

# Effect of erosive and abrasive challenges on the bond strength and marginal degradation of composite restorations

## Efeito dos desafios erosivos e abrasivos na resistência de união e degradação marginal em restaurações de resina composta

### Abstract

**Purpose:** To evaluate the microtensile bond strength ( $\mu$ TBS) and the presence of marginal gaps (MG) on composite restorations submitted to erosive and abrasive challenges.

**Methods:** Seventy bovine incisors were sectioned in two halves; one half was used for the  $\mu$ TBS assay and the other for the MG measurement. For  $\mu$ TBS, the dentin on the incisal edge was exposed, the adhesive system was applied (Single Bond 2), and composite blocks were built (Filtek Z250). For MG, two rounded-shaped cavities were prepared and restored with the same materials. Specimens ( $n=10/\mu$ TBS and  $n=10$ /MG) were distributed into 7 groups: G1 – control; G2 – abrasive challenge (2 brushing cycles, for 35 days); G3 – erosive challenge (two 90 s immersions into 20 mL of a citric solution, for 35 days); G4, G5, G6, and G7 – abrasive challenge performed 0, 15, 30 or 60 min before the erosive challenge. Beam-shaped specimens were tested for  $\mu$ TBS until failure. MG was measured by means of qualitative scores, recorded in 8 regions from the margin.

**Results:** No significant differences in  $\mu$ TBS means or MG scores were detected among the tested experimental groups.

**Conclusion:** The abrasive and erosive challenges were not able to affect the bond of a conventional two-step adhesive system to tooth substrates.

**Key words:** Composite; dentin; enamel; dentin-bonding agents; erosion; tooth abrasion

### Resumo

**Objetivo:** Avaliar a resistência de união pela microtração ( $\mu$ TBS) e a presença de fendas marginais (FM) em restaurações de resina composta submetidas a desafios erosivos e abrasivos.

**Metodologia:** Setenta incisivos bovinos foram divididos ao meio; metade foi utilizada na avaliação da  $\mu$ TBS e a outra para a presença de FM. Para  $\mu$ TBS, a dentina no bordo dos fragmentos foi exposta, o sistema adesivo (Single Bond 2) foi aplicado, e blocos de compósito (Filtek Z250) foram construídos. Para FM, duas cavidades circulares foram preparadas e restauradas com os mesmos materiais. Espécimes ( $n=10/\mu$ TBS e  $n=10$ /FM) foram distribuídos em 7 grupos: G1 – controle, G2 – desafio abrasivo (2 ciclos de escovação, por 35 dias), G3 – desafio erosivo (2 imersões de 90 s em 20 ml de solução cítrica, por 35 dias), G4, G5, G6 e G7 – desafio abrasivo realizado 0, 15, 30 ou 60 min após o desafio erosivo. Espécimes em formato de “palitos” foram testados sob tração até a fratura. A FM foi avaliada por meio de escores qualitativos, medidos em 8 regiões da margem.

**Resultados:** Não foram detectadas diferenças significativas entre as médias de  $\mu$ TBS, nem entre os escores de FM.

**Conclusão:** Os desafios erosivos e abrasivos não foram capazes de afetar a união do sistema adesivo convencional de dois passos aos substratos dentais.

**Palavras-chave:** Resinas compostas; dentina; esmalte; adesivos dentinários; erosão; abrasão dentária

Paula Mathias <sup>a</sup>  
Angela Guimarães Lessa <sup>b</sup>  
Andrea Nóbrega Cavalcanti <sup>c</sup>

<sup>a</sup> Faculdade de Odontologia da Universidade Federal da Bahia, Salvador, BA, Brazil

<sup>b</sup> Private practice, Salvador, BA, Brazil

<sup>c</sup> Fundação Bahiana para Desenvolvimento das Ciências, Curso de Odontologia, Salvador, BA, Brazil

### Correspondence:

Paula Mathias  
Faculdade de Odontologia da  
Universidade Federal da Bahia  
Av. Araújo Pinho, 62, Canela  
Salvador, BA – Brazil  
40110-060  
E-mail: pmathias@yahoo.com

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## Introduction

The consumption of citric beverages has increased in the last years partially because of the search for a healthy diet (1). Nevertheless, *in vitro*, *in situ*, and *in vivo* studies have demonstrated that such beverages might damage the surface of dental hard tissues by erosion (2-6). Erosion lesions are defined as the local, chronic, pathologic, and painless loss of tooth structure, throughout a chemical process, with no bacteria involvement (7).

The direct contact of acidic solutions is able to demineralize tooth surfaces, reducing enamel and dentin hardness and increasing surface wear (2,3,8). The acidic challenge is more aggressive to dentin surfaces, since this dental substrate is less mineralized than the dental enamel (3). The frequent ingestion of acidic substances, especially those containing citric acid, promotes the reduction of salivary pH, resulting in a decrease of the buffering effect of saliva against demineralization (5). In addition, the association of acidic demineralization by dietary solutions and routine mechanical abrasive processes, such as toothbrushing, might increase the surface loss of tooth structure (9), especially when brushing is performed right after the contact with acidic substances (3,10).

The negative action of low pH solutions is not restricted to dental surfaces, but it is also able to accelerate the degradation of resin-based materials (11-13). Considering the possible damage of adhesive interfaces by acidic solutions associated with abrasion, the aim of the present study was to evaluate the bond strength and gap formation in resin composite restorations submitted to an erosive (citric juice)/abrasive (toothbrushing) challenge. The experimental hypothesis was that the erosive and abrasive challenges decrease the bond strength and increase gap formation around adhesive composite restorations.

## Methods

Seventy recently extracted bovine incisors were stored in 0.5% thymol solution. After cleansing and removal of organic debris, teeth were longitudinally sectioned into two halves according to the mesial-distal direction. The microtensile bond strength ( $\mu$ TBS) test was conducted on the buccal half, and gap formation (GF) was analyzed on the lingual half.

### Specimen preparation for $\mu$ TBS

The incisal edge of each fragment was flattened with silicon carbide papers (100 and 200-grit) under constant refrigeration, until dentin exposure. The complete removal of enamel structure was verified under 4X magnification (Bioart, São Carlos, SP, Brazil). Afterwards, surfaces were abraded with silicon carbide paper of decreased abrasiveness under constant water irrigation (220, 320, 400-grit for 10 s; 600-grit for 60 s) in order to standardize the smear layer.

After surface preparation, specimens were conditioned with 37% phosphoric acid (Condac 37, FGM, Joinville, SC,

Brazil) for 15 s. Surfaces were then washed with water for the same period, and dried with absorbent papers, keeping dentin humidity. The adhesive system (Adper Single Bond Plus, 3M ESPE, St. Paul, MN, USA) was applied in two consecutive layers, with a brief air spray between them. After solvent evaporation, the adhesive layer was light cured for 10 s using a halogen light source (OptiPlus, Gnatus, Ribeirão Preto, Brazil), emitting approximately 550 mW/cm<sup>2</sup> of irradiance. Resin composite build-ups (Filtek Z250, 3M ESPE, St. Paul, MN, USA) of approximately 5×5×2 mm were incrementally placed on the dentin surface at the incisal region. Specimens were individually stored in distilled water at 37°C during the 35 days of this study; distilled water was daily changed.

### Specimen preparation for the evaluation of GF

Two cylindrical cavities with 2×1.5 mm (diameter×depth), with margins in enamel (middle third of the crown) and dentin (1 mm below the enamel-cement junction) were prepared with coarse diamond burs (#2294, KG Sorensen, Barueri, SP, Brazil), in a high-speed handpiece with constant water irrigation. Burs were replaced after each 10 preparations to guarantee their uniformity. Cavities were restored with the same materials and following the instructions previously described except for composite insertion, which was conducted with a single increment. After 24 h, restorations were polished with aluminum oxide disks of decreased abrasiveness (Sof-lex, 3M ESPE, St. Paul, MN, USA). Each disk was used for 30 s with no refrigeration; disks were replaced after each 10 procedures. Specimens were washed between changes on disks granulation.

### Erosive and abrasive challenges

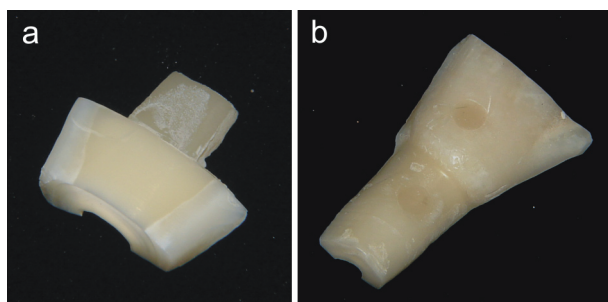
Specimens prepared for  $\mu$ TBS testing and GF were randomly assigned into seven experimental groups (n=10/ $\mu$ TBS and n=10/GF):

- G1 – Control: storage in distilled water during the 35 days period;
- G2 – Abrasive challenge: simulation of two daily cycles of toothbrushing for 20 s using an electric brushing device with no dentifrice (Oral B CrossAction Power, São Paulo, SP, Brazil). During the brush cycle, the brush part of the electric device was placed in contact with the specimens surface and no hand pressure was made in order to standardize the load applied during this procedure. Brushing cycles were performed for 35 days;
- G3 – Erosive challenge: specimens were immersed in 20 mL of orange juice, twice a day, for 90 s, during 35 days (pH=4.7; Ades, São Paulo, SP, Brazil). The orange juice contained, according to its manufacturer: water, soy extract, sugar, concentrated orange juice, dextrin, vitamins and minerals (B1, B2, B3, B6, B12, C, folic acid, iron, and zinc), citric and malic acid, citric pectin, calcium chloride, sucralose, pigments;
- G4 – Abrasive challenge performed right after the erosive challenge;

- G5 – Abrasive challenge performed 15 min after the erosive challenge;
- G6 – Abrasive challenge performed 30 min after the erosive challenge;
- G7 – Abrasive challenge performed 60 min after the erosive challenge.

**μTBS testing**

Specimens were sectioned perpendicular to the bonded interface of each tooth in the “x” and “y” directions, resulting in 0.7 mm-thick slabs (n=3 per restoration) with a slow speed diamond wafering blade (Isomet 1000, Buehler, Lake Buff, IL, USA) and constant water coolant. Specimens were then mounted in a testing apparatus with a cyanocrylate adhesive (Super Bonder; Henckel Loctite, Itapevi, SP, Brazil), and debonded using a universal testing machine (DL 500; EMIC Ltd., Sao Jose dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min until failure. Means and standard deviations were calculated and expressed in MPa. Premature debonded specimens were not considered for statistical analysis. Statistical analysis was performed using one-way ANOVA and Tukey test, at a significance level of 5%. After debonding, specimens were viewed in a stereoscope microscope to classify their failure modes into the following categories: adhesive failure, cohesive failure in dentin, cohesive failure in composite and mixed failure.



**Fig. 1.** (a) Specimen for μTBS testing presenting a composite build-up on the incisal region of the tooth; (b) Specimen presenting two cylindrical cavities for the evaluation of gap formation on dentin and enamel margins.

**Evaluation of GF**

The tooth-restoration interfaces were viewed in a stereoscope microscope under 40X magnification, by a single examiner in a randomized and blinded sequence. Eight regions on cavity margins were evaluated: 1) the most incisal area; 2) area between incisal and mesial surfaces; 3) mesial area; 4) area between mesial and cervical surfaces; 5) the most cervical area; 6) area between cervical and distal surfaces; 7) distal area; and 8) area between distal and incisal surfaces. These regions were classified by using qualitative scores: 1 – absence of marginal gap and staining; 2 – absence of GF and presence of some staining; 3 – presence of GF and absence of staining; 4 – presence of GF and some staining. Statistical analysis was conducted with non-parametric tests

with a 95% confidence level: the regions around the enamel and dentin margins were compared with the Friedman test; group comparison was performed with the Kruskal-Wallis test, using the median of scores at the eight regions in each margin; enamel and dentin cavities were compared with the Wilcoxon test.

**Results**

Means and standard-deviations of the bond strength are depicted in Table 1. The statistical analysis did not observed significant differences among experimental groups (P=0.40). Table 2 presents the failure modes obtained. In all groups, most specimens presented adhesive failures. The other mode frequently observed was the mixed one. Medians obtained were predominantly from score 1 (approximately 96%), regardless of the marginal area, the substrate and the experimental group. Therefore, it is possible to affirm that no gap formation and stained is provided under the experimental conditions tested. Statistical analysis did not detect any significant differences (P>0.05).

**Table 1.** Mean (standard deviation) of the bond strength of the control and experimental groups.

Group	Bond strength (MPa)
G1 – Control	17.31 (14.40) pf=2
G2 – Abrasive challenge	10.83 (8.12) pf=3
G3 – Erosive challenge	13.75 (8.26) pf=2
G4 – Abrasive challenge performed right after the erosive challenge	8.71 (3.66) pf=4
G5 – Abrasive challenge performed 15 min after the erosive challenge	13.79 (7.98) pf=5
G6 – Abrasive challenge performed 30 min after the erosive challenge	14.74 (3.55) pf=2
G7 – Abrasive challenge performed 60 min after the erosive challenge	16.25 (6.75) pf=4

pf - number of premature failures.

**Table 2.** Distribution of the failure mode.

Group	A	CD	CC	M
G1 – Control	100%	–	–	–
G2 – Abrasive challenge	71%	–	–	29%
G3 – Erosive challenge	55%	5%	–	40%
G4 – Abrasive challenge performed right after the erosive challenge	69%	–	6%	25%
G5 – Abrasive challenge performed 15 min after the erosive challenge	85%	–	–	15%
G6 – Abrasive challenge performed 30 min after the erosive challenge	79%	–	–	21%
G7 – Abrasive challenge performed 60 min after the erosive challenge	73%	–	–	27%

Abbreviations: A - Adhesive; CD - Cohesive in dentin; CC - Cohesive in composite; M - Mixed.

## Discussion

Adhesive restorations are frequently submitted to different challenges in the oral environment, such as thermal alterations, mechanical loading through mastication and toothbrushing, and erosive attacks by intrinsic or extrinsic acidic sources (5,8). In the present study, the erosive challenge placed by citric juice (pH=4.7) and the abrasive challenge through simulated toothbrushing were investigated individually or combined to test if the complex adhesive interface obtained with simplified systems allied to their greater permeability by the presence of hydrophilic monomers (14) would increase the susceptibility to erosive and abrasive challenges.

Previous studies have demonstrated that adhesive interfaces aged in aqueous mediums present significant decrease on bond strength and marginal sealing between adhesive system and dental structures (15,16). Because of the possible penetration of fluids through adhesive interfaces, the dietary and oral hygienic habits of patients should be analyzed. Not only patients with poor habits of oral hygienic should be in concern given the possibility of secondary caries lesions around restorations margins, but also patients with greater frequency of toothbrushing should be carefully followed due to the possibility of loss of substance and polymer degradation if the action is conducted right after drinking juices or eating citric fruits (1).

The erosive potential of citric juices on enamel surfaces was evaluated in a previous study (5). This potential was determinate by the pH of each fruit juice and through micro structural alterations on enamel surfaces. All fruit juices present some erosive potential with pH varying from 1.64 to 4.0. The enamel demineralization depends not only on the concentration of acid in each solution but also on the ionic dissociation of each solution (presence of ions H<sup>+</sup>). Along with the irreversible loss of dental hard tissue, acidic solutions might provide progressive softening of tooth surfaces, rendering them more susceptible to mechanical forces, such as abrasion (17).

In a clinical situation, toothbrushing is associated with a number of variables, such as the abrasiveness, pH, buffer capacity, and fluoride content on toothpastes (17). A previous *in situ* study on the effect of different dentifrices on eroded dentin surfaces subjected or not to abrasion has indicated that abrasion increases wear of acid-softened tooth surfaces (17). Fluoride concentrations around 1,100 ppm in dentifrices can reduce but not inhibit dentin wear by erosion and erosion plus abrasion (17).

Nonetheless, under the conditions tested in the present study, results of bond strength and gap formation indicated that erosive and abrasive challenges were not able to modify the adhesion to tooth structures. Even though some studies have indicated that erosive challenges make tooth tissues more susceptible to abrasion and modify the surface of some restorative materials (2,3,9,19), the resin composite proved to be unaffected by the experimental conditions

tested in the present study. A previous study evaluated the effect of erosive pH cycling on the percentage of surface microhardness change and wear of different restorative materials (resin-modified glass-ionomer, conventional glass ionomer, composite, and amalgam) (6). Those authors concluded that surface changes after the erosive challenge were discrete, although longer periods of erosive pH cycling and the use of other types of beverages would add supplementary information. Similar findings were also noted in another investigation on the erosive potential of some food on the surface hardness of enamel, dentin, and restorative materials (universal and microfilled composite, conventional and resin-modified glass ionomer cement, and composite modified by polyacids) (11). In that study, the orange juice reduced the hardness of dental enamel, while the other substrates were not affected (11).

It should be considered that, in order to prepare specimens for the bond strength test, the most outer area of the adhesive interfaces was removed. Consequently, areas that directly received the action of erosive and abrasive challenges were not tested for bond strength. For this reason, one can infer that if some alteration on the bonded interface has occurred, it might have been superficial, and thus, insufficient to allow a greater compromise of the interface. This effect could be probably detected through longitudinal evaluations, and using different methods to measure the bond strength, which should be performed in further studies.

Differently from bond strength testing, gap formation was investigated on the most superficial area of the adhesive interface, which was directly exposed to the erosive and abrasive challenge. The results are in accordance with the bond strength findings, indicating absence of marginal alterations after contact with orange juice and toothbrushing. These findings suggest that an adequately formed adhesive interface between restorative material and tooth structure might be able to resist against some erosive and abrasive challenges. Previous studies demonstrated the deleterious effect of acidic substances on dental substrates and adhesive interfaces but such studies have used longer periods of contact between solution and substrates, and lower pHs were tested in comparison with the present work.

## Conclusions

Accordingly to the method used and results obtained, it can be concluded that erosive and abrasive regimens were not able to reduce the strength of the adhesive interface or to induce gap formation along enamel and dentin bonded interfaces.

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