles, generally less than 2 µm thick. It has been generally accepted that the composition

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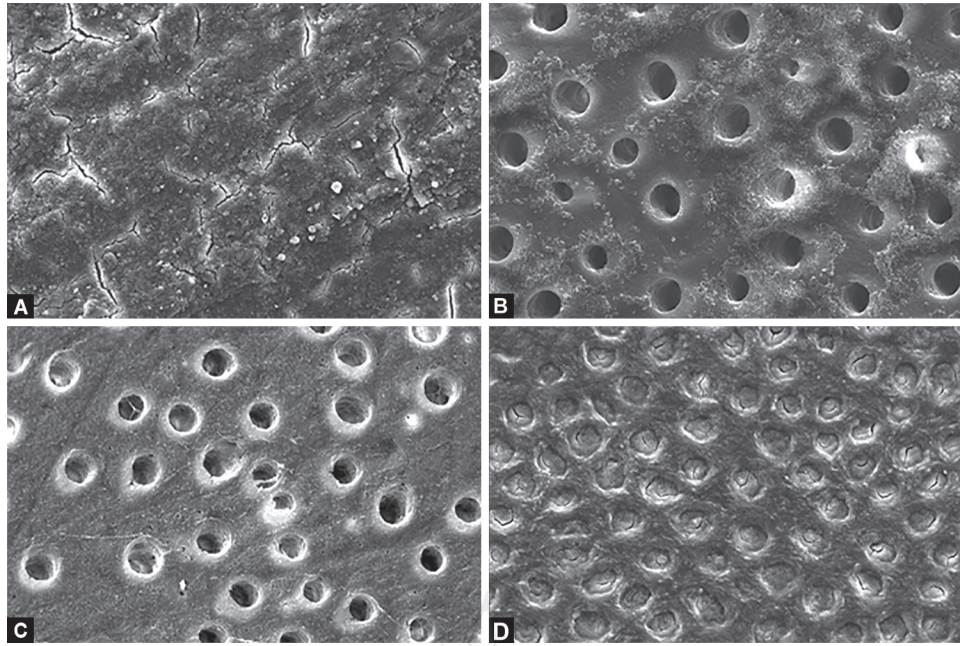
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of the smear layer is a mixture of partly denatured collagen and mineral.^{5–7} The most common disadvantage of the smear layer is that it diminishes bonding to underlying dentin.⁴ Durability and effective bonding between the dentin and an adhesive resin are essential for the longevity of restorations.⁸ Previous studies have shown that the efficiency of dentin adhesives depends on the removal of the smear layer and the generation of the resin–dentin inter-diffusional area.⁹ Acidic conditioners are most commonly used to treat the dentin surface. They eliminate the smear layer, open dentinal tubules, expose collagen fibers, and alter dentin



Figs 1A to D: (A) SEM micrograph of dentin surface showing complete closure of the dentin tubule entry points by the smear layer; (B) Dentin surface etched with phosphoric acid gel 37% for 15 seconds. Open tubules on the surface with numerous silica particles; (C) Dentin surface etched with phosphoric acid liquid 37% for 15 seconds. Open tubules on the surface with no silica particles present; (D) SEM micrograph of dentin surface etched with polyacrylic acid 25%. Dentin tubules are visible, but not open, because of plugging from the smear layer covering dentin surfaces

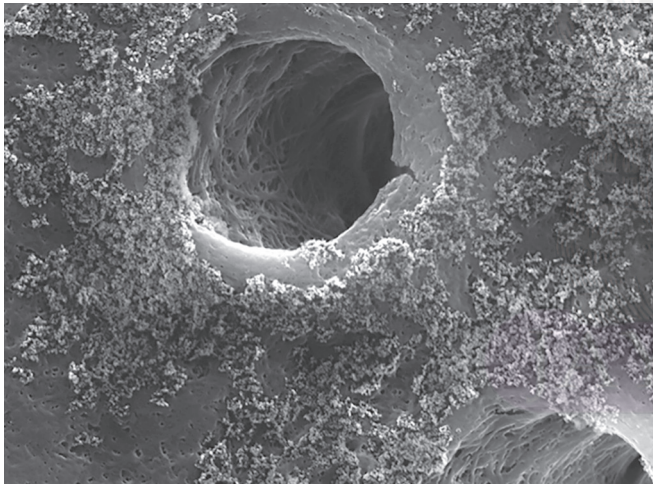


Fig. 2: Higher magnification of dentin etched with phosphoric acid gel 37%. Amounts of small debris (silica) are clearly observed

Table 1: Mass percentage of calcium (mean ± standard deviation) on dentin surfaces etched with the three acids (groups II–IV), and for the control group (GI), measured in 10 zones of 443 μm² each

	Group I	Group II	Group III	Group IV
Ca mass (%)	23.48 ± 2.45	6.95 ± 1.64	5.25 ± 1.17	17.19 ± 3.54

DISCUSSION

Tooth preparation processes create important changes in dentin thickness, and density leading to different resin bonding results.²⁰

For this reason, smear layers produced clinically with abrasive papers (P320-grit silicon carbide paper) were used to produce a denser smear layer in the present study.²¹

Dentin surfaces covered with a smear layer (GI) typical of those produced by abrasive papers were used as the fully mineralized dentin control, as they have the highest mineral content, along with closed dentin tubules.

Hydroxyapatite crystals in the smear layer are loosely compressed, interconnected debris delicately attached to the tooth structure.⁶

In the light of these findings, the acid conditioning of dentin must be considered as an essential step in achieving clean dentin, and consequently reliable bond strength.²² The thick smear layer on the dentin surface that forms during diamond bur use is removed by etching with phosphoric acid liquid and gel 37% for 15 seconds (Figs 1B and C). SEM imaging indicated that dentin surfaces exhibited different morphological appearances: dissolution of both the smear layer and peritubular dentin, removal of smear plug, and exposure of collagen fibers. Similar results have been found for dentin and enamel by Perdigão²³ and Tao et al.,²⁴ who noted that the use of 30–40% phosphoric acid remove the smear layer and smear plugs. All groups showed the presence of a demineralized collagen matrix, except for the non-etched dentin control group (GI) and when polyacrylic acid was used (GIV). The application of phosphoric acid for 15 seconds revealed demineralized collagen matrix by SEM and dissolutions of both smear plugs and peritubular dentin. There was no obvious breakup in dentin. The demineralized collagen matrix was only seen after etching with phosphoric acid, but not after polyacrylic acid. Therefore, conditioning the dentin with polyacrylic acid removes the smear layer structure except for smear plugs. Moreover, this acid partially demineralizes the dentin surface in accordance with Summitt.²⁵ This confirms our study which shows the poor efficacy of polyacrylic acid in etching through relatively denser smear layers.²⁶ Peutzfeldt reported that the dentinal tubules were opened totally and cleared to a considerable depth by using 25% polyacrylic acid solution for 30 seconds.²⁷

16. Jeng YR, Lin TT, et al. Human enamel rod presents anisotropic nanotribological properties. *J Mech Behav Biomed Mater* 2011 May;4(4):515–522. DOI: 10.1016/j.jmbbm.2010.12.002.
17. Stape THS, Wik P, et al. Selective dentin etching: a potential method to improve bonding effectiveness of universal adhesives. *J Mech Behav Biomed Mater* 2018 Oct;86:14–22. DOI: 10.1016/j.jmbbm.2018.06.015.
18. Moyaho-Bernal M, Contreras-Bulnes R, et al. Morphological and chemical changes in human deciduous dentin after phosphoric acid, self-etching adhesive and Er:YAG laser conditioning. *Microsc Res Tech* 2018 May;81(5):494–501. DOI: 10.1002/jemt.23003.
19. Stape THS, Wik P, et al. Selective dentin etching: a potential method to improve bonding effectiveness of universal adhesives. *J Mech Behav Biomed Mater* 2018 Oct;86:14–22. DOI: 10.1016/j.jmbbm.2018.06.015.
20. Sattabanasuk V, Vachiramon V, et al. Resin-dentin bond strength as related to different surface preparation methods. *J Dent* 2007 Jun;35(6):467–475. DOI: 10.1016/j.jdent.2007.01.002.
21. Armstrong S, Breschi L, et al. Academy of Dental Materials guidance on *in vitro* testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (μ TBS) approach. *Dent Mater* 2017 Feb;33(2):133–143. DOI: 10.1016/j.dental.2016.11.015.
22. Zhang L, Huang L, et al. Effect of post-space treatment on retention of fiber posts in different root regions using two self-etching systems. *Eur J Oral Sci* 2008 Jun;116(3):280–286. DOI: 10.1111/j.1600-0722.2008.00536.x.
23. Perdigão J. Dentin bonding-variables related to the clinical situation and the substrate treatment. *Dent Mater* 2010 Feb;26(2):e24–e37. DOI: 10.1016/j.dental.2009.11.149.
24. Tao L, Pashely DH, et al. Effect of different types of smear layers on dentin and enamel shear bond strengths. *Dent Mater* 1988;4:208–216. DOI: 10.1016/S0109-5641(88)80066-6.
25. Summitt JB, Santos JD. *Fundamentals of operative dentistry: a contemporary approach*, 3rd ed. Chicago: Quintessence; 2006.
26. El-Askary FS, Nassif MS, et al. Shear bond strength of glass-ionomer adhesive to dentin: effect of smear layer thickness and different dentin conditioners. *J Adhes Dent* 2008 Dec;10(6):471–479.
27. Peutzfeldt A, Asmussen E. Effect of polyacrylic acid treatment of dentin on adhesion of glass ionomer cement. *Acta Odontol Scand* 1990 Oct;48(5):337–341. DOI: 10.3109/00016359009033626.
28. Oyarzún A, Rathkamp H, et al. Immunohistochemical and ultrastructural evaluation of the effects of phosphoric acid etching on dentin proteoglycans. *Eur J Oral Sci* 2000 Dec;108(6):546–554. DOI: 10.1034/j.1600-0722.2000.00912.x.
29. Besinis A, van Noort R, et al. Infiltration of demineralized dentin with silica and hydroxyapatite nanoparticles. *Dent Mater* 2012 Sep;28(9):1012–1023. DOI: 10.1016/j.dental.2012.05.007.
30. Perdigão J, May KN Jr, et al. The effect of depth of dentin demineralization on bond strengths and morphology of the hybrid layer. *Oper Dent* 2000 May-Jun;25(3):186–194.
31. Eick JD, Gwinnett AJ, et al. Current concepts on adhesion to dentin. *Crit Rev Oral Biol Med* 1997;8(3):306–335. DOI: 10.1177/10454411970080030501.
32. Pashley DH, Carvalho RM. Dentine permeability and dentine adhesion. *J Dent* 1997 Sep;25(5):355–372. DOI: 10.1016/S0300-5712(96)00057-7.

