UNIVERSIDADE ESTADUAL DO OESTE DO PARANÁ
CENTRO DE CIÊNCIAS BIOLÓGICAS E DA SAÚDE
PROGRAMA DE PÓS-GRADUAÇÃO EM
ODONTOLOGIA (PPGO) - MESTRADO

MARIANA MACEDO RIBAS

Efeito do plasma atmosférico não-térmico, do condicionamento ácido e do envelhecimento na resistência de união de um adesivo universal ao esmalte dental humano

Cascavel-PR
2020

MARIANA MACEDO RIBAS

Efeito do plasma atmosférico não-térmico, do condicionamento ácido e do envelhecimento na resistência de união de um adesivo universal ao esmalte dental humano

Dissertação apresentada ao Programa de Pós-Graduação em Odontologia, Centro de Ciências Biológicas e da Saúde, Universidade Estadual do Oeste do Paraná, como requisito parcial para obtenção do título de Mestre em Odontologia

Área de concentração: Odontologia

Orientador: Prof. Dra. Fabiana Scarparo Naufel

Cascavel-PR
2020

Ficha de identificação da obra elaborada através do Formulário de Geração Automática do Sistema de Bibliotecas da Unioeste.

Ribas, Mariana Macedo
Efeito do plasma atmosférico não-térmico, do condicionamento ácido e do envelhecimento na resistência de união de um adesivo universal ao esmalte dental humano / Mariana Macedo Ribas; orientador(a), Fabiana Scarparo Naufel , 2020.
22 f.

Dissertação (mestrado), Universidade Estadual do Oeste do Paraná, Campus de Cascavel, Centro de Ciências Biológicas e da Saúde, Programa de Pós-Graduação em Odontologia, 2020.

1. Esmalte dentário. 2. Condicionamento ácido. 3. Gases em plasma. I. Naufel , Fabiana Scarparo. II. Título.

unioeste
Universidade Estadual do Oeste do Paraná
Campus de Cascavel CNPJ 78680337/0002-65
Rua Universitária, 2069 - Jardim Universitário - Cx. P. 000711 - CEP 85819-110
Fone:(45) 3220-3000 - Fax:(45) 3324-4566 - Cascavel - Paraná

PARANÁ
GOVERNO DO ESTADO

MARIANA MACEDO RIBAS

Efeito do plasma atmosférico não-térmico, do condicionamento ácido e do envelhecimento na resistência de união de um adesivo universal ao esmalte dental humano

Dissertação apresentada ao Programa de Pós-Graduação em Odontologia em cumprimento parcial aos requisitos para obtenção do título de Mestra em Odontologia, área de concentração Odontologia, linha de pesquisa Materiais Dentários Aplicados À Clínica Odontológica, APROVADO(A) pela seguinte banca examinadora:

Orientador(a) - Fabiana Scarparo Naufel

Universidade Estadual do Oeste do Paraná - Campus de Cascavel (UNIOESTE)

Maria Daniela Basso de Souza

Universidade Estadual do Oeste do Paraná - Campus de Cascavel (UNIOESTE)

Larissa Pinceli Chaves

UNIVERSIDADE PARANAENSE - UNIPAR

Cascavel, 19 de fevereiro de 2020

DEDICATÓRIA

Dedico este trabalho aos meus pais Leonel e Eliene, que me ensinaram desde cedo a valorizar os estudos, apoiaram e incentivaram minha trajetória acadêmica. Sou muito grata pela oportunidade de aprimorar meus conhecimentos. À minha irmã Isabela, por estar ao meu lado e oferecer apoio em todos os momentos. Ao meu amor, Michael, pelo companheirismo, incentivo, carinho e compreensão.

AGRADECIMENTOS

Primeiramente sou grata a Deus por mais essa conquista, por despertar todos os dias e ter uma vida tão privilegiada.

À minha orientadora, professora Dra. Fabiana Scarparo Naufel, pela paciência, incentivo e apoio, não somente durante a realização deste trabalho, mas desde a graduação, sou grata por sua grande contribuição para o meu crescimento pessoal, profissional e científico. Obrigada por acreditar em mim e me guiar até aqui.

À professora Dra. Maria Daniela Basso de Souza, por compartilhar seu vasto conhecimento e contribuir para a realização desta dissertação.

À professora Dra. Vera Lucia Schmitt, por ensinar mais do que conhecimentos científicos, transmitir alegria e gratidão pela vida.

À professora Dra. Larissa Pinceli Chaves, pela disponibilidade e disposição para contribuir no aprimoramento deste trabalho.

À minha “tutora” na Iniciação Científica, Kevelin Poliana, obrigada por todas as lições e por sempre exigir o melhor de mim.

Efeito do plasma atmosférico não-térmico, do condicionamento ácido e do envelhecimento na resistência de união de um adesivo universal ao esmalte dental humano

RESUMO

Este estudo *in vitro* avaliou o efeito do plasma atmosférico não térmico sobre a resistência de união (RU) de um adesivo universal ao esmalte humano, com ou sem condicionamento ácido, quando submetido ao envelhecimento. Vinte molares foram seccionados no sentido vestíbulo-lingual para obtenção de 40 superfícies proximais de esmalte. Essas foram planificadas, polidas e distribuídas aleatoriamente em 4 grupos (n = 10) de acordo com o condicionamento ácido ([C], [n-C]) e tratamento com plasma ([P], [n-P]): C/P, C/n-P, n-C/P, n-C/n-P. Empregou-se o gel de ácido fosfórico a 37% para o condicionamento do esmalte e o plasma foi aplicado em forma de varredura por 20 s. Após, o adesivo Single Bond Universal [SBU] foi aplicado de forma ativa e um bloco de resina composta foi construído de forma incremental (Filtek Z100, 3M ESPE). Após 24 horas de estocagem em água deionizada, os espécimes foram seccionados em palitos (área 1.0±0.2 mm; 10 por grupo). Então, metade dos palitos de cada grupo foi submetida imediatamente ao teste µTBS (0,5 mm/ s) [24h] e a restante após 12 meses de envelhecimento [12m]. Os dados foram submetidos a ANOVA três critérios com parcelas subdivididas (p<0,01) seguido pelo teste de Tukey (<0,05). As superfícies fraturadas foram analisadas por meio de microscopia óptica digital (aumento de 100%) para determinar o modo de falha. Os três fatores isolados mostraram diferença estatística: tempo (p<0,001), plasma (p=0,04) e condicionamento ácido (p=0,006). Houve interação significativa apenas para tempo-condicionamento ácido (p=0,013). O condicionamento aumentou os valores de RU após tração imediata, efeito não observado para o plasma. O maior valor de RU foi observado para o grupo C/n-P/24h. O envelhecimento após 12 meses reduziu significativamente a RU de todos os grupos sem diferença estatística entre eles. Os autores concluíram que, ao contrário do condicionamento com ácido fosfórico, a aplicação do plasma não aumentou a RU do esmalte. O envelhecimento das amostras teve efeito negativo sobre os valores de RU.

Palavras-chave: Esmalte dentário, gases em plasma, ataque ácido dentário.

Effect of non-thermal atmospheric plasma, acid etching, and aging on the bond strength of a universal adhesive to dental enamel

ABSTRACT

This study in vitro evaluated the effect of non-thermal atmospheric plasma on the bond strength (BS) of a universal adhesive to human enamel, with or without acid etching, when subjected to aging. Twenty molars were sectioned in the buccolingual direction to obtain 40 proximal enamel surfaces. These were planned, polished and randomly assigned to 4 groups (n = 10) according to acid etching ([Et], [no-Et]) and plasma treatment ([P], [no-P]): Et/P, Et/no-P, no-Et/P, no-Et/no-P. 37% phosphoric acid gel was used for conditioning and plasma was applied on the enamel surface for 20 seconds by scanning mode. Afterwards, Scothbond Universal Adhesive [SBU] was actively applied and a composite resin block was incrementally built up (Filtek Z100, 3M ESPE). After 24 hours of storage in deionized water, the specimens were sectioned into sticks (area 1.0 ± 0.2 mm; 10 per group). Then, half of the sticks in each group were immediately submitted to the µTBS test (0.5 mm / s) [24h] and the remaining half after 12 months of aging [12m]. Data were submitted to three-way ANOVA with subdivided plots ($p < 0.01$) followed by Tukey test (<0.05). Fractured surfaces were analyzed by digital optical microscopy (100% magnification) to determine the failure mode. The three isolated factors showed statistical difference: time ($p < 0.001$), plasma ($p = 0.04$) and acid etching ($p = 0.006$). There was significant interaction only for time-acid etching ($p = 0.013$). The acid conditioning of enamel increased the BS values after immediate traction, effect not observed for plasma. The highest BS value was observed for the Et/no-P/24h group. Aging after 12 months significantly reduced the BS of all groups without statistical difference between them. The authors concluded that, unlike phosphoric acid etching, plasma application did not increase enamel BS. Aging of the samples had a negative effect on the BS values.

***Keywords*:** Dental enamel, plasma gases, acid etching dental.

Dissertação elaborada e formatada conforme as normas das publicações científicas: *Journal of Biomedical Materials Research: Part B – Applied Biomaterials.* Disponível em: <https://onlinelibrary.wiley.com/page/journal/15524981/homepage/forauthors.html>

SUMÁRIO

Effect of non-thermal atmospheric plasma, acid etching, and aging on the bond strength of a universal adhesive to dental enamel

1. Introduction

Despite the immediate and short-term bonding efficacy of dental adhesives, their durability and stability[10,17] remain under investigation, particularly when multimode universal adhesive systems (UA)[10,15] is used by the self-etch adhesion strategy[7]. Indeed, the weak action of Scotchbond Universal Adhesive (SBU) monomer on enamel compromised the in vitro long-term bonding between them[29].

As known, changes in physical and chemical properties of dental surfaces enable its compatibility with the adhesive[11]. In this field, the non-thermal atmospheric plasma (NTAP) gas[6,8] has been used to modify enamel/dentin surfaces[5,14,18,19]in restorative procedures to improve the adhesion, as well for other purposes[1,3,14,23]. Plasma (found naturally in the sun, stars, auroras and rays) contains many highly reactive species, including electronically excited ions, electrons, free radicals and neutrals[5]. It is generated when a gas becomes ionized[16] and is composed of a low ionization degree of electrons[26].

Although a better wetting (by increasing active species, such as carboxylic groups) and surface free energy had facilitated the adhesive distribution when human dentin was NTAP pre-treated[24], the knowledge about enamel is scarce to date[2, 18, 25]. A recent study showed that NTAP increased the hydrophilicity of enamel and improved the marginal sealing of sealants by reduction the voids in the interface[25]; this result suggests an advantage of applying NTAP when using a mild UA to make a composite resin restoration.

Considering the selective etching of enamel margins with phosphoric acid (SEE) is still need when using SBU[29] because the acidity of self-etching adhesives monomers[7,27], is possible that pre-treating enamel with NTAP would a) make SEE an unnecessary step, b) increase the bond strength even in long-term aging if the adhesive is applied by self-etch protocol, c) decrease the number of critical clinical steps (humidity control in acid etching and clinical time), and d) reduce the probability of failure. Based on the aforementioned, this in vitro study aimed to assess the effect of NTAP treatment on the bond strength of a universal adhesive system to human enamel, with or without enamel etching, when submitted to aging, simulating what occurs clinically with the restorations over time. The null hypotheses tested were NTAP, acid etching, and aging do not increase the bond strength of universal adhesives to human dental enamel.

2. Materials and Methods

The present study was approved by Research Ethics Committee of the State University of Western Paraná (UNIOESTE) (CAAE 48197115.3.0000.0107). Twenty extracted unerupted human third molars were collected, cleaned, and stored in a 0.1% thymol solution under refrigeration before the experiment. Then, the roots were sectioned using a low-speed diamond saw.

Sample calculation

Sample size was calculated in terms of a minimum detectable difference in a mean BS (MPa) of 0.62 (30 human teeth, 8 per group), using the same adhesive system with and without enamel acid etching[29]. However, for predicting eventual losses, 10 samples were included.

Specimen preparation

The crowns were sectioned in the buccolingual direction using a 4"x0.012"x0.5" diamond cutting disc (Erios Equipamentos Ltda - EPP, São Paulo SP, Brazil) attached to a precision saw (Labcut 1010, Erios Equipamentos Ltda - EPP, São Paulo, SP, Brazil) at low speed to obtain two enamel proximal surfaces (samples 40 in total; 10 per group, 10 stick each, on average). First, the samples was flattened and polished with sequential silicon carbide abrasive papers up to the 2000 grit under water cooling, (Figure 1) and randomly assigned to two groups (n=20) according to the acid etching treatment (etched [Et], no-etched [no-Et]). For etching, 37% phosphoric acid gel (Condac 37, FGM, Joinville, SC, Brazil) was applied for 15 s, rinsed, and gently air-dried. Next, each group was subdivided into two (n=10) according to the NTAP treatment (plasma [P], no-plasma [no-P]) that consist of an argon unit as operating gas (Surface Plasma Tool Model, Surface Engineering and Plasma Solution, Campinas, SP, Brazil). The plasma torch (which emerged at the nozzle of 1 mm in diameter) was applied by scanning mode over the entire enamel surface for 20 s at room temperature of 22° C. The nozzle-enamel surface distance was kept in 10 mm (allowed by a movable base). Thus, the following groups were designed: Et/P, Et/no-P, no-Et/P, no-Et/no-P. Finally, the Scothbond Universal [SBU] was actively applied in all samples with a microbrush (20 s), air-dried (5 s), and light-cured (10 s) using a Valo Curing Light (Ultradent Products, INC., South Jordan, UT, USA) with an irradiance of 1000 mW/cm^2 and a composite resin block (Filtek Z100, 3M ESPE, St Paul, MN, USA) was incrementally built up (4 mm in thickness). The samples were kept for 24-hour in deionized water (37° C). Table 1 shows the compositions and manufactures of the materials.

Microtensile bond strength (µTBS)

After that, they were longitudinally sectioned in both 'x' and 'y' directions across the adhesive interface using a low-speed diamond saw attached to a precision cutting machine to obtain bonded sticks (1.0 mm±0.2 mm^2; 10 per sample) (Figure 1). Then, five sticks of each group were immediately [24h] submitted to the µTBS test and the other half after 12 months of aging [12m]. Sticks were attached to a testing jig with cyanoacrylate glue (Super Bonder Gel, Loctite, Henkel Ltda, São Paulo, SP, Brazil) and tested in tension in a universal testing machine (Emic-Instron, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/s. The results of µTBS values were expressed in MPa. Statistical analysis was performed using the SigmaPlot 12.0 software at a 5% significance level, considering the plasma, etching, and aging as variation factors. The means of µTBS values among the specimens in each group were used for the statistical analysis. The variable passed the normality and homogeneity tests (Shapiro-Wilk/Kolmogorov-Smirnov) and was submitted to a three-way ANOVA with subdivided plots ($p<0.01$). Fractured surfaces were analyzed for failure mode by digital optical microscopy (Dino-Lite Premier, Anmo Electronics Corporation, New Taipei City, Taiwan) (magnification of 100%) and categorized according to the predominant remaining structure: adhesive in enamel (A); cohesive in enamel (CE); cohesive in resin (CR); or mixed (failure between adhesive/resin [M-AR] or among adhesive/resin/enamel [M-ARE]). The failure modes were expressed as percentages for each group.

3. Results

The mean values of μTBS are shown in Table 2. The three isolated factors showed statistical difference: time (24h x 12m) ($p<0.001$), plasma (P x non-P) ($p=0.04$), and acid etching (Et x no-Et) ($p=0.006$). There was significant interaction only for the factor of time-acid etching ($p=0.013$).

The enamel acid etching increased μTBS values when sticks were submitted to immediate traction testing, whereas plasma did not, even when combined with phosphoric acid. Overall, the highest μTBS value, which differed from all other groups, was observed for Et/no-P (52.7±13.8 MPa), followed by Et/P (40.9± 5.5 MPa), no-Et/no-P (37.2±16.9 MPa), and no-Et/P (32.9±11.8 MPa); the latter presented the lowest μTBS value. The 12-month aging reduced μTBS values for all groups, with no statistical difference among them. The μTBS ranged from 20.3±3.8 MPa (for Et/no-P) to 17.68±15.1 MPa (for no-Et/P). Table 3 presents the failure rates of all groups.

4. Discussion

The present study was planned to advance the understanding of the effect of NTAP, etching and aging on enamel adhesion by an established methodology[4,6,19,26]. It was expected plasma would increase surface wettability and consequently the bond strength between enamel and adhesive[1,14], as well acid etching. A direct comparison between the μTBS means values of Et and non-Et groups showed that etching increased μTBS significantly in samples tested immediately after 24-hour storage. Besides, those values were significantly reduced after 12 months of aging for all groups, illustrated by the percentage of A and M failures when the samples were not subjected to both treatments. The absence of CR failures for the no-Et/no-P groups indicates that there was a lack of ability of the adhesive to bind to the substrate, when applied without previous surface treatment. Thus, the null hypotheses of acid etching and aging would not affect the μTBS of universal adhesives to human dental enamel was rejected. The present results are corroborated by other studies[9,13,28], except for the universal adhesive, which differ by trademark. According to studies[9], adhesive system exhibited such a performance when evaluated in vitro. Indeed, a systematic review and meta-analisys[7] showed a lower bond strength to enamel with self-etch adhesives than that of etch-and-rinse adhesives. So, dentists should etch the enamel margins with 37% phosphoric acid technique to achieve better clinical results when using universal adhesives.

The way samples were affected by aging needs to be mentioned. Investigation of all dental adhesives categories showed mechanical and morphological evidence of degradation that resembles the effects of in vivo aging, after three months[10]. In the present study, samples tested after 12 months of aging in water indicated a significant reduction of μTBS, regardless of etching and NTAP treatments, as found others[28,21].

The high percentage of A/M failures for the no-Et/P groups, regardless of aging time for both aging times. The no-Et/P group showed the positive effect of the etching procedure, considering that the μTBS values were high enough for the loss of composite resin rather than adhesive.

In respect of NTAP, this treatment did not increase the immediate μTBS values. It means the enamel-resin bonding was not favoured, despite the high hydrophilicity of both SBU and enamel surface[19]. Thus, the first null hypothesis was accepted. Based on the present results and to the best knowledge about the dense crystalline structure and mineral composition of enamel, the authors suggest NTAP is not able to remove calcium phosphate from enamel enough to create microporosities and to promote a higher μTBS (as those well-documented for phosphoric acid etching)[27]. Besides, other study showed insignificant change in Ca/P ratio of enamel when plasma was applied on surface[18].

So, the effect of this treatment in enamel is quite different from the benefit observed in dentin[1,11,12,14,18]. Favourable effect of NTAP for dentin/composite resin interfaces[18], the increased μTBS to dentin[11,12] as well the superficial changes induced by plasma (30 s) in dentin previously exposed to sodium hypochlorite which improved bond strength for a self-etch adhesive system[1] were demonstrated. Recently, a scanning electron microscopy analysis showed NTAP increased hydrophilicity of the dentin surface (compared to the untreated one), favoured the primer penetration and led a longer and more abundant resin tags[14].On the other hand, NTAP produced no apparent micro-morphological changes on dentin surface comparable to acid etching[24].

To conclude, NTAP treatment is considered unnecessary for enamel bonding. The phosphoric acid etching is still the best strategy to improve bond strength of universal adhesives to enamel.

5. Conclusion

It was concluded that the phosphoric acid etching of enamel increased the immediate bond strength of the universal adhesive, whereas non-thermal atmospheric plasma did not increase bond strength. After 12 months of storage in water, the bond strength significantly reduced in all groups.

6. References

1. ABREU JLB, PRADO M, SIMÃO RA, SILVA EM, DIAS KRHC. Effect of Non-Thermal Argon Plasma on Bond Strength of a Self-Etch Adhesive System to NaOCl-Treated Dentin. Braz Dent J 2016;446-451.
2. ALMOAMMAR S, ALSHAHRANI I, MOHHABAB AA, DUARTE S, JANAL M, KHOO E. Non-thermal plasma treatment to enhance the adhesion between enamel surface and orthodontic bracket. Bio-Medical Materials and Engineering 2019;439-448.
3. AYRES AP, BONVENT JJ, MOGILEVYCH B, SOARES LES, MARTIN AA, AMBROSANO GM, NASCIMENTO FD, VAN MEERBEEK B, GIANNINI M. Effect of non-thermal atmospheric plasma on the dentin-surface topography and composition and on the bond strength of a universal adhesive. Eur J Oral Sci 2018;53-65.
4. AYRES AP, FREITAS PH, DE MUNCK J, VANANROYE A, CLASEN C, DOS SANTOS DIAS CT, GIANNINI M, VAN MEERBEEK B. Benefits of Nonthermal Atmospheric Plasma Treatment on Dentin Adhesion. Oper Dent, 2018;43-6.
5. CHEN M, ZHANG Y, SKY DRIVER M, CARUSO AN, YU Q, WANG Y. Surface modification of several dental substrates by non-thermal, atmospheric plasma brush. Dent Mater 2013; 871-880.
6. CHERUTHAZHEKATT S, CERNÁK M, SLAVICEK P, HAVEL J. Gas plasmas and plasma modified materials in medicine. J Appl Biomed 2010;55-66.
7. CUEVAS-SUÁRES CE, DA ROSA WLO, LUND RG, SILDA AF, PIVA E. Bonding Performance of Universal Adhesives: An Updated Systematic Review and Meta-Analysis. J Adhes Dent 2019;7-26.
8. D'AGOSTINO R, FAVIA P, OEHR C, WERTHEIMER MR. Low-Temperature Plasma Processing of Materials: Past, Presente and Future. Plasma Process. Polym 2005;7-15.
9. DA ROSA WL, PIVA E, SILVA AF. Bond strength of universal adhesives: A systematic review and meta-analysis. J Dent 2015;765-776.
10. DE MUNCK J, VAN LANDUYT K, PEUMANS M, POITEVIN A, LAMBRECHTS P, BRAEM M, VAN MEERBEEK B. A Critical Review of the Durability of Adhesion to tooth Tissue: Methods and Results. J Dent Res 2005;118-132.
11. DONG X, RITTS AC, STALLER C, YU Q, CHEN M, WANG Y. Evaluation of plasma treatment effects on improving adhesive/dentin bonding by using the same tooth controls and varying cross-sectional surface areas. Eur J Oral Sci 2013;355-362.

12. DONG X, LI H, CHEN M, WANG Y, YU Q. Plasma treatment of dentin surfaces for improving self-etching adhesive/dentin interface bonding. Clin Plasma Med 2015;10-16.
13. GORACCI C, RENGO C, EUSEPI L, JULOSKI J, VICHI A, FERRARI M. Influence of selective enamel etching on the bonding effectiveness of a new "all-in-one" adhesive. Am J Dent 2013;99-104.
14. HAN GJ, KIM JH, CHUNG SN, CHUN BH, KIM CK, SEO DG, SON HH, CHO BH. Effects of non-thermal atmospheric pressure pulsed plasma on the adhesion and durability of resin composite to dentin. Eur J Oral Sci 2014;1-7.
15. HANABUSA M, MINE A, KUBOKI T, MOMOI Y, VAN ENDE A, VAN MEERBEEK B, DE MUNCK J. Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine. J Dent 2012;475-481.
16. HIRATA R, TEIXEIRA H, AYRES APA, MACHADO LS, COELHO PG, THOMPSON VP, GIANNINI M. Long-term Adhesion Study of Self-etchingSystems to Plasma-treated Dentin. J Adhes Dent 2015;227-233.
17. INOUE S, VARGAS MA, ABE Y, YOSHIDA Y, LAMBRECHTS P, VANHERLE G, SANO H, VAN MEERKEEK B. Micro-tensile Bond strength of eleven modern adhesives to dentin. J Adhes Dent 2001;237-46.
18. LEHMANN A, RUEPPELL A, SCHINDLER A, ZYLLA IM, SEIFERT HJ, NOTHDURFT F, HANNIG M, RUPF S. Modification of Enamel and Dentin Surfaces by Non-thermal Atmospheric Plasma. Plasma Process. Polym 2013;262-270.
19. LIU Y, LIU Q, YU QS, WANG Y. Nonthermal Atmospheric Plasmas in Dental Restoration. J Dent Res 2016;496-505.
20. LORENZO B, MAZZONI A, RUGGERI A, CADENARO M, DI LENARDA R, DORIGO ES. Dental adhesion review: Aging and stability of the bonded interface. Dent Mater 2007;90-101.
21. MAKISHI P, ANDRÉ CB, AYRES A, MARTINS AL, GIANNINI M. Effect of Storage Time on Bond Strength and Nanoleakage Expression of Universal Adhesives Bonded to Dentin and Etched Enamel. Oper Dent 2016;305-317.
22. MCLEAN DE, MEYERS EJ, GUILLORY VL, VANDEWALLE KS. Enamel Bond Strength of New Universal Adhesive Bonding Agents. Oper Dent 2015;410-417.
23. RITTS AC, LI H, YU Q, XU C, YAO X, HONG L, WANG Y. Dentin surface treatment using a non-thermal argon plasma brush for interfacial bonding improvement in composite restoration. Eur J Oral Sci 2010;510-516.

24. STASIC JN, SELAKOVIC N, PUAC N, MILETIC M, MALOVIC G, PETROVIC ZLJ, VELJOVIC DN, MILETIC V. Effects of non-thermal atmospheric plasma treatment on dentin wetting and surface free energy for application of universal adhesives. Clin Oral Invest 2018;1-14.
25. TEIXEIRA H, COELHO P, DUARTE S, JANAL M, SILVA N, THOMPSON V. Influence of atmospheric pressure plasma treatment on mechanical proprieties of enamel and sealant bond strength. J Biomed Mater Res Part B 2014;1-10.
26. TENDERO C, TIXIER C, TRISTANT P, DESMAISON J, LEPRINCE P. Atmospheric pressure plasmas: A review. Spectrochimica Acta Part B 2006;2-30.
27. VAN MEERBEEK B, YOSHIHARA K, YOSHIDA Y, MINE A, DE MUNCK J, VAN LANDUYT KL. State of the art of self-etch adhesives. Dent Mater 2011;17-28.
28. VERMELHO PM, REIS AF, AMBROSANO GMB, GIANNINI M. Adhesion of multimode adhesives to enamel and dentin after one year of water storage. Clin Oral Investig 2017;1707-1715.
29. VICENZI B, RIBAS MM, SCHMITT VL, NAUFEL FS, THIESEN KPP, SOUZA MDB. Effect of acid etching and aging on bond strenght of a universal adhesive on dental enamel. J Dent Health Oral Disord Ther. 2019;299-303.

7. Annexes

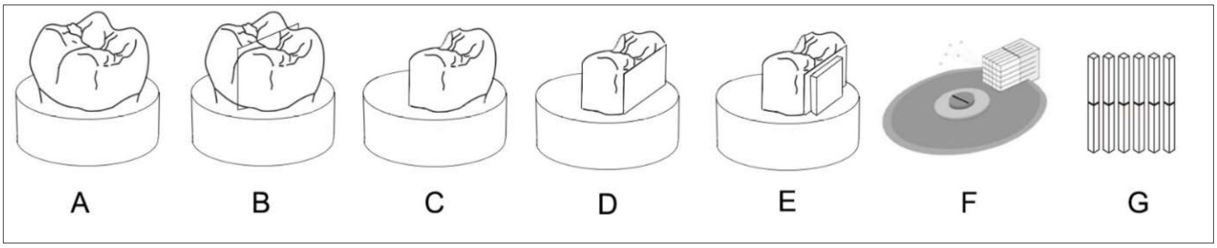

Figure 1. Schematic representation of the preparation of the specimens for the microtensile test. From a healthy tooth (A), sections were made in the VL direction (B, C). A flat enamel surface (D) was obtained, for which the restoration of composite resin was carried out according to the experimental groups (E). After serial cuts (F), toothpick specimens (G) were produced.

Table 1. Composition and manufacturers of the materials used in this study.

Materials, manufacturer	Composition
Scotchbond Universal Adhesive, 3M Oral Care, St Paul, MN, USA	Adhesive: 10-MDP, phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid, copolymer, filler, ethanol, water, initiators, silane
Z100, 3M Oral Care, St Paul, MN, USA	Silane treated ceramic, triethylene glycol dimethacrylate (TEGDMA), bisphenol A diglycidyl A ether dimethacrylate (BISGMA), 2-Benzotriazolyl-4-methylphenol
Condac 37, FGM, Joinville, SC, Brazil	37% phosphoric acid, thickener, pigment, deionized water

Table 2. Microtensile bond strength means (MPa) obtained at 24 hours or after 12 months of aging.

		Treatment			
		Et/no-P	no-Et/no-P	Et/P	no-Et/P
Evaluation Time	24 hours	52.7 (13.8)^{Aa}	37.2 (16.9)^{Bca}	40.9 (5.5)^{Ba}	32.9 (11.8)^{Ca}
	12 months	19.3 (5.3)^{Ab}	20.1 (3.8)^{Ab}	19.6 (3.8)^{Ab}	17.6 (15.1)^{Ab}

Mean (SD), n=20. Different superscript capital letters indicate significant difference among the means and compare treatments within the evaluation time. Different superscript lower-case letters indicate significant difference among the means and compare evaluation times within the same treatment. Three-way ANOVA ($p<0.01$). Plasma [P], non-plasma [no-P]), etched [Et], non-etched [no-Et], 24-hour aging [24h], 12-month aging [12m].

Table 3. Failure pattern incidence for the experimental groups.

		Non-NTAP	NTAP
24 hours	Non-etched	A – 37% M – 63%	A - 53,5% M – 46.5%
	Etched	A – 6,5% M – 56.5% Cr – 37%	A – 9.5% M – 65% Cr –25.5%
12 months	Non-etched	A – 49% M – 51%	A – 64% M – 36%
	Etched	A – 35% M – 58% Cr – 7%	A – 12.5% M – 64% Cr –23.5%

Adhesive (A); Cohesive in resin (Cr); Mixed (M).