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DEGRADAÇÃO DE MATERIAIS RESTAURADORES RESINOSOS E DO ESMALTE DECÍDUO E PERMANENTE EM SOLUÇÕES SIMULADORAS DA DIETA

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, como parte dos requisitos para obtenção do título de Doutor em Materiais Dentários.

Orientadora: Profa. Dra. Regina Maria Puppin Rontani

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RESUMO

A cavidade bucal é considerada um ambiente agressivo onde a estrutura dentária e os materiais restauradores estão constantemente sujeitos a desafios térmicos, mecânicos e químicos. A associação destes fatores está diretamente relacionada à longevidade/estabilidade das restaurações, bem como à preservação da estrutura dentária. A exposição da estrutura dentária e dos materiais restauradores resinosos aos agentes da dieta pode causar erosão dental, amolecimento e aumento da rugosidade de restaurações resinosas, deixando-as mais susceptíveis ao desgaste. Dessa forma, os objetivos desta tese fundamentada em três capítulos foram: (1) avaliar o efeito do armazenamento em longo prazo em diferentes soluções simuladoras da dieta na rugosidade superficial de materiais restauradores resinosos; (2) avaliar o efeito da associação dos desafios erosivo e abrasivo no desgaste do esmalte decíduo e permanente; (3) avaliar o efeito da associação dos desafios corrosivo e abrasivo no desgaste de materiais restauradores resinosos. No capítulo 1, sessenta amostras de diferentes materiais restauradores (Filtek Z250, Esthet X, Filtek Flow, Dyract AP e Vitremer) foram armazenadas em 5 líquidos simuladores da dieta (água destilada, ácido lático, ácido cítrico, refrigerante a base de cola (Coca-Cola) e álcool) por até 6 meses. A rugosidade superficial das amostras foi mensurada após o polimento (*baseline*), 1 semana, 1 mês, 3 meses e 6 meses de armazenamento. Houve aumento significativo na rugosidade superficial apenas para Esthet X armazenado em Coca-Cola e em ácido cítrico após 1 e 3 meses, respectivamente; e para Dyract AP armazenado em água destilada após 6 meses. No capítulo 2, foram obtidas amostras planas de esmalte decíduo e permanente (n=10), as quais foram submetidas ao desgaste (100.000 ciclos a 1,9 Hz) em meio neutro (água deionizada) e meio ácido (ácido cítrico). Antes e após o desgaste, as amostras foram analisadas em perfilômetro (MTS 3D Profiler). A alteração do volume e a profundidade máxima do desgaste foram quantificadas utilizando o software Ansur 3D. O esmalte decíduo apresentou maior desgaste que o esmalte permanente, independentemente do meio. O meio ácido proporcionou maior desgaste do que o meio neutro ($p<0,05$). Diferença significativa entre meio neutro e ácido foi observada apenas para o esmalte decíduo. No capítulo 3, dez amostras de cada material [Filtek Supreme (S), Point 4 (P), Dyract AP (D) e Fuji II LC (F)] foram confeccionadas e submetidas ao desgaste (100.000 ciclos a 1,9 Hz) em meio neutro (água) e meio ácido (ácido cítrico). Antes e após o desgaste, as amostras foram analisadas em perfilômetro (MTS 3D Profiler). A alteração do volume e a profundidade máxima do desgaste foram quantificadas utilizando o software Ansur 3D. Diferença significativa no desgaste dos materiais foi observada (S=P<D<F), independentemente do meio. O meio ácido gerou maior desgaste nos materiais comparado ao meio neutro ($p<0{,}05$). Não houve diferença significativa entre o meio neutro e meio ácido para todos os materiais quando analisados individualmente. Concluindo, a degradação química dos materiais restauradores resinosos é material e solução dependente. O desafio ácido aumenta a susceptibilidade ao desgaste dos materiais restauradores resinosos e do esmalte dental, especialmente esmalte decíduo.

Palavras-chave: Biodegradação, desgaste, erosão dentária, esmalte decíduo, materiais restauradores.

ABSTRACT

The oral cavity can be considered a hostil environment. The dental structure and the restorative materials are constantly subjected to thermal, mechanical and chemical stresses. The interplay of these factors is directly related with the longevity/stability of the restorative treatment, and also with the dental structure preservation. The effect of diet constituents on dental structure and restorative materials can be dental erosion, softening and roughening of resin-based restorations. These phenomena increase the susceptibility of tooth and material wear. This way, the objectives of this work are: (1) to evaluate the effect of long-term storage in different food-simulating media on surface roughness of resin-based materials; (2) to evaluate the effect of the association of erosive and abrasive challenges on deciduous and permanent wear; (3) to evaluate the effect of the association of corrosive and abrasive challenges on resin-based materials wear. In the first chapter, sixty specimens of different material categories (Filtek Z250, Esthet X, Filtek Flow, Dyract AP, and Vitremer) were stored in five food-simulating solutions (distilled water, lactic acid, citric acid, carbonated soft drink (Coca-Cola), and ethanol) for 6 months. The surface roughness was measured after polishing (baseline), 1 week, 1, 3, and 6 months of storage. Significant difference was observed for Esthet X specimens stored in Coca-Cola and in citric acid after 1 and 3 months, respectively; and for Dyract AP specimens stored in distilled water after 6 months. In the second chapter, deciduous and permanent flat enamel specimens (n=10) were obtained. The specimens were subjected to the wear test (100,000 cycles at 1.9 Hz) in neutral (water) or acidic (citric acid) media. Specimens were profiled before and after the wear test using the MTS 3D Profiler. Volume loss and maximum depth were quantified by the Ansur 3D software. Deciduous enamel showed more wear than permanent enamel, regardless the media. The acidic media produced more wear than the neutral media ($p<0.05$). Significant difference was observed between neutral and acidic conditions only for deciduous enamel. In the third chapter, ten specimens were produced from each material [Filtek Supreme (S), Point 4 (P), Dyract AP (D), and Fuji II LC (F)]. The specimens were submitted to the wear test (100,000 cycles at 1.9 Hz) in neutral (water) and acidic (citric acid) media. Specimens were profiled before and after the wear test using the MTS 3D Profiler. Volume loss and maximum depth were quantified by the Ansur 3D software. Significant differences were observed for the material wear ($S=P<D<F$),

regardless the media condition. Acidic media produced more wear than neutral media (p<0,05). There were no significant differences between neutral and acidic condition for all materials, when individually analyzed. Concluding, the chemical degradation of resin-based materials is material and solution dependent. The acidic media produced more material and tooth wear, especially for deciduous enamel.

Key Words: Biodegradation, wear, dental erosion, deciduous enamel, restorative materials.

SUMÁRIO

INTRODUÇÃO

A estrutura dentária e as restaurações estão constantemente sujeitas aos desafios térmicos, mecânicos e químicos na cavidade bucal. A associação destes fatores está diretamente relacionada à longevidade/estabilidade das restaurações, bem como a preservação da estrutura dentária (Oilo, 1982).

A degradação química pode ser causada por desafios ácidos, incluindo aqueles produzidos pelo biofilme cariogênico (Asmussen, 1984), dieta ácida (Yap *et al.*, 2000a, 2000b, 2001 e 2002) e enzimas salivares (Larsen & Munksgaard, 1991; de Gee *et al.*, 1996). A dieta é a fonte externa mais comum de ácidos relacionados a biodegradação na cavidade bucal. O consumo de bebidas ácidas tem aumentado drasticamente em diversos países, especialmente entre crianças e adolescentes (Shaw & Smith, 1994; Yip *et al.*, 2003). Os ácidos mais frequentemente consumidos são ácido fosfórico, presente nos refrigerantes, e ácido cítrico, presente em alguns sucos de frutas (West *et al.*, 2000, 2001).

Os efeitos provocados pela dieta ácida são amolecimento (Wu & McKinney, 1982; Asmussen, 1984; Yap *et al.*, 2000a), corrosão (Sarkar, 2000) e aumento da rugosidade de materiais restauradores resinosos (Yap *et al.*, 2000a; Turssi *et al.*, 2001 e 2002) e erosão da estrutura dentária (Eisenburger & Addy, 2002; Eisenburger *et al.*, 2003; Lussi *et al.*, 2004), tornando-os, mais susceptíveis ao desgaste.

A rugosidade das superfícies intraorais duras (dentes e materiais restauradores) está diretamente relacionada à retenção de microorganismos. Demonstrou-se que um aumento na rugosidade superficial leva a uma mais rápida colonização e maturação do biofilme, aumentando assim o risco de desenvolvimento de cárie dentária e doença periodontal (Bollen *et al.*, 1997). Além disso, uma superfície rugosa pode aumentar a susceptibilidade ao manchamento e corrosão dos materiais restauradores (Hachiya *et al.*, 1984; Dietchi *et al.*, 1994; Bagheri *et al.*, 2005) ou gerar maior desgaste do dente ou restauração antagonista (Turssi *et al.*, 2003b).

O desgaste pode ser definido como conseqüência da interação entre superfícies que se movem em contato, o que resulta na remoção gradual de material dessas superfícies (Mair *et al.*, 1996). Este processo é cumulativo e multi-fatorial. Sua etiologia envolve a combinação de forças mecânicas e degradação química (de Gee *et al.*, 1996a, 1996b; Yip *et al.*, 2004). Os tipos de desgaste mais freqüentemente observados são: abrasivo, corrosivo ou erosivo e desgaste por fadiga (Mair *et al.*, 1996; Turssi *et al.*, 2003a). A associação destes diferentes processos é o principal fator para o entendimento do mecanismo de desgaste dos dentes e restaurações (Mair *et al.*, 1996; Turssi *et al.*, 2003a; Yip *et al.*, 2004).

O desgaste abrasivo ocorre quando superfícies ásperas e duras entram em contato com superfícies de menor dureza (Mair *et al.*, 1996). Este tipo de desgaste pode ser distinguido em dois processos, abrasão e atrição. Atrição está relacionada à perda de substância resultante do contato direto entre as superfícies antagonistas (desgaste por dois corpos), enquanto abrasão envolve a presença de um terceiro corpo, o abrasivo, agindo entre as superfícies antagonistas (desgaste por três corpos) (Condon & Ferracane, 1996). O desgaste provocado por fadiga é causado pela aplicação de uma carga repetitiva sobre a estrutura dentária e/ou restaurações, resultando na nucleação e propagação de microtrincas subsuperficiais.

Em Odontologia, o desgaste corrosivo está relacionado às reações químicas ocorridas na superfície das restaurações, tornando-as mais susceptíveis à remoção mecânica pelo contato com o antagonista (Turssi *et al.*, 2003a; Yip *et al.*, 2004). Já o termo desgaste erosivo geralmente é empregado para denominar o processo ocorrido na estrutura dentária. Erosão é definida como a perda da estrutura dentária pela dissolução química sem envolvimento de bactérias (ten Cate & Imfeld, 1996).

Dentes decíduos e permanentes apresentam diferenças estruturais e químicas (Featherstone & Mellberg, 1981; Amaechi *et al.*, 1999). Dentre essas diferenças encontram-se a menor espessura do esmalte e da dentina, a presença de uma camada mais espessa de esmalte aprismático, a maior porosidade do esmalte, o menor grau de mineralização, o maior conteúdo de dióxido de carbono e carbonato, bem como o menor conteúdo de fósforo no esmalte de dentes decíduos comparado aos dentes permanentes (Najouks *et al.*, 1967; Stack *et al.*, 1953). Essas diferenças morfo-fisiológicas entre os esmaltes decíduo e permanente podem desempenhar um papel importante na progressão do desgaste abrasivo e erosivo nestes substratos.

Alguns estudos demonstram maior susceptibilidade do esmalte decíduo à erosão, comparado ao esmalte permanente (Amaechi *et al.*, 1999; Johansson *et al.*, 2001). Entretanto, as diferenças entre o esmalte decíduo e permanente não são claras, uma vez que outros estudos não encontraram diferenças entre estes substratos quando analisada a resistência à erosão (Lussi *et al.*, 2000; Hunter *et al.,* 2000a e 2000b). Estes estudos avaliaram apenas um aspecto relacionado ao desgaste da estrutura dentária. Não se tem dados na literatura sobre a susceptibilidade do esmalte decíduo ao desafio simultâneo abrasivo e erosivo.

O desgaste dentário é um achado comum na dentição decídua, fazendo parte de um processo fisiológico de maturação e transição para a dentição permanente. Knight *et al.* 1997 demonstraram que o desgaste dentário na dentição decídua está significativamente relacionado com o subseqüente desgaste da dentição permanente, sugerindo uma etiologia comum para ambos. Assim, a identificação do desgaste dentário (não fisiológico) na infância pode ajudar a preveni-lo na dentição permanente (Warren *et al.*, 2002).

Deve-se lembrar que os mesmos processos de desgaste estão ativos em todas as estruturas bucais, incluindo os dentes e os materiais restauradores. O estudo dos materiais restauradores deve considerar não só a resistência dos mesmos ao desgaste, mas o efeito destes materiais no esmalte antagonista (Mair *et al.,* 1996). Materiais selecionados para restaurações em dentes decíduos devem sofrer desgaste similar ao dente. Entretanto, maior desgaste do esmalte decíduo adjacente às restaurações em compósito tem sido observado clinicamente (da Cunha *et al.*, 2006). O desgaste diferencial entre o esmalte decíduo e os materiais restauradores pode causar efeitos deletérios na estética e função do sistema mastigatório (Yip *et al.*, 2004). Dessa forma, observa-se que o entendimento dos diferentes processos de desgaste é fundamental para garantir a realização do procedimento restaurador o mais próximo possível da condição fisiológica.

Dessa forma, os objetivos desta tese[1], dividida em três capítulos, foram: 1. Avaliar o efeito do armazenamento em longo prazo em diferentes soluções simuladoras da dieta na rugosidade superficial de materiais restauradores resinosos; 2. Avaliar o efeito da associação dos desafios erosivo e abrasivo no desgaste de dentes decíduos e permanentes e correlacionar o desgaste dos dentes ao desgaste do antagonista; 3. Avaliar o efeito da associação dos desafios corrosivo e abrasivo no desgaste de diferentes materiais restauradores resinosos e correlacionar o desgaste dos materiais e o desgaste do esmalte antagonista.

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[1] Este trabalho foi realizado no formato alternativo, com base na deliberação da Comissão Central de Pós-Graduação (CCPG) da Universidade Estadual de Campinas (UNICAMP) n$^{o.}$ 001/98.

CAPÍTULO 1

In vitro long-term degradation of aesthetic restorative materials in acidic solutions[2]

Degradation of aesthetic restorative materials

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[2] Manuscrito submetido para publicação no periódico Acta Odontológica Scandinavica.

Abstract

Objective: The aim of this study was to evaluate the effect of long-term food-simulating media storage on degradation of restorative materials through roughness measurements. **Material and Methods:** Sixty cylindrical specimens of each material (Filtek Z250, Esthet X, Filtek Flow, Dyract AP and Vitremer) were prepared, stored for 24 h, and polished. The surface roughness analysis was conducted using Surfcorder (SE1700) roughness-measuring instrument. Three traces were recorded on each specimen at three different locations. The specimens were randomly distributed into 5 groups (n=12) according to the storage media: (water, ethanol, Coca-Cola®, citric acid, and lactic acid). Roughness measurements were recorded after 1 week, 1, 3 and 6 months. The storage solutions were montly changed. Data were submitted to ANOVA and Tukey test ($p<0.05$). **Results:** There was no significant increase on roughness means for Filtek Z250, Filtek Flow, and Vitremer over time, regardless the storage media. Significant increase on surface roughness was observed for Esthet X after 1 month of storage in Coca-Cola® and after 3 months of storage in citric acid, and for Dyract AP after 6 months in water. There were no significant differences on surface roughness between resin composites and compomer. Resin-modified glass ionomer showed significant higher surface roughness mean than the other materials. **Conclusion:** Food-simulating media affects the surface roughness of the materials. Biodegradation is material and solution dependent.

Key words: compomer, composite, degradation, long-term, roughness

Introduction

Long-term clinical performance of restorative materials is related to a number of factors, including biodegradation in the oral environment [1]. The chemical degradation can be caused by acid challenges, including those produced by the cariogenic biofilm [2], acidic diet (soft drinks and acidic beverages) [3-6], and salivary enzymes [7,8].

Studies have shown the effect of the chemical environment of the oral cavity on the degradation of resin-based materials [2-8]. The effects are surface softening [2,3,9] and roughening [3,10,11] that can decrease the long-term durability of the restorations. As a consequence, degraded restorations encourage plaque accumulation, which may result in gingival inflammation, superficial staining and secondary caries [12,13].

The chemical degradation is material and solution dependent. The nature of resin matrix, and size, type, and distribution of filler particles, and the resin-filler coupling agent regulated the biodegradation of the resin-based materials [3,10]. Oral fluids can promote disintegration of the silane-coupling agent at the resin-filler interface what leaches the filler particles to the oral environment, reducing the physical properties of the material [13,14]. In addition, organic acids and various food and liquid constituents can soften the resin matrix [2,3,9].

Each solution will mainly degrade one component of the resin-based material. Alcohol solutions act on the matrix. According to Yap et al.[3], Bis-GMA matrix are susceptible to the softening effect of the food simulating liquids. Moreover, the inorganic filler can be damaged by the water and weak intraoral acids, as citric and lactic acids [15].

Considering that it is important not only to compare the performance of different restorative materials but also to estimate their chemical durability on the oral environment, this study aims to evaluate the long-term effect of food-simulating liquids on degradation of resin-based materials through roughness measurements and Scanning Electron Microscopy (SEM) observation. The hypothesis tested is that materials will have different behavior in long-term, depending on the acidic storage solutions.

Material and Methods

Specimen Preparation

Three resin composites (Filtek Z250, Esthet-X, and Filtek Flow), one polyacid-modified resin composite (Dyract AP), and one resin-modified glass ionomer cement (Vitremer) were selected for this study. The technical profiles of the materials are described on Table I.

Table I – Technical profiles and manufacturers of the materials evaluated

Material	**Category**	**Composition***	**Mean filler size (µm)***	**Manufacturer**
Filtek Z250	Hybrid resin composite	Bis-GMA, Bis-EMA, UDMA, Inorganic filler – Zirconia/sílica (60 vol. %); Photoinitiator	0.60	3M Dental Products, St. Paul, MN 55144, USA
Esthet-X	Hybrid resin composite	Bis-GMA, TEGDMA, barium-fluoro-alumino-silicate glass, silica, initiators/stabilizers.	<1 - glass 0.04 - silica	Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil
Filtek Flow	Flowable composite	Bis-GMA, TEGDMA, dimetacrylate polymer, Inorganic filler – Zirconia/sílica (47 vol%); Photoinitiator	0.60	3M Dental Products, St. Paul, MN 55144, USA
Dyract AP	Polyacid-modified resin composite	Cetyllamine hydrofluoride acetone; UDM resin TCB resin; Polymerizable resins; Strontium fluoro-silicate glass (47 vol%); Strontium fluoride; Initiators/stabilizers.	0.80	Dentsply Indústria e Comércio Ltda, Petrópolis, RJ, Brazil
Vitremer	Resin-modified glass-ionomer cement	Powder: fluoraluminosilicate glass, redox catalyst system, pigments Liquid: aqueous solution of a polycarboxylic acid modified with pedant methacrylate groups, Vitrebond copolymer, water, HEMA, photoinitiators. Primer: Vitrebond copolymer, HEMA, ethanol, photoinitiators.	6.25**	3M Dental Products, St. Paul, MN 55144, USA

* as disclosed by the manufacturers; ** according to Gladys *et al.*[19]

Materials were handled according to manufacturer's instructions and inserted in a single increment into a stainless-steel mold (4 mm in diameter x 2 mm thick). A Centrix syringe (Centrix Inc., Shelton, CT 06484, USA) was used to insert Vitremer, whereas syringes supplied by the manufacturers were used for Dyract AP and Filtek Flow, and a metal spatula was used for the other materials.

Sixty specimens of each material were made at 23°C at 50% relative humidity. First, the mold was filled with the material and then a polyester strip (Probem Ltda, Catanduva, SP 15800-000, Brazil) was placed over the mold and the excess of the material was pressure extruded through a glass plate of 2 mm thickness. The restorative materials were photoactivated for the recommended exposure times through the polyester strip using the photocuring unit Elipar Tri-light (ESPE – America Co., Seefeld 82229 – Germany) with power density of 800 mW/cm^2.

After polymerization, specimens were individually stored for 24 h at 37°C at 100% relative humidity. Vitremer specimens were protected using Finishing Gloss, supplied by the manufacturer, to avoid water sorption during the first 24 h. Thereafter, specimens were submitted to standard finishing and polishing procedures, using medium, fine and superfine aluminum oxide abrasive disks, Sof-Lex (3M Dental Products, St Paul, MN 55144, USA) mounted in a low-speed hand piece (Kavo, Joinville, SC, Brazil). Each instrument was applied in a single direction for 15 s. After the finishing and the polishing steps, specimens were flushed with air-water spray and ultrasonically cleaned (Ultrasonic Cleaner, Model USC1400, UNIQUE Ind. e Com. Ltda., São Paulo SP 04709-111, Brazil) in distilled water for 10 minutes to remove polishing debris.

Baseline Surface Roughness Measurements

Each specimen was gently dabbed dry with absorbent paper and the surface roughness analyses conducted using the Surfcorder SE1700 (Kosaka Corp., Tokyo, Japan) surface roughness-measuring instrument equipped with a diamond needle of 2-µm radius. To record roughness measurements, the needle moved at a constant speed of 0.5 mm/second with a load of 0.7 mN. The cut-off value was set at 0.25 mm to maximize filtration of surface waviness. The surface roughness was characterized by the average roughness (Ra) that is the arithmetical average value of all absolute distances of the roughness profile from the centerline within the measuring length. Ra values for each specimen were taken across the diameter over a standard length of 0.25 mm. Three traces were recorded on each specimen at three different locations – parallel, perpendicular, and oblique to the finishing and the polishing scratch directions. The average of these three traces was used as the value for each specimen.

Storage in acidic solutions

Each group of 60 specimens was randomly subdivided into five groups (n=12) and stored at 37°C as follows: Group 1 – distilled water; Group 2 – 0.1M lactic acid; Group 3 – 0.1M citric acid; Group 4 – Coca-Cola®; Group 5 – 50% ethanol-water solution [3]. The materials were immersed into 3 ml of these solutions immediately after the baseline surface roughness measurement and evaluated for surface roughness after one-week, one-, three- and six-month-storage period. The specimens were lightly rinsed in water and gently dabbed dry with absorbent paper before each measurement, which was carried out as described before for the baseline surface roughness measurement. Solutions were montly changed.

The pH values of each storage solution were determined using a pH meter (Orion Model 420A, Analyzer Com. e Ind. Ltda., São Paulo SP 03638-030, Brazil) immediately before the immersion of the specimens and repeated after each roughness measurement. The pH values of the solution were 5.5 for distilled water, 5 for citric acid, 5 for lactic acid, 2.6 for Coca-Cola®, and 7 for 50% ethanol-water solution.

SEM evaluation

Polished specimens and representative specimens of each material on each solution after 6 months storage were subjected to SEM (JEOL- JSM 5600LV, Tokyo, Japan) evaluation at X3000 magnification to determine micro-structural surface alterations.

Statistical Analysis

Analysis of variance with repeated measures (ANOVA) was carried out on roughness data, with restorative materials, storage solution, and time as main effects and, all possible combination of these variables as interaction effects in the ANOVA model, with repeated measures, followed by Tukey-Kramer´s test to carry out pairwise comparisons among these factors at 5% of significance. Statistical analysis was carried out by SAS/LAB (SAS Institute Inc. The SAS system release 8.2, software SAS/LAB. SAS Institute Inc, Cary:NC, 1999).

Results

Results of surface roughness are shown on Table II and Figure 1 and 2.

ANOVA showed significant difference for materials, storage solutions and time, and for the interactions among these factors. As the interactions were significant, a comparison of the behavior under each storage condition and also the effect of different storage regimes on each material over time could be made. Within the material factor, significant difference was found between Vitremer and the other materials. Vitremer showed the most significant rough surface on baseline, maintaining this behavior over time. No significant differences were observed for Ra values of Filtek Z250, Filtek Flow, Esthet X, and Dyract AP on baseline (Table II).

There was no significant increase on surface roughness over time for Filtek Z250, Filtek Flow, and Vitremer, regardless the storage solution. There was significant increase in surface roughness of Esthet X after 1 month of storage in Coca-Cola® and the roughness values continue to significantly increase after 3 and 6 months (Figure 1). Esthet X after 3 months of storage in citric acid showed significant increase on surface roughness, which was maintained after 6 months (Figure 1). Dyract AP showed a significant increase on surface roughness after 6 months of storage in water (Figure 2).

Within the storage solution factor, statistical differences were not observed over time in lactic acid and 50% ethanol-water solution storage, regardless the material. Coca-Cola® led to an increase on Ra values only for Esthet X after 1 month. Citric acid storage increased Ra values only for Esthet X after 3 months, and water storage increased Ra values only for Dyract AP after 6 months.

Table II – Means of roughness (Ra - µm) for restorative materials stored in different solutions over time.

Time	Storage media	Material									
		Filtek Z250		Esthet-X		Filtek Flow		Dyract AP		Vitremer	
Baseline	Water	0.11(0.02)	Aa	0.15(0.04)	Aa	0.19(0.02)	Aa	0.18(0.05)	Aa	0.35(0.13)	Ab
	Lactic acid	0.17(0.05)	Aa	0.17(0.03)	Aa	0.17(0.02)	Aa	0.13(0.03)	Aa	0.36(0.09)	Ab
	Citric acid	0.17(0.07)	Aa	0.18(0.05)	Aa	0.16(0.01)	Aa	0.12(0.03)	Aa	0.34(0.11)	Ab
	Coca-Cola®	0.11(0.04)	Aa	0.14(0.06)	Aa	0.20(0.03)	Aa	0.24(0.10)	Aa	0.39(0.15)	Ab
	50% ethanol	0.17(0.09)	Aa	0.13(0.02)	Aa	0.17(0.02)	Aa	0.22(0.06)	Aa	0.35(0.10)	Ab
1 week	Water	0.15(0.09)	Aa	0.19(0.18)	Aa	0.18(0.02)	Aab	0.14(0.02)	Aa	0.32(0.13)	Ab
	Lactic acid	0.19(0.14)	Aa	0.14(0.02)	Aa	0.17(0.07)	Aa	0.13(0.04)	Aa	0.33(0.11)	Ab
	Citric acid	0.17(0.06)	Aa	0.19(0.06)	Aa	0.16(0.05)	Aa	0.16(0.03)	Aa	0.47(0.15)	Ab
	Coca-Cola®	0.13(0.03)	Aa	0.23(0.10)	Aa	0.20(0.08)	Aa	0.22(0.09)	Aa	0.40(0.13)	Ab
	50% ethanol	0.17(0.05)	Aa	0.22(0.10)	Aa	0.19(0.02)	Aa	0.19(0.07)	Aa	0.41(0.12)	Ab
1 month	Water	0.16(0.06)	Aa	0.21(0.09)	ABab	0.19(0.02)	Aa	0.17(0.07)	Aa	0.35(0.10)	Ab
	Lactic acid	0.20(0.11)	Aa	0.13(0.01)	Aa	0.17(0.03)	Aa	0.18(0.06)	Aa	0.39(0.12)	Ab
	Citric acid	0.17(0.04)	Aa	0.16(0.06)	Aba	0.17(0.03)	Aa	0.17(0.04)	Aa	0.46(0.16)	Ab
	Coca-Cola®	0.14(0.03)	Aa	0.26(0.11)	Bb	0.21(0.02)	Aab	0.20(0.07)	Aab	0.36(0.11)	Ab
	50% ethanol	0.18(0.08)	Aa	0.22(0.07)	ABab	0.20(0.07)	Aa	0.20(0.09)	Aa	0.38(0.11)	Ab
3 months	Water	0.16(0.07)	Aa	0.15(0.07)	Aa	0.22(0.05)	Aab	0.18(0.07)	Aa	0.38(0.16)	Ab
	Lactic acid	0.18(0.03)	Aa	0.17(0.04)	ABa	0.22(0.05)	Aa	0.17(0.05)	Aa	0.59(0.27)	Ab
	Citric acid	0.18(0.03)	Aa	0.31(0.22)	CBa	0.20(0.03)	Aa	0.17(0.04)	Aa	0.50(0.19)	Ab
	Coca-Cola®	0.15(0.06)	Aa	0.34(0.21)	Cb	0.23(0.04)	Aab	0.18(0.05)	Aab	0.56(0.22)	Ac
	50% ethanol	0.19(0.08)	Aa	0.19(0.12)	ABCa	0.20(0.02)	Aa	0.22(0.07)	Aa	0.42(0.14)	Ab
6 months	Water	0.15(0.04)	Aa	0.15(0.06)	Aa	0.17(0.03)	Aab	0.30(0.17)	Abc	0.35(0.16)	Ac
	Lactic acid	0.16(0.04)	Aa	0.23(0.11)	ABa	0.18(0.04)	Aa	0.19(0.04)	Aa	0.57(0.25)	Ab
	Citric acid	0.17(0.05)	Aa	0.32(0.19)	BCab	0.16(0.01)	Aa	0.19(0.07)	Aa	0.48(0.16)	Ab
	Coca-Cola®	0.19(0.10)	Aa	0.40(0.13)	Cb	0.19(0.02)	Aa	0.24(0.10)	Aab	0.40(0.15)	Ab
	50% ethanol	0.23(0.04)	Aab	0.17(0.07)	Aa	0.17(0.01)	Aa	0.32(0.09)	Abc	0.42(0.12)	Ac

Statistical differences are expressed by upper case letters in columns (within each time interval), and by lower case letters in rows ($p<0.05$). Standard deviation is inside the parentheses.

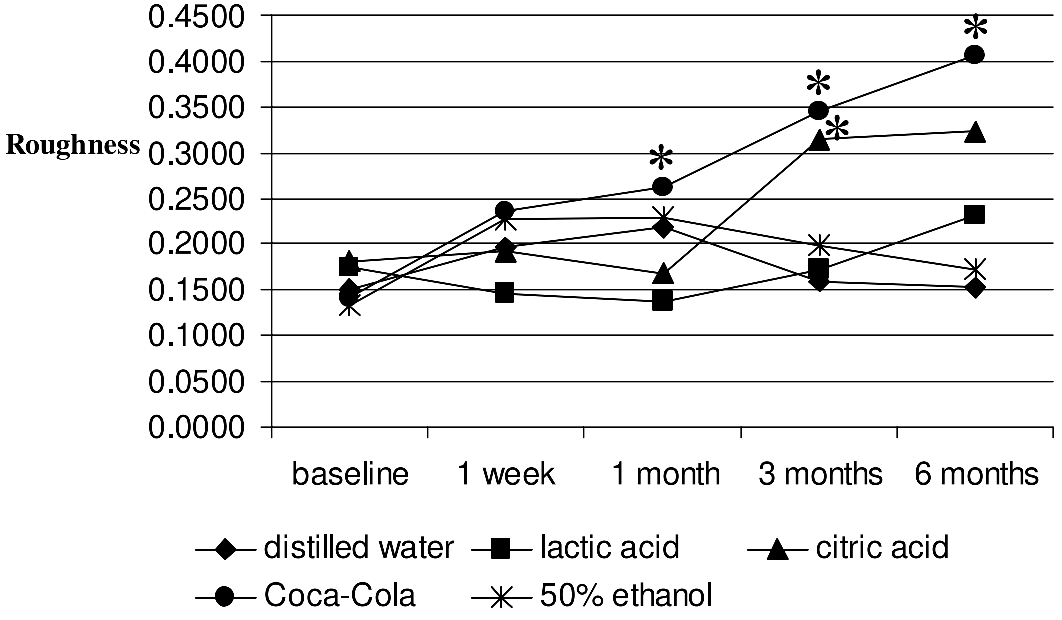

Figure 1 – Means of roughness (Ra - µm) for Esthet X stored in different solutions over time. Statistical differences are expressed by * over the mean value in different periods.

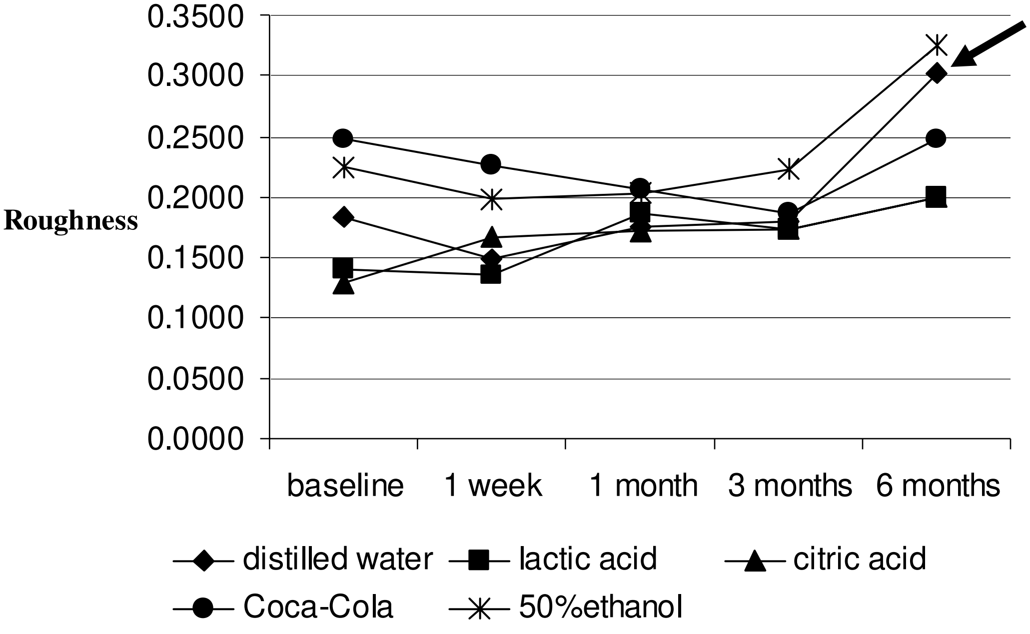

Figure 2 – Means of roughness (Ra - µm) for Dyract AP stored in different solutions over time. Statistical differences are expressed by an arrow over the mean value in different periods.

Figures 3 and 4 show the SEM micrographs of Esthet X and Dyract AP. Micrographs of polished Esthet X and Dyract AP surfaces are shown on Figures 3A and 4A, respectively. Esthet X specimen 6 months stored in citric acid shows that a generalized roughening occurred with formation of pits (filler debonding) on material surface (Figure 3B). Protrusion of filler particles can be observed on Figure 1C that shows Esthet X surface after 6 months in Coca-Cola®. Figure 4B shows Dyract AP surface after 6 months in water, a generalized roughening was observed.

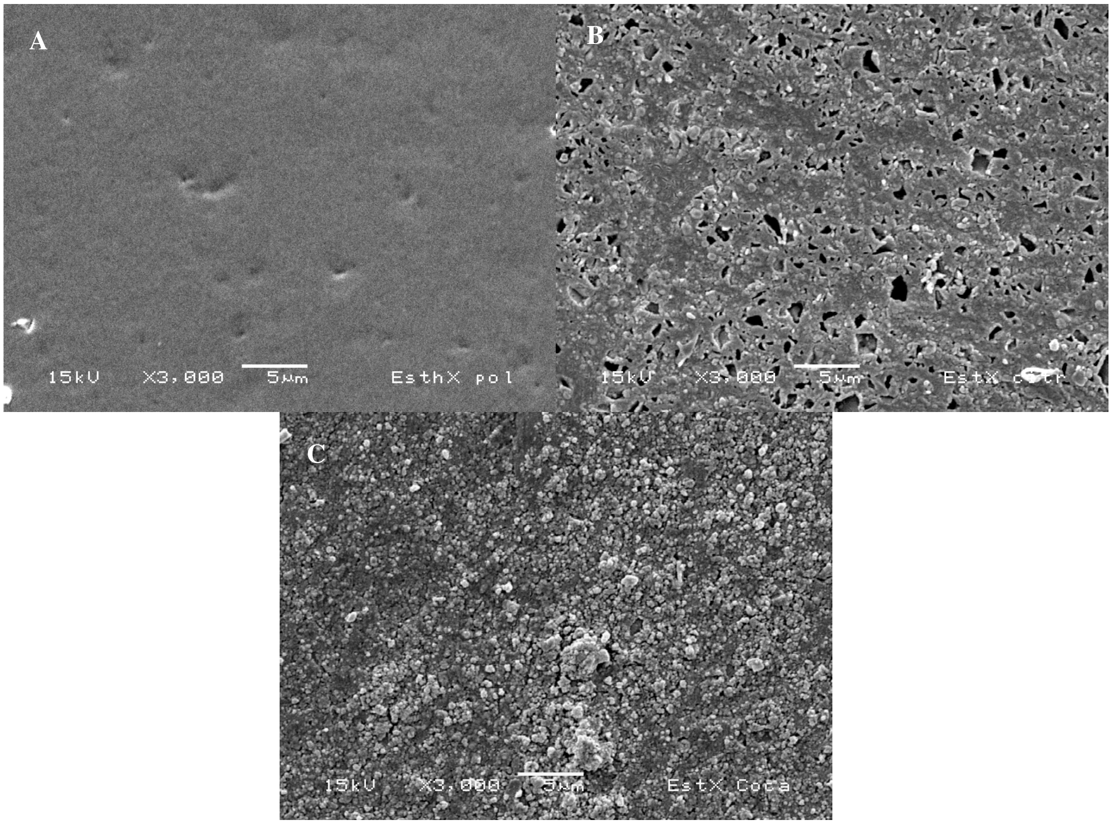

Figure 3 – Scanning Electron Micrograph of Esthet X. (A – polished surface; B – 6 months storage in citric acid; C – 6 months storage in Coca-Cola®).

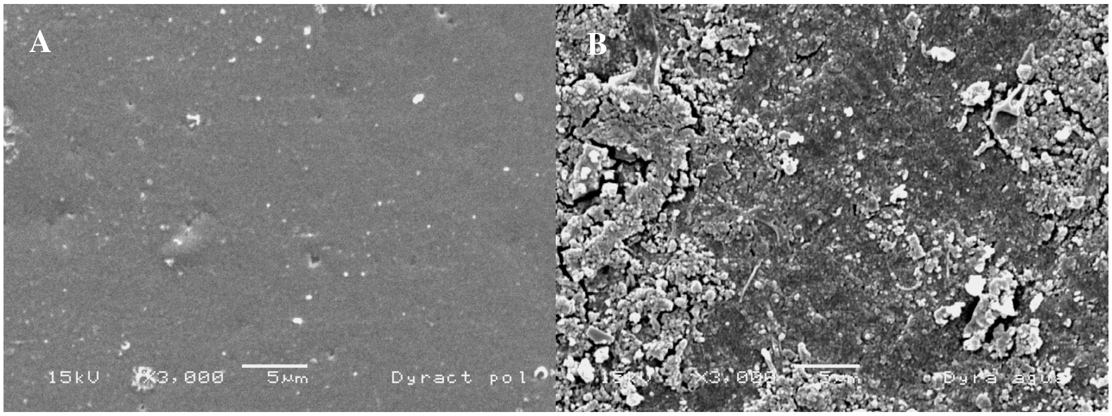

Figure 4 - Scanning Electron Micrograph of Dyract AP. (A – polished surface; B – 6 months storage in distilled water).

Discussion

The roughness of intraoral hard surfaces (teeth or restorative materials) had a major impact on the retention of oral microorganisms, increasing the risk for caries and periodontal inflammation [12]. In addition, a rough surface affects the teeth and restorative material wear[16] and increases the susceptibility to staining [13,17,18].

According to Bollen *et al.* [12], the roughness of all intraoral hard surfaces should approximate a Ra value of 0.2 µm or lower to reduce bacterial retention. In this study, the resin-based materials Filtek Z250, Filtek Flow, Esthet X, and Dyract AP showed initial Ra values lower than, or very close to (Dyract), 0.2 µm (baseline – Table II). These materials showed no significant difference on mean surface roughness, corroborating with Gladys *et al.*[19] and Turssi *et al.*[10]. Vitremer was the roughest material, showing Ra values higher than 0.2 µm in baseline, maintaining a higher roughness over time, regardless the storage solution, which concurs with el-Kalla and Garcia-Godoy [20]. The differences among materials regarding their means of surface roughness on baseline are mainly related to differences in their filler particles size, shape, volume, and distribution [19].

Roughening can be a consequence of the chemical dissolution of resin-based materials by the exposure of the surfaces to chemicals from drinks, food, microorganisms, and saliva [2-8,16]. Some of the food-simulating liquids used to store the restorative materials in this investigation are among those recommended in FDA Guidelines to be used as food simulators. The ethanol-water solution and the citric acid simulate certain beverages, including alcoholics, and vegetables, fruits, candy, and syrup. Distilled water was included to simulate the wet intraoral environment. Lactic acid is the main acid produced by the plaque microorganisms, and is a product of *Lactobacillus* fermentation process in fermented milk beverages. Coca-Cola® was selected because it is the soft drink frequently consumed by the youth population.

In this study, most of the materials were not affected by the food-simulating media storage. However, the long-term storage of resin-based restorative materials upon the acidic media trended toward an increase on surface roughness over time, especially for Esthet X, Dyract AP and Vitremer.

Composite resins Filtek Z250 and Filtek Flow had a similar behavior, not showing a significant increase on roughness over time, regardless the storage solution. This is in agreement with Yap *et al.* [3,4]. Both materials have a similar composition, similar organic matrix and same filler size and type. The major difference between these materials is the filler content, lower in the flowable composite Filtek Flow. But this difference seems to have no influence on the material roughness. The appearance of the specimens of Filtek Z250 and Filtek Flow did not change in any solution after 6 months storage, when viewed with naked eye and by SEM observation. These specimens showed the same polished surface shine.

The analysis of the pH of the solutions that stored these materials did not show any marked change on the pH over time, compared with the pH of recently made solutions. This confirms that these materials were not affected by the different solutions, not leaching their components in the solutions. Karantakis *et al.*[21] demonstrated that composites are resistant to dissolution in acidic conditions.

The zirconia/silica filler particles from Filtek Z250 and Filtek Flow seem to be more inert in the different solutions compared to the barium-fluoro-alumino-silicate glass filler particles from Esthet X. In this study, the composite Esthet X showed a significant increase on surface roughness after 3-month storage in citric acid and after 1-month storage in Coca-Cola® (Figure 3B and 3C). Esthet X specimens showed loss of polished surface shine and the specimens stored in Coca-Cola® showed a slightly brown hue.

It has been established that the corrosive potential of an acidic solution is related to their pH, titratable ability, buffer capacity, and the solution degree of saturation [22]. Coca-Cola® has low titratable ability and low buffer capacity, reducing its erosive potential. However, in this study the pH of this solution was very low (pH ~ 2.5), what is related with the erosive potential [22]. In addition, this soft drink has in its composition an inorganic and strong acid, phosphoric acid. Thus, the association of a low pH, and the presence of a strong inorganic acid could have caused sooner (one month) and more aggressive attack of Esthet X surface by this solution. Figure 1C shows some filler protrusions. This could be related to the resin matrix degradation. In addition, most specimens stored in Coca-Cola® showed a slightly brown hue, more evident in the case of the RMGI group.

Citric and lactic acids have high titratable ability and buffer capacity. However, in this study their baseline pH was around 5.0, which is higher than that of Coca-Cola®. This could be related to the lack of significant effect on surface roughness for most materials tested in this study. The pH of these solutions (citric and lactic acids) was selected based on the pH to produce active caries [23].

The significant increase on Esthet X surface roughness after 3 months storage in citric acid could be related to dissolution of the inorganic fillers, as suggested by McKinney [15]. Figure 3B shows some dark pits, filler lost, on Esthet X surface after 6 months storage in citric acid solution, what is an indicative of filler dissolution. In addition, the hydrolysis of the silane-coupling agent could have taken place, leading to the leaching of these fillers on the acidic solution, increasing the surface roughness of this material.

The compomer Dyract AP had a similar behavior to composites, regarding the surface roughness. This is in agreement with other studies [10,11,19]. Dyract AP surface roughness was not affected by the storage on different acidic solutions over time, except for a significant increase on Ra values after 6 months of storage in water. Dyract AP specimens also showed loss of polished surface shine.

Dyract is anhydride, which could react with water of the storage medium, showing the development of a carboxylate rich surface on their uppermost layer, turning this material less resistant to mechanical forces (wear) [24]. Conversely, in the presence of low pH aqueous environment, water-soluble salts formed as a result of the increased solubility and selective dissolution of filler particles may retard or completely inhibit the carboxylate salt yields, increasing the abrasion resistance of the surface [24]. Thus, when Dyract AP specimens were subjected to an acidic condition the mean of surface roughness was lower than that observed for those immersed in distilled water after 6 months.

Also, the increased surface roughness after 6 months of Dyract AP stored in water could have been caused by a hydrolysis of the silane-coupling agent, or even the plasticizing process of the resin matrix. The SEM photomicrograph of Dyract AP after 6 months storage in water shows a generalized rough surface (Figure 4B). The poorer silanization of the filler particles could increase the potential for filler particle debonding.

The analysis of the pH of the solutions containing Dyract AP showed an increase on pH over time, especially in more acidic solutions, like Coca-Cola®, citric and lactic acids. This increase could be related to the release of the acid-base reaction products (fluoride and other ions) of this material that increases the degree of saturation of the solutions, increasing the pH, and decreasing the chemical degradation of this material. Nicholson *et al.* [25] demonstrated that Dyract AP are able to increase pH of lactic acid solution and this reduction on the pH was accompanied by a decrease on material mass. The more acidic the solution the higher is the fluoride release.

The resin-modified glass ionomer Vitremer had the roughest surface of all the materials. This is related to the highest filler particle size (Table I). However, the roughness of this material was not increased over time in any solution. The same process of increasing pH was observed for Vitremer. There was a marked change on the pH of the solutions, especially the more acidic ones. This increase was greater after 1 week, maintaining the pH over time. The fluoride and/or acid-base reaction products release in the acidic solution increased the saturation of the solution, decreasing the chemical attack of this material [26].

It should be considered that for both RMGI and compomer, the chemical degradation of material was accompanied by an increase in pH of the acidic solution [25,26]. Such buffering effect is likely to be beneficial in protecting restored teeth from the development of secondary caries.

In conclusion, different food-simulating solutions affect the long-term degradation of resin-based materials, depending on the material composition and type of solution. The tested hypothesis must be accepted; most resin-based materials were resistant to chemical degradation over time, except for the composite Esthet X stored in citric acid and in Coca-Cola® and for the compomer Dyract AP stored in water.

Although chemical degradation is an important factor on resin-based material surface characteristics, other aspects must be considered, such as the presence of mechanical forces (brushing and mastication). The association of chemical and mechanical challenges would determine the ultimate surface characteristics of resin-based materials in the oral environment.

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CAPÍTULO 2

In vitro wear of deciduous and permanent enamel – Simultaneous erosion and abrasion[3]

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[3] Manuscrito aceito para publicação no periódico American Journal of Dentistry

In vitro wear of deciduous and permanent enamel – Simultaneous erosion and abrasion

Abstract

Purpose: The aim of this study was to evaluate the wear differences between deciduous and permanent enamel caused by the association of abrasion and erosion. **Material and Methods:** Fragments of forty human teeth (20 deciduous and 20 permanent) were cast in acrylic rings, polished to expose a flat enamel area, and profiled with MTS 3D Profiler. Antagonists were made from deciduous and permanent molars. The specimens were distributed into 4 groups (n=10) according to type of substrate and slurry (neutral and acidic), and cycled 100,000 times in the OHSU oral wear simulator. Specimens were cleaned and re-profiled. Volume loss and maximum depth were determined on the polished specimens. Data were submitted to ANOVA and Tukey's test. The area of the wear facet on the antagonist was measured. **Results:** Deciduous enamel showed more wear than permanent enamel, regardless of the slurry type. The acidic environment increased the wear rate compared to the neutral slurry. The association of abrasion and erosion increased the wear rate for the deciduous teeth only. There was no significant difference among groups for cusp wear and no correlation between cusp wear and the enamel wear.

Clinical Significance

Deciduous teeth are more susceptible to wear than permanent teeth, especially in an acidic environment, and limited exposure to acidic food and drink should be recommended to prevent accelerated tooth wear in children.

INTRODUCTION

Tooth wear is a cumulative multi-factorial process, whose aetiology involves a combination of mechanical forces and chemical dissolution.[1-4] The usual and most frequent types of wear are abrasion, attrition and erosion. Erosion is defined as loss of tooth structure by chemical dissolution without involvement of bacteria.[5] Attrition is related to the tooth surface loss by tooth-to-tooth contact; whereas abrasion involves the presence of an abrasive third body between both antagonizing surfaces.[2,5,6]

The synergistic interplay among erosion, abrasion and attrition is an important factor in understanding the mechanism of tooth wear.[2,4,7] The chemical dissolution caused by acid attack promotes the softening of the enamel surface, making it more susceptible to physical forces (abrasion and attrition).[2,4,7,8]

The etiology of erosion can be extrinsic or intrinsic.[9] Extrinsic etiology can be divided into environmental, diet, medication, and lifestyle.[10] The second type, diet, is the most common cause of external erosion. It was proven that erosion is strongly correlated with frequency and amount of soft drink intake.[11] Consumption of soft drinks has increased dramatically in several countries, especially among children.[11-13]

The most frequently consumed erosive acids are phosphoric together with fruit acid, predominantly citric and malic acid.[14,15] Citric acid is the major organic hydroxy acid found in fruit juices and soft drinks. Beverages have concentrations around 0.3% (w/v) citric acid in ready to drink juices.[15] The erosive effect of dietary acids can be influenced by various factors such as pH, pKa, titratable acidity, temperature, concentration, buffering capacity, and chelating potential.[2,14-19] These factors are related to the acid solution characteristics. But the effect of these acids on the enamel surface can also be related to the type of substrate upon which they are acting.

It has been established that deciduous and permanent teeth have structural and chemical differences.[20,21] The enamel and dentine layers of the deciduous teeth are thinner than those of permanent teeth.[22] Deciduous teeth often have a thicker aprismatic enamel on the surface, and demonstrate a higher degree of enamel porosity,[13] lower degree of mineralization, higher content of carbon dioxide and carbonate in the enamel, as well as a lower content of phosphorous than in its permanent successor.[23,24] These differences in the structure and chemical composition between deciduous and permanent enamel may play a role in the development and progression of erosion and abrasion wear.

Some studies show that deciduous teeth are more susceptible to erosion than permanent teeth.[21,25] But the differences between deciduous and permanent teeth are not totally clear, since other studies do not show significant differences between them in terms of erosion.[13,26,27] Also, the differences in mechanical wear (abrasion and attrition) between deciduous and permanent teeth have not been precisely demonstrated.

No published study has specifically examined the relative susceptibility of deciduous teeth to a simultaneous abrasion and erosion challenge. The aim of this study was to evaluate the wear differences between deciduous and permanent teeth caused by the association of abrasion and erosion, developed at the same time, with the use of the OHSU oral wear simulator. The working hypotheses for this study were that deciduous teeth have a greater wear than permanent teeth, and that the extent of wear can be increased in acidic media for both substrates.

MATERIALS AND METHODS

Specimens preparation

Forty human teeth (20 deciduous and 20 permanent molars) were selected, cleaned and stored in a 0.5% Chloramine T solution for up to 2 months after extraction. Specimens of permanent enamel were derived from caries free, erupted third molars, from patients aged between 18 and 25 years of either gender. This study was conducted after the approval of the Ethical Committee of the School of Dentistry of Oregon Health and Science University (OHSU).

Specimens of deciduous enamel were derived from caries free deciduous first and second molars, from children aged between 9 and 11 years of either gender. At the time of extraction, donors were resident in areas with water fluoridation (about 0.07 ppm of fluoride). Also, all the patients reported the use of fluoride-containing toothpastes.

The roots of the teeth were sectioned 1 mm below the cement-enamel junction [Figure 1 – (a)], and the crowns were sectioned in a mesio-distal direction with a slow speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA), obtaining two teeth fragments from each tooth [Figure 1 – (b)].

The teeth fragments were then cast in acrylic rings using metallographic epoxy (Buehler), wet sanded with 400- and 600-grit silicon carbide paper and polished with 1000-grit aluminium oxide and 1-μm diamond paste, and then placed into water in an ultrasonic cleaner for 5 min, to expose a flat enamel area on the lingual, buccal, or palatine surfaces of the molars [Figure 1 – (c)]. Because deciduous enamel is thinner than permanent enamel, the teeth that presented the most flat enamel surface were selected, i.e., lingual surface of deciduous inferior second molars. Care was taken to remove the minimum amount of tissue, just the amount sufficient to expose an 8 mm length x 2 mm width flat area in both permanent and deciduous enamel. Each specimen was checked by eye, and in a stereomicroscope to ensure that no dentine exposure was present. Those specimens in which dentine had been exposed were discarded.

Next, the polished enamel surfaces were profiled with MTS 3D Profiler (MTS Systems Corporation, Eden Prairie, MN, USA) [Figure 1 – (d)] using Capture software (MTS Systems Corporation, Eden Prairie, MN, USA) to obtain a baseline.

The antagonists were made from extracted human deciduous and permanent molars [Figure 1 – (a)]. Enamel cusps were sectioned with a slow-speed diamond saw [4 enamel fragments – Figure 1 – (b1)] (Isomet, Buehler) and mounted onto the heads of nylon screws using a flowable resin [Figure 1 – (c1)] and next embedded in metallographic epoxy within rubber cups [Figure 1 – (d1)]. The embedded enamel cusps were milled on a lathe into a 12 mm diameter hemispherical shape (including enamel cusp + epoxy resin) using a diamond bur in a custom-pivoting fixture [Figure 1 – (e1)]. The enamel was finished with 600-grit silicon carbide paper and polished with 1000-grit silicon carbide slurry, and then ultrasonically cleaned for 5 min.

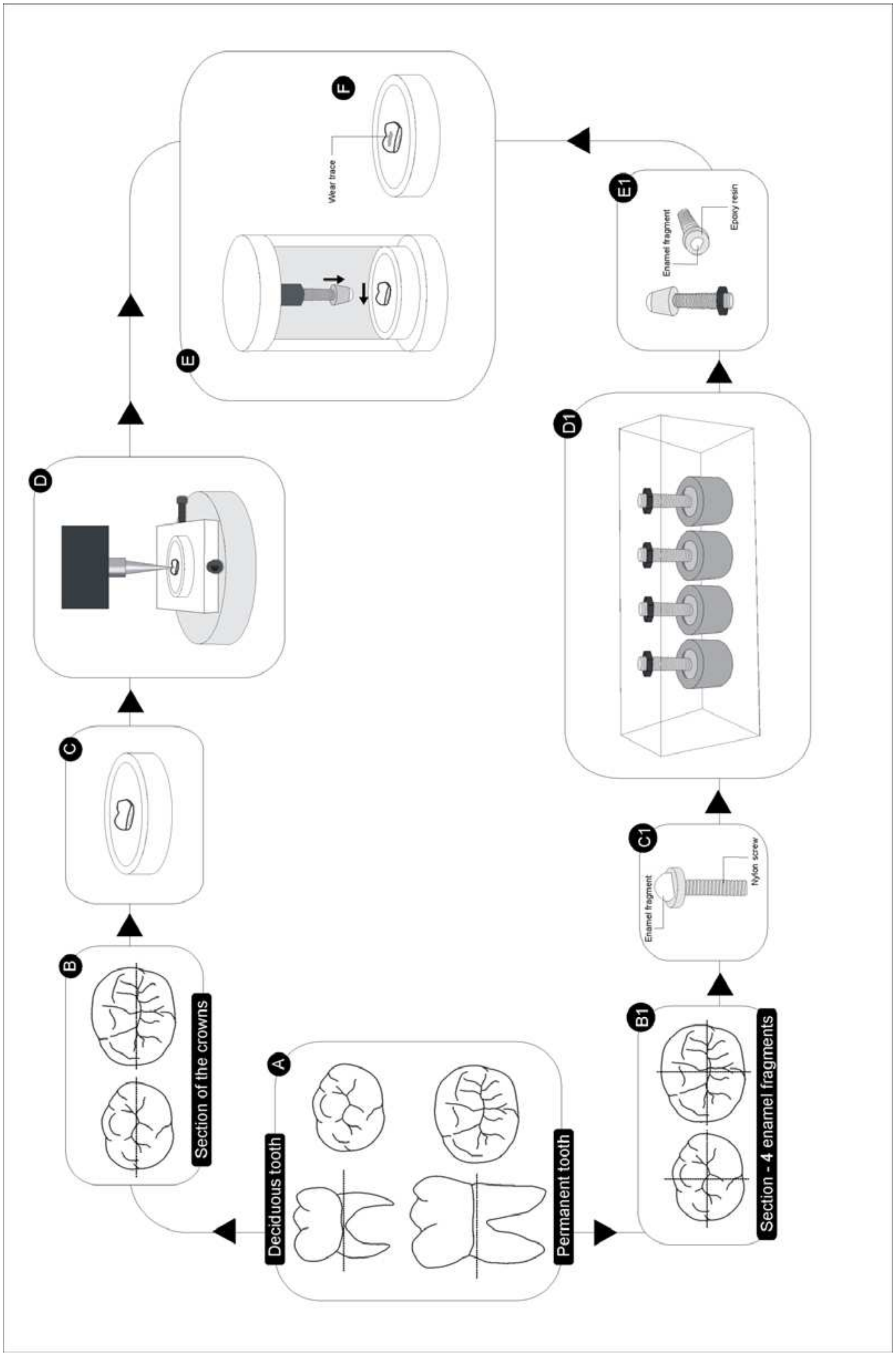

Figure 1 – Diagram of the experimental setup. (a) Selected deciduous and permanent molars; section of the roots or root remains. (b) Section of the crowns of inferior deciduous and permanent molars (mesio-distal direction), obtaining two fragments. (c) Embedded flat enamel surface (~ 8

mm). (d) Specimen profiling before wear (MTS 3D Profiler). (b1) Section of permanent and deciduous superior molars into 4 enamel fragments. (c1) Enamel fragment mounted, using a flowable composite, on the head of a nylon screw. (d1) Mounted enamel fragments secured by embedding in metallographic epoxy inside rubber cups . (e1) Embedded enamel cusps milled on a lathe into a 12 mm diameter (enamel + epoxy resin) hemispherical shape (final aspect). (e) Specimen and enamel cusp prepared for the wear test (100,000 cycles

The abrasive slurry, which served as the third body in the wear simulator, consisted of 6 g of lightly ground poppy seeds, 3 g of polymethylmethacrylate (PMMA) beads and 30 ml of water. To simulate the simultaneous erosion and abrasion challenge, instead of water, a 0.3% citric acid solution (Merck & Co. Inc., Rahway, NJ, USA) was used to produce the slurry. The citric acid solution was adjusted to a pH of 3.2 using 1 N NaOH. The pH of the solution was measured using a pH electrode connected to a pH meter (model 710A, Orion, Boston, MA, USA).

The specimens were then distributed into 4 groups (n=10) according to the type of substrate (permanent or deciduous teeth) and the type of slurry (neutral – water and acidic - citric acid solution).

Wear Procedure

The new version of the OHSU oral wear simulator (Prototech, Portland, OR, USA) was used in this study. The basic mechanism for producing wear with this device is similar to the original instrument described previously.[28] Briefly, an enamel-tipped stylus is brought into contact with the specimen and loaded to 20 N in the presence of mildly abrasive slurry. The stylus is driven 8 mm horizontally across the surface of the specimen. At the end of the pass, the vertical load is increased to 80 N and released. The resulting wear pattern contains two distinct regions corresponding to the abrasion and attrition events. But, in this study, the same load of 20N was applied in both abrasion and attrition regions.

The specimens were placed into the wear machine chambers and 3 ml of well-stirred, fresh slurry was added to each of the chambers for each experiment. The specimens (n=10) were cycled for 100,000 times at 1.9 Hz [Figure 1 – (e)].

Following the wear challenge, the specimens were ultrasonically cleaned in water, and then re-profiled in the MTS Profiler. Using Ansur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA) the digitized surface maps were compared. Volume loss in mm^3 and maximum depth in µm were determined by subtracting differences observed between the baseline and worn data sets. A two-way ANOVA and Tukey's test ($p < 0.05$) were used for data analysis.

The area of the wear facet on the antagonist was painted with a pencil for better visualization, imaged using a stereomicroscope (Nikon SMZ-10, Japan) with a digital camera (Nikon Coolpix 990, Japan), and measured with commercial software (Scion Image for Windows, Scion Corporation, Frederick, MD, USA). The cusp wear values were compared by ANOVA and Tukey test ($p < 0.05$).

The surface texture of the wear patterns was examined by sputter-coating representative worn specimens with a 40 nm film of Au-Pd and imaging in the scanning electron microscope (6400, JEOL, Tokyo, Japan).

RESULTS

The values of volume loss (mm^3) and maximal depth (µm) are shown in Table 1.

Table 1 – Means of tooth wear expressed by volume loss and Maximal Depth.

Substrate	Volume loss (mm^3)		Max Depth (µm)	
	Neutral	Acidic	Neutral	Acidic
Permanent	0.016 (0.010) Aa	0.023 (0.022) Aa	21.14 (11.86) Aa	26.98 (19.52) Aa
Deciduous	0.027 (0.014) Ba	0.057 (0.028) Bb	28.69 (16.48) Aa	39.11 (23.04) Aa

Statistical differences are expressed by upper case letters in columns, and by lower case letters in rows ($p<0.05$). Standard Deviation is inside the parenthesis.

The ANOVA test for volume loss detected a significant difference between substrates (deciduous and permanent teeth) and between type of slurry (neutral or acidic), but no significant interaction between these factors.

Regarding the factor substrate, deciduous enamel showed more wear than the permanent enamel, regardless of the slurry type used. Concerning the factor type of slurry, wear was increased in the acidic environment compared to that in the neutral slurry. The association of abrasion and erosion only increased the wear rate for the deciduous teeth. There was no significant difference between neutral and acidic conditions for the permanent teeth.

The ANOVA test for maximal depth did not show any significant difference for either of the factors ($p>0.05$).

The wear of the enamel antagonists for all groups is shown in Figure 2. No statistically significant difference was found among the groups ($p>0.05$). There was no correlation between cusp wear and the wear of the samples.

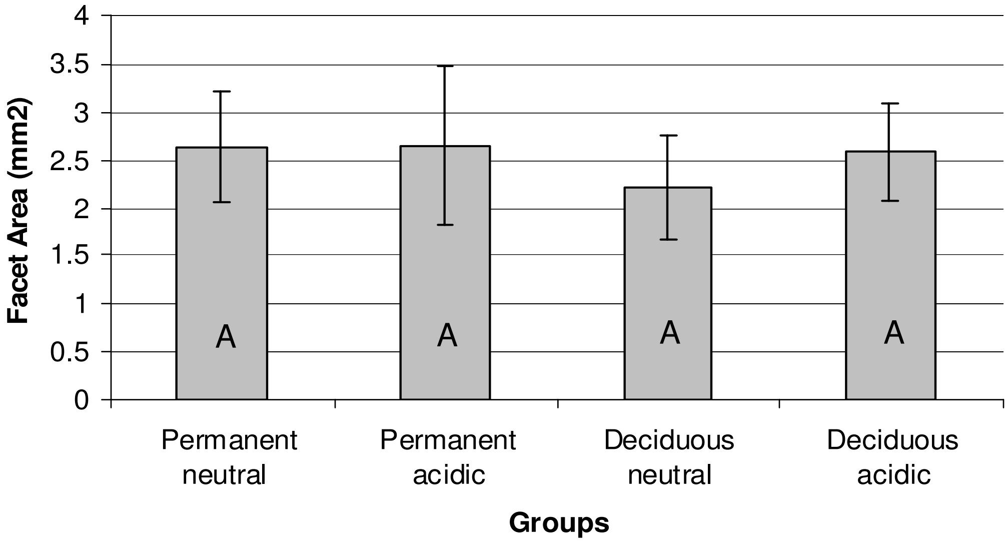

Figure 2 - Area of the wear facet (mm^2) formed on the enamel antagonist worn against the enamel surface. There were no differences between the groups. (Statistical differences are expressed by upper case letters inside the bars ($p<0.05$). Standard Deviations are over the bars).

The SEM micrographs for deciduous and permanent groups are shown in Figures 3 and 4. Differences between neutral and citric acid worn samples were only observed in non-wear regions (Figure 3a and 3d; and Figure 4a and 4d), where a slight demineralization of the enamel surface was evident. Similar patterns for both neutral and citric acid slurry were seen in the wear region, independent of the substrate (deciduous or permanent teeth), except for cusp wear (Figures 3c and 3f), where deciduous cusps (Figure 3c) showed fractures in the enamel, not observed on permanent cusps (Figure 4f).

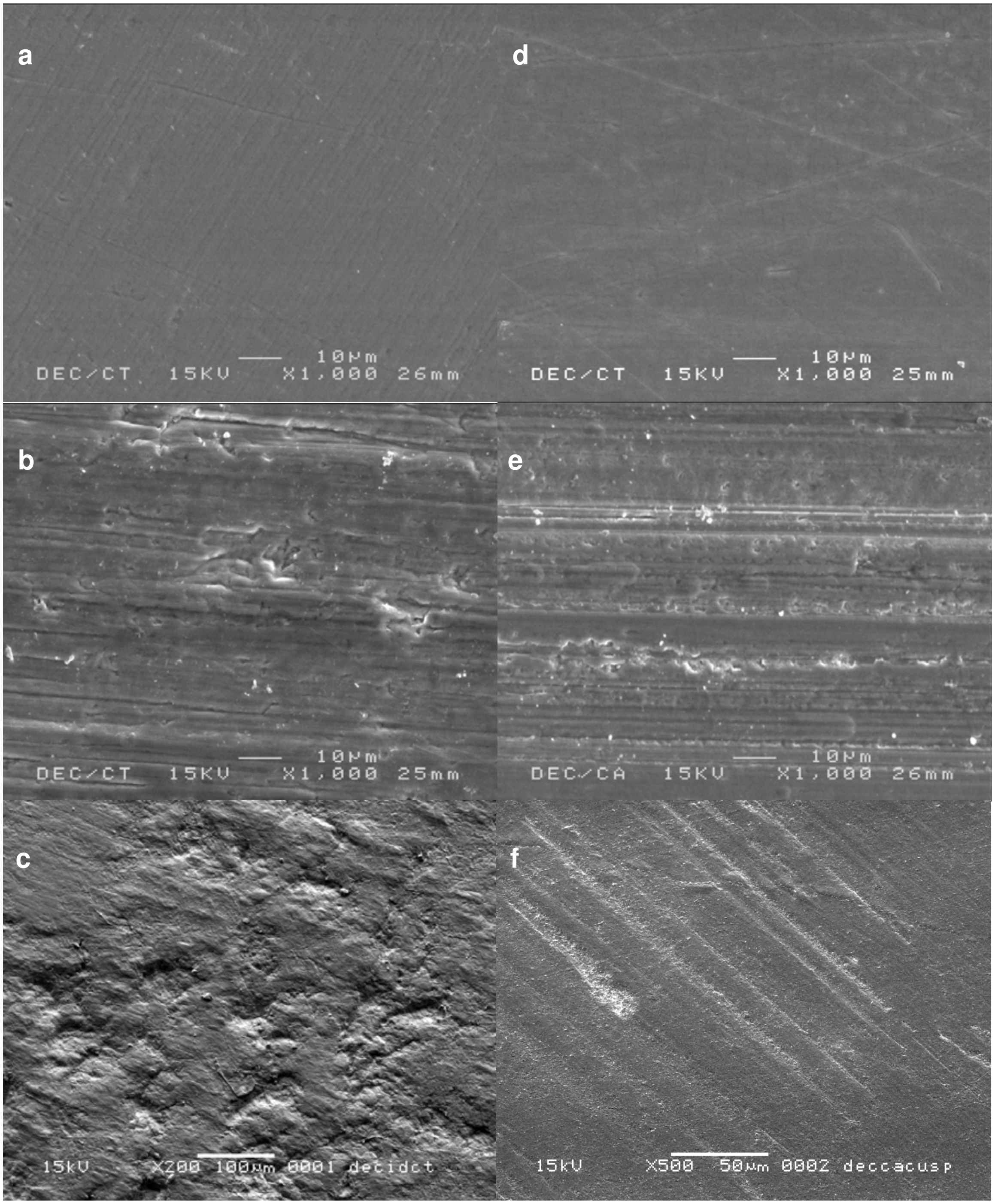

Figure 3 - SEM micrograph of deciduous teeth, demonstrating: a – non-wear region neutral; b – wear trace neutral; c – cusp wear neutral; d – non-wear region citric acid; e – wear trace citric acid; f – cusp wear citric acid.

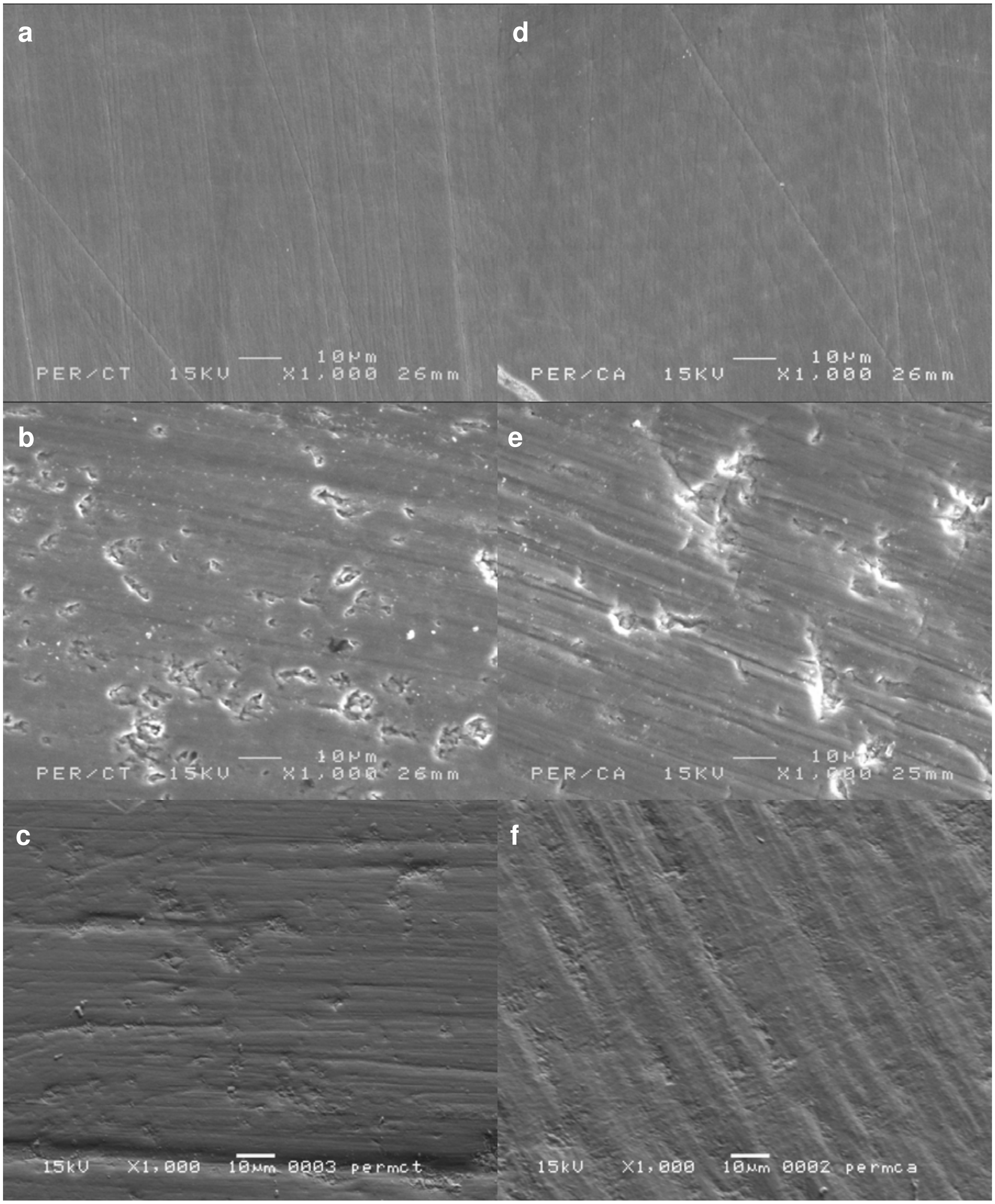

Figure 4 - SEM micrograph of permanent teeth, demonstrating: a – non-wear region neutral; b – wear trace neutral; c – cusp wear neutral; d – non-wear region citric acid; e – wear trace citric acid; f – cusp wear citric acid.

DISCUSSION

Tooth wear is a common finding in the deciduous dentition and it is part of the physiological process of maturation and transition to the permanent dentition. Knight *et al.*[29] found that tooth wear in deciduous dentition was significantly related to subsequent tooth wear in adults, suggesting a common aetiology. Thus, identifying wear in children may help to prevent it later in life.[30]

In this study, deciduous teeth showed more wear than permanent teeth, which is in agreement with Nelson *et al.*[22] who also found higher wear rates in deciduous enamel. The structural and chemical composition of deciduous teeth make them more susceptible to the physical forces induced during the wear test (abrasion in this case), especially when they were submitted to erosion and abrasion challenges at the same time. [20,21,23,24]

The wear in this study was greater in an acidic environment (citric acid solution) than in the neutral (water) groups (only for deciduous enamel). The 0.3% citric acid solution with pH 3.2 was chosen because it has commonly been used in similar studies [14,15] and is often an ingredient of soft drinks.

Amaechi *et al.*[21] suggested that erosion in a citric acid solution involves two processes; including the dissolution of the hydroxyapatite with formation of calcium citrate, and the chelating action of the citric acid which withdraws calcium ions from the acid/saliva mixture in contact with the enamel mineral, resulting in increased dissolution of the enamel. Thus, the erosion process produces a softening of the enamel surface and slight subsurface mineral loss. This process is diffusion limited and can be reversed by remineralization if the softened enamel is not subsequently submitted to physical forces during this period.[2,8,31]

In this study, the simultaneous challenge of erosion and abrasion increased the wear of enamel, mainly because the superficial enamel was abraded away as soon as it reached a critical degree of softening, allowing contact of the underlying sound enamel with the acid solution.[8] This process would be continuous until the dentin surface was exposed. However, this did not happen in this study due to a number of factors, including the limited number of wear cycles and changes in the pH and consistency of the slurry during the test.

It has been shown that the erosion process is related, among other factors, to the pH of the solution.[15-17, 32] Meurman & Ten Cate[33] stated that any solution with a pH value lower than 5.5 may cause erosion. However Hughes *et al.*[17] showed that no erosion was found at pH 4.4. West *et al.*[15] found minimal erosion with acidic drinks whose pH exceeded 3.5.

The pH of the solution in this study was measured before (pH ~ 3.2) and after mixing with the seeds and PMMA (pH ~ 3.8), and after the wear test (pH ~ 4.3). As the enamel is eroded by the acid, calcium and phosphate are released into the acid solution. This increase in the calcium and phosphate of the solution produces an increase of the degree of saturation, and consequently, increasing the pH.[16,17] The overall low erosion observed in the samples, and the lack of difference between the neutral and acidic environment on the permanent teeth, could be related to the saturation of the solution that both increases the pH of the solution during the test and changes in the ion balance for continued dissolution. Therefore, not only does the pH have to be taken into consideration, but also the amount of mineral dissolved by the erosive acid solution, which has been shown to be associated with the type of the acid, the titratable amount of the acid, exposure time, temperature, and concentration of the acid.[21,32] This would be consistent with previous studies that showed lower erosion at pH values of 3.5 and 4.5 than that found in lower pH.[15,17] Also, the slurry could offer some protection to the enamel surface, reducing the exposure of the surface to the acid, and making the erosion less severe.

Hannig *et al.*[32] demonstrated that during a short-term exposure of enamel to acidic environments, erosive effects mainly depended on pH and type of acid, but not on amount of titratable acid. They observed a linear rate of phosphate dissolution in the citric acid solution in a time and pH dependent manner. The phosphate release was lower in the solution with pH 3.0 than in the solution with pH 2.0. However, they could not measure the calcium release in the citric acid solution because of the capacity of citric acid to form chelating complexes with calcium. The chelating processes are influenced by pH and tend to increase as pH rises since the metal ions compete directly with hydrogen ions.

The SEM micrographs for the deciduous teeth demonstrated a similar wear pattern for both neutral and acidic conditions, showing a rough, scratched surface (Figure 3). Apparently, the wear process followed the same pattern in both groups (neutral and citric acid), but to a slightly increased extent in the acidic environment. This may be explained by a faster demineralization of the deciduous teeth in the citric acid solution, especially during the beginning stages of the test before the pH began to rise.

The SEM micrographs of the permanent teeth (Figure 4) demonstrated that there was no difference in the wear pattern under both neutral and acidic conditions. In both cases, the wear surface demonstrated a scratched surface with some fractures in the enamel, indicating an abrasive wear pattern with some regions of fatigue wear (fractured enamel), caused by subsurface cracking, commonly found in hard tissues.[6]

Deciduous teeth presented a rough surface with groves running parallel to the direction of the wear movement. In contrast, fine scratches with some delamination regions (cracks in the enamel), characterizing fatigue wear, where evident on the permanent teeth. This evidence supports the fact that deciduous teeth are softer and wear to a greater extent than permanent teeth. The surface hardness of both substrates before and after exposure to erosion challenge were measured to confirm this, and the following results were obtained using Knoop Indenter and 100 g load after storing the teeth in citric acid for 7 h: deciduous enamel (baseline - 266.14±52.88 KHN; after 7 h - 66.65±10.20 KHN) showed lower hardness than permanent enamel (baseline - 349.98±33.07 KHN; after 7 h - 64.76±9.54 KHN) in the baseline, decreasing the hardness values over time, until both substrates reach the same level (after 7 h).

Despite the lack of difference in the wear pattern for both deciduous and permanent teeth under different conditions, subtle differences were observed in the non-wear regions, where a slight demineralization took place, exposing the enamel prisms. This was evidence that the citric acid had a significant effect on the surface of both substrates (deciduous and permanent).

No significant differences were found between the cusp wear values for the different groups. Also, no correlation was found between the cusp and the sample wear ($p>0.05$). The lack of difference in the cusp wear could be due to structural causes, lack of homogeneity in natural enamel, and variations inherent in the measuring technique. The SEM micrographs showed no difference in the cusp wear pattern for permanent teeth in both conditions (neutral or acidic). The cusp contained some fine scratches, characterizing a three-body type of wear (Figure 4c and 4f). The only difference noticed in the micrographs was for the deciduous teeth neutral group (Figure 3c) that presented evidence for a fatigue type of wear, with the presence of some fractured enamel.

The statistical differences presented in this study were only observed when the volume loss was considered. No statistical differences among the groups could be found when the maximum depth was analysed. Thus, the maximum depth was not a good discriminator of the total wear of the teeth in this study.

The hypotheses for this study could not be totally rejected. Deciduous teeth wore to a greater extent than permanent teeth; and the extent of wear was increased in acidic media, but only for the deciduous teeth.

Based on the results of this study, wear damage (abrasive and erosive) in early childhood may compromise the dentition and require expensive and extensive restoration therapy in the future. Early diagnosis and adequate preventive measures, such as reducing acid exposure by decreasing the frequency of ingestion of potentially harmful drinks and foodstuffs with low pH, are therefore very important.

It could be concluded that deciduous teeth were more susceptible to wear than permanent teeth under both conditions (neutral and acidic), when volume loss was considered. In addition, the association of erosion and abrasion increased the extent of wear (volume) for the deciduous teeth. The wear evaluation based on maximum depth was not a good discriminator of the wear behaviour of the two types of teeth.

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CAPÍTULO 3

In vitro wear of resin-based materials – Simultaneous corrosive and abrasive wear[4]

Corrosive and abrasive wear of restorative materials

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[4] Manuscrito publicado no periódico Journal Biomedical Material Research. Part B: Applied Biomaterials v. 78, n. 1, p. 105-14, 2006.

ABSTRACT

The aim of this study was to evaluate the wear of resin-based materials caused by the association of abrasive and corrosive processes. Twenty specimens were prepared for each material, cast in epoxy in acrylic rings, polished, and profiled with an MTS 3D Profiler. Antagonists were made from deciduous molars. Specimens were distributed into 8 groups (n=10) according to the material (Filtek Supreme, Point 4, Dyract AP, and Fuji II LC) and the type of slurry (neutral and acidic), and then cycled 100K times in the OHSU oral wear simulator. The specimens were cleaned and re-profiled. Volume loss and maximum depth were determined. ANOVA and Tukey's test were used for data analysis ($p<0.05$). The area of the wear facet on the antagonist was also measured. Composites displayed less wear than the compomer and the resin-modified glass ionomer (RMGI). Significant differences also were found for cusp wear, with a significant positive correlation shown between cusp and material wear. The acidic slurry significantly increased the wear of the materials compared to the neutral slurry. Exposure to acidic slurry accelerated the wear of resin-based materials.

Keywords: Wear, enamel, resin-based materials, corrosive wear, abrasive wear.

INTRODUCTION

Wear can be defined as the consequence of the interaction between surfaces, moving in contact, that results in the gradual removal of material.[1] This process is cumulative and multi-factorial, and its etiology involves a combination of mechanical forces and chemical dissolution.[2-4] The most frequently observed wear processes in dentistry are abrasive, fatigue and corrosive.[1,5] The synergistic interplay among these types of wear phenomena is the most important factor in understanding the mechanism of tooth and material wear.[1,4,5]

Abrasive wear occurs when hard asperities plough into softer surfaces.[1] This type of wear can be distinguished by two different processes, abrasion and attrition. Attrition is related to the material or tooth surface loss resulting from direct contact between both surfaces; whereas abrasion involves the presence of an abrasive third body acting between both antagonist surfaces.[6] Fatigue wear is caused by subsurface cracks that nucleate and propagate as a result of repeated load cycles. Corrosive wear is related to a chemical reaction that produces a surface layer that can be scraped away through contact with the antagonist.[4,5]

The chemical dissolution occurring in corrosive wear can be caused by the exposure of the surfaces (material or tooth) to chemicals from drinks, food, microorganisms, and saliva.[2,3,5,7] On exposure to theses chemicals, resin-based materials can undergo softening[8-10] and roughening,[11] making their surfaces more susceptible to the physical forces occurring during abrasion and attrition.

The wear of resin-based materials can be influenced by various factors, such as applied load,[12] aging, degree of polymerization, type of organic matrix, type and content of filler particles,[13-16] and environmental conditions (pH).[2,3,17,18] The diet is the most common external resource of acids that can cause corrosive wear in the oral cavity. Consumption of soft drinks has increased dramatically in several countries, especially among children.[19,20] The most frequently consumed acids are phosphoric acid together with fruit acids, predominantly citric and malic acid.[21,22] Citric acid is the major organic hydroxy acid found in fruit juices and soft drinks.[22]

The same fundamental processes of wear are active on all dental structures, including restorative materials and the natural teeth. The study of dental materials must therefore consider not only the wear resistance of the materials, but also the likely effect of the materials on the opposing teeth.[1] A natural and continuous changing relationship between the form and function is observed in deciduous dentition that shows physiological wear. Restorative materials for use in children should undergo similar wear as these teeth. However, resin composites have shown a higher wear resistance than deciduous teeth. The differential wear of deciduous teeth and restorative materials may have significant deleterious effects on the biologic, functional, and esthetic conditions of the masticatory system.[4] The understanding of the wear process is therefore important to ensure that the outcome of clinical dental procedures is as similar as possible to the physiological condition.

The aim of this study was to evaluate in vitro the wear differences between resin-based materials caused by the simultaneous association of abrasive and corrosive wear processes in the OHSU oral wear simulator, and to correlate the material and the deciduous enamel cusp wear. The first hypothesis for this study was that the glass ionomer and compomer materials would have greater wear than the composites, and the second hypothesis was that the extent of wear would be increased in an acidic environment for the least acid resistant materials, i.e. glass ionomers.

MATERIALS AND METHODS

Materials

Four direct restorative materials, one resin-modified glass ionomer, one compomer, one nanofilled resin composite, and one hybrid resin composite were evaluated in this study. Their brand names, type, composition, mean filler size, manufacturers, and batch numbers are listed in Table 1.

Table 1 – Description of the restorative materials used in the present study.

Materials	Category	Composition*	Mean particle size (µm)*	Filler load (vol%)*	Manufacturers and Batch No.
Fuji II LC	Resin modified glass ionomer	Aluminum-fluoro-silicate glass, HEMA, tartaric acid, polyacrylic acid, water.	1.80	Non-manufacturer offered	GC Corp., Tokyo, Japan Batch # 0403227
Dyract AP	Compomer	Cetylamine hydrofluoride acetone; UDM resin TCB resin; polymerizable resins; strontium-fluoro-silicate glass; strontium fluoride; Initiators/stabilizers.	0.80	47	Dentsply DeTrey, GmbH, Konstanz, Germany Batch # 0402000773
Filtek Supreme	Nanofilled resin composite	Nanosilica and zirconia/silica nanoclusters, UDMA, Bis-EMA, TEGDMA	Nanoclusters 0.6 to 1.4 nanosilica 20 nm	59.9	3M/ESPE, St. Paul, MN, USA Batch # 20040625 3910A2D
Point 4	Hybrid resin composite	Barium alumino silicate glass, Bis-GMA, TEGDMA, EBADM	0.4	57.2	Kerr Corporation, Orange, CA, USA Batch # 207A55

* As disclosed by the manufacturers.

Specimen Preparation

Twenty bar-shaped specimens (12 mm long x 5 mm wide x 2.5 mm thick) were formed from each material in a stainless steel mold. The top and bottom of each bar were covered with clear matrix strips. Each bar was cured in a light-curing unit (Triad II Dentsply, York, PA, USA) for 40 seconds each from both the top and the bottom surfaces. All specimens were aged in deionized water for 24 h at 37^{o}C. The specimens were cast in acrylic rings using metallographic epoxy (Buehler, Lake Bluff, IL, USA), wet sanded with 400- and 600-grit silicon carbide paper and polished with 1000-grit aluminum oxide and 1-µm diamond paste, and then placed into water in an ultrasonic cleaner for 5 min.

Next, the specimens were profiled at 80 µm intervals with a carbide stylus (50 µm diameter) moving at 1 mm/s on a MTS 3D Profiler (MTS Systems Corporation, Eden Prairie, MN, USA) using Capture software (MTS Systems Corporation, Eden Prairie, MN, USA) to obtain baseline data.

The wear antagonists were made from extracted human deciduous molars. Enamel cusps were sectioned with a slow-speed diamond saw (Isomet, Buehler) and mounted onto the heads of nylon screws using a flowable composite, and then embedded using metallographic epoxy. The embedded cusps were milled on a lathe into an approximately 12 mm diameter hemispherical shape using a diamond bur in a custom-pivoting fixture. The enamel was finished with 600-grit silicon carbide paper and polished with 1000-grit silicon carbide slurry, and then ultrasonically cleaned for 5 min.

The abrasive slurry that served as the third body in the wear simulator, consisted of 6 g of lightly ground poppy seeds, 3 g of polymethylmethacrylate (PMMA) beads and 30 ml of water. To simulate the simultaneous corrosion and abrasion challenge, a 0.3% citric acid solution (Merck & Co. Inc., Rahway, NJ, USA) was used to produce the slurry instead of water. The citric acid solution was adjusted to a pH of 3.2 using 1 N NaOH. The pH of the solution was measured using a pH electrode connected to a pH meter (model 710A, Orion, Boston, MA, USA).

The specimens were then distributed into 8 groups (n=10) according to the four materials tested and the two types of slurry (neutral - water or acidic – citric acid solution).

Wear Procedure

The new version of the OHSU oral wear simulator (Prototech, Portland, OR, USA) was used in this study. The basic mechanism for producing wear with this device is similar to the original instrument described previously.[6] Briefly, an enamel-tipped stylus is brought into contact with the specimen and loaded to 20 (±2) N in the presence of mildly abrasive slurry. The stylus is driven approximately 8 mm horizontally across the surface of the specimen. At the end of the pass, the vertical load is increased to 80 (±5) N and released. The resulting wear pattern contains two distinct regions corresponding to the abrasion and attrition events.

The specimens were placed into the chambers of the wear machine and 3 ml of well-stirred, fresh slurry was added to each of the chambers for each experiment. The specimens were cycled 100,000 times at 1.9 Hz.

Following the wear challenge, the specimens were ultrasonically cleaned in water and then re-profiled in the MTS Profiler. Using Ansur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA) the digitized surface maps were compared. Volume loss in mm^3 and maximum depth in µm were determined by subtracting the baseline and worn data sets. A two-way ANOVA and Tukey's test ($p < 0.05$) were used for data analysis.

The area of the wear facet on the antagonist was painted with a pencil for better visualization, imaged using a stereomicroscope (Nikon SMZ-10, Japan) with a digital camera (Nikon Coolpix 990, Japan), and measured with commercial software (Scion Image for Windows, Scion Corporation, Frederick, MD, USA). The cusp wear values were compared by ANOVA and Tukey's test ($p < 0.05$).

The surface texture of the wear patterns was examined by sputter-coating representative worn specimens with a 40 nm film of Au-Pd and imaging in a scanning electron microscope (6400, JEOL, Tokyo, Japan).

RESULTS

The values of volume loss (mm^3) and maximum depth (µm) are shown in Table 2. The ANOVA test for volume loss and for maximum depth detected a significant difference among materials and between type of slurry (neutral or acidic), with no significant interaction between these factors.

Table 2 – Means of material wear expressed by Volume loss (mm^3) and Maximal Depth (μm).

Material	Volume loss (mm^3)		Max Depth (μm)	
	Neutral	Acidic	Neutral	Acidic
Filtek Supreme	0.140 (0.07) Aa	0.163 (0.09) Aa	58.69 (15.54) Aa	65.00 (19.69) Aa
Point 4	0.182 (0.08) Aa	0.247 (0.14) Aa	68.59 (15.00) Aa	85.31 (35.34) Aa
Dyract AP	0.843 (0.36) Ba	1.113 (0.44) Ba	156.62 (45.43) Ba	197.75 (50.09) Ba
Fuji II LC	3.019 (0.83) Ca	3.284 (0.61) Ca	366.32 (107.34) Ca	336.44 (51.72) Ca
Total	1.045 (1.27) a	1.201 (1.33) b	165.05 (140.25) a	171.12 (116.36) a

Statistical differences are expressed by upper case letters in columns, and by lower case letters in rows ($p<0.05$). Standard Deviation is inside the parenthesis**.**

The composites (Filtek Supreme and Point 4) showed less wear than the compomer (Dyract AP) and the resin-modified glass ionomer (Fuji II LC), regardless of the slurry type used. The compomer presented intermediate wear resistance, showing more wear than the composites but less wear than RMGI (Fuji II LC). The RMGI showed the most wear, regardless of the environmental condition.

According to the ANOVA, the slurry was significant with the wear being greater in the acidic environment (citric acid) compared to that in the neutral environment, regardless of the material. However, there was no significant difference between the neutral and acidic conditions for any of the materials when individual comparisons were made with the Tukey's test.

There were significant differences in the wear of the enamel antagonists among the groups (Figure 1). The composites (Supreme/neutral (S); Supreme/citric acid (SCA); Point 4/neutral (P); and Point 4/citric acid (PCA)) showed the lowest cusp wear, differing from the other groups. The Dyract/neutral group (D) showed the most wear of all the groups, not differing from Fuji II LC/neutral. Fuji II LC/neutral (F) did not differ from Dyract/neutral

(D), Dyract/citric acid (DCA), and Fuji II LC/citric acid (FCA). There was a weak (R=0.56) but significant correlation between wear on the antagonist cusp and wear of the material samples, with higher material wear relating to higher cusp wear.

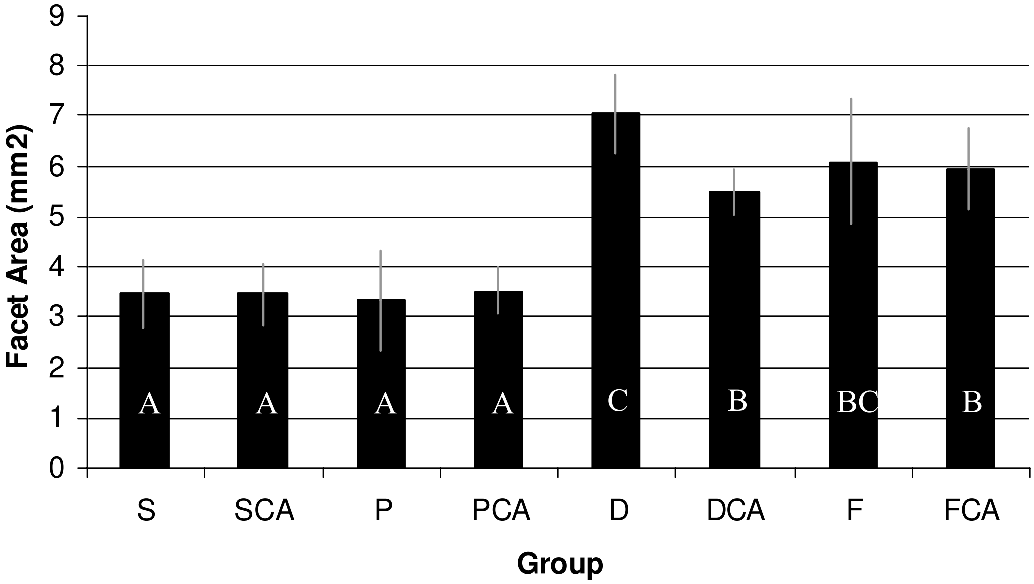

Figure 1 - Area of the wear facet (mm^2) formed on the enamel antagonist worn against the material surface (Statistical differences are expressed by upper case letters inside the bars ($p<0.05$). Standard Deviations are over the bars).

The SEM micrographs (Figures 2 to 5) revealed differences in the material and cusp wear pattern for the different materials. The wear traces of the nanofill resin composite, Filtek Supreme, showed only slightly roughened surfaces in the abrasion region, with no major differences apparent between the abrasion and attrition regions in the acidic slurry (Figure 2D and 2E). However, in the neutral slurry, the attrition region for the Filtek Supreme showed a rougher surface with some evidence of filler delamination that may be more typical of fatigue wear (Figure 2B).

The wear of the composite Point 4 in the abrasion region using the neutral slurry showed many scratches with minimal evidence of surface pitting (Figure 3A). The abrasion region produced in the acidic slurry showed less defined scratches, but more evidence of surface pitting (Figure 3D). In contrast, in the attrition zone, the heightened load generated severe fatigue-like pitting in both slurries (Figures 3B and 3E). The attrition wear surface from the acid slurry showed evidence of large particles (40-50 µm) that appeared to be only loosely attached to the surface, and that may have been composed of deformed pieces of the slurry particles themselves.

The RMGI, Fuji II LC, showed similar rough wear patterns in both the abrasion and attrition regions for the neutral condition (Figures 5A and 5B), while fractures were observed in the attrition region in the acidic condition (Figure 5E). However, much of the attrition surface from the acidic condition for the RMGI was covered by spherical-shaped particles (approximately 50 µm) that appeared to be impacted into the surface and resembled deformed PMMA spheres from the slurry.

The enamel wear facets from the neutral condition revealed fine scratches on the surface, typical of abrasive wear (Figures 2C, 3C, 4C, and 5C). In the acidic environment the wear facets showed smoother, more polished surfaces (Figures 2F, 3F, 4F, and 5F).

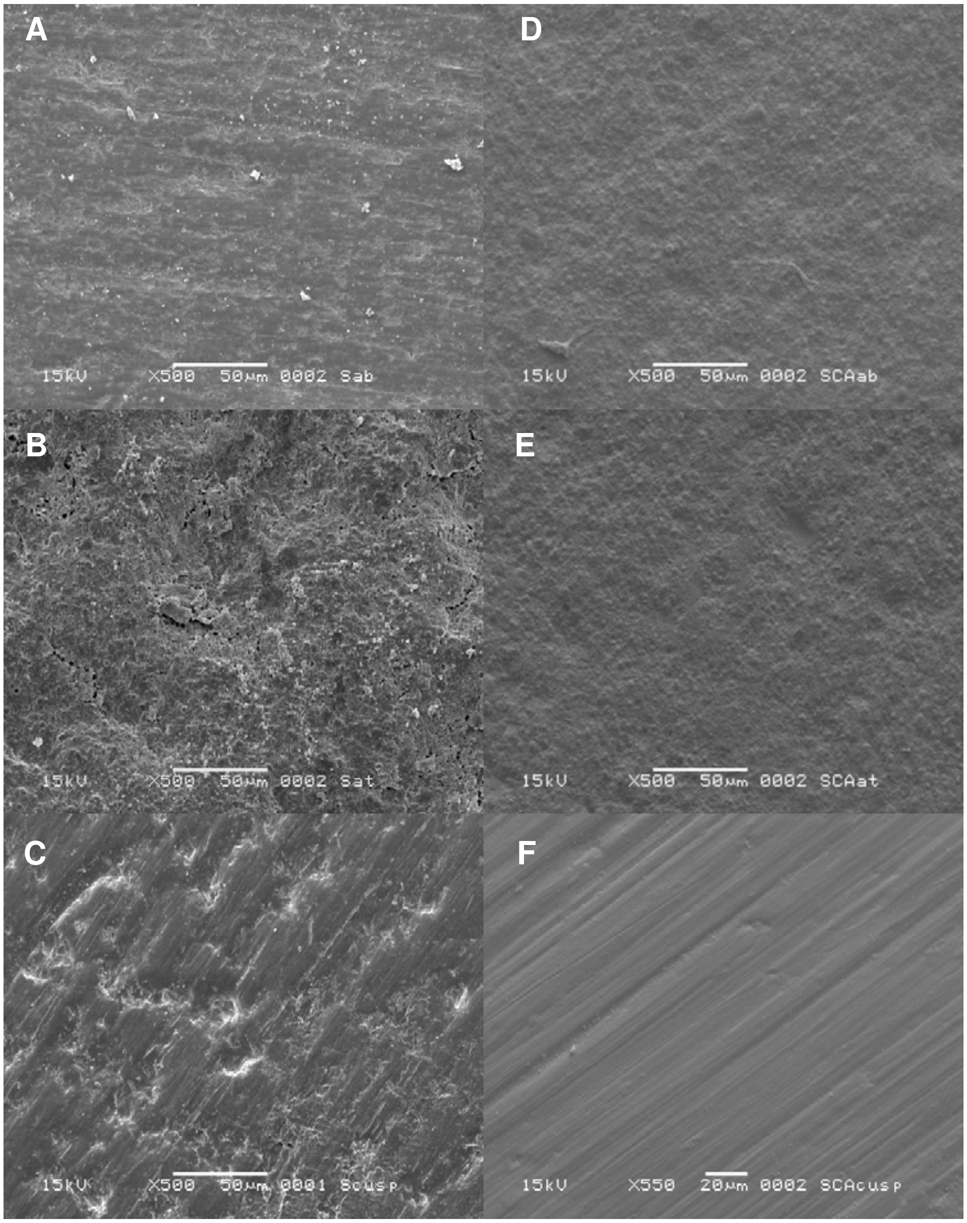

Figure 2 - SEM micrograph of Filtek Supreme, demonstrating: A – abrasion wear region control; B – attrition wear region control; C – cusp wear control; D – abrasion wear region citric acid; E –attrition wear region citric acid; F – cusp wear citric acid.

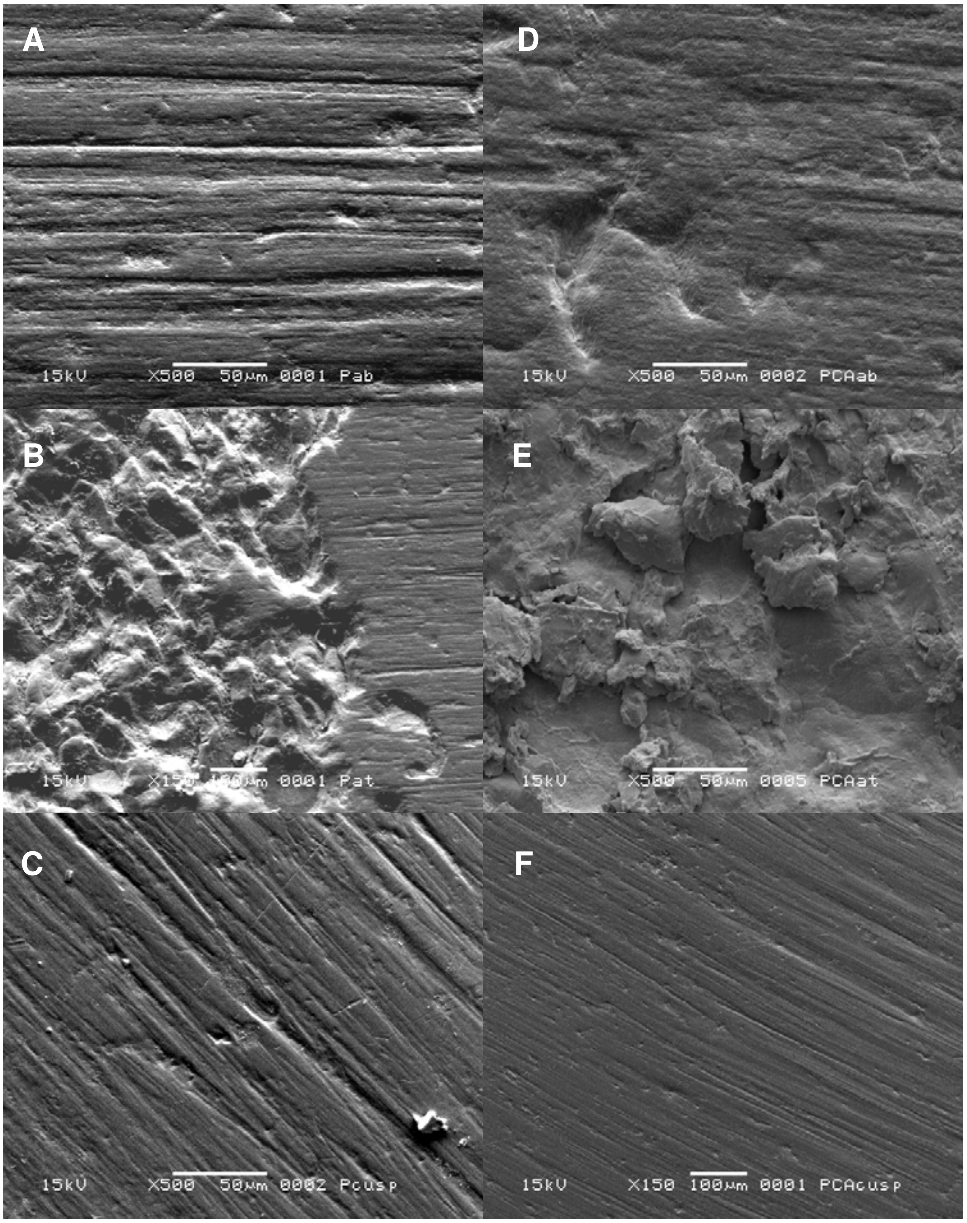

Figure 3 - SEM micrograph of Point 4, demonstrating: A – abrasion wear region control; B – attrition wear region control; C – cusp wear control; D – abrasion wear region citric acid; E –attrition wear region citric acid; F – cusp wear citric acid.

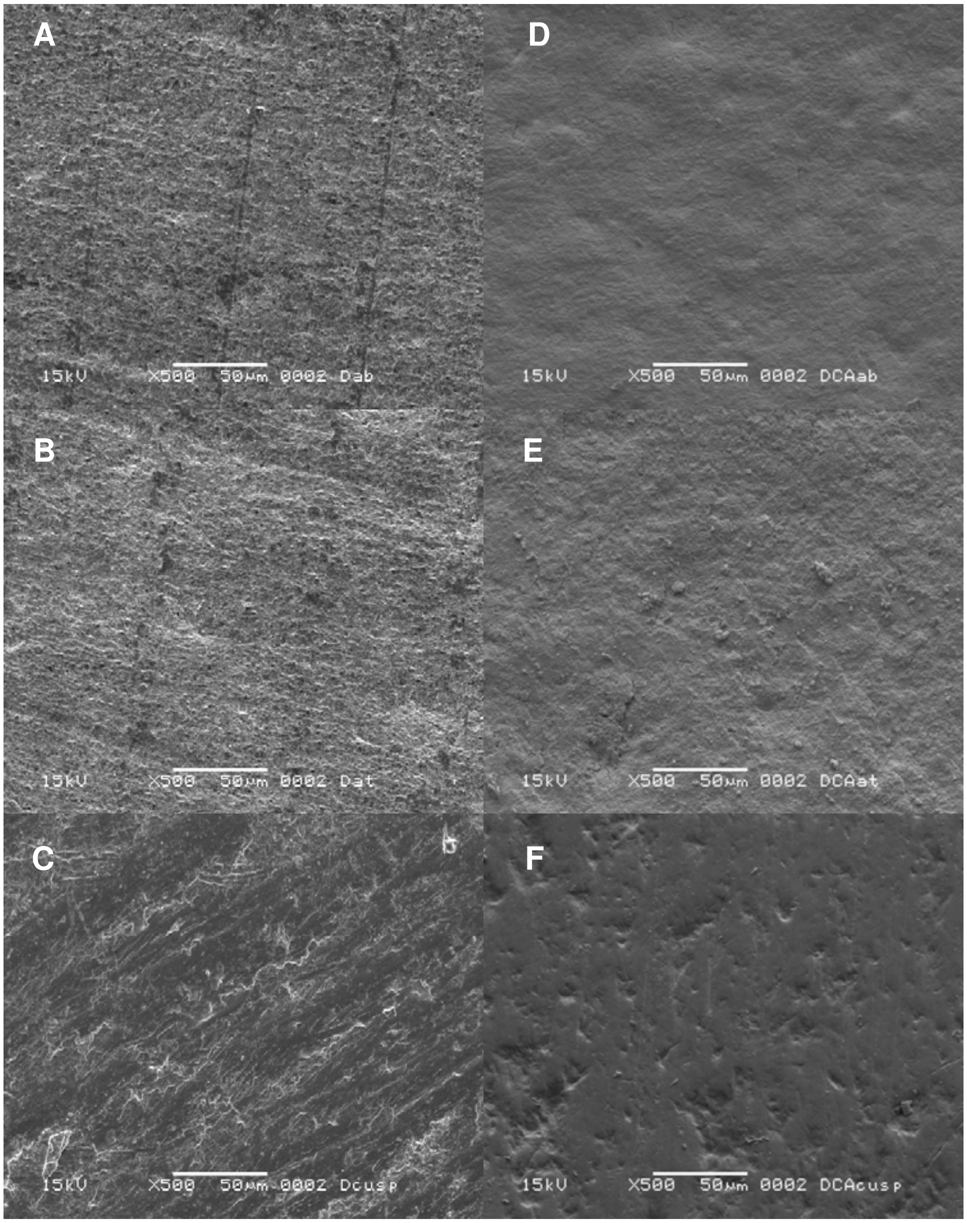

Figure 4 - SEM micrograph of Dyract AP, demonstrating: A – abrasion wear region control; B – attrition wear region control; C – cusp wear control; D – abrasion wear region citric acid; E –attrition wear region citric acid; F – cusp wear citric acid.

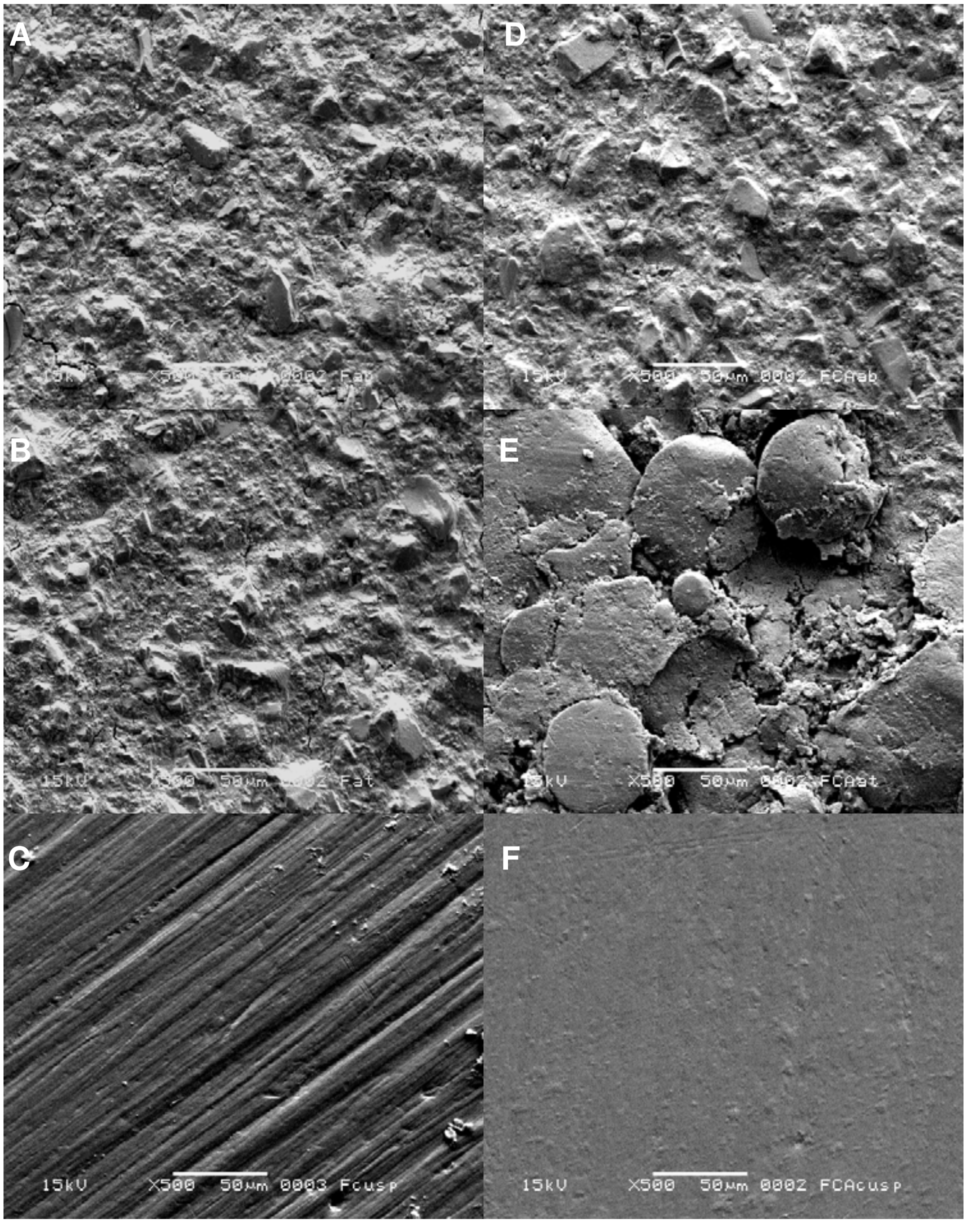

Figure 5 - SEM micrograph of Fuji II LC, demonstrating: A – abrasion wear region control; B – attrition wear region control; C – cusp wear control; D – abrasion wear region citric acid; E –attrition wear region citric acid; F – cusp wear citric acid.

DISCUSSION

The search for the ideal restorative material to replace natural tooth tissue and the demand for products with adhesive and caries-protective properties together with a simple clinical application procedure have led to the development of restorative materials that combine conventional glass ionomers and light-cured resin composites, like the resin-modified glass ionomers and the polyacid-modified resin composites.[23] Resin composites, polyacid-modified resin composites (compomers) and resin-modified glass ionomers (RMGI) have been shown to rank in the following order in terms of their mechanical properties: Resin composites ≥ compomers > RMGI. [11, 23-26] In this study, the same ranking was observed for the wear resistance of these materials. The resin composites showed less wear than the compomer and the RMGI. This result was similar to that observed in other studies.[26-28]

The currently available composites offer improvements in wear resistance by using different fillers of smaller average filler particle size, effectively reducing the incidence of filler exfoliation during abrasion.[13,27] In this study, no significant difference in wear volume was observed between the resin composites Filtek Supreme and Point 4. Both composites have fillers of relatively small size and high filler volume loading (Table 1), conferring to them a high wear resistance.

The wear traces of the nanofill resin composite, Filtek Supreme, showed less evidence of surface roughness and pitting than did the worn surfaces of the microhybrid, Point 4. This difference might be due to the small individual particle size of the nanofill composite. While there was evidence for surface pitting due to fatigue in the attrition region for both composites, it was more extensive for Point 4, again possible related to the larger filler size. The presence of the loose particles on the attrition surface of the Point 4 specimens worn in the acidic slurry is not readily explained. While similar particles were also noted on the RMGI, there is a big difference in the particle size between these two materials that cannot explain this result.

The compomer showed intermediate wear values, being lower than the RMGI and higher than the composites. Unlike the RMGI, anhydrous compomers are said to undergo an acid-base reaction after the initial polymerization that is dependent on exogenous water.

It has been suggested that the development of a carboxylate-rich surface, formed by the acid-base reaction once the surface absorbs water, may contribute to lower wear resistance of these materials compared to composites. This layer would be expected to be similar in composition and properties to that of the RMGI, which has been shown in this and other studies to be less wear resistant than composite. It is also possible that the absorbed water has a plasticizing effect that reduces the wear resistance of the surface of the compomer.[27,29] This difference in wear between composites and compomers also may be related to the different polymers present in their organic phases,[27] the typically lower overall filler content of the compomers, and the higher mean filler particle size compared to those of the composites (Table 1).

The RMGI, Fuji II LC, showed the least wear resistance of the materials evaluated in this study. This relatively high wear is likely due to the characteristics of this material, which include inferior mechanical properties and higher roughness compared to compomers and composites.[7,11,23,25] According to Peutzfeld *et al.* [26], in RMGI crosslinks are formed between the chains of the polyacid, often *via* a HEMA molecule. The crosslink has a certain length and may prevent the two joined polyacid chains from coming close together in the vicinity of the crosslink. In this region, carboxylate groups in the two polyacid chains may be too far apart to be crosslinked via Ca^{+2} as would normally happen in the absence of the organic crosslink. Thus, the reaction of the carbon double bond may physically separate the chains sufficiently to minimize the ionic interactions that would provide the greater hardening and stiffening of the matrix. The result of this destabilizing effect produced by the methacrylate crosslinking is decreased resistance to surface indentation and wear. This difference between Fuji II LC and Dyract could be also attributed to the higher roughness and the presence of a greater amount of porosities in the RMGI. Also, it is likely that contributing factors to the greater wear of the RMGI are the lower filler load, the larger filler particle size (Table 1), and the poorer interaction between filler and matrix in the RMGI as compared to the compomer, all of which are readily visible in the comparison of the wear surfaces of the compomer (Figures 4-A,B,D, and E) and the RMGI (Figures 5-A,B,D, and E).

The wear resistance of a material can be negatively affected by acidic conditions.[2,3,7,30,31] The 0.3% citric acid solution with pH 3.2 was chosen for this study because it is often an ingredient of soft drinks. According to West *et al.* [21,22] beverages ready to drink have an acid concentration of around 0.3% and pH~3.2. It was expected that wear would be heightened in this solution, especially for the more acid soluble materials, such as the RMGI.

According to Sarkar [32] corrosive wear involves a sequence of events, beginning with water absorption that causes a hydrolytic degradation of the silane coupling and reinforcing fillers in the resin-based materials. The absorbed water diffuses internally through the resin matrix, filler interfaces, pores, and other defects, slowly dissolving the filler particles, compromising their reinforcing effect. The result is the formation of a corroded subsurface layer that is softer and more porous, and one that can more easily be removed by masticatory forces. Therefore, any alteration of the surface of resin-based materials at low pH should predominantly be due to hydrolytic degradation.[18,30,32]

On exposure to plaque acids and food simulating media, resin-based materials have been shown to undergo softening [8,9] and loss of surface integrity.[11,32] An increase in their wear rate has also been demonstrated.[2,3,17,18,31] In this study, the simultaneous challenge of corrosive and abrasive wear produced a generalized increased in the wear of resin-based materials, likely because the superficial exposed layer of the material was abraded away as soon as it reached a critical degree of softening. This would be a continuous process occurring during the lifetime of the restoration.

In this study, the two-way ANOVA suggested a trend for increased wear for all materials in the acid environment. However, no significant difference was observed between the neutral and acidic environment for the materials when individually analysed using the post-hoc multiple comparison test. While unexpected, this lack of difference could be related to a number of factors, including the limited number of wear cycles conducted in this study, and changes in the pH and consistency of the slurry during the test.

The pH of the solution in this study was measured before (pH ~ 3.2) and after mixing with the seeds and PMMA (pH ~ 3.8), and after the wear test (pH ~ 4.0 – composites; pH ~ 4.5 to 4.8 – compomers and RMGI). As the acid corrodes the materials, portions of the organic matrix and filler particles are released into the acid solution, buffering the acid and increasing the pH.[33] The overall low erosion observed in the samples, and the lack of difference between the neutral and acidic environment on the materials when individually analysed, could be related to this increase in the pH of the solution during the test, increasing the degree of saturation of the solutions, especially for the compomer and the RMGI. In these materials, the acid-base reaction products (salts) could have buffered the acidic solution due to the salt matrix dissolution and material breakdown. Also, the slurry could have offered some protection to the material surfaces,[15] likely imparted by the oils in the seeds, reducing the exposure of the surface to the acid and making the corrosive process less severe.

In general, the wear of the antagonist enamel cusps reflected the wear of the materials, with significant differences found between the groups. Dyract AP/neutral group (D) produced the highest enamel cusp wear, though this was not different from the Fuji II LC/neutral group (F). Also, a significant, but weak (R=0.56) positive correlation was found between cusp wear and wear of the material, with higher material wear correlating to higher cusp wear. The composites were not different from each other and produced lower cusp wear than the compomer and the RMGI. These results are likely explained by the superior mechanical properties and smaller filler particle size of the composites that make it more difficult to dislodge abrasive fillers from the resin matrix, thus increasing the wear resistance of the material and decreasing the generation of abrasive particles that wear on the antagonist cusp.

Microscopic examination of the enamel wear facets of all the materials from the neutral condition revealed a pattern of fine scratches, typical of abrasive wear (Figures 2C, 3C, 4C, and 5C). However, the in the acidic environment the wear facets showed a more polished surface (Figures 2F, 3F, 4F, and 5F). This was especially true for the compomer and the RMGI, and may be explained by a solubilization of the tips of the scratches and the polishing effect of the citric acid [34] resulting in a more even wear surface on the enamel antagonist. This effect was also seen on the surface of some of the worn composites, which overall showed less distinct surface features.

Based on the results of this study, the first hypothesis tested was proven. The resin-based materials presented a different wear pattern and extent of wear, the compomer and the resin-modified glass ionomer had greater wear than the resin composites. The second hypothesis tested cannot be proven, as individual comparison of the wear for each material in water and in the acidic environment were not significantly different.

It should be remembered that a material with very high wear resistance may not be the most ideal for restoring deciduous teeth. It is a common clinical finding for the composite restoration to be exposed above the margin as the surrounding tooth wears with time to a greater extent and at a faster rate. A mismatch of wear rates between the deciduous teeth and the restorative material could result in an occlusal destabilization, especially in a mixed dentition.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. Resin-modified glass ionomer was more susceptible to wear than compomer, which was more susceptible to wear than the two resin composites under both conditions (neutral and acidic);
2. The association of abrasive and corrosive wear trended toward an increase in the extent of wear, but the difference was not significant when the materials were evaluated individually;
3. The higher the material wear, the higher the wear on the antagonist enamel cusp. The compomer and the resin modified glass ionomer produced higher wear than the composites on the antagonist enamel cusp in both conditions (neutral or acidic).

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CONSIDERAÇÕES GERAIS

A biodegradação da estrutura dentária e dos materiais restauradores é decorrente dos desafios químicos, térmicos e mecânicos aos quais estas estruturas estão constantemente submetidas (de Gee *et al.*, 1996a e 199b; Turssi *et al.*, 2003a e 2003b). Algumas conseqüências da biodegradação podem ser: diminuição da dureza (Wu & McKinney, 1982; Asmussen, 1984; Yap *et al.*, 2000a) o aumento da rugosidade (Yap *et al.*, 2000a; Turssi *et al.*, 2001 e 2002), descoloração (Bollen *et al.*, 1997; Bagueri *et al.*, 2005) e perda de estrutura, ou seja, desgaste tanto da estrutura dentária como dos materiais restauradores.

O objetivo desta tese foi avaliar a degradação de diferentes materiais restauradores e do esmalte decíduo e permanente frente aos desafios químico e/ou mecânico. Pode-se observar que tanto a estrutura dentária (esmalte decíduo e permanente) quanto os diferentes materiais restauradores (compósitos, resina modificada por poliácidos e cimento de ionômero de vidro modificado por resina) foram afetados diferentemente pelo desafio químico (exposição a diferentes soluções).

No capítulo 1, observou-se que a rugosidade superficial dos materiais restauradores foi aumentada após armazenamento, embora este aumento não tenha sido estatisticamente significativo para todos os materiais. As amostras foram submetidas apenas ao armazenamento em soluções normalmente contidas na dieta (água, ácido cítrico, ácido lático, álcool 50% e Coca-Cola), sem, contudo, serem submetidas a qualquer outro desafio térmico ou mecânico, para que apenas um dos fatores relacionados à biodegradação, o efeito da solução na superfície do material, pudesse ser avaliado.

O aumento da rugosidade observado neste estudo foi material e solução dependente. Os materiais restauradores selecionados (compósitos, resina modificada por poliácido e cimento de ionômero de vidro modificado por resina) apresentaram comportamentos diferentes.

A rugosidade dos compósitos Filtek Z250 e Filtek Flow não foi afetada pelo armazenamento independentemente da solução de armazenamento ou do tempo em que as amostras ficaram imersas, indicando que estes compósitos foram resistentes à degradação química. De acordo com Karantakis *et al.* (2000) e Yap *et al.* (2000a e b), os compósitos são, em geral, resistentes à degradação química. Isto pode ser atribuído ao alto grau de conversão, alto conteúdo de partículas de carga e inércia química destas partículas, além da eficiente silanização das partículas de carga, o que permite que a estrutura final do compósito polimerizado permaneça estável frente à biodegração. Entretanto, neste estudo o compósito Esthet X apresentou aumento significativo da rugosidade após 1 mês de armazenamento em Coca-Cola e 3 meses de armazenamento em solução de ácido cítrico. Comparando os resultados do compósito Esthet X aos dos demais compósitos utilizados neste estudo observamos que a diferença entre eles está principalmente relacionada à sua composição. O compósito Esthet X apresenta partículas de carga de vidro de bário enquanto os demais compósitos possuem partículas de zircônia-sílica. De acordo com McKinney (1985), as partículas de vidros de metais pesados são mais suscetíveis à degradação em ácidos fracos, o que pode ser comprovado pela imagem da superfície do Esthet X após 6 meses de armazenamento em ácido cítrico, onde espaços vazios, típicos de dissolução de partículas de carga, foram observados.

Outra diferença entre estes materiais está relacionada à composição da matriz orgânica (tipo e quantidade de diferentes monômeros, sistema de iniciação, etc), o que poderia estar relacionado à maior suscetibilidade do Esthet X à degradação quando armazenado em Coca-Cola. Isto foi evidenciado na imagem da superfície do Esthet X após 6 meses de armazenamento em Coca-Cola, na qual observou-se protrusão das partículas de carga do material.

Com relação à resina modificada por poliácido Dyract, aumento significativo da rugosidade somente foi observado após 6 meses de armazenamento em água. Este material apresentou rugosidade similar aos compósitos (Turssi *et al.*, 2001 e 2002; Gladys *et al.*, 1997) embora apresente partículas de carga de tamanho superior. A água desempenha um papel importante para a resina modificada por poliácido, uma vez que este material utiliza a água do meio externo para a reação do tipo ácido-base do seu componente ionomérico (Eliades *et al.*, 1998). A reação da superfície deste material com a água, forma uma camada rica em carboxilato, a qual é menos resistente a degradação (Eliades *et al.*, 1998). Além disso, o aumento da rugosidade generalizado, como observado na imagem da superfície deste material após 6 meses de armazenamento em água, poderia estar relacionado à hidrólise do agente de união silano ou até mesmo à um amolecimento da matriz resinosa.

Dessa forma, se avaliado o efeito das soluções ácidas sobre os materiais restauradores observa-se que as características físicas e químicas como: o tipo, conteúdo, tamanho da partícula de carga, tipo de monômero, silanização das partículas de carga, grau de conversão, etc, desempenham papel importante na resistência destes materiais a biodegradação.

Com relação à solução, algumas características estão relacionadas principalmente à capacidade da mesma em produzir efeitos deletérios sobre a estrutura dentária e materiais restauradores, como a concentração, o pH, a capacidade de manutenção do pH baixo, etc. Como observado neste estudo, o ácido cítrico, o qual apresenta alta capacidade de manter-se com pH baixo, resistindo ao tamponamento provocado pela lixiviação dos produtos dos materiais na solução, apresentou efeitos significativos (aumento da rugosidade) sobre Esthet X, e a Coca-Cola®, a qual apresentou o pH mais baixo deste estudo, também produziu efeitos similares sobre o mesmo material.

Deve-se considerar que o desafio ao qual as amostras foram submetidas neste estudo (capítulo 1) pode ser considerado extremo e não poderia ser transportado para o ambiente clínico. Entretanto, este estudo mostra que os materiais devem ser adequadamente selecionados, especialmente para utilização em pacientes que apresentam dieta ácida, evitando assim um aumento excessivo da rugosidade superficial do mesmo com o tempo, o que por sua vez, levaria ao aumento no acúmulo de placa sobre a restauração e necessidade de troca da mesma.

Além do aumento da rugosidade provocado pela dieta ácida, os materiais restauradores e a estrutura dentária podem apresentar redução da dureza superficial e, conseqüentemente, aumento da suscetibilidade ao desgaste, como observado nos capítulos 2 e 3, nos quais o desgaste abrasivo foi aumentado significativamente em meio ácido comparado ao meio neutro.

As características físicas e mecânicas de cada substrato, esmalte decíduo ou permanente, têm papel importante no desgaste abrasivo dos mesmos. O esmalte decíduo apresentou maior desgaste em ambas as condições do meio (neutro ou ácido) comparado ao esmalte permanente, confirmando a hipótese testada no capítulo 2. Esta diferença estaria relacionada às diferenças morfo-fisiológicas entre o esmalte decíduo e o permanente, como por exemplo, menor dureza e menor grau de mineralização do esmalte decíduo (Featherstone & Mellberg, 1981; Amaechi *et al.*, 1999).

No capítulo 3, os diferentes materiais restauradores (compósitos, resina modificada por poliácido e cimento de ionômero de vidro modificado por resina) apresentaram também comportamento diferenciado frente ao desafio químico-mecânico. Os compósitos foram mais resistentes ao desgaste em ambos os meio (ácido ou neutro), seguido pela resina modificada por poliácido e pelo cimento de ionômero modificado por resina, o qual apresentou o maior desgaste nesses mesmos meios. Novamente neste caso, como no capítulo 1, as características físicas e químicas (composição) dos materiais tiveram influência no comportamento dos mesmos frente ao desafio erosivo/abrasivo.

Um aspecto importante dos estudos dos capítulos 2 e 3 foi a possibilidade de submeter as amostras tanto do substrato dentário quanto dos materiais restauradores, ao desafio simultâneo de abrasão e erosão, através da utilização da máquina de ensaio OHSU Oral Wear Simulator. Esta máquina simula os movimentos de abrasão e atrição executados durante a mastigação, além disso, o desgaste é realizado na presença de um terceiro corpo, uma pasta, a qual é constituída de água, pó de polimetilmetacrilato (PMMA) e sementes de papoula. Para reproduzir o meio ácido, e simular o desafio químico erosivo, a água foi substituída por ácido cítrico. Isto permitiu a realização de estudos em condições mais próximas possíveis da situação clínica (cavidade bucal).

Adicionalmente, a análise do desgaste nos capítulos 2 e 3 foi realizada através de dois parâmetros: profundidade máxima de desgaste (bidimensional) e alteração do volume (tridimensional). Observou-se que não houve diferença significativa em ambos os capítulos quando o parâmetro profundidade máxima foi avaliado. Já quando o parâmetro alteração de volume foi utilizado, diferenças significativas foram observadas. Dessa forma, observa-se que a somente a análise da profundidade do desgaste não é um bom parâmetro para determinar diferenças entre o desgaste dos tipos de substrato e/ou materiais restauradores, porque o desgaste ocorre em três dimensões, que devem ser consideradas na avaliação, como é realizado na análise da alteração do volume.

Além da análise do desgaste do material/substrato também foi observado o desgaste do antagonista. A análise do desgaste do antagonista é um fator importante no processo de abrasão, pois dependendo das superfícies em contato pode haver maior ou menor desgaste destas. Portanto, a análise da correlação entre desgaste da superfície e do antagonista foi realizada nos capítulos 2 e 3. No capítulo 2, não houve correlação entre desgaste do esmalte decíduo e/ou permanente e da respectiva cúspide antagonista. Já no capítulo 3, houve significativa correlação positiva entre desgaste do material e da cúspide antagonista em esmalte decíduo. A ausência de correlação no capítulo 2 e a fraca correlação encontrada no capítulo 3 estão relacionadas à limitação da avaliação do desgaste da cúspide antagonista. Esta avaliação foi realizada por meio da determinação da área de desgaste, método bidimensional. Como dito anteriormente, a análise da alteração do volume da cúspide após o desgaste seria o parâmetro mais indicado. Entretanto, a cúspide antagonista em formato de hemi-esfera dificultou o reposicionamento da amostra após o ensaio de desgaste, na exata posição da leitura inicial, impossibilitando nestes estudos a determinação da alteração do volume obtida através da análise no perfilômetro MTS utilizando o *software* Ansur 3D. Além disso, deve-se considerar que nestes estudos o desgaste apresentado pela cúspide antagonista foi muito pequeno, especialmente nas amostras do capítulo 2 (esmalte vs. esmalte).

Em síntese, esta tese contribuiu para o estudo do comportamento de materiais restauradores resinosos e do esmalte decíduo e permanente, submetidos à exposição em soluções simuladoras da dieta, enfatizando dois aspectos importantes, a rugosidade e o desgaste. Os estudos apresentados nos capítulos 1, 2 e 3, nos permitiram verificar as diferenças no comportamento de dentes decíduos e permanentes submetidos ao desgaste abrasivo e erosivo simultâneo, ajudando na identificação do mesmo e selecionando melhores alternativas de tratamento em cada dentição, ou seja, os materiais mais adequados para a realização de restaurações nestes substratos.

CONCLUSÕES

Baseado nos resultados dessa tese pôde-se concluir que:

A degradação dos materiais restauradores resinosos é material e solução dependente. Os materiais restauradores estudados foram em geral resistentes à degradação química (aumento da rugosidade superficial), exceto Esthet X armazenado em ácido cítrico e Coca-Cola®, e Dyract AP armazenado em água destilada.

O esmalte decíduo é menos resistente ao desgaste do que o esmalte permanente. O meio ácido propiciou maior desgaste comparado ao meio neutro e a associação dos desafios abrasivo e erosivo aumentou a taxa de desgaste apenas para o esmalte decíduo.

Os materiais restauradores resinosos apresentaram diferente comportamento de acordo com sua composição. O cimento de ionômero de vidro modificado por resina foi mais susceptível ao desgaste, seguido pela resina modificada por poliácido, e pelos compósitos em ambas condições do meio (neutra ou ácida). A associação dos desafios abrasivo e corrosivo promoveu uma tendência ao aumento do desgaste dos materiais, embora esse aumento não tenha sido significativo para os materiais quando analisados individualmente.

Existe correlação entre o desgaste do material e o desgaste das cúspides em esmalte. Quanto maior desgaste do material, maior desgaste da cúspide em esmalte decíduo. Entretanto, a mesma correlação não foi observada entre o esmalte decíduo e permanente e suas respectivas cúspides antagonistas.

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[5] De acordo com a norma da FOP/ UNICAMP, baseada na norma do International Committee of Medical Journal Editors - Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o MEDLINE.

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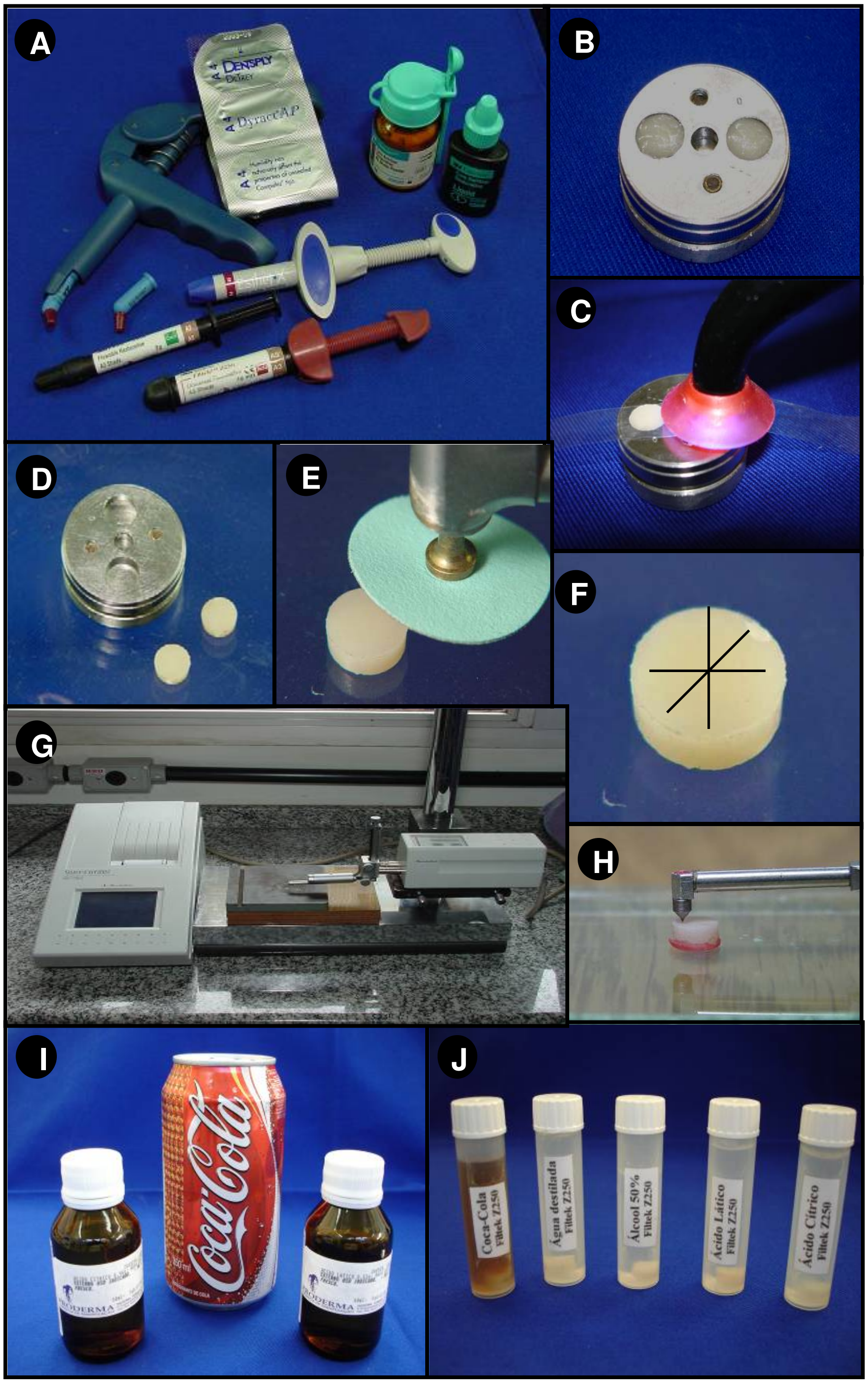

Figura 1 - Ilustrações do Capítulo 1. A. Materiais utilizados para confecção das amostras: Compósitos – Esthet X (Dentsply), Filtek Z250 e Filtek Flow (3M/ESPE); Resina modificada por poliácido – Dyract AP (Dentsply); Cimento de ionômero de vidro modificado por resina – Vitremer (3M/ESPE). B. Material restaurador inserido em matriz metálica. C. Fotoativação das amostras (Elipar Tri-Light – 3M/ESPE). D. Amostras após remoção da matriz. E. Polimento das amostras (Discos Sof-Lex – 3M/ESPE). F. Amostra polida, indicação do local das três leituras. G. Rugosímetro (Surfcorder SE 1700, Japan). H. Agulha posicionada para leitura na amostra. I. Soluções utilizadas para armazenamento (Coca-Cola®, Ácido cítrico, Ácido lático). J. Amostras armazenadas e identificadas: Coca-Cola®, água destilada, álcool 50%, ácido lático, ácido cítrico.

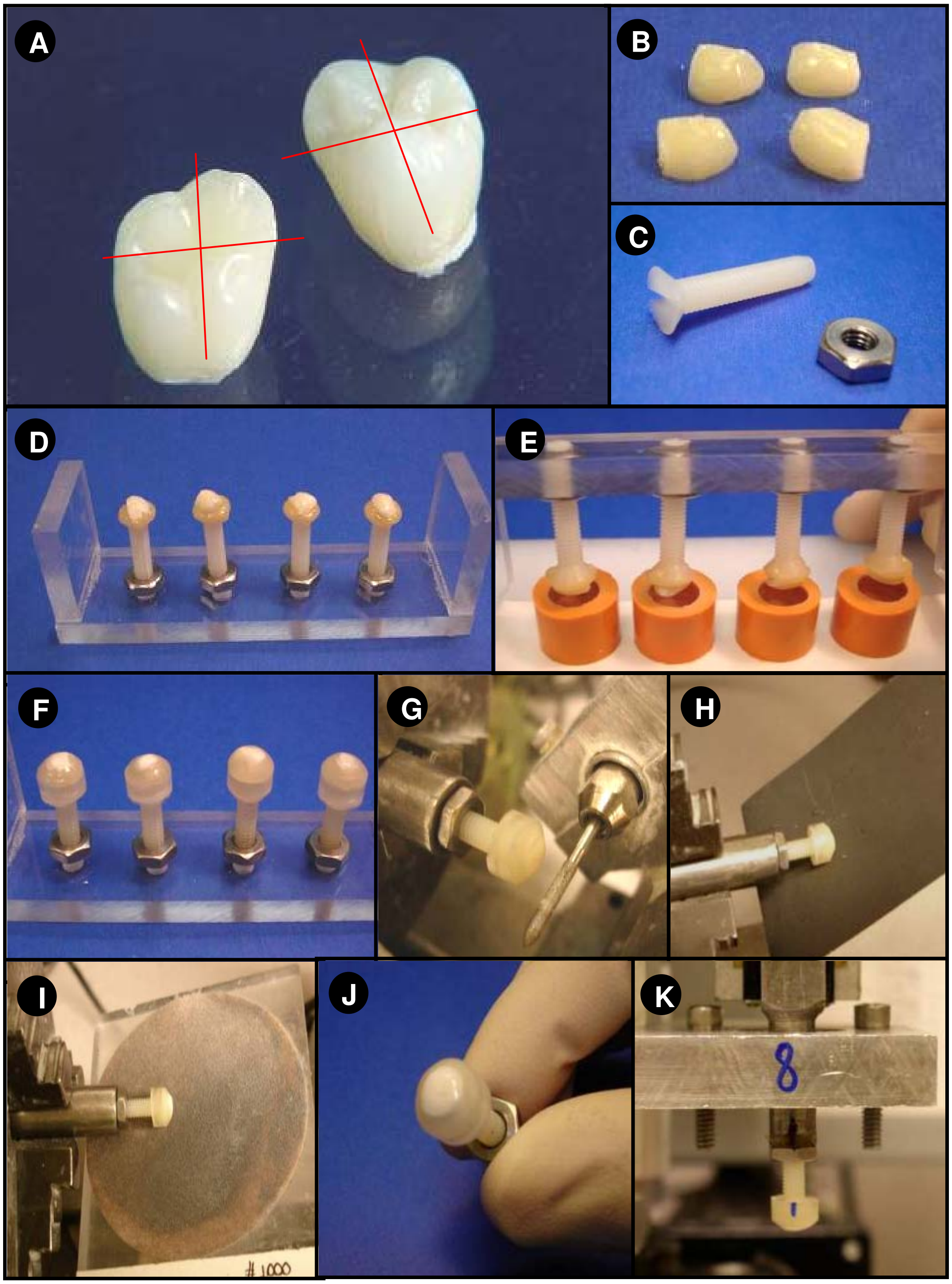

Figura 2 - Ilustrações do Capítulo 2 e 3. Confecção das cúspides. A. Molares superiores decíduos. Localização dos cortes para obtenção dos fragmentos em esmalte. B. Quatro fragmentos dentários obtidos após o corte. C. Parafuso de nylon e rosca. D. Fragmentos em esmalte posicionados nos parafusos utilizando resina fluida. E. Inclusão dos parafusos com os fragmentos dentais, por meio do dispositivo em acrílico, em cilindros ocos de silicona de adição utilizando resina epóxica. F. Fragmentos em esmalte após inclusão em resina epóxica. G. Amostra (cúspide antagonista) posicionada para torneamento. H e I. Polimento da cúspide em esmalte. (Lixas # 600 e feltro com pó de óxido de alumínio granulação 1000, respectivamente). J. Cúspide antagonista em esmalte após polimento (forma de hemi-espera de aproximadamente 12 mm de diâmetro). K. Cúspide antagonista posicionada na máquina de desgaste.

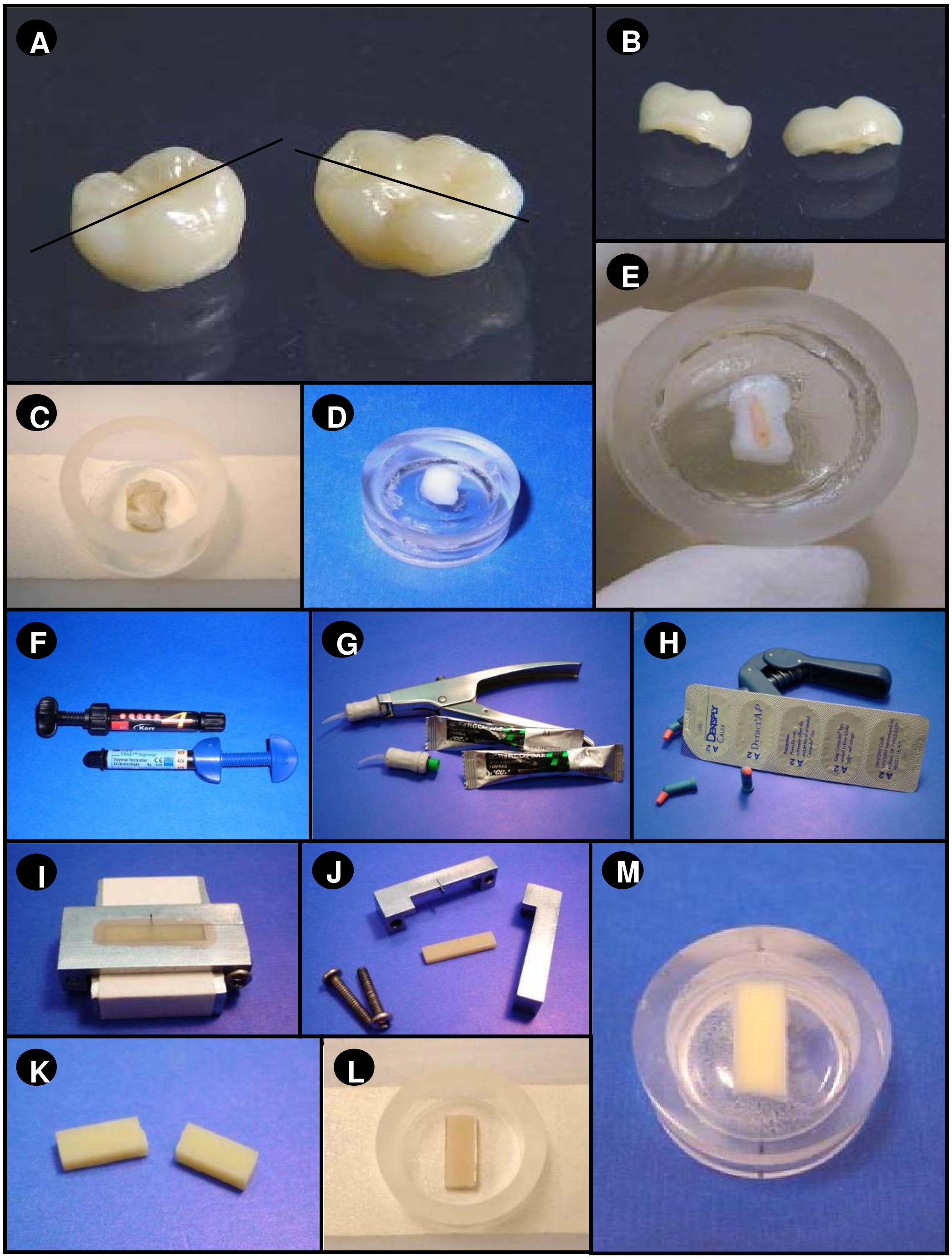

Figura 3 - Ilustrações Capítulos 2 e 3. Confecção das superfícies para desgaste (esmalte – capítulo 2 e materiais restauradores – capítulo 3). A. Molares inferiores decíduos. Localização do corte (sentido mésio-distal). B. Fragmentos dentários após o corte. C. Amostra posicionada sobre fita adesiva dupla-face para inclusão em resina epóxica. D. Amostra incluída após polimento (lixas de carbeto de silício granulação 400, 600 e 1200; feltro com pó de óxido de alumínio granulação 1000, 1 e 0,5 µm). E. Amostra com marca do traço de desgaste (teste para verificação da localização do desgaste). F. Compósitos utilizados para confecção das amostras (Capítulo 3): Filtek Supreme (3M/ESPE) e Point 4 (Kerr). G. Resina modificada por poliácidos utilizada para confecção das amostras (Dyract AP – Dentsply). H. Ionômero de vidro modificado por resina (Fuji II LC – GC). I. Compósito inserido em matriz metálica bipartida. J. Amostra finalizada após remoção da matriz. K. Amostra fraturada para obtenção de dois corpos-de-prova (12 x 5 x 2 mm). L. Amostra posicionada sobre fita adesiva dupla-face para inclusão em resina epóxica. M. Amostra incluída após polimento (lixas de carbeto de silício granulação 400, 600 e 1200; feltro com pó de óxido de alumínio granulação 1000).

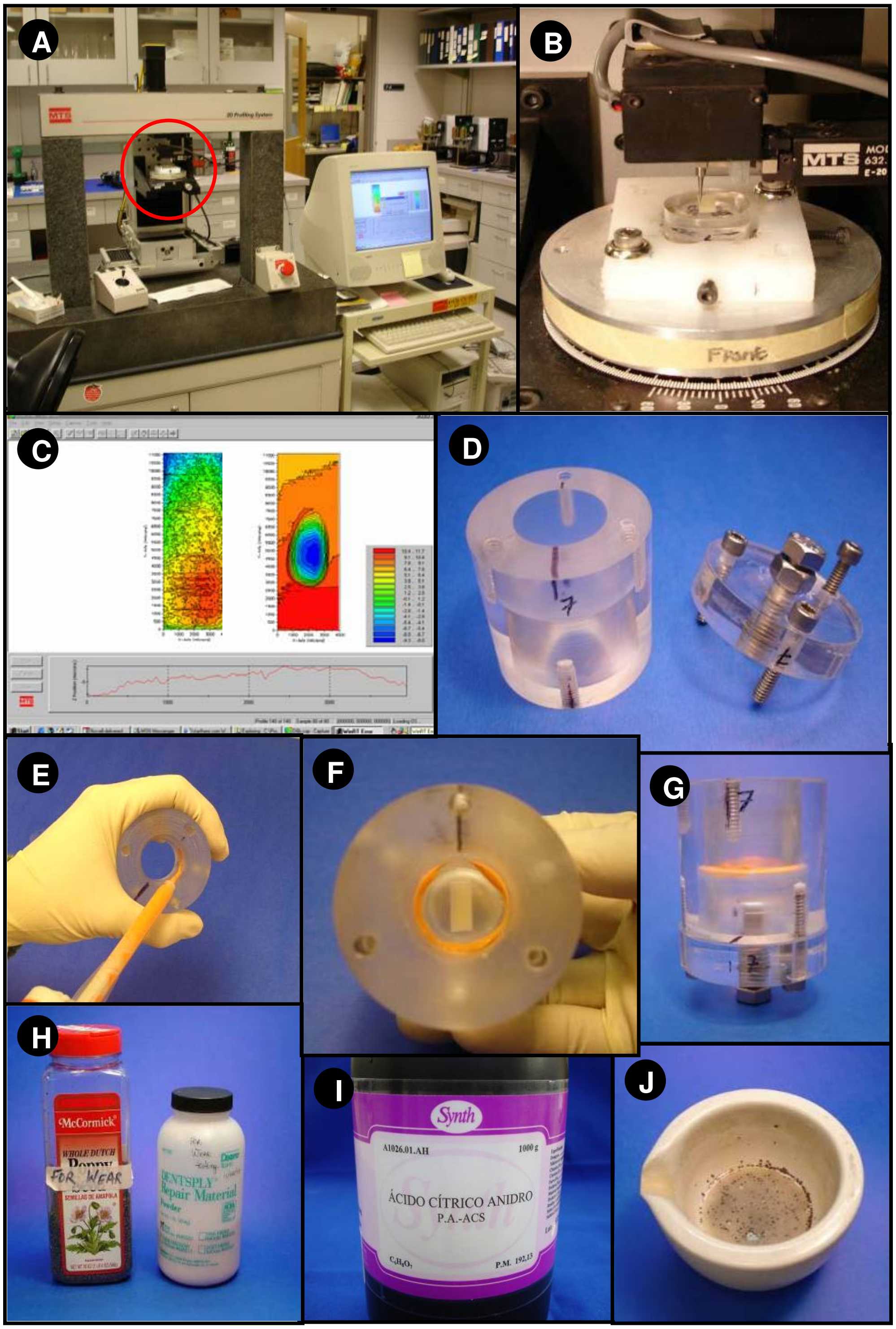

Figura 4 - Ilustrações dos Capítulos 2 e 3, preparo da máquina de ensaio e programa para leitura do desgaste. A. Perfilômetro – MTS 3D Profiler. B. Vista aproximada da agulha posicionada para leitura. C. Software Capture – leitura do perfil da amostra antes e após o ensaio de desgaste. D. Câmara de acrílico da máquina de desgaste. E. Vedamento da câmara de desgaste com silicona de adição (Express – 3M/ESPE). F. Amostra (material restaurador) posicionada na câmara de desgaste. G. Câmara montada para ensaio de desgaste. H. Semente de papoula e pó da resina acrílica (Polimetilmetacrilato) utilizados para obtenção da pasta de desgaste (terceiro corpo). I. Ácido cítrico anidro utilizado para preparar a solução utilizada para obtenção da pasta (meio ácido). J. Pasta de desgaste após mistura dos componentes.

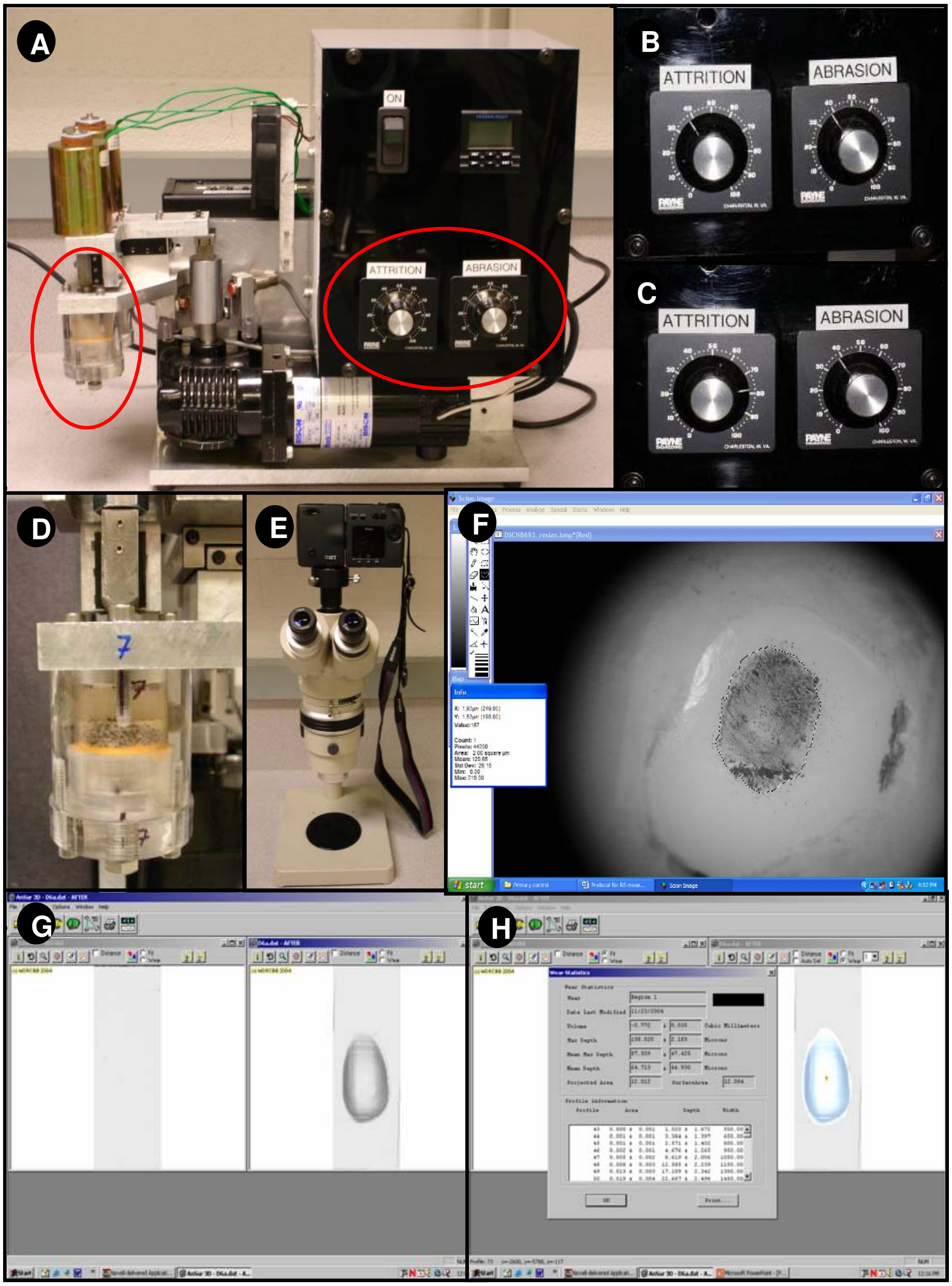

Figura 5 - Ilustrações dos Capítulos 2 e 3, máquina de desgaste e programas para análise dos resultados. A. Máquina de ensaio OHSU Oral Wear Simulator. B. Vista aproximada da carga utilizada para ensaio mecânico de desgaste: Capítulo 2 – regiões de abrasão e atrição – aproximadamente 20 N. C. Vista aproximada da carga utilizada para ensaio mecânico de desgaste: Capítulo 3 – região de abrasão ~ 20 N e atrição ~ 80 N. D. Vista aproximada da câmara de desgaste pronta para ensaio mecânico. E. Máquina digital (Coolpix, Nikon, Japan) acoplada a lupa estereoscópica para captura da imagem da cúspide antagonista em esmalte após ensaio de desgaste. F. Software Scion Image. Área de desgaste da cúspide antagonista em esmalte pintada com lápis preto e demarcada utilizando ferramenta do programa. G. Software Ansur 3D. Imagem da amostra antes e após ensaio de desgate. Sobreposição das regiões. H. Software Ansur 3D. Imagem da região de desgaste individualizada por ferramenta do programa. Resultados da alteração do volume, máxima profundidade de desgaste, profundidade média e área de desgaste da amostra.

APÊNDICE 2

Análise Estatística Capítulo 1

Delineamento experimental: Rugosidade Superficial

Transformação das observações segundo função logaritmo

Tabela 1 – Análise de Variância dos dados de rugosidade, em parcelas subdivididas.

Causa de variação	**G.L.**	**S. Q.**	**Q. M.**	**Valor de F**	**Pr >F**
Material	4	126.0060188	31.5015047	127.61	<0.0001
Meio	4	3.9600368	0.9900092	3.38	0.0104
Material*Meio	16	11.6509480	0.7281843	2.49	0.0016
Resíduo (A)	225	65.8650584	0.2927336	3.97	<0.0001
Tempo	4	8.8428693	2.2107173	29.99	<0.0001
Material*tempo	16	4.5618589	0.2851162	3.87	<0.0001
Meio*tempo	16	2.1521712	0.1345107	1.82	0.0242
Material*meio*tempo	64	12.9301246	0.2020332	2.74	<0.0001
Resíduo (B)	900	66.3414046	0.0737127		
Total corrigido	1249	302.3104907			
R-Quadrado	Coef. Var	Raiz QMR	Média de t_rug		
0.780552	-17.29884	0.271501	-1.569474		

Tabela 2 - Alteração do pH das soluções de imersão com o tempo.

Material	Solução	Tempo				
		Imediato	1 dia	1 semana	1 mês	6 meses
Filtek Z250	Água	5.54	6.66	7.2	5.98	5.44
	Ácido lático	5.00	5.41	5.41	5.4	4.83
	Ácido cítrico	5.00	5.38	5.22	5.21	5.13
	Coca-Cola®	2.59	2.69	2.46	2.38	2.35
	Álcool 50%	6.93	7.22	6.90	6.14	6.29
Esthet X	Água	5.54	6.65	6.17	5.97	5.44
	Ácido lático	5.00	5.46	5.41	5.54	5.03
	Ácido cítrico	5.00	5.24	5.19	5.2	5.3
	Coca-Cola®	2.59	2.79	2.53	2.59	2.58
	Álcool 50%	6.93	7.07	6.95	6.31	6.48
Filtek Flow	Água	5.54	6.54	5.92	5.96	5.53
	Ácido lático	5.00	5.43	5.46	5.5	6.21
	Ácido cítrico	5.00	5.23	5.09	5.02	5.44
	Coca-Cola®	2.59	2.65	2.46	2.45	2.45
	Álcool 50%	6.93	6.71	6.64	6.07	6.41
Dyract AP	Água	5.54	6.84	6.27	6.08	6.31
	Ácido lático	5.00	6.01	6.01	6.35	6.16
	Ácido cítrico	5.00	5.64	5.64	5.83	7.12
	Coca-Cola®	2.59	3.07	3.07	2.98	2.99
	Álcool 50%	6.93	6.35	6.35	6.24	6.08
Vitremer	Água	5.54	7.29	6.45	6.45	6.31
	Ácido lático	5.00	6.19	6.74	7.12	7.38
	Ácido cítrico	5.00	5.69	5.91	6.18	7.02
	Coca-Cola®	2.59	3.04	3.28	3.07	3.10
	Álcool 50%	6.93	6.8	6.53	6.37	5.29

Análise Estatística – Capítulos 2

Delineamento experimental: Desgaste do esmalte
Dados não transformados

Tabela 3 – Análise de Variância para a variável alteração do volume de desgaste de dentes decíduos e permanentes em diferentes condições do meio.

Causa de variação	**G.L.**	**S.Q.**	**Q. M.**	**Valor de F**	**Pr>F**
Substrato	1	0.00497	0.00497	12.600	0.001*
Meio	1	0.00324	0.00324	8.209	0.007*
Subst*Meio	1	0.00132	0.00132	3.351	0.075
Resíduo	36	0.0142	0.000395		
Total	39	0.0237	0.000609		

Tabela 4 - Teste de Tukey para médias de degaste para o fator substrato.

Grupo	**Repetições**	**Médias originais**	**5%**	**1%**
Esmalte permanente	20	-0.0195	a	A
Esmalte decíduo	20	-0.0418	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 5 - Teste de Tukey para médias de desgaste para o fator condição do meio.

Grupo	**Repetições**	**Médias originais**	**5%**	**1%**
Meio neutro	20	-0.0216	a	A
Meio ácido	20	-0.0396	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 6 - Teste de Tukey para médias de desgaste do esmalte decíduo nas diferentes condições do meio.

Grupo	**Repetições**	**Médias originais**	**5%**	**1%**
Decíduo neutro	10	-0.0270	a	A
Decíduo ácido	10	-0.0565	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 7 - Teste de Tukey para médias de desgaste do esmalte permanente nas diferentes condições do meio.

Grupo	**Repetições**	**Médias originais**	**5%**	**1%**
Permanente neutro	10	-0.0162	a	A
Permanente ácido	10	-0.0227	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 8 – Análise de Variância para a variável profundidade máxima do desgaste de dentes decíduos e permanentes em diferentes condições do meio.

Causa de variação	G.L.	S.Q.	Q.M.	Valor de F	Pr>F
Substrato	1	190.984	190.984	1.004	0.323
Meio	1	439.428	439.428	2.311	0.138
Subst*Meio	1	9.026	9.026	0.047	0.829
Resíduo	34	6464.601	190.135		
Total	37				

Tabela 9 – Análise de Variância para o desgaste da cúspide de dentes decíduos e permanentes em diferentes condições do meio.

Causa de variação	G.L.	S.Q.	Q.M.	Valor de F	Pr>F
Substrato	1	0.590	0.590	1.498	0.229
Meio	1	0.350	0.350	0.887	0.353
Subst*Meio	1	0.313	0.313	0.795	0.379
Resíduo	36	14.189	0.394		
Total	39				

Análise Estatística – Capítulos 3

Delineamento experimental: Desgaste de materiais restauradores
Dados não transformados

Tabela 10 – Análise de Variância para a variável alteração do volume de desgaste de materiais restauradores em diferentes condições do meio.

Causa de variação	G.L.	S.Q.	Q.M.	Valor de F	Pr>F
Material	3	106.349	35.450	249.245	<0.00001*
Meio	1	0.8207	0.8207	5.7708	0.0189*
Mater*Meio	3	0.5849	0.1949	1.3710	0.2586
Resíduo	71	10.0982	0.1422		
Total	78	118.9406	1.52488		

Tabela 11 - Teste de Tukey para médias de degaste (alteração do volume) para o fator material.

Grupo	Repetições	Médias originais	5%	1%
Filtek Supreme	20	-0.15160	a	A
Point 4	20	-0.21465	a	A
Dyract AP	20	-0.97770	b	B
Fuji II LC	20	-3.05528	c	C

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 12 - Teste de Tukey para médias de desgaste (alteração do volume) para o fator condição do meio.

Grupo	Repetições	Médias originais	5%	1%
Meio neutro	40	-0.99781	a	A
Meio ácido	40	-1.20180	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 13 - Teste de Tukey para médias de desgaste (alteração do volume) de Filtek Supreme nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Filtek Supreme neutro	10	-0.13990	a	A
Filtek Supreme ácido	10	-0.16330	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 14 - Teste de Tukey para médias de desgaste (alteração do volume) de Point 4 nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Point 4 neutro	10	-0.18210	a	A
Point 4 ácido	10	-0.24720	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 15 - Teste de Tukey para médias de desgaste (alteração do volume) de Dyract AP nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Dyract AP neutro	10	-0.84260	a	A
Dyract AP ácido	10	-1.11280	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 16 - Teste de Tukey para médias de desgaste (alteração do volume) de Fuji II LC nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Fuji II LC neutro	10	-2.82666	a	A
Fuji II LC ácido	10	-3.28390	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 17 – Análise de Variância para a variável profundidade máxima do desgaste de de materiais restauradores em diferentes condições do meio.

Causa de variação	G.L.	S.Q.	Q.M.	Valor de F	Pr>F
Material	3	927780.1511	309260.0503	200.0308	<0.00001*
Meio	1	5007.7340	5007.7340	3.2390	0.0496*
Mater*Meio	3	4905.7972	1635.2657	1.0576	0.3726
Resíduo	71	109770.3702	1546.0615		
Total	78	1049157.5427	13450.7377		

Tabela 18 - Teste de Tukey para médias de degaste (profundidade máxima) para o fator material.

Grupo	Repetições	Médias originais	5%	1%
Filtek Supreme	20	61.843350	a	A
Point 4	20	76.951950	a	A
Dyract AP	20	177.188450	b	B
Fuji II LC	20	336.655511	c	C

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 19 - Teste de Tukey para médias de desgaste (profundidade máxima) para o fator condição do meio.

Grupo	Repetições	Médias originais	5%	1%
Meio neutro	40	155.193256	a	A
Meio ácido	40	171.126375	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 20 - Teste de Tukey para médias de desgaste (profundidade máxima) de Filtek Supreme nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Filtek Supreme neutro	10	58.694400	a	A
Filtek Supreme ácido	10	64.992300	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 21 - Teste de Tukey para médias de desgaste (profundidade máxima) de Point 4 nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Point 4 neutro	10	68.589900	a	A
Point 4 ácido	10	85.314000	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 22 - Teste de Tukey para médias de desgaste (profundidade máxima) de Dyract AP nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Dyract AP neutro	10	156.625500	a	A
Dyract AP ácido	10	197.751400	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 23 - Teste de Tukey para médias de desgaste (profundidade máxima) de Fuji II LC nas diferentes condições do meio.

Grupo	Repetições	Médias originais	5%	1%
Fuji II LC neutro	10	336.863222	a	A
Fuji II LC ácido	10	336.447800	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 24 – Análise de Variância para o desgaste da cúspide de dentes decíduos por diferentes materiais restauradores em diferentes condições do meio.

Causa de variação	GL	Soma dos quadrados	Quadrados médios	Valor de F	P
Material	3	151.2472	50.4157	90.6872	<0.00001*
Meio	1	1.9881	1.9881	3.5762	0.0626
Mater*Meio	3	11.0562	3.6854	6.6292	0.0005*
Resíduo	71	39.4710	0.5559		
Total	78	203.3316	2.6068		

Tabela 25 - Teste de Tukey para médias de desgaste das cúspides para o fator material.

Grupo	Repetições	Médias originais	5%	1%
Filtek Supreme	20	3.4565	a	A
Point 4	20	3.2994	a	A
Dyract AP	20	6.2720	b	B
Fuji II LC	20	6.0080	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

Tabela 26 - Teste de Tukey para médias de desgaste das cúspides para o fator condição do meio.

Grupo	Repetições	Médias originais	5%	1%
Meio neutro	40	4.9177	a	A
Meio ácido	40	4.6002	a	A

Médias seguidas por letras iguais não diferem entre si ao nível de significância indicado

Tabela 27 - Teste de Tukey para médias de desgaste das cúspides em esmalte relacionadas a cada material e condição do meio.

Grupo	Repetições	Médias originais	5%	1%
Filtek Supreme neutro	10	3.4670	a	A
Filtek Supreme ácido	10	3.4460	a	A
Point 4 neutro	10	3.0688	a	A
Point 4 ácido	10	3.5300	a	A
Dyract AP neutro	10	7.0480	c	C
Dyract AP ácido	10	5.4960	b	B
Fuji II LC neutro	10	6.0870	bc	BC
Fuji II LC ácido	10	5.9290	b	B

Médias seguidas por letras distintas diferem entre si ao nível de significância indicado

ANEXO 1

OREGON HEALTH & SCIENCE UNIVERSITY
Research Integrity Office, L106-RI (503) 494-7887

MEMO

Date: 09/21/2004

To: Jack Ferracane

From:
Gary T. Chiodo, DMD, Chair, Institutional Review Board, L106-RI
Susan Hansen, MD, MPH, Co-Chair, Institutional Review Board, L106-RI
Charlotte Shupert, PhD, Manager, Research Integrity Office, L106-RI
Bradley T. Noren, MA, CIP, Assistant Manager, Research Integrity Office, L106-RI

Subject: IRB00000272, IN VITRO WEAR EVALUATION IN PRIMARY AND PERMANENT TEETH AND IN RESTORATIVE MATERIALS

Special Communication

This protocol meets the requirements for Exemption from IRB review and approval in accordance with 45CFR46.101(b)(***4***); data collected from existing specimens and recorded in such a manner that participants can not be identified, directly or through identifiers linked to the participants.

You are required to submit any future revisions to this research activity for prospective IRB review via Modification Request. The IRB will determine whether or not the revision affects the study's Exempt status.

Additionally, the requirement to obtain informed consent has been waived or its elements altered in accordance with 45CFR46.116(d)(1-4) as:

(1) the research involved no more than minimal risk to subjects;
(2) the waiver or alteration will not adversely affect the rights and welfare of the subjects;
(3) the research could not practicably be carried out without the waiver or alteration; and
(4) whenever appropriate, the subjects will be provided with additional pertinent information after participation.

Analyst: Wendy Doggett/4

ANEXO 2

Faculdade de Odontologia de Piracicaba
Universidade Estadual de Campinas

Declaração

As cópias de artigo de minha autoria ou de minha co-autoria, já publicados ou submetidos para publicação em revistas científicas ou anais de congressos sujeitos a arbitragem, que constem da minha Tese de Doutorado, intitulada “Degradação de materiais restauradores resinosos e do esmalte decíduo e permanente em soluções simuladoras da dieta”, não infringem os dispositivos da Lei n$^{o.}$ 9.610/98, nem o direito autoral de qualquer editora.

Piracicaba, 07 de novembro de 2006

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