Cecilia Pedroso Turssi

MICROMORFOLOGIA SUPERFICIAL DE MATERIAIS RESTAURADORES ESTÉTICOS SUBMETIDOS A DIFERENTES PROCESSOS DE DEGRADAÇÃO

Dissertação apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do título de Mestre em Clínica Odontológica, área de concentração em Dentística.

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3. Prof. Dr. MARIO ALEXANDRE COELHO SINHORETI

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>
> *Richard Bach*

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SUMÁRIO

RESUMO

Na cavidade bucal, materiais restauradores estão sujeitos a diversos fenômenos de degradação, desencadeados por processos físicos, químicos e mecânicos, que acometem a micromorfologia superficial das restaurações. Assim, este trabalho, composto por quatro artigos, teve como objetivos: 1) avaliar a micromorfologia superficial de materiais restauradores estéticos imersos em diferentes meios; 2) verificar a influência do meio de armazenamento sobre a rugosidade superficial de materiais restauradores submetidos à escovação; 3) avaliar a rugosidade superficial de materiais estéticos ao longo da simulação de procedimentos de higienização, representados pela escovação, subseqüente a desafios ácidos; 4) analisar o efeito da aplicação de géis fluoretados na micromorfologia superficial de cimentos de ionômero de vidro modificados por resina e de resinas compostas modificadas por poliácidos. Com base nos protocolos experimentais adotados, concluiu-se que materiais restauradores estéticos apresentaram alterações micromorfológicas, quando submetidos a uma condição de alto desafio ácido. A degradação proporcionada por esse modelo, quando seguido pela escovação, desencadeou a um ionômero de vidro modificado por resina e a uma resina composta modificada por poliácidos maior lisura superficial do que quando armazenados em água destilada e deionizada ou saliva artificial, enquanto a textura dos compósitos, conseqüente à escovação, não foi influenciada pelo meio de armazenamento. Ao longo dos ensaios de escovação, precedidos por ciclos de pH, a rugosidade superficial de resinas compostas e de resinas compostas modificadas por poliácidos apresentou-se inalterada. Aumento progressivo, intercalado por períodos de estabilização, foi observado na rugosidade superficial de um ionômero de vidro modificado por resina. Como resultado da aplicação de géis fluoretados, ionômeros de vidro modificados por resina e resinas compostas modificadas por poliácidos apresentaram comportamentos peculiares.

ABSTRACT

In the oral cavity, restorative materials are subject to several degradation phenomena provided by physical, chemical and mechanical processes, which may impair the surface micromorphology of restorations. This study, composed of four manuscripts, had the following objectives: 1) to evaluate the surface micromorphology of aesthetic restorative materials immersed in different media; 2) to verify the influence of the storage medium on the surface roughness of restorative materials subjected to brushing; 3) to evaluate the surface roughness of aesthetic materials throughout brushing, in a highly acidic simulation; 4) to analyze the effect of fluoride gel applications on the surface micromorphology of resin-modified glass ionomers and polyacid-modified composite resins. Under the experimental protocols adopted, it was concluded that aesthetic restorative materials demonstrated micromorphological damage when subjected to a highly acidic simulation. The degradation provided by this model, when followed by brushing, produced smoother surfaces in a resin-modified glass ionomer and a polyacid-modified composite resin than when they were stored either in distilled-deionised water or in artificial saliva, whereas the texture of composites consequent to brushing were not influenced by the storage media. Throughout the brushing simulation preceded by pH-cycling composite resins and polyacid-modified composite showed steady surface textures. A progressive increase in surface roughness, interspersed with periods of stabilization was observed for a resin-modified glass ionomer. As a result of fluoride gel applications both resin-modified glass ionomers and polyacid-modified composite resins showed erratic behaviors.

1. INTRODUÇÃO

A expressividade com que vêm sendo enfocadas filosofias preventivas tem contribuído para a difusão de abordagens que visam à manutenção ou restabelecimento da saúde bucal ao paciente, através da atuação junto aos fatores etiológicos da doença cárie (ELDERTON, 1993; WINSTON & BHASKAR, 1998). Entretanto, procedimentos restauradores, alicerçados em um correto diagnóstico e plano de tratamento, constituem uma manobra clínica que viabiliza a recuperação morfo-funcional e estética das estruturas dentais (TYAS *et al.*, 2000).

As resinas compostas, cimentos ionoméricos e materiais híbridos[1] representam as classes de materiais estéticos utilizadas para a confecção de restaurações diretas, sendo a determinação da aplicação clínica de cada um deles devida, principalmente, às considerações de suas propriedades cariostáticas, mecânicas, físicas e estéticas (BURGESS, 1995; MAGALHÃES *et al.*, 1999; TYAS *et al.*, 2000).

Restaurações confeccionadas com resina composta e materiais híbridos têm demonstrado um desempenho clínico satisfatório (ABDALLA *et al.*, 1997; BROWNING *et al.*, 2000). No entanto, ao longo do tempo, processos de biodegradação podem acarretar alterações na textura superficial desses materiais (ROULET & WÄLTI, 1984; VAN GROENINGEN *et al.*, 1986; SIDHU *et al.*, 1997). A despeito da carência de informações

[1] Denominação genérica atribuída aos cimentos de ionômero de vidro modificados por resina e às resinas compostas modificadas por poliácidos

concretas acerca do limiar de lisura superficial capaz de impedir o favorecimento da colonização e maturação do biofilme bacteriano (STEINBERG *et al.*, 1999), o aumento da rugosidade de restaurações pode implicar maior risco do desenvolvimento de lesões de cárie e inflamação periodontal (QUIRYNEN & BOLLEN, 1995; BOLLEN *et al.*, 1997), sobretudo em restaurações cervicais (DUNKIN & CHAMBERS, 1983). Ademais, há a possibilidade de pigmentação da restauração, acarretando comprometimento estético da mesma (HACHYA *et al.*, 1984).

Dentre os fatores relacionados com o incremento da rugosidade superficial das restaurações localizadas em áreas cervicais, ressaltam-se processos de degradação química (VAN GROENINGEN *et al.*, 1986) e de desgate abrasivo determinados por procedimentos de higiene bucal (ASMUSSEN, 1985; MOMOI *et al.*, 1997; ATTIN *et al.*, 1998), os quais podem agir isolada ou mutuamente (ØILO, 1992, SÖDERHOLM & RICHARDS, 1998).

Embora, em estudos laboratoriais, a sorção de água esteja relacionada com o relaxamento da tensão gerada na interface dente-restauração pela fotopolimerização (FEILZER *et al.*, 1995; SEGURA & DONLY, 1993), em meio aquoso os materiais restauradores podem ser acometidos por erosão superficial, hidrólise ou dissolução de seus componentes (SÖDERHOLM *et al.*, 1984; SÖDERHOLM *et al.*, 1996; CATTANI-LORENTE *et al.*,1999). Além disso, acredita-se que na cavidade bucal tais fenômenos ocorram de maneira mais acentuada (SÖDERHOLM *et al.*, 1996), em função da composição iônica da saliva, da ingestão de alimentos e bebidas ácidas e da presença de ácidos provenientes do metabolismo bacteriano (GEURTSEN *et al.*, 1999; YAP *et al.*, 2000).

Considerando que, com a ingestão de carboidratos fermentáveis, há o declínio do pH do biofilme bacteriano pela produção de ácidos (ERICSON & HARDWICK, 1978; KIDD, 1995), existe a possibilidade de degradação da superfície e subsuperfície de materiais resinosos (WU *et al.*, 1984; CHADWICK *et al.*, 1990). Para avaliar a textura desses materiais sob uma condição de alto desafio ácido, em comparação ao armazenamento em água destilada e deionizada ou saliva artificial, foi realizado o trabalho *"Effect of storage media upon the surface micromorphology of resin-based restorative materials"*, apresentado no Capítulo 1.

Uma maneira de minimizar a duração desses adventos de queda de pH é através da escovação dental (ERICSON & HARDWICK, 1978). Estudos têm sido conduzidos com a finalidade de se verificar a influência dessa interação no incremento da degradação de restaurações (ATTIN *et al.*, 1998; CHADWICK *et al.*, 1990). Contudo, resultados contraditórios têm sido apresentados, o que justificou a condução de uma pesquisa visando à definição da influência do meio de armazenamento – água destilada e deionizada, saliva artificial e soluções que simulam um modelo dinâmico de ciclos de pH – na rugosidade superficial de materiais resinosos submetidos à escovação, intitulada "*Influence of storage media on roughness of aesthetic materials subjected to brushing*", constante no Capítulo 2.

Tendo em vista a possibilidade da lisura superficial de restaurações submetidas à escovação ser influenciada por uma condição de alto desafio ácido [Capítulo 2], através de um estudo laboratorial, propôs-se a avaliação do comportamento de resinas compostas – híbrida e de micropartículas – e de materiais híbridos – um cimento de ionômero de vidro modificado por resina e duas resinas modificadas por poliácidos – ao longo de ensaios de

escovação, que retrataram dez anos de uso clínico desses materiais (GOLDSTEIN & LERNER, 1991), subseqüentes à simulação de um modelo dinâmico de ciclos de pH. O artigo "*Surface roughness assessment of resin-based materials throughout brushing preceded by pH-cycling simulations*" [Capítulo 3] apresenta a metodologia e resultados desse estudo.

Procedimentos de higienização, no entanto, podem não ser adequadamente realizados pelo paciente, representando fator de risco ao desenvolvimento de cárie adjacente à restaurações (MJÖR & TOFFENETTI, 2000). Nesse contexto, diferentes fontes de fluoreto podem ser utilizadas de forma vantajosa em pacientes com moderado e elevado risco de cárie (ANUSAVICE, 1998; STOOKEY, 1998; WINSTON & BHASKAR, 1998). Uma dessas fontes são os géis fluoretados, cujos mecanismos de ação os tornam coadjuvantes na prevenção de lesões primárias e secundárias de cárie (ØGAARD *et al.*, 1994).

Comparado ao fluoreto de sódio neutro, a administração de géis acidulados promove a formação de maior quantidade de fluoreto de cálcio sobre o esmalte (RETIEF, 1983; ØGAARD, 1990), bem como incrementa a aquisição de flúor[2] e conseqüente padrão de liberação desse íon pelos cimentos de ionômero de vidro convencionais e materiais híbridos (DIAZ-ARNOLD *et al.*, 1995). Porém, tem sido relatado que ácidos presentes na composição dos géis acidulados podem desencadear a erosão química e degradação superficial desses materiais (EL-BADRAWY *et al.*, 1993; EL-BADRAWY & MCCOMB, 1998). Entretanto, nesses estudos não se tem considerado condições inerentes aos pacientes de alto risco ou

[2] Termo genérico para definir as formas iônica e ionizável do elemento flúor

atividade de cárie – para os quais estaria indicada a administração de géis fluoretados – que podem desencadear, por si sós, alterações micromorfológicas aos materiais restauradores, como será apresentado no Capítulo 1. Assim, o artigo "*Effect of fluoride gels on micromorphology of resin-modified glass ionomer cements and polyacid-modified composite resins*" [Capítulo 4] viabilizou a avaliação das características superficiais de materiais híbridos frente à aplicação de géis fluoretados, sob uma condição experimental que simulou alto desafio cariogênico.

Embora experimentações controladas não substituam pesquisas clínicas, as mesmas permitem inferir a respeito de mecanismos básicos envolvidos na complexidade inerente aos processos de biodegradação (MAIR *et al.*, 1996). Além disso, modelos laboratoriais podem ser considerados mais condizentes com a constante evolução dos materiais restauradores, à medida que permitem a verificação de suas propriedades de maneira mais breve e menos onerosa.

2. PROPOSIÇÃO

O presente trabalho, composto por quatro artigos, teve como objetivo geral avaliar, através de modelos experimentais *in vitro*, a micromorfologia superficial de materiais restauradores estéticos submetidos a diferentes processos de degradação. Os objetivos específicos foram:

1) avaliar, através de rugosimetria, a micromorfologia superficial de materiais restauradores estéticos imersos em diferentes meios;

2) verificar a influência do meio de armazenamento sobre a rugosidade superficial de materiais restauradores estéticos submetidos à escovação;

3) analisar a rugosidade superficial de materiais estéticos ao longo da simulação de procedimentos de higienização, representados pela escovação, subseqüente a desafios ácidos;

4) avaliar, através de microscopia eletrônica de varredura, o efeito da aplicação de géis fluoretados na micromorfologia superficial de cimentos de ionômero de vidro modificados por resina e de resinas compostas modificadas por poliácidos.

Effect of Storage Media upon the Surface Micromorphology of Resin-based Restorative Materials

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Summary

The aim of this study was to evaluate the effect of different storage media upon the surface micromorphology of resin-based restoratives. One resin-modified glass-ionomer (Fuji II LC Improved), one polyacid-modified composite resin (Dyract AP), one microfilled composite resin (Durafill VS), and one hybrid composite resin (Filtek Z250) were tested. For each material, forty-five standardized cylindrical specimens were randomly made. After 24 h, samples were finished and polished, and their surface roughness measured to obtain Ra (μm) baseline values *(Bv)*. Fifteen specimens of each material were then stored at 37 ± 1° C, for 24 hours, either in distilled deionised water or in artificial saliva, or else subjected to a pH-cycling regimen. At the end of 10 days of storage, final readings *(Fv)* of surface roughness were obtained. The Analysis of Covariance ($\alpha = 0.05$), considering the covariate *Bv* showed a significant interaction between Restorative Materials and Storage Media (p_{value}=0.0000). Multiple Comparison Tukey's Test revealed that the surface roughness of resin-based restoratives subjected to a pH-cycling model was significantly higher compared with both distilled deionised water and artificial saliva. Micromorphological changes were noticed in a situation of acid challenge.

Introduction

One factor, which has an appreciable influence on the satisfactory clinical performance of dental restorations, is their resistance to biodegradation (Øilo, 1992). In the oral cavity, this process includes diverse phenomena, such as sliding, abrasion, chemical degradation, and fatigue (Söderholm & Richards, 1998). These mechanisms may operate either alone or in combination with others and, considering the intricacy of the oral environment, the breakdown of dental materials mediated by biological activity is very complicated (Øilo, 1992).

A detrimental effect on the physical properties of resin-based restorative materials as a consequence of water sorption has been reported (Sarret & Ray, 1994; Cattani-Lorente *et al.*, 1999a, b), since water erodes the surface and causes hydrolysis and dissolution of some of their components (Söderholm *et al.*, 1984; Söderholm, Mukherjee & Longmate, 1996; Cattani-Lorente *et al.*, 1999a). However, it has been suggested that the oral environment is likely to cause more pronounced filler degradation than indicated by storage in distilled water (Söderholm *et al.*, 1996). Thus, the changes observed occurring in distilled water do not necessarily take place to the same extent in the mouth (Anstice & Nicholson, 1992).

On exposure to plaque acids, food-simulating constituents, and enzymes, resin-based restorative materials can undergo softening (Asmussen, 1984; Ferracane & Marker, 1992). An increase of their wear rate has also been demonstrated (Wu & McKinney, 1982; Chadwick *et al.*, 1990; de Gee *et al.*, 1996). Moreover, under oral conditions, in the absence of mechanical forces, chemical processes or dissolution can produce an increase of surface roughness (Roulet & Wälti, 1984; van Groeningen, Jongebloed & Arends, 1986). These

undesirable damages have been associated with chemical degradation of the surface and subsurface (Wu *et al.*, 1984; Chadwick *et al.*, 1990), which may involve either the resin matrix, the filler, or the matrix-filler interface (Söderholm & Richards, 1998).

In view of the degradability of resin-based restorative materials by chemical processes (Roulet & Wälti, 1984; van Groeningen *et al.*, 1986; Lavis *et al.*, 1997; Nicholson, Czarnecka & Limanowska-Shaw, 1999a), research is required to evaluate the interaction of these materials with their surrounding medium. In this context, although clinical trials are the ideal method of predicting restoration behavior, laboratory studies may be useful in providing information on the fundamental mechanisms of degradation (Mair *et al.*, 1996). In this way, this *in vitro* study aimed to assess the effect of storage media upon the surface micromorphology of resin-based restorative materials.

Materials and methods

Experimental design. The factors under study were Restorative Materials at four levels and Storage Media at three levels. The association between restorative materials and storage media resulted in twelve groups. The experimental sample comprised 180 specimens (n=15), made in a random sequence. The response variable was surface micromorphology evaluated by means of a roughness measuring instrument.

Specimen Preparation. The restorative materials used in this study are given in Table 1. Each material was handled as outlined by the manufacturers and was bulk placed into a cylindrical stainless steel matrix (4.0 mm inside diameter and 2.0 mm thick). The surface of the restorative materials was covered with a polyester strip and a glass slab. For 30 seconds, a load of 500 g was applied to expel excess material from the matrix. The polymerization

procedure was carried out through the polyester strip for the manufacturers' recommended exposure time, using a light-curing device (Optilux 401*). The light intensity was monitored periodically with a Curing Radiometer† and it ranged from 400 to 520 mW/cm^2. After 24 hours of storage at 37 ± 1° C in 100% relative humidity, specimens were finished and polished using medium, fine, and superfine aluminum oxide abrasive disks (Sof-Lex Pop On‡). Each instrument was applied in only one direction for 15 seconds. All samples were flushed with air-water spray between each disk, and at the end of the procedure they were kept in an ultrasonic§ bath in distilled deionised water for 10 minutes to remove polishing debris.

Baseline surface roughness measurements. Surface roughness analyses were done by means of a surface roughness measuring instrument€, using a diamond stylus tip of 2 μm radius, which traversed the surface at a constant speed of 0.05 mm/s with a force of 0.7 mN. The cut-off value was set at 0.08 mm (Gauss Filter). The surface roughness was characterized by the height parameter, Ra (μm), the arithmetical mean of the absolute values of the profile departures within the evaluation length. For mean surface roughness, the pick-up of the measuring instrument tracked over the surface at three locations in each direction – parallel, perpendicular, and oblique to the finishing and polishing scratch directions – amounting to

* Demetron / Kerr Corp, Danburry, USA
† Model 100, Demetron / Kerr Corp, Danburry, USA
‡ 3M, St Paul, MN, USA
§ Model T1440D, Odontobrás Ltda, Ribeirão Preto, São Paulo, Brazil
€ Model Surfcorder SE- 1700, Kosaka Corp, Tokyo, Japan

nine tracings per sample. An average of these nine mean surface roughness values was used as a measurement for each sample.

Storage protocols. According to the assigned group, specimens were stored either in distilled deionised water or artificial saliva, or else subjected to a severe acid challenge, as proposed by Featherstone *et al,* 1986 and modified by Serra & Cury (1992). The artificial saliva consisted of 1.5 mM of calcium, 0.9 mM of phosphate, and 150.0 mM of potassium chloride in a buffer solution of 0.1 mM of Tris (hydroxymethyl-aminomethane) at pH 7.0. The acid solution contained 2.0 mM of calcium and 2.0 mM of phosphate in a buffer solution of 74.0 mM of acetate at pH 4.3. For groups stored either in distilled deionised water or in artificial saliva, the specimens were immersed in an individual vial containing 75 ml of these respective media for 24 hours at 37 ± 1° C. For groups subjected to the dynamic pH-cycling model, the samples were firstly immersed in 75 ml of the acid solution for 6 hours at 37 ± 1° C, rinsed with distilled deionised water, and then stored in 75 ml of the artificial saliva by 18 hours at 37 ± 1° C. These regimens of storage were repeated for 10 uninterrupted days.

Final surface roughness measurements. Specimens were subjected to final readings of surface roughness. The measurement procedure was similar do that for the baseline condition.

Environmental scanning electron microscope examination. Representative samples of each experimental group were selected for microscopic examination. Specimens were

mounted on aluminum stubs and observed without any preparation at X3000 magnification using an environmental scanning electron microscope[¥] (ESEM).

Statistical Analysis. For each specimen, the mean of the nine baseline measurements (*Bv*) and the mean of the nine final measurements (*Fv*) were considered for statistical analysis. The Analysis of Covariance (ANCOVA) at the 95% confidence level was used to compare means among experimental groups, considering the covariate *Bv*. A study of the interaction among the factors analyzed (Restorative Materials and Storage Media) was made. Multiple Comparison Tukey's test was used to identify significant difference in means of Material-Media interaction. The software Statgraphics® Plus[¤] was used to perform the statistical calculations.

Results

The adjusted means and standard errors of the final values of surface roughness (*Fv*) of each one of the twelve groups are given in Table 2. The Analysis of Covariance showed a significant interaction between Restorative Materials and Storage Media ($p_{value} = 0.0000$). Since the interaction was significant a comparison of the behavior of the different materials in each medium and the effect of the different media on each material could be made. A Multiple Comparison Tukey's test showed that all restorative materials stored both in distilled deionised water and in artificial saliva had significant lower surface roughness than they had done when they were subjected to the pH-cycling model. No statistically significant difference in surface roughness existed between distilled deionised water and artificial saliva

[¥] Model Leo 435 VP, Leo Microscopy Ltd., Cambridge, England
[¤] Manugistics, Rockville, Maryland, USA

groups. In Table 2, surface roughness values showing no statistical differences are connected by brackets. The Figs 1 through 4 illustrate the surface characteristics of each group.

Within the factor Storage Media, in distilled deionised water, Durafill VS presented a smoother texture than Filtek Z-250, which did not differ from Dyract AP, whereas Fuji II LC showed the roughest surface. As a consequence of both the storage in artificial saliva and performing pH-cycling, all restorative materials showed statistically significant difference from each other regarding their surface roughness. Durafill VS had the lowest surface roughness, followed by Filtek Z-250, which had a smoother texture than Dyract AP. Fuji II LC presented the roughest surface. These results are indicated in Table 2, where means designated with different superscript letters are statistically different.

Discussion

In attempting to understand the phenomena involved in the biodegradation of resin-based restorative materials, *in vitro* (Chadwick *et al.*, 1990; Söderholm *et al.*, 1996; de Gee *et al.*, 1996; Gao *et al.*, 1997; Cattani-Lorente, 1999b), *in situ* (Roulet & Wälti, 1984), and *in vivo* (van Groeningen *et al.*, 1986; Sidhu, Sherriff & Watson, 1997) experiments have been carried out. In spite of the mouth being the ultimate testing environment for predicting the behavior of restorations (Mair *et al.*, 1996), because of the complexity and diversity of intra-oral conditions, *in vitro* models may be most important in providing an insight into the fundamental mechanisms of biodegradation. Hence, considering that water or other chemicals available in the oral cavity can account for the breakdown of restorations (Roulet & Wälti, 1984), this study was performed under controlled experimental conditions in order

to highlight the influence of the surrounding media upon the micromorphology of resin-based restoratives.

Some *in vitro* investigations have been made to predict the effect of plaque acids on degradation of resin-based restorative materials (Chadwick *et al.*, 1990; Gao *et al.*, 1997; Lavis *et al.*, 1997). However, in such experiments these materials have been immersed in acid media during extensive and uninterrupted periods, overestimating the amount of time the plaque remains acid. In this study, in an attempt to simulate the clinical situation more closely, a dynamic pH-cycling model was employed, as proposed by Featherstone *et al.* (1986) and modified by Serra & Cury (1992). This regimen incorporates six hours of acid challenge daily, which is a reasonable estimate for subjects who snack sugar frequently (Featherstone *et al.*, 1986).

The degradability of resin-based restorative materials by chemicals has been evaluated by various methods such as microhardness (van Groeningen *et al.*, 1986; Chadwick *et al.*, 1990), surface roughness (Roulet & Wälti, 1984), profile tracings (de Gee *et al.*, 1996), weight change (Lavis *et al.*, 1997; Nicholson *et al.*, 1999a,b), leaching of filler elements (Söderholm et al., 1996), scanning electron microscopy (Gao *et al.*, 1997), and polarized light microscopy (Wu *et al.*, 1984). In the present study, surface roughness assessment was chosen because it is well documented that surface micromorphology can play a role in bacterial colonization and in maturation of plaque on restorative materials (Quirynen & Bollen, 1995). Although the effect of surface properties on these phenomena have been reported as contradictory (Quyrinen & Bollen, 1995), these interactions may predispose a restoration to the development of secondary caries and may lead to periodontal inflammation (Bollen, Lambrechts & Quirynen, 1997).

The surface roughness of restorative materials subjected to chemical attacks has been previously reported by Roulet & Wälti (1984) and van Groeningen *et al.* (1986). Since it is a non-destructive method, repeated measurements of surface roughness – before and after specimen storage – could be performed. Thus, in the statistical analysis the final surface roughness measurement (*Fv*) of each specimen was adjusted by its baseline roughness value (*Bv*), that is, *Bv* was considered a covariate. This was possible since a relatively strong relationship (ρ = 0.98) between the response variable (*Fv*) and the covariate (*Bv*) was verified. Analysis of Covariance provided substantial increase in precision of this randomized investigation through residual variance reduction (Cox & McCullagh, 1982).

According to the results of this study, all restoratives investigated became significantly rougher after they have been subjected to the pH-cycling regimen. This can be ascribed to the capability of acid media to soften resin-based restorative materials (Asmussen, 1984; Chadwick *et al.*, 1990). Furthermore, it has been previously reported that acids can provoke loss of surface integrity (Gao *et al.*, 1997; Nicholson *et al.*, 1999b). It was hence expected that an increase in their surface roughness would occur, which was substantiated by Figs. 1 to 4.

Based on ESEM, following the regimen of acid challenge, the surface of the microfilled composite resin (Fig. 1c) revealed protruding particles probably as a result of matrix degradation. On the other hand, for the hybrid composite and the polyacid-modified composite resin, as can be seen from Fig. 2c and 3c, the resin matrixes showed particles voids, which might be attributed to degradation of the surrounding resin matrix or silane coupling agent. Unlike the other materials, for the resin-modified glass-ionomer cement

matrix dissolution was detected peripheral to the glass particles, which could be due to dissolution of the siliceous hydrogel layer (El-Badrawy & Mc Comb, 1998).

In spite of acids adversely prejudicing the surface integrity of resin-based restoratives, it must be pointed out that for both resin-modified glass-ionomer and polyacid-modified composite resin, this erosive loss of material may be accompanied by an increase in pH of the acid solution (Nicholson *et al.*, 1999a,b; Nicholson *et al.*, 2000). Such buffering effect is likely to be beneficial in protecting restored teeth from the development of secondary caries. Moreover, clinically, within a short time, restorations will be covered by a biofilm, which probably changes the diffusion on their surfaces (Øilo, 1992).

Different methodologies have been used to evaluate the degradability of resin-based restoratives stored in artificial saliva. It has been suggested that storage in this solution causes more pronounced filler degradation of composite resins than does distilled water (Söderholm *et al.*, 1996). In addition, resin-modified glass ionomers immersed in artificial saliva have presented significantly lower surface microhardness than the same materials stored in distilled water (Kanchanavasita, Anstice & Pearson, 1998). By contrast, Lavis *et al.* (1997) reported that specimens of a polyacid-modified composite resin immersed in either distilled water or in a solution that roughly approximates the concentrations of calcium and phosphate ions in saliva at pH 7, showed similar weight changes and surface disintegration under SEM examinations. Similarly, in the present study the surface roughness results of distilled deionised water and artificial saliva groups did not differ from each other, as can be seen from Figs. 1a and b, 2a and b, 3a and b, and 4a and b.

Independently of the storage conditions, the lowest surface roughness was measured for the microfilled composite resin. This may be attributed to its larger pre-polymerized

blocks, as can be seen in Figs 1. The hybrid composite showed lower surface roughness than the polyacid-modified composite resin, but in distilled deionised water they did not differ from each other. Actually, Dyract AP has been regarded as most closely approximating composite resins (Gladys *et al.*, 1997). In addition, the filler particles of composite resins and polyacid-modified composite are treated during manufacturing, with a silane coupling agent to bond the filler chemically to the resin matrix (Gladys *et al.*, 1997; Meyer, Cattani-Lorente & Dupuis, 1998), which may account for their hydrolytic stability (Söderholm *et al.*, 1984). Unlike, the entanglement of the cross-linked polyacrylate network and the polymer chain of the resin-modified glass ionomer cement may not be sufficiently coherent (Kanchanavasita *et al.*, 1998), favoring its micromorphological damages (Fig. 4).

Under the experimental conditions adopted in this study, it was concluded that the resin-based restoratives underwent greater micromorphological damages following the regimen of acid challenge than after storage either in distilled deionised water or artificial saliva.

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Table 1. Restorative materials studied, their respective manufacturers, compositions, mean particle size, and batch #

Material	Brand Name	Manufacturer	Basic Composition*	Mean particle size (μm)*	Batch #
microfilled composite resin	Durafill VS (Df)	Heraeus Kulzer	Colloidal silica, BIS-GMA, TEGDMA, UDMA	0.02 – 0.04	40
hybrid composite resin	Filtek Z250 (Z2)	3M	Zirconia, silica, TEGDMA, UDMA, BIS-EMA	0.60	9BP and 9BX
polyacid-modified composite resin	Dyract AP (Dy)	Dentsply DeTrey	Strontium-fluoro-silicate glass, strontium fluoride, polymerizable resins, TCB resin	0.80	9812000362
resin-modified glass-ionomer cement	Fuji II LC (Fj)	GC Corp	Aluminum-fluoro-silicate glass, HEMA, tartaric acid, polyacrylic acid	1.80	020781

*As disclosed by the manufacturers
BIS-GMA = bisphenol glycidyl methacrylate; TEGDMA = tetraethyleneglycol dimethacrylate; UDMA = urethane dimethacrylate; BISEMA = Bisphenol A polyetheylene glycol diether dimethacrylate; HEMA =hydroxyethyl methacrylate

Table 2. Results of Multiple Comparison Tukey's test for Ra (μm) to identify differences between material-storage media interaction.

Medium \ Material	Du	Z2	Dy	Fj
Distilled deionised water	0.0477 (0.0047) [a]	0.0760 (0.0024) [b]	0.0819 (0.0021) [b]	0.1671 (0.0074) [c]
Artificial saliva	0.0489 (0.0045) [a]	0.0713 (0.0022) [b]	0.0792 (0.0020) [c]	0.1681 (0.0070) [d]
Acid challenge condition	0.0614 (0.0045) [a]	0.0913 (0.0023) [b]	0.1019 (0.0022) [c]	0.2075 (0.0074) [d]

Standard errors are given between parentheses

Means connected by vertical brackets did not differ from each other. Statistically significant differences within rows are shown by different superscript letters ($p < 0.05$)

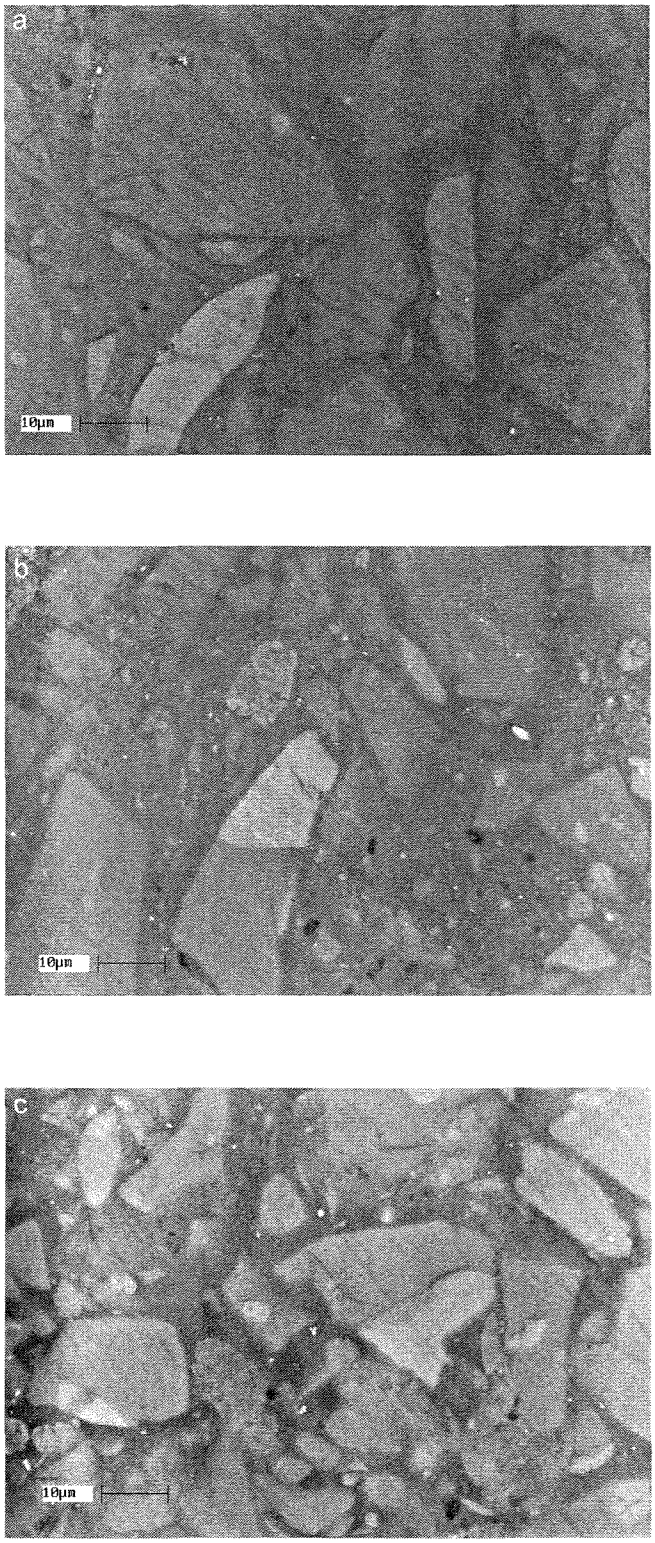

Fig. 1. Surface appearance of Durafill VS after storage in distilled deionised water (a), immersion in artificial saliva (b), and pH-cycling regimen (c)

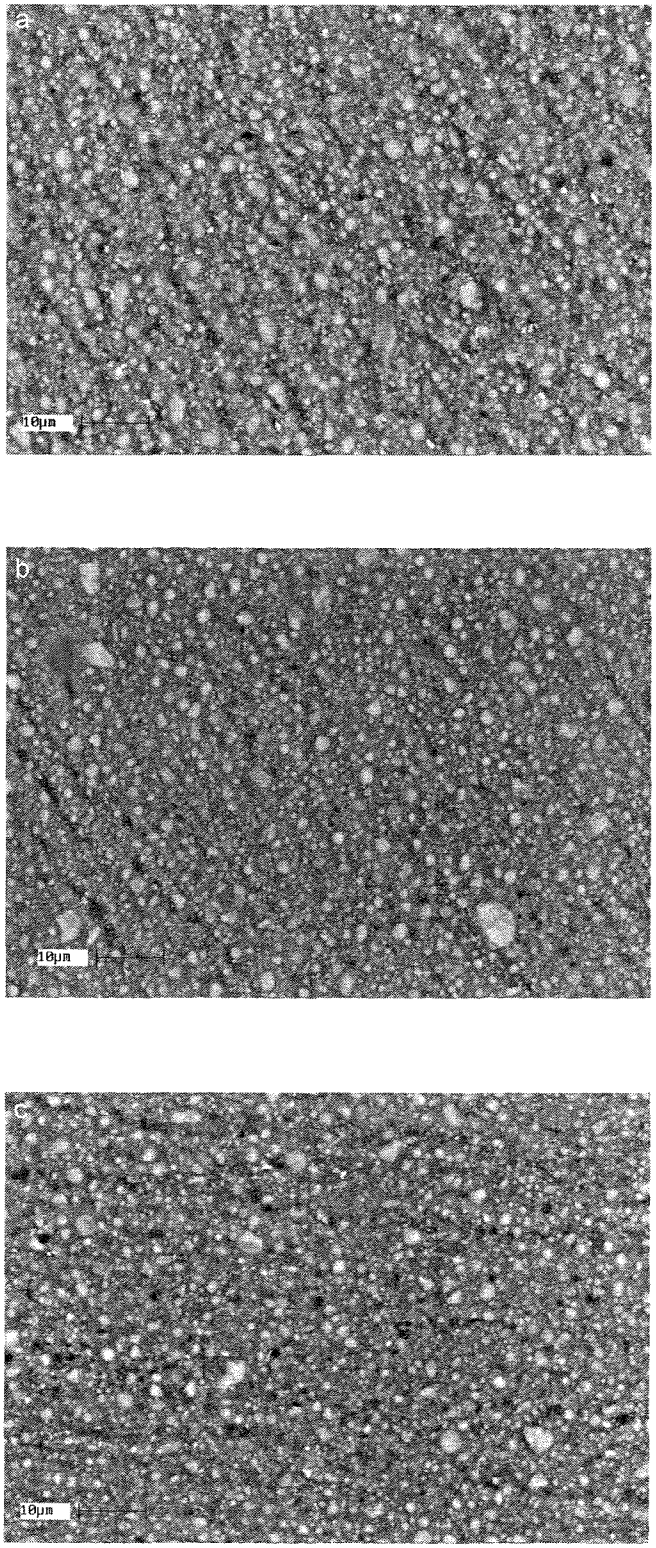

Fig. 2. Micromorphologycal pattern of Filtek Z-250 after storage in distilled deionised water (a), immersion in artificial saliva (b), and pH-cycling model (c)

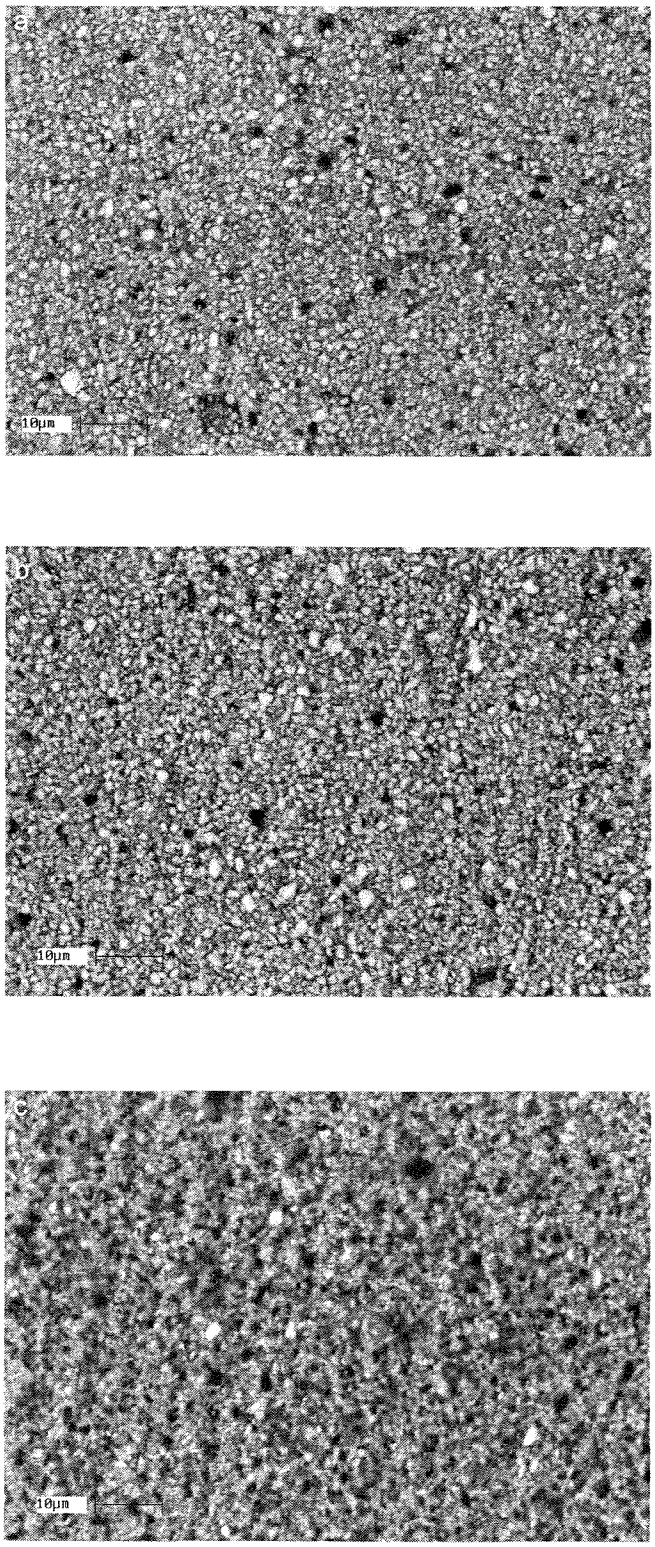

Fig. 3. Surface roughness pattern of Dyract AP after storage in distilled deionised water (a), immersion in artificial saliva (b), and pH-cycling regimen (c)

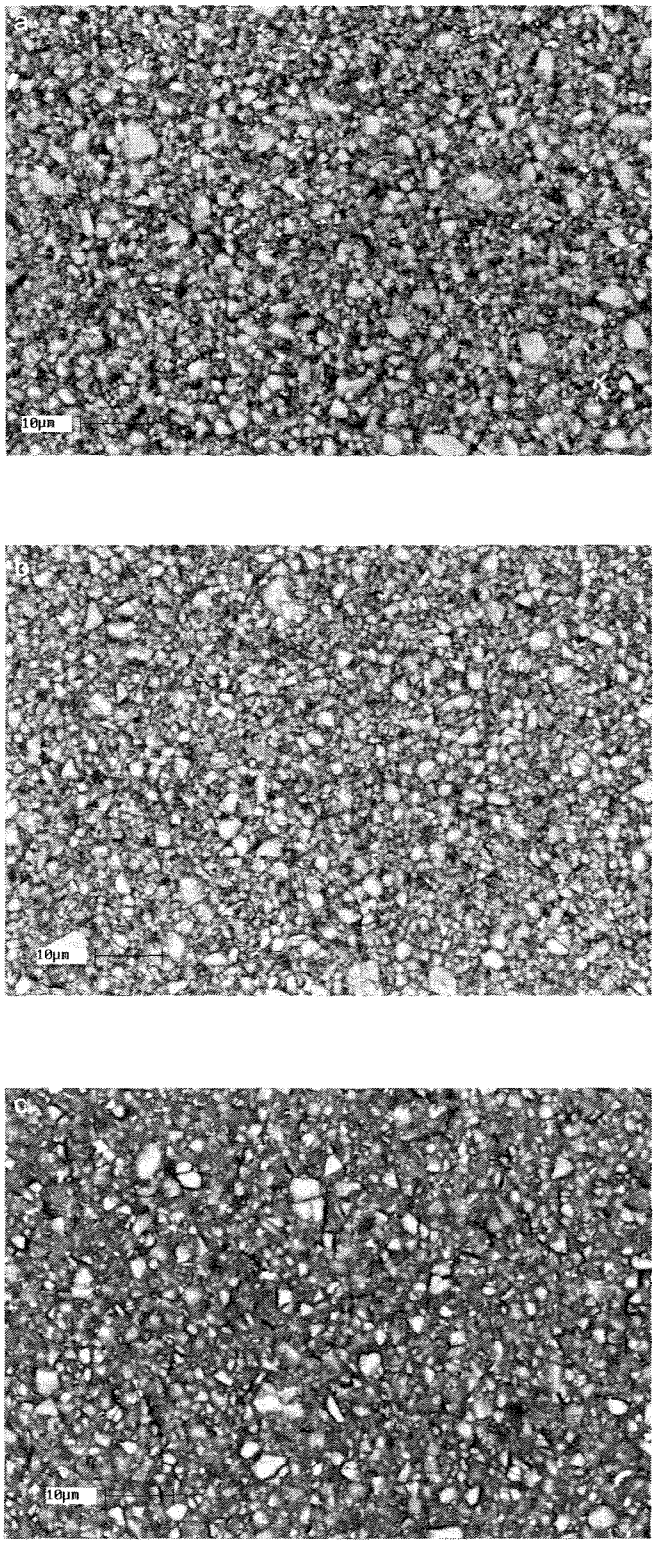

Fig. 4. Surface micromorphology of Fuji II LC following storage in distilled deionised water (a), immersion in artificial saliva (b), and pH-cycling model (c)

Influence of storage media on roughness of aesthetic materials subjected to brushing

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SHORT TITLE: Brushing of aesthetic materials stored in various media

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ABSTRACT

Objective. This study evaluated the surface roughness of direct restorative materials and composite resins subjected to brushing after storage in various media.

Methods. One resin-modified glass-ionomer (Fuji II LC Improved/GC Corp), one polyacid-modified composite resin (Dyract AP/Denstply De Trey), one microfill composite (Durafill VS/Kulzer GmbH), and one hybrid composite (Filtek – Z250/3M) were tested. Forty-five standardized cylindrical specimens of each material were randomly prepared and assigned into 3 groups according to the storage media: distilled de-ionized water (Dw), remineralizing solution (Re) and, de- and remineralizing solutions (De-Re). After 24 h, the specimens were finished and polished and surface roughness was measured to obtain Ra baseline values *(Bv)*. Storage in media was done for 24 h, after which samples were subjected to 10,000 brushing strokes. By the end of 10 repetitions of this protocol, final readings *(Fv)* of surface roughness were carried out.

Results. The Analysis of Covariance (α=0.05), considering the covariate *Bv* showed a significant interaction between Restorative Material and Storage Media (p_{value}=0.0002). Multiple Comparison Tukey's Test used to identify the material-media interaction revealed that for Fuji II LC and Dyract AP, De-Re cycling resulted in a significantly lower surface roughness, in comparison with the other media. For both composites no significant difference among groups was detected.

Significance. After storage in De-Re, surface roughness of the resin-modified glass ionomer and the polyacid-modified composite resin subjected to brushing is lower than that provided by storage in Dw and Re, whereas this pH-cycling model does not affect the roughness of composites.

KEYWORDS

Brushing, storage media, composite resins, resin-modified glass-ionomer cements, polyacid-modified composite resin, surface roughness

INTRODUCTION

Advances in the scientific approach to dental caries have led to an appreciation of the need for a reappraisal of the role of restorative dentistry in caries management. Based on the diagnosis of caries risk or activity, preventive care rather than the traditional surgical model can be established [1, 2]. Nevertheless, restorative interventions may be necessary and therefore integrated as part of the treatment planning [3].

The clinical longevity of any restorative materials may be attributed to various factors, including its resistance to wear in the oral environment [4]. In spite of the mechanism of *in vivo* wear is highly complex, it is suggested that interactions between several material-specific features and biochemical, biophysical, and mechanical processes are involved [5-8].

Although it is not the only wear mechanism in the oral cavity, abrasion probably constitutes an important consideration in the total wear process [9] and occurs in different ways in the mouth, depending on the site of the restoration [6]. Class III and V restorations, for instance, may be jeopardized predominantly by toothbrushing abrasion [10-12].

Various devices have been developed in order to enable prediction of brushing abrasion of restorative materials [13-15], however many of the tests performed do not adequately simulate the conditions existing in the mouth. Furthermore, there is a lack of information on their behavior after exposure to neutral and acidic media, since in the oral environment chemicals can change the surface and subsurface integrity of restorative materials [16].

Considering that microstructure changes may be shown by restorative materials after toothbrushing abrasion, various methods, such as weight change monitoring [14, 17, 18],

surface-profile evaluations [11, 12], and surface roughness measurements [19] have been employed to make possible the assessment of their performance.

It was the purpose of this investigation to evaluate the surface roughness of direct restorative materials subjected to brushing after storage in various media.

MATERIALS AND METHODS

Experimental design. The factors under study were restorative materials at four levels and storage media at three levels. The experimental sample were 180 specimens randomly assigned into twelve groups of 15 samples each. The order in which the samples were made was randomly determined. The response variable was surface micromorphology evaluated by means of roughness measurements.

Specimen Preparation. The restorative materials used in this study are indicated in Table 1. Stainless steel matrixes (4.0 mm inside diameter and 2.0 mm thick), marked with a bur on their external side allowing for polishing and brushing in a standardized manner, were used. Manufacturers' directions were followed for materials handling. The matrixes were slightly overfilled with the materials, in one increment, covered with a polyester strip and a glass slab, and pressed with a weight of 500 g for 30 seconds to extrude any excess. After the load and the slab had been removed, the specimens were cured using a visible light-curing unit (Optilux 401, Demetron / Kerr Corp, Danburry, USA) for the manufacturers' recommended exposure time (*Fig 1A*). The light intensity was monitored periodically in a radiometer (Curing Radiometer, Model 100, Demetron / Kerr Corp, Danburry, USA), and it ranged from 400 to 520 mW/cm^2. Subsequently the samples were wrapped up in gauze

soaked with 2.5 ml of distilled de-ionized water and stored at 37 ± 1° C. Twenty-four hours after, finishing and polishing procedures were performed using the sequence of medium, fine, and superfine aluminum oxide disks (Sof-Lex Pop On, 3M, St Paul, MN, USA). Each disk was applied in only one direction for 15 seconds (*Fig 1B*). All samples were flushed with air-water spray between each grit, and at the end of the procedure were cleaned ultrasonically (T1440D, Odontobrás Ltda, Ribeirão Preto, São Paulo, Brazil) in distilled de-ionized water during 10 minutes for removal of any remaining debris.

Surface roughness measurements. Surface roughness analyses were done quantitatively by means of a surface roughness-measuring instrument (Surfcorder SE- 1700, Kosaka Corp, Tokyo, Japan), using a diamond stylus tip of 2 μm radius, which traversed the surface at a constant speed of 0.05 mm/s with a force of 0.7 mN. The surface roughness was characterized by the height parameter, Ra (μm), the arithmetical mean of the absolute values of the profile departures within the evaluation length. For mean surface roughness, the pick-up of the measuring instrument tracked over the surface at three locations in each one of the directions, parallel, perpendicular, and oblique to the finishing and polishing scratch directions, amounting to nine tracings for each sample (*Fig 1C*). An average of these nine mean surface roughness values was used as a measurement for that sample. The cut-off value was set at 0.08 mm (Gaus Filter).

Storage and brushing protocols. The samples were subjected to three different regimen of storage, according to the assigned group: distilled de-ionized water (Dw), remineralizing solution (Re), and a dynamic pH-cycling model with de- and remineralizing solutions (De–Re). The remineralizing solution consisted of 1.5 mM of calcium, 0.9 mM of

phosphate, and 150.0 mM of potassium chloride in a buffer solution of 0.1 mM of Tris (hydroxymethyl-aminomethane) at pH 7.0. The demineralizing solution contained 2.0 mM of calcium and 2.0 mM of phosphate in a buffer solution of 74.0 mM of acetate at pH 4.3. For Dw and Re groups, the specimens were individually stored in 5.0 ml of the respective media for 24 hours at 37 ± 1° C. For De-Re group, the samples were firstly immersed in the 5 ml of demineralizing solution for 6 hours at 37 ± 1° C, rinsing with distilled de-ionized water, and then stored in 5ml of remineralizing solution for 18 hours at 37 ± 1° C, simulating a high cariogenic challenge, as proposed by Featherstone *et al.* [20] (*Fig 1D*). Afterwards, all samples were taken from their storage media and wrapped up in gauze soaked with distilled de-ionized water.

Subsequently, brushing abrasion of the specimens was performed with an automatic toothbrushing machine (*Fig 2*), which consisted of a motor that imparted reciprocating motion to ten soft nylon bristle toothbrushing heads (Oral-B 40, Gillette do Brasil Ltda., Manaus, Amazonas, Brazil). The machine was equipped with a stainless-steel base with ten independent appliances for positioning the specimens. Special devices, which fixed the head of the toothbrushes, allowed alignment of the base parallel to the head of the toothbrush. The stroke length was adjustable according to sample dimension. Moreover, simulated brushing was performed in a thermostatically controlled environment to simulate the temperature encountered in the mouth (37 ± 0.5° C). The abrasive slurry (dentifrice plus distilled de-ionized water) was independently and simultaneously injected beside each brush. The frequency and volume of these injections were programmable according to sample dimensions.

For brushing simulation, the specimens were positioned in the appliances with the bur mark exactly in the same direction used for finishing and polishing (*Fig 1D*). Since the appliances were specially designed for the samples used in this study, no material was necessary to fix the specimens and to obtain the alignment with the base. The brush heads were identified and used for brushing only their respective specimens for 50,000 strokes. The stroke length was calibrated for 20 mm. Ten thousand strokes were performed at a speed of 4.5 strokes per second and at a load of 300 g, in the presence of an abrasive slurry. The slurry consisted of a dentifrice (Colgate MFP, Colgate Palmolive – Division of Kolynos do Brasil Ltda, Osasco, São Paulo, Brazil) and distilled de-ionized water in the rate 1:3 by weight. This slurry was independently injected beside each brush at a frequency of 0.4 ml at two-minute intervals. After the brushing abrasion, specimens were again wrapped up in gauze soaked with distilled and de-ionized until they were subjected to a new storage period, performed like the first one. Storage regimens interspersed with brushing abrasion simulations were performed 10 times consecutively, amounting to 100,000 brushing strokes. Afterwards, surface roughness measurements were again obtained, as described for baseline condition (*Fig 1E*).

Statistical Analysis. For each specimen, the mean of the nine baseline measurements (*Bv*) and the mean of the nine final measurements (*Fv*) were considered for statistical analysis. The Analysis of Covariance (ANCOVA) at the 95% confidence level was used to compare means among experimental groups, considering the covariate *Bv*. A study of the interaction among the factors analyzed (restorative materials and storage media) was made. Multiple Comparison Tukey's test was used to identify significant difference in means of

material-media interaction. The software Statgraphics® Plus (Manugistics, Rockville, Maryland, USA) was used to perform the statistical calculations.

RESULTS

Since a relatively strong relationship between *Bv* and *Fv* ($\rho = 0.94$) was detected, the Analysis of Covariance provided substantial increase in precision of this randomized study through residual variance reduction [21]. The adjusted means (standard error) of the surface roughness *Fv* of each one of the twelve groups are given in table 2 and depicted in figure 3. The Analysis of Covariance showed a significant interaction between Restorative Material and Storage Media ($p_{value} = 0.0002$). Since the interaction was significant a comparison of the behavior of the different materials in each medium and the effect of the different media in each material could be made. Within the factor storage media, all restorative materials showed statistically significant difference from each other regarding their surface roughness irrespectively of the media in which they had been immersed. Durafill VS had the lower surface roughness, followed by Dyract AP, which had smoother texture than Filtek Z-250. Fuji II LC presented the roughest surface. These statistical differences are depicted in table 2 by different lower cases at the right side of the means (per row).

Within the factor restorative material, Multiple Comparison Tukey's test showed that for Durafill VS and Filtek-Z250 there were no statistical significant difference among their surface roughnesses as a result of storage in different media (Dw, Re, and De-Re). On the other hand, Dyract AP and Fuji II LC Improved immersed in De-Re presented lower surface roughness than they had done when they were stored in Dw and Re, which did not differ

from each other. These results can be seen in the table 2, where different capital letters at the left side of the means denote statistically significant difference (per column).

DISCUSSION

A long-term clinical trial of a material or technique is the ideal method of predicting restoration behavior [7, 22]. However, such trials require considerable resources spread over a significant amount of time [23]. In this manner, laboratory-based methodologies have been designed to be predictors of clinical service. Thus, it is important that these studies simulate the clinical situation as closely as possible so that realistic results be obtained [6].

In order to appropriately simulate abrasion due to brushing, parameters observed *in vivo* and previously described [13, 14, 24] were incorporated into the design of the toothbrushing abrasion device used in this study. In this case 100,000 strokes were used, which is equivalent to 10 years of brushing in the clinical situation [25]. These extensive number of brushing strokes were used considering that many restorations continued to function adequately after long survival times [26].

Based on the finding that the results of wear test conducted at room temperature can not be reliably extrapolated to the temperature encountered in the mouth [13], the thermostatically controlled environment of the toothbrushing machine provides brushing simulation closer to oral conditions.

Considering that the wear pattern of Class III and V dental restorations can be attributed not only to abrasion process [10-12] but also to chemical degradation [6], the de-

and remineralization model used provides an attempt to assess the behavior of restorative materials under a severe acid challenge at a pH value observed *in vivo* [20].

Although both mass-loss and changes in surface profile have been determined as being useful for measuring the effects of abrasion-resistance of restorative materials [18], the first methodology may disguise the true mass-loss of the resin-modified glass-ionomer cement since it exhibits marked weight changes on exposure to dry or wet conditions [27, 28]. In order to avoid the complications of dehydration and rehydration associated with weighting methods, as pointed out by Momoi *et al.* [11], and considering that changes in roughness are often used to determine the wear of a material [4], a surface roughness measuring instrument were used.

Due to the complexity of the real surface, a multiparameter representation has been advocated as a method capable of providing a more complete description [29]. However, the most often used parameter is Ra, the arithmetic deviation of the surface height from the mean line through the profile. One way of increasing the surface roughness measurement accuracy is to increase the number of tracings [30]. For this reason, in this study nine measurements were taken from each sample.

As a result of brushing abrasion as well as of corrosion process, the resin-based materials can become rougher [16, 19]. Since an increase in surface roughness was found capable to result in faster colonization of the surfaces and a faster maturation of the plaque, thereby increasing the risk of caries and periodontal inflammation, its occurrence can represent a concern [31]. Furthermore surface stains can be more easily retained on rough surfaces [32].

The rate of abrasion depends on several factors such as the type of dentifrice, the water to dentifrice ratio, the type of brush, and the speed and pressure used during brushing [13]. However, since in this investigation these parameters were standardized for all groups, the abrasion resistance of restorative materials studied seems to be dependent on their inherent properties [7]. Actually, the variations in wear among the materials have been reported as a combination of factors [4], such as the size, hardness and percent of the surface area occupied by filler particles [9, 33]; the degree of conversion of the polymer matrix of the resin matrix [34]; and the filler/matrix interaction, as well as, the stability of the silane coupler [34].

In this study, irrespective of the media in which restorative materials were stored Durafill VS had the lower surface roughness, followed by Dyract AP, which had smoother texture than Filtek Z-250, whereas Fuji II LC presented the roughest surface. The finding that Dyract AP was situated between the microfill composite resin, Durafill VS, and the hybrid composite, Filtek Z-250, is supported by Gladys et al. [19] who reported that this polyacid-modified composite resin has small filler particles and approximates composites. Actually, the wear resistance of materials can be improved by decreasing the size of their particles [7].

Resin-modified glass ionomer, polyacid-modified composite resin, and composite resins behaved differently with regard to their surface roughness as result of the different media used. It was found that De-Re regimen plus brushing abrasion of the resin-modified glass ionomer and the polyacid-modified composite resin resulted in a lower surface roughness in comparison with the other media (Dw and Re). On the other hand, no differences were observed among surface microstructures provided by Dw, Re or De-Re media for composite resins subjected to brushing. It is likely that the difference between both

the resin-modified glass ionomer and polyacid-modified composite resin and resin composites regarding their surface roughness results from differences in their inherent chemical and physical characteristics, which may affect their acid and abrasion resistance.

An insufficient coherent entanglement between the ionic cross-linked polyalkenoate network and the polymeric chains has been reported for the resin-modified glass-ionomer cements [19]. Therefore, based on this shortcoming, an increase in their erosion susceptibility is expected, above all in acidic environment [35]. However, according to the present results the De-Re regimen did not increase, but caused a decrease in the surface roughness as a result of brushing abrasion, in comparison with the other storage media. An explanation is that the demineralizing solution has reacted with the polymeric matrix of Fuji II LC to form a cohesive film on their surfaces, which can only be removed by a sliding movement over these surfaces [6]. Since the softened matrix probably stayed on the surface until brushing simulation had been performed, it is hypothesized that the softened matrix may have mixed with the abrasive slurry, decreasing its abrasive potential.

For polyacid-modified composite resins, the development of a carboxylate-rich surface on the uppermost layer of these materials after water storage may explain their lower wear resistance [36]. Moreover, according to Eliades *et al.* [36] it is possible that in low pH aqueous environments, polyacid-modified composites demonstrate increased solubility and selective dissolution of filler particles, by forming water-soluble salts which may further retard or completely inhibit the carboxylate salt yields on material surfaces. Therefore, it was expected that the brushing abrasion resistance of these materials after De-Re regimen would be higher than that presented after Dw storage, as was observed in this study. This disagree with the results of Attin *et al.* [12], who found that abrasion in acidic environment was

significantly higher compared with neutral conditions for polyacid-modified composites. However, considering that in their study the brushing abrasion was assessed by vertical loss, the comparison with the present investigation may be limited.

Concerning the surface roughness of composite resin, no significant difference among storage media was shown. This may be attributed to the presence of TEGDMA, a diluting monomer that can decrease the surface softening by acids [37]. In addition, this monomer increases the degree of polymerization of resin-based materials, improving their physical properties and thereby minimizing their abrasion rate [34].

In summary, it may be concluded that the surface roughness of the investigated resin-modified glass ionomer and polyacid-modified composite resin as a result of brushing abrasion was reduced after De-Re regimen, but this pH-cycling model did not affect the micromorphology of both composite resins. However, changes in surface roughness in isolation cannot entirely predict the abrasive wear resistance of these materials. Other aspects, such as the wear depth can account for their clinical performance since cavosurface marginal discrepancies of restorations can occur. Further investigations must be conducted in order to prove whether the present findings are also true for *in vivo* situation and to draw accurate conclusions on the performance of these restorative materials.

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TABLE 1. RESTORATIVE MATERIALS STUDIED, THEIR RESPECTIVE MANUFACTERS, COMPOSITIONS, MEAN PARTICLE SIZE, AND BATCH #

Restorative Material	Manufacturer	Basic composition*	Mean particle size (μm)*	Batch #
Fuji II LC (Fj)	GC Corp, Tokyo, Japan	Aluminum-fluoro-silicate glass, HEMA, tartaric acid, polyacrylic acid	1.80	020781
Dyract AP (Dy)	Dentsply DeTrey GmbH, Konstanz, Germany	Strontium-fluoro-silicate glass, strontium fluoride, polymerizable resins, TCB resin	0.80	9812000362
Durafill VS (Df)	Heraeus Kulzer GmbH, Wehrheim, Germany	Colloidal silica, BIS-GMA, TEGDMA, UDMA	0.02 – 0.04	40
Filtek Z250 (Z2)	3M, St Paul, MN, USA	Zirconia, silica, TEGDMA, UDMA BIS-EMA	0.60	9BP

*As disclosed by the manufacturers
HEMA = BIS-GMA = bisphenol glycidyl methacrylate; TEGDMA = tetraethyleneglycol dimethacrylate; UDMA = urethane dimethacrylate; BIS-EMA = Bisphenol A polyetheylene glycol diether dimethacrylate

TABLE 2: RESULTS OF MULTIPLE COMPARISON TUKEY'S TEST FOR RA (μM)TO IDENTIFY DIFFERENCES BETWEEN MATERIAL-STORAGE MEDIA INTERACTION.

	Du	Dy	Z2	Fj
De-Re	^{A}0.1025 (0.0072) ^{a}	^{A}0.1371 (0.0048) ^{b}	^{A}0.2040 (0.0052) ^{c}	^{A}0.2986 (0.0112) ^{d}
Dw	^{A}0.1061 (0.0072) ^{a}	^{B}0.1647 (0.0050) ^{b}	^{A}0.2022 (0.0050) ^{c}	^{B}0.3331 (0.0116) ^{d}
Re	^{A}0.1050 (0.0072) ^{a}	^{B}0.1607 (0.0046) ^{b}	^{A}0.2037 (0.0050) ^{c}	^{B}0.3229 (0.0114) ^{d}

Standard errors in brackets
Groups that were statistically different from each other are indicated by different superscript capital letters (per column) and lower cases (per row)

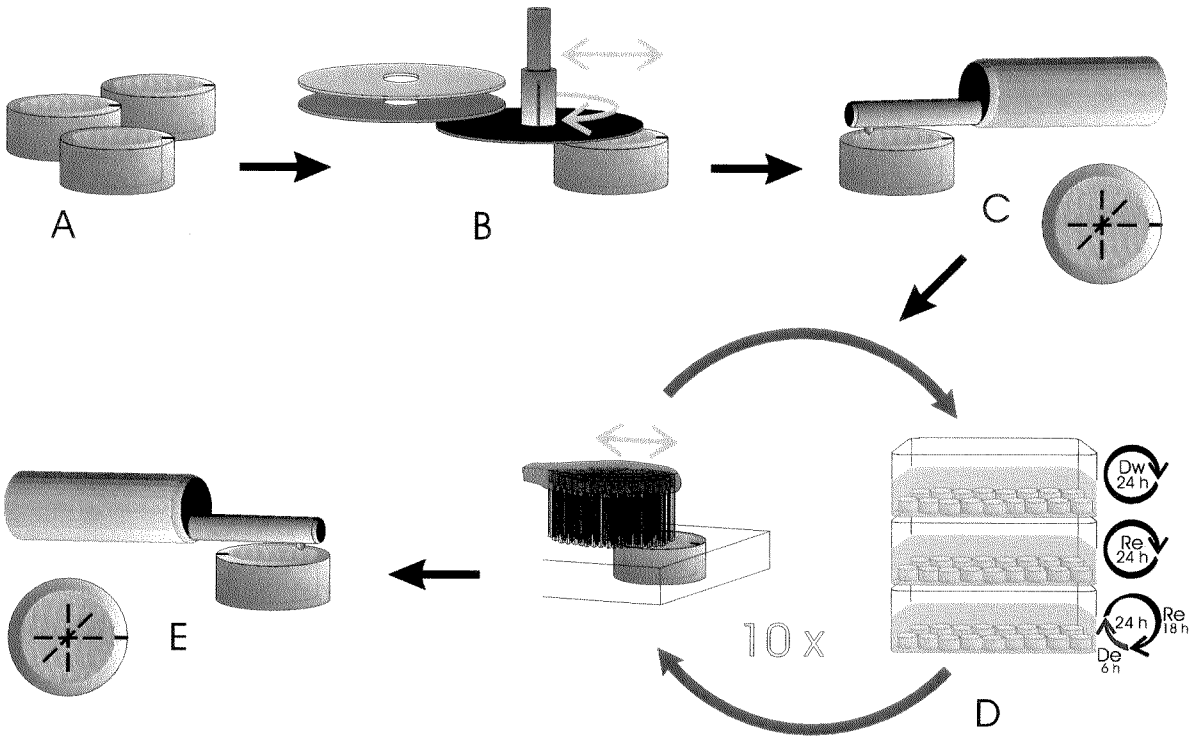

Fig. 1. **Diagram of the experimental set-up: (A) the materials were inserted into the marked matrixes, covered with a polyester strip and a glass slab, pressed with a weight and cured by a light-activation unit; (B) finishing and polishing were performed using medium, fine, and superfine aluminum oxide disks, in only one direction; (C) baseline surface roughness measurements were carried out at three locations in each one of the directions, parallel, perpendicular, and oblique to the finishing and polishing scratches; (D) storage in Dw, Re or De-Re media followed by 10,000 brushing strokes were repeated ten times; and (E) the final readings of surface roughness were obtained as described for baseline condition.**

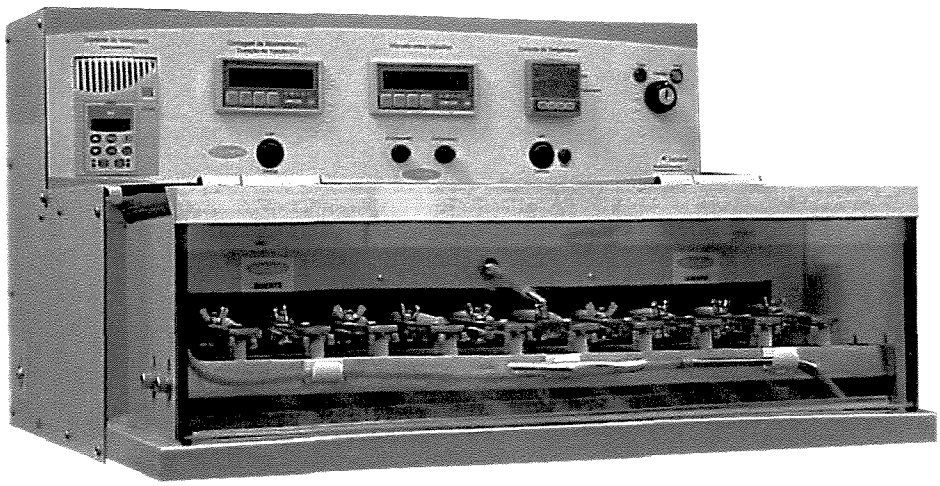

***Fig. 2.* A view of the device designed for the brushing abrasion test.**

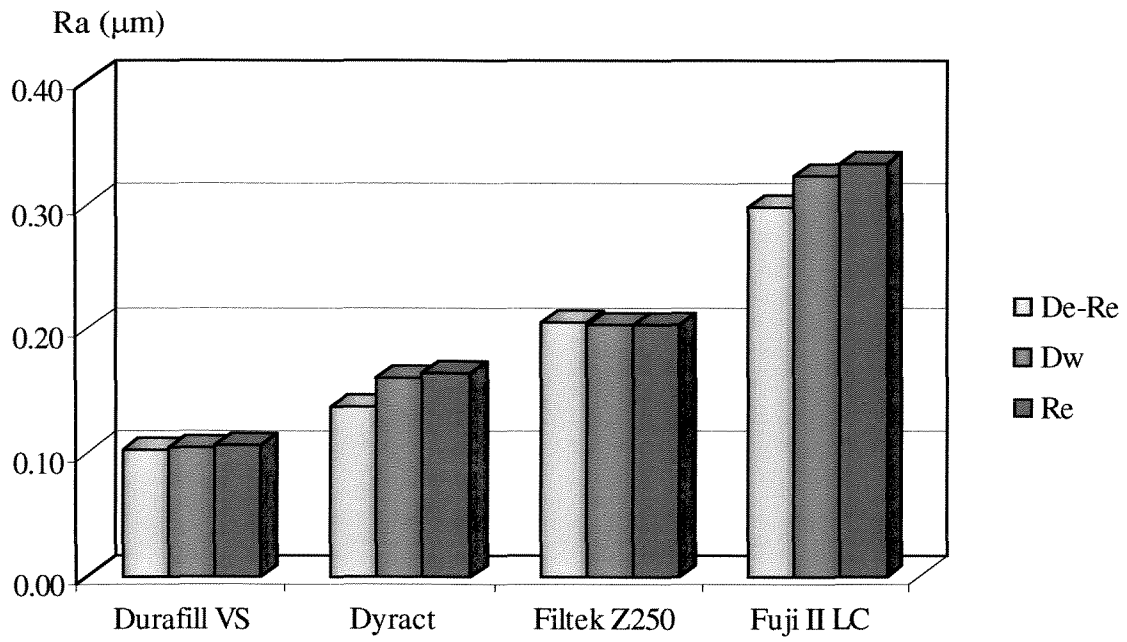

Fig. 3. **Graphic presentation of surface roughness of aesthetic materials after storage in different media plus brushing simulation.**

Surface roughness assessment of resin-based materials throughout brushing preceded by pH-cycling simulations

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SUMMARY

This study was intended to evaluate the surface roughness pattern of resin-based restorative materials throughout brushing preceded by a regimen that simulated a dynamic pH-cycling. Restoratives included two composite resins (Renamel Microfill and Charisma), two polyacid-modified composite resins (Compoglass-F and Dyract AP), and one resin-modified glass-ionomer cement (Fuji II LC Improved). Twenty standardized cylindrical specimens of each material were prepared according to a randomized complete block design. After finishing and polishing, the average surface roughness (Ra) and the profile-length ratio (LR) of the specimens were determined. The experimental units were subjected to a pH-cycling regimen, and then to 10,000 brushing strokes. New readings of both the Ra and LR parameters were obtained. The same protocol of pH-cycling, brushing simulation and, surface roughness measurements were repeated ten times. Data was analyzed using ANOVA according to split-plot design and Tukey's test. Results showed that the pH-cycling followed by brushing provided a remarkable increase in Ra for all restorative materials except for Renamel after 10,000 brushing strokes. Throughout the brushing simulation, Renamel, Charisma, Compoglass-F and Dyract AP showed steady textures, whereas Fuji II LC Improved exhibited a progressive increase in surface roughness. Among the materials tested, Renamel presented the smoothest surface, followed by Charisma and Compoglass-F, which did not differ from each other, except at the baseline. Dyract AP was different from both these materials, except at the baseline. Fuji II LC Improved had the roughest surface texture.

Clinical Relevance

Depending on the restorative material, brushing under a pH-cycling condition provided a remarkable initial increase in surface roughness which, thereafter remained unaltered or showed further increase.

INTRODUCTION

For a restorative material, long term clinical performance may be impaired by a number of factors, including degradation in the oral environment (Øilo, 1992). Among the aspects contributing to this breakdown are wear processes which include diverse phenomena, such as sliding, abrasion, chemical degradation, and fatigue (Söderholm & Richards, 1998). The outcome of these phenomena may be submargination (Gladys & others, 1997; Browning, Brackett & Gilpatrick, 2000) and changes in surface roughness (Sulong & Aziz, 1990; Sidhu, Sherriff & Watson, 1997). As a consequence, restorative materials may be a source of increased plaque retention and gingival irritation (Dunkin & Chambers, 1983). Moreover, these restorations may become susceptible to staining (Hachiya & others, 1984).

Among the wear processes, toothbrushing abrasion constitutes an important consideration in nonstress-bearing areas like cervical locations (Asmussen, 1985). Hence, considering that resin-based restoratives are often the materials of choice for restorations of carious and noncarious lesions located in these surfaces, studies have been undertaken to predict their clinical performance (Heath & Wilson, 1977; Goldstein & Lerner, 1991; Frazier, Rueggeberg & Mettenburg, 1998). It has been reported that various material-related features, such as their inter-particle filler spacing, filler particle size, and the degree of cure of the resin, influence resistance to abrasion (Draughn & Harrison, 1978; Bayne, Taylor & Heymann, 1992; Söderholm & Richards, 1998). Further, direction, and magnitude of acting forces and time may also be strongly related to the abrasion process (Milleding & others, 1998). On the other hand, certain physical characteristics of abrasive particles, including their

size, hardness and shape, have been shown to have a pronounced effect on their ability to wear surfaces (Mair & others, 1996).

In addition to abrasive processes, in the oral cavity numerous factors like low pH due to cariogenic microorganisms or acidic food, ionic composition and ionic strength of the saliva are important parameters that may play a role in the physical and mechanical characteristics of a restorative material (Geurtsen, Leyhausen & Garcia-Godoy, 1999). The chemical environment is, therefore, one aspect of the oral cavity that can have an appreciable influence on the *in vivo* wear process of resin-based restoratives (Roulet & Wälti, 1984; van Groeningen, Jongebloed & Arends, 1986). Only very limited information, however, is available about the abrasion resistance of these materials under conditions presumably encountered in the mouth (Attin & others, 1998).

Considering these facts, it seems important not only to compare the performance of different restorative materials, but also to estimate their long-term behavior. Hence, this study was undertaken to assess the surface roughness pattern of resin-based materials throughout brushing under a condition that simulated a dynamic pH-cycling.

METHODS AND MATERIALS

Experimental Design

The present study observed surface roughness as a response variable in relation to two explanatory factors: (1) restorative materials and (2) pH-cycling followed by brushing strokes. The average roughness (Ra) and the profile-length ratio (LR) values were taken from each experimental unit and were evaluated separately. The restorative materials factor were

taken into five levels (Renamel; Charisma; Compoglass-F; Dyract AP; and Fuji II LC Improved) and pH-cycling followed by brushing strokes into eleven levels (baseline; 10,000; 20,000; 30,000; 40,000; 50,000; 60,000; 70,000; 80,000; 90,000; and 100,000). This study used 20 experimental units for each restorative material made in 10 blocks, with 2 replicates each. The randomized complete block design was used to reduce the experimental error arising from known and controlled nuisance sources of variability (Montgomery, 1991). The split-plot design was employed supported by repeated measurements taken from the same experimental unit at different pH-cycling followed by brushing strokes levels.

Specimen Preparation

The restorative materials used in this study are listed in Table 1, together with other information on their basic composition, particle size, and filler content. The materials were handled according to the manufacturers' instructions and inserted into stainless steel matrixes of internal dimensions of 4 mm diameter by 2 mm thickness. A Centrix syringe (Centrix Inc., Shelton, CT, 06484) was used for inserting Fuji II LC Improved, whereas syringes supplied by the manufacturers were employed for Dyract AP and for Compoglass-F, and a metal spatula was used for the other materials. The surface of the restorative materials was covered with a polyester strip (Probem Ltda, Catanduva, SP, Brazil, 15800-000), which was pressed using a glass slide with a load of 500 g for 30 seconds to remove the excess material. The restoratives were then polymerized for the recommended exposure times through the polyester strip with a light unit (Optilux 401, Demetron / Kerr Corp, Danburry, CT, USA, 06810). The output from the curing light was monitored periodically using a light meter

(Curing Radiometer, Model 100, Demetron / Kerr Corp, Danburry, CT, USA, 06810) and it ranged from 400 to 520 mW/cm^2.

After setting, specimens were individually stored for 24 hours at 37(± 1)° C at 100% relative humidity. Thereafter, samples were finished and polished using medium, fine, and superfine aluminum oxide abrasive disks (Sof-Lex Pop On, 3M Dental Products, St Paul, MN, USA, 55144-1000). Each instrument was applied in a single direction for 15 seconds. Following each finishing and polishing step, specimens were flushed with air-water spray. Afterwards, samples were ultrasonically cleaned (Model T1440D, Odontobrás Ltda, Ribeirão Preto, SP, Brazil, 14075-060) in distilled-deionized water for 10 minutes to remove polishing debris and stored at 100% relative humidity.

Baseline Surface Roughness Measurements

Each sample was gently dabbed dry with absorbent paper and the surface roughness analyses were done using the Surfcorder SE1700 surface roughness measuring instrument equipped with a diamond needle of 2 μm radius (Kosaka Corp, Tokyo, Japan, 125). To record roughness measurements, the needle moved at a constant speed of 0.05 mm/s with a force of 0.7 mN. The cut-off value was set at 0.08 mm to maximize filtration of surface waviness. The surface roughness was characterized by the average roughness (Ra) and by the profile-length ratio (LR). Ra is the arithmetical average value of all absolute distances of the roughness profile from the center line within the measuring length. LR is defined as the ratio between the true profile length, i.e., the length of the profile being drawn out into a straight line, and the measuring distance. An ideal, smooth surface has an LR value of 1; the rougher the surface becomes, the greater the LR-value will be. Three traces were recorded on each

specimen at three different locations in each direction – parallel, perpendicular, and oblique to the finishing and polishing scratch directions – amounting to nine tracings per sample. The average of these nine mean surface roughness measurements was used as the score for each sample.

pH-cycling Protocol

The specimens were subjected to a pH-cycling regimen, as proposed by Featherstone & others (1986) and modified by Serra & Cury (1992). The samples were immersed in 5 ml of demineralizing solution for 6 hours at 37(± 1)° C, followed by rinsing with distilled-deionized water, and storage in 5ml of remineralizing solution (artificial saliva) by 18 hours at 37(± 1)° C. The artificial saliva consisted of 1.5 mM of calcium, 0.9 mM of phosphate, and 150 mM of potassium chloride in a buffer solution of 0.1 mM of Tris (hydroxymethyl-aminomethane) at pH 7.0. The acid solution contained 2.0 mM of calcium and 2.0 mM of phosphate in a buffer solution of 74.0 mM of acetate at pH 4.3. After this protocol, the specimens were rinsed with distilled-deionized water and stored at 37(± 1)° C in 100% relative humidity.

Brushing Abrasion Protocol

Brushing abrasion of the specimens was performed with an automatic toothbrushing abrasion testing machine (Marcelo Nucci, São Carlos, SP, Brazil, 13574-080) which consisted of a motor that produced a reciprocating motion on ten soft nylon bristle toothbrush heads (Oral-B Indicator 40, Gillette do Brasil Ltda., Manaus, AM, Brazil, 69075-900) in a thermostatically controlled environment at 37(± 0.5)° C. The experimental units were aligned so that the brushing heads moved parallel to their surfaces, loaded with a 300g weight and

traveled horizontally for 20 mm at a speed of 4.5 strokes per second. Specimens were brushed with 10,000 strokes. An abrasive slurry was prepared by mixing dentifrice (Colgate MFP, Colgate Palmolive – Division of Kolynos do Brasil Ltda, Osasco, SP, Brazil, 06020-170) and distilled-deionized water at a ratio of 1:3 by weight respectively, which was independently injected beside each brush at a frequency of 0.4 ml at 2-minute intervals. By means of this intermittent regimen of injection it was possible to reduce sedimentation of the abrasive and to avoid a decrease in the amount of slurry.

After testing, specimens were removed from the machine, rinsed with tap water, and cleaned ultrasonically in distilled-deionized water for 10 minutes. Samples were again stored at 100% relative humidity and data was collected using the surface roughness instrument as previously described.

Repeated Measurements Throughout the pH-cycling Followed by Brushing Strokes

The same protocols of pH-cycling and brushing simulation were subsequently repeated ten times. After every pH-cycling followed by 10,000 strokes, the specimens were again subjected to surface roughness analysis. Data were also obtained after 20,000; 30,000; 40,000; 50,000; 60,000; 70,000; 80,000; 90,000; and 100,000 brushing strokes. The brush heads were replaced after simulating 50,000 brushing strokes.

Statistical Analysis

The statistical evaluation of the data was made by Analysis of Variance, according to split-plot design, followed by Tukey's test to perform pairwise comparisons between restorative materials and pH-cycling followed by brushing strokes at the level of 5% of significance. The regression method was used to fit a mathematical function of surface

roughness (dependent variable) by pH-cycling followed by brushing strokes (independent variable) using a quartic-order additive model. Statistical analysis were performed by Statgraphics® Plus (Manugistics, Rockville, Maryland, USA, 20852).

RESULTS

The Analysis of Variance revealed significant effect for restorative materials, pH-cycling followed by brushing strokes and for the interaction between these factors for both Ra and LR response variables. Since the interaction was significant for each response variable, the comparisons of different materials at baseline and within each 10,000 brushing strokes could be made. Tables 2 and 3 show the mean and standard deviation for Ra and for LR, respectively.

For both Ra and LR, the surface roughness pattern for each material as a function of pH-cycling followed by brushing strokes is shown in Figure 1 and Figure 2, respectively. This was done by fitting the data according to a quartic-order mathematical equation. In regards to Ra, an increase in surface roughness for every restorative material except for Renamel was shown after pH-cycling followed by 10,000 brushing strokes (Table 2 and Figure 1). Throughout the brushing simulation, i.e., from 10,000 to 100,000 strokes, Renamel, Charisma, Compoglass-F and Dyract AP showed steady textures, whereas Fuji II LC Improved exhibited a progressive increase in surface roughness, interspersed with periods of stabilization. Comparing the restorative materials within each level of the pH-cycling followed by brushing strokes factor (comparisons by rows in Table 2), Renamel presented the smoothest surface, followed by Charisma and Compoglass-F which did not

differ from each other except at the baseline. Dyract AP was different from both these materials, except at the baseline. Fuji II LC Improved had the statistically roughest surface texture.

Regarding the LR response in each restorative material, a remarkable increase in surface roughness for Charisma, Dyract AP, and Fuji II LC Improved was observed, whereas Renamel and Compoglass-F showed a steady state throughout all the experimental period in relation to the baseline (Figure 2). Considering the levels of the pH-cycling followed by brushing strokes factor shown in Table 3, there were no statistical differences among Renamel, Charisma, Dyract AP, and Compoglass-F, except when Compoglass-F was compared to Renamel. From the first pH-cycling followed by 10,000 brushing strokes to the last, Renamel showed the statistically smoothest surface; Charisma and Compoglass-F did not differ from each other; Dyract AP was rougher than both Charisma and Compoglass-F; Fuji II LC Improved showed the greatest results of roughness than any material, since the baseline.

DISCUSSION

Based on studies, which examined various behavioral aspects of adult toothbrushing (Heath & Wilson, 1974; van der Weijden & others, 1996), attempts have been made to develop *in vitro* abrasion tests intended to predict clinical abrasion reliability. In this study, factors previously described like the brushing load, the stroke rate (Heath & Wilson, 1974), the temperature and the schedule in which the test may simulate more closely oral conditions (Heath & Wilson,1977) were incorporated into the abrasion procedure.

In previous laboratory studies, the load applied to the toothbrushes during brushing amounted to 100-576 g (Montes & Draughn, 1986; Hotta & Hirukawa, 1994; Wülknitz, 1997; Attin 1998; Kaway, 1998; Tanoue, Matsumura & Atsuta, 2000b). Considering this wide variation, in this investigation the load chosen – 300 g – simulated a medium brushing force. Moreover, there has been controversy over the number of strokes that have been used to simulate 1 year's brushing. Numbers have ranged from 4,320 (Kanter, Koski & Don Martin, 1982) to 16,000 (Aker, 1982). In the present study the assignment of 10,000 brushing strokes, which is equivalent to 1 year of brushing in clinical situation, was based on Goldstein & Lerner (1991).

The abrasion resistance of resin-based restorative materials to brushing has been evaluated by various methods, such as surface roughness (Kanter & others, 1982; de Gee, ten Harkel-Hagenaar & Davidson, 1985; Goldstein & Lerner, 1991; Whitehead & others, 1996; Gladys & others, 1997; Momoi & others, 1997; Tanoue & others, 2000b), profilometrical tracings (Heath & Wilson, 1976; de Gee & others, 1985; Goldstein & Lerner, 1991; Momoi & others, 1997, Attin & others, 1998; Tanoue & others, 2000b), weight loss (Aker, 1982; Kanter & others, 1982; Kaway, Iwami & Ebisu, 1998; Frazier & others, 1998), and photomicrographs (Draughn & Harrison, 1978; Ehrnford, 1983). In the present study, surface roughness assessment was chosen because it is documented that surface roughness can play a role in bacterial colonization on restorative materials (Dunkin & Chambers, 1983). Although contradictory results with regard to the effect of surface properties on these phenomena have been reported in the literature (Quirynen & Bollen, 1995), it is well known that adherence and metabolic activities of microorganisms in the mouth are the primary causes of a variety

of conditions including dental caries and inflammatory diseases of the gingival and periodontal tissues (Bollen & others, 1997).

Although the arithmetical mean of the absolute values of the profile departures within the evaluation length (Ra) is the most common roughness parameter used for describing surface texture (Sidhu & others, 1997), it has been advocated that the roughness height is merely one estimator of surface quality (Nowicki, 1985). As stated by Jung (1997), the horizontal aspect of roughness remains largely unconsidered. In order to deal with this limitation, the same author proposed the measurement of the profile-length ratio (LR), which take into account both the vertical and horizontal dimensions of roughness at the same time. For this reason, Ra and LR parameters were recorded.

In addition to brushing simulation, in the present investigation the specimens were subjected to pH cycles comprising alternating storage in de- and remineralizing solutions. Although this model has been introduced to simulate the caries process in cariology research, it incorporates a severe acid challenge at a pH value that has been reported to occur in vivo (Featherstone & others, 1986). For this reason it was used with the intent to mimic chemical processes or dissolution already reported to occur in the mouth (Roulet & Wälti, 1984; van Groeningen & others, 1986).

With regard to the performance of the restorative materials, the microfilled composite, Renamel, showed the lowest surface roughness, followed by Charisma. These findings might be ascribed to the filler particles size of these materials, which is lower for Renamel, as shown in Table 1. It was suspected that the presence of small fillers could result in decreased interparticle space and reduced wear (Söderholm & Richards, 1998). Throughout the brushing strokes preceded by pH-cycling, the hybrid composite and the polyacid-modified

composite resin Compoglass-F were not different from each other. It was hypothesized that although Compoglass-F was less filled, it was as smooth as Charisma, probably due to the presence of a wide range of small particlesThese findings might be explained by the presence in both composites of smaller filler particles, as showed in Table 1, which would result in decreased interparticle space and reduced wear (Söderholm & Richards, 1998). Throughout the brushing strokes preceded by pH-cycling, the hybrid composite and the polyacid-modified composite resin Compoglass-F were not different from each other. It was hypothesized that although Compoglass-F was less filled, it was as wear-resistant as Charisma probably due to the presence of a wide range of small particles. Although Compoglass-F and Dyract AP are both polyacid-modified composite resins, they differ significantly from each other throughout brushing. This is probably due to the dissimilarities in their microstructure. Fuji II LC Improved was rougher than the other materials under evaluation. Unlike polyacid-modified composite resins, the coherence between the cross-linked polyacrylate network and the polymer chain of resin-modified glass ionomers seems insufficient (Kanchanavasita, Anstice & Pearson, 1998). Moreover, in an aqueous environment, this material may take up great amounts of water, swell, became plastic and mechanically less resistant than other resin-based materials (Meyer & others, 1998; Cattani-Lorente & others, 1999).

Although the dissimilarity in surface roughness of materials may be mainly attributable to the differences in their properties, such as size and content of filler particles, these restoratives differ in many other aspects, e.g., type of fillers, degree of conversion of the polymer matrix , and silane coupler, which may also influence their abrasion resistance (Jaarda, Wang & Lang, 1996; Kaway, Iwami & Ebisu, 1998; Tanoue & others, 2000a).

In the attempt to show the process taking place, it was considered more convenient to show the behavior of the restorative materials as a function of pH-cycling followed by brushing strokes. For both response variables, Ra and LR, the best approximations of the behaviors of these restoratives were obtained when a quartic-order mathematical equation was used. In the description of Ra, the surface roughness of all restorative materials increased throughout brushing simulation except for Renamel. Throughout the pH-cycling followed by brushing strokes, Renamel, Charisma, Compoglass-F and Dyract AP exhibited steady textures. These findings may be attributed to the difference between the abrasion of the matrix and the filler particles. This discrepancy is large enough to provide an anisotropic degradation, which gradually slows down the more the filler becomes exposed to the surface (de Gee & others, 1985). Fuji II LC Improved exhibited a progressive increase in surface roughness, interspersed with stable periods. One possible explanation is the deficient coherence between the matrix and the fillers of this material, which may cause exfoliation of some of its particles as the matrix is being worn away (Aker; 1982; Condon & Ferracane, 1996).

When evaluating the LR, throughout the experimental period both Renamel and Compoglass-F showed steady surface textures. Since the profile-length ratio taking into account both the vertical and horizontal dimensions of roughness at the same time, it is likely that the overall aspect of the microstructure of these materials remained unconsidered when recording the Ra values. However, considering that after 10,000 brushing strokes preceded by pH-cycling the Ra and LR results were very similar and that there is a lack of equipment suitable for its direct measurement (Nowicki, 1985), it may be inferred that under the

conditions adopted in this study the surface roughness description may be determined on the basis of Ra.

In laboratory-based experiments, the inherent complexity of the oral environment is disregarded with the intent to highlight the main factor in analysis. In the present investigation in order to evaluate the surface roughness pattern of resin-based materials throughout brushing, only an approach that simulated an abrasive wear preceded by a dynamic pH-cycling was considered. Other aspects, such as thermal stress (Montes & Draughn, 1985; Sulong & Aziz, 1990) and cuspal flexure resulting from occlusal loading (Rees & Jacobsen, 1998), which may alter the process of wear of restorations located in cervical areas, were not included. Therefore, *in vitro* models may not necessarily give a full realistic indication of what goes on in the mouth. However, considering the high turnover of new restorative materials, they are important together with other laboratory studies for predicting the behavior of dental restorations.

CONCLUSIONS

Under the conditions in which this study was undertaken, it may be concluded that:

1. pH-cycling followed by brushing provided a remarkable increase in Ra for all restorative materials strokes except for Renamel after 10,000 brushing.
2. Throughout the brushing strokes preceded by pH-cycling, Renamel, Charisma, Compoglass-F and Dyract AP showed steady textures, whereas Fuji II LC Improved exhibited a progressive increase in surface roughness.
3. Among the materials tested, Renamel presented the smoothest surface, followed by Charisma and Compoglass-F, which did not differ from each other, except at baseline. Dyract AP was different from both these materials except at the baseline. Fuji II LC Improved had the roughest surface texture.

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Table 1. *Resin-based materials under investigation and their technical profiles*

Material	***Manufacturer***	***Type***	***Basic Composition****	***Filler size (μm)****	***Filler Content (% by volume)****	***Batch #***
Renamel	Cosmedent Inc., Chicago, USA, 60640	microfilled composite resin	Pyrogenic silic acid; highly molecular multi-functional methacrylate ester	0.02-0.04	80	993460 G
Charisma	Heraus Kulzer GmbH, Wehrheim, Germany, D-61273	hybrid composite resin	Barium aluminum fluoride glass; highly dispersive siliciumdioxide, bisGMA	0.02-2.0	64	74
Compoglass-F	Vivadent Ets, Liechtenstein, Germany, FL-9494	polyacid-modified composite resin	YF_3, Ba-Al-fluorosilicate glass; bisGMA, UDMA; TEGDMA; cyclo-aliphatic dicarboxylic acid dimethacrylate	0.2-3.0	55	B0069
Dyract AP	Dentsply De Trey GmbH, Konstanz, Germany, 78467	polyacid-modified composite resin	Strontium-AL-Na-fluoro-P-silicate-glass; strontium fluoride; UDMA; TCB resin; highly cross-linking methacrylate-monomer	0.8	47	9904001505
Fuji II LC Improved	GC America Inc. Tokyo, Japan, 174	resin-modified glass-ionomer cement	Al, fluorosilicate glass; HEMA; tartaric acid; polyacrylic acid	0.1-25	60	Powder: 120191 Liquid: 060191

*As disclosed by the manufacturers
BisGMA = Bisphenol-A-glycidyl methacrylate; UDMA = Urethane dimethacrylate; TEGDMA = Triethylene glycol dimethacrylate;HEMA = hydroxyethyl methacrylate

Table 2. *Mean and standard deviations for Ra, showing the results from the Tukey's test*

Condition	Renamel	Charisma	Compoglass-F	Dyract AP	Fuji II LC Improved
Baseline	0.0270 (0.0036) [a]	0.0517 (0.0046) [b]	0.0723 (0.0053) [c]	0.0650 (0.0084) [bc]	0.1747 (0.0100) [d]
10,000 brushing strokes	0.0390 (0.0036) [a]	0.0914 (0.0062) [b]	0.0952 (0.0062) [b]	0.1433 (0.0078) [c]	0.3216 (0.0228) [d]
20,000 brushing strokes	0.0407 (0.0024) [a]	0.0944 (0.0049) [b]	0.0937 (0.0087) [b]	0.1459 (0.0072) [c]	0.3774 (0.0252) [d]
30,000 brushing strokes	0.0429 (0.0036) [a]	0.0959 (0.0050) [b]	0.0945 (0.0062) [b]	0.1499 (0.0061) [c]	0.3968 (0.0324) [d]
40,000 brushing strokes	0.0449 (0.0108) [a]	0.0923 (0.0043) [b]	0.0930 (0.0037) [b]	0.1494 (0.0116) [c]	0.3922 (0.0855) [d]
50,000 brushing strokes	0.0428 (0.0024) [a]	0.0927 (0.0039) [b]	0.0895 (0.0055) [b]	0.1473 (0.0072) [c]	0.4310 (0.0379) [d]
60,000 brushing strokes	0.0447 (0.0027) [a]	0.0911 (0.0042) [b]	0.0913 (0.0063) [b]	0.1527 (0.0049) [c]	0.4367 (0.0255) [d]
70,000 brushing strokes	0.0485 (0.0036) [a]	0.0900 (0.0031) [b]	0.0916 (0.0048) [b]	0.1543 (0.0059) [c]	0.4624 (0.0281) [d]
80,000 brushing strokes	0.0528 (0.0045) [a]	0.0924 (0.0048) [b]	0.0961 (0.0080) [b]	0.1575 (0.0057) [c]	0.4750 (0.0178) [d]
90,000 brushing strokes	0.0524 (0.0043) [a]	0.0943 (0.0048) [b]	0.0948 (0.0061) [b]	0.1555 (0.0067) [c]	0.4768 (0.0270) [d]
100,000 brushing strokes	0.0523 (0.0030) [a]	0.0922 (0.0061) [b]	0.0950 (0.0073) [b]	0.1583 (0.0059) [c]	0.4700 (0.0226) [d]

Standard deviations are given between parentheses
Means connected by vertical brackets did not differ fromeach other. Values with the same superscript letter were not statistically different ($p < 0.05$) by row

Table 3. *Mean and standard deviations for LR, showing the results from the Tukey's test*

Condition	Renamel	Charisma	Compoglass-F	Dyract AP	Fuji II LC Improved
Baseline	1.0031 (0.0009) [a]	1.0040 (0.0012) [ab]	1.0051 (0.0023) [b]	1.0047 (0.0010) [ab]	1.0078 (0.0021) [c]
10,000 brushing strokes	1.0028 (0.0007) [a]	1.0070 (0.0013) [b]	1.0067 (0.0016) [b]	1.0092 (0.0015) [c]	1.0218 (0.0028) [d]
20,000 brushing strokes	1.0031 (0.0010) [a]	1.0072 (0.0015) [b]	1.0058 (0.0014) [b]	1.0106 (0.0018) [c]	1.0259 (0.0029) [d]
30,000 brushing strokes	1.0029 (0.0011) [a]	1.0077 (0.0011) [b]	1.0062 (0.0017) [b]	1.0103 (0.0017) [c]	1.0279 (0.0021) [d]
40,000 brushing strokes	1.0027 (0.0011) [a]	1.0074 (0.0020) [b]	1.0057 (0.0012) [b]	1.0104 (0.0018) [c]	1.0265 (0.0067) [d]
50,000 brushing strokes	1.0029 (0.0009) [a]	1.0070 (0.0012) [b]	1.0058 (0.0011) [b]	1.0103 (0.0021) [c]	1.0289 (0.0028) [d]
60,000 brushing strokes	1.0023 (0.0006) [a]	1.0062 (0.0006) [b]	1.0054 (0.0015) [b]	1,0103 (0.0013) [c]	1.0297 (0.0040) [d]
70,000 brushing strokes	1.0023 (0.0007) [a]	1.0062 (0.0006) [b]	1.0049 (0.0009) [b]	1,0101 (0.0014) [c]	1.0303 (0.0021) [d]
80,000 brushing strokes	1.0030 (0.0017) [a]	1.0075 (0.0023) [b]	1.0063 (0.0022) [b]	1,0110 (0.0024) [c]	1.0301 (0.0032) [d]
90,000 brushing strokes	1.0029 (0.0008) [a]	1.0062 (0.0006) [b]	1.0049 (0.0009) [b]	1,0099 (0.0010) [c]	1.0308 (0.0021) [d]
100,000 brushing strokes	1.0029 (0.0008) [a]	1.0075 (0.0023) [b]	1.0063 (0.0022) [b]	1,0108 (0.0014) [c]	1.0297 (0.0037) [d]

Standard deviations are given between parentheses
Means connected by vertical brackets did not differ from each other. Values with the same superscript letter were not statistically different ($p < 0.05$) by row

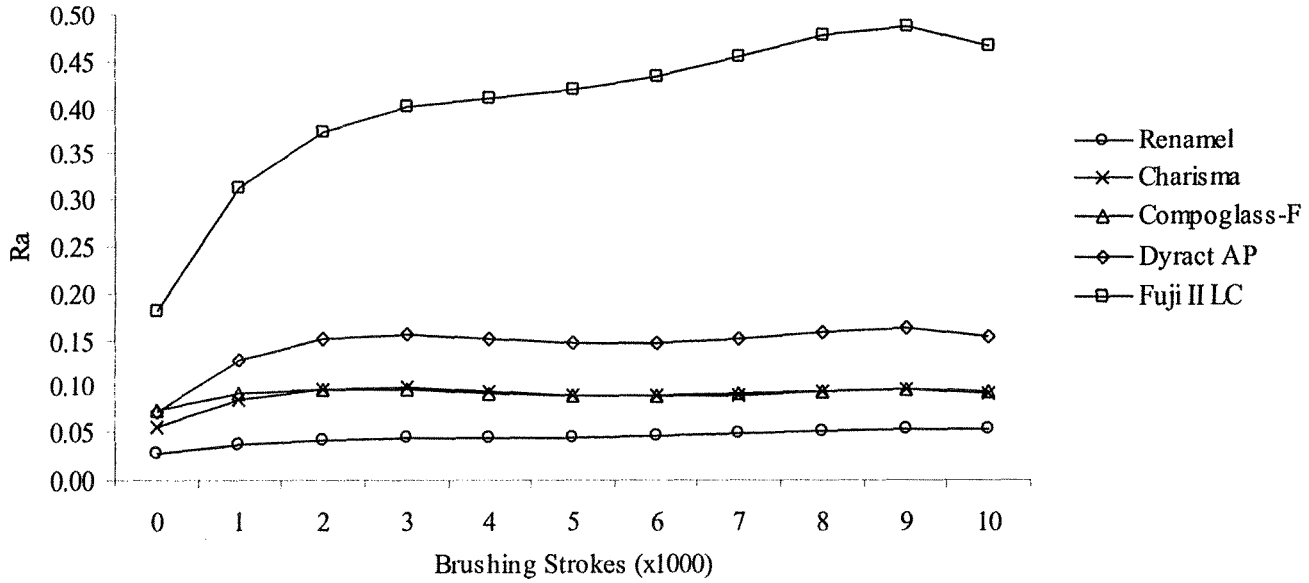

Figure 1. *Ra response of the different materials as a function of pH-cycling followed by brushing strokes. The experimental data were fitted according to a quartic-order mathematical equation*

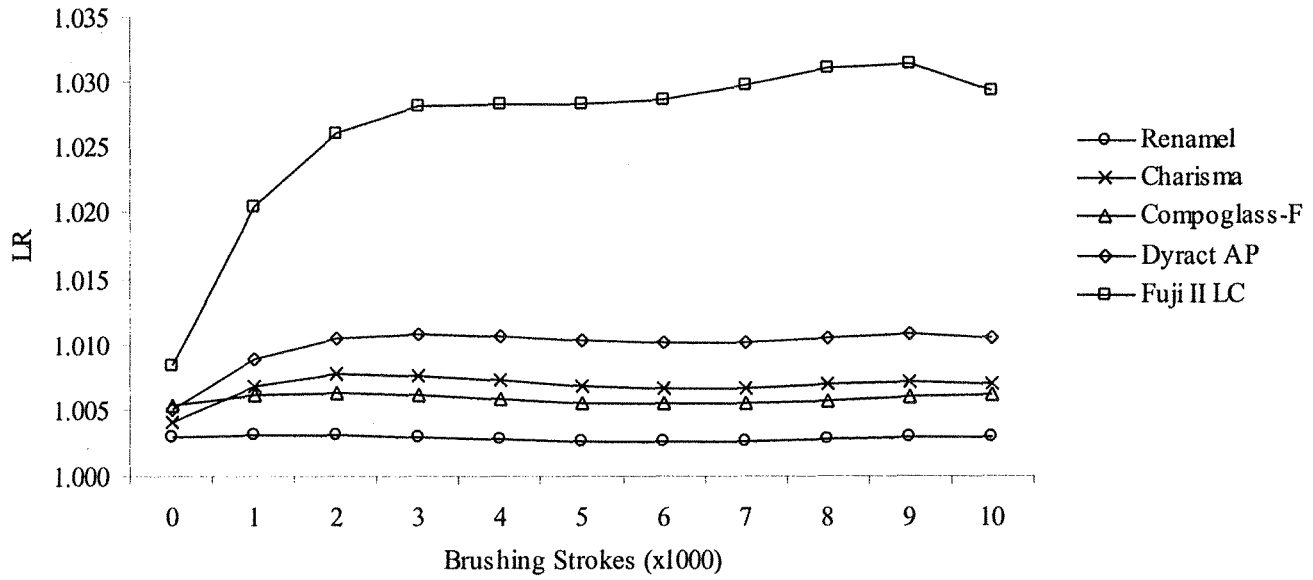

Figure 2. *LR response of the different materials as a function of pH-cycling followed by brushing strokes. The experimental data were fitted according to a quartic-order mathematical equation*

Effect of fluoride gels on micromorphology of resin-modified glass ionomer cements and polyacid-modified composite resins

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ABSTRACT

Objective: *To evaluate the surface micromorphology of resin-modified glass ionomer cements and polyacid-modified composite resins subjected to a neutral (NNaF) and an acidulated fluoride (APF) gel application.* **Method and Materials:** *Thirty standardized cylindrical specimens were randomly obtained from each resin-modified glass ionomer cements – Fuji II LC Improved/GC (Fj) and Photac-Fil Aplicap/Espe (Po) – and polyacid-modified composite resins – Dyract/Dentsply (Dy) and F2000/3M (F2), amounting to 120 samples. After a week, the specimens were finished and polished with aluminum oxide disks. Surface treatments with fluoride gels or distilled water (DW) as control were performed four times, interspersed with 8 pH cycles, simulating high cariogenic challenges. Five calibrated evaluators assessed the surface micromorphology through photomicrographs.* **Results:** *The Kruskal-Wallis test showed no significant difference between the control and experimental groups for Fj and Dy. Po showed less micromorphological change as a result of DW application, unlike the NNaF and APF treatments, which revealed no significant difference from each other. For F2, there was no significant difference between the surfaces treated by NNaF and DW; the highest degradation occurred with the APF.* **Conclusion :** *Both the resin-modified glass ionomer cements and the polyacid-modified composite resins showed erratic behaviors concerning their micromorphology when subjected to fluoride gel applications.*

KEY WORDS: fluoride gels, resin-modified glass ionomer cement, polyacid-modified composite resin, scanning electron microscopy, surface degradation

CLINICAL RELEVANCE STATEMENT: Due to the erratic behavior pattern demonstrated by the resin-modified glass ionomer cements and the polyacid-modified composite resins as a result of fluoride gel treatments, care must be taken when using such gels over these restorations.

INTRODUCTION

Scientific knowledge concerning caries has led to the establishment of preventive and therapeutic strategies in accordance with the individual characteristics of each patient in regards to the risk and activity of caries.[1,2] Measures to increase protective factors, such as fluoride therapy, may be adopted as part of these approaches,[3-5] inhibiting tooth demineralization and enhancing remineralizing potential.[6,7] Although the ideal concentration and time during which fluoride remains available for interfering in the demineralization process and for inducing the remineralization phenomenon have not been established,[8-10] different sources of fluoride can be used to benefit patients with a moderate or a high risk of developing caries.[11]

Since glass ionomer cements release fluoride ions, they are particularly useful where cariostatic action is clinically needed.[12-15] However, because the setting of these materials relies on the completion of acid-base reactions, sensitivity to dehydration and susceptibility to moisture contamination can result in a decline in their physical and aesthetic properties. With the introduction of resin-modified glass ionomer cements, some of these inherent shortcomings of conventional glass ionomers were overcome while partially retaining their clinical advantages.[16]

Studies have demonstrated that the fluoride release from newly set restorations of resin-modified glass ionomer cements and polyacid-modified composites is elevated during the first few days, and a relatively considerable amount continues for a month or more thereafter.[17-19] Moreover, these materials are capable of acquiring further fluoride ions following their exposure to fluoridated products, such as toothpaste,[20] solutions,[21] and gels,[22]

thereby acting as a rechargeable fluoride release system during eventual cariogenic challenges.[23]

Considering the composition and pH values of some fluoride gels, the surface integrity of resin-based restorative materials subjected to these products represents a concern.[24,25] For this reason, the effect of fluoride gel applications has been assessed, although the conditions have not simulated the dynamics of the oral environment.

The aim of this study was to evaluate the effect of fluoride gel applications on surface micromorphology of resin-modified glass ionomer cements and polyacid-modified composite resins, in a highly cariogenic challenging simulation.

METHOD AND MATERIALS

Experimental design

The factors under study were restorative materials at four levels and surface treatments at three levels, amounting to 12 groups. The experimental sample was comprised of 120 specimens (n=10), fabricated in a random sequence. The response variable was surface micromorphology which was independently and blindly evaluated through photomicrographs by five examiners using an ordinal scale from 0 to 2.

Specimen Preparation

Two resin-modified glass ionomers and two polyacid-modified composite resins were used in this experiment (Table 1). These materials were handled according to manufacturers' directions (Table 2) and inserted into acrylic rings (5.0 mm inside diameter and 2.0 mm thick). The rings were overfilled with the materials, covered with a polyester strip and a glass

slab and, pressed with a weight of 500 g for 30 seconds to extrude any excess and to create uniform surface texture. When the load and the slab were removed, the specimens were photopolymerized (Optilux 500, Demetron Research Corp) for the manufacturers' recommended exposure times (Table 2). After removing the polyester strip, the surfaces of the resin-modified glass ionomer cement specimen surfaces were protected by a bonding resin (Heliobond, Vigodent S.A.), which was light-cured for 20 seconds. Then each sample was stored in 5ml of distilled water at 37 ± 1° C for a week.

Finishing and polishing were carried out using aluminum oxide disks (Sof-Lex Pop On, 3M Dental) at medium, fine, and superfine grits, while keeping the material surface wet. Each grit was applied in only one direction for 30 seconds. All samples were flushed with air-water spray between each instrument, and at the end of the procedure they were cleaned ultrasonically (Inpec Eletrônica) in distilled water for 10 minutes to remove any remaining debris.

Surface Treatment Applications

Surface treatments consisted of fluoride gel and distilled water (DW) applications. The fluoride gels used were a neutral sodium fluoride (NNaF) and an acidulated phosphate fluoride (APF) which are described in table 2. Depending on the group, 0.4 ml of either DW, NNaF or, APF was applied over the specimen upper surface for 4 minutes. Immediately after the treatment, samples were flushed with water and a dynamic model of pH-cycling was initiated, simulating a high cariogenic challenge, as proposed by Featherstone et al.[26] and described by Serra & Cury.[12] This model consisted of immersing samples in 5 ml of demineralizing solution for 6 hours at 37 ± 1° C, followed by rinsing specimens with water,

and storing them in 5ml of remineralizing solution (artificial saliva) for 17 hours at 37 ± 1° C. This protocol was applied during 2 consecutive days. Afterwards, the specimens remained immersed in 5 ml of artificial saliva at 37 ± 1° C for a week. The surface treatment of samples, followed by de- and remineralization cycles, was performed during a period of 4 weeks, amounting to 4 surface treatment applications interspersed with 8 pH-cycles.

Photomicrograph Evaluation

Specimens were again ultrasonically cleaned for 10 minutes and examined in a environmental scanning electron microscope (SM-300, Topcon) without any preparation. Photomicrographs were obtained at 2000x magnification and then assessed by five calibrated evaluators. The surface micromorphology of the specimens was rated according to El-Badrawy et al. modified criteria,[27] the degradation of glass particles and resinous matrix were jointly evaluated: (0) both the particles and the matrix appear intact, with no evidence of etching, and glass particles are level with and embedded in the matrix; (1) moderate degradation with pitting or slight cracking of the glass particles, the resinous matrix showing irregular surface with particles partially protruding, and a limited number of voids present, (2) severe cracking and pitting of glass particles, little or no matrix around particles, and a considerable number of voids in the matrix. For calibration, representative photomicrographs of each one of the scores were used. Due to the differences in characteristics among the materials under evaluation, it was necessary to perform the calibration process independently for each material.

Statistical Analysis

The responses of the 5 evaluators were used for establishing a rough estimate for the median function. To analyze the difference among the medians of the ordinal qualitative responses (scores 0 to 2) assigned by the examiners, the nonparametric Kruskal-Wallis test at 95% confidence level was used. Subsequently, by means of the least significant difference (lsd), pairwise comparisons of the average ranks were carried out, in order to check the hypothesis of equality among the groups studied. The Statgraphics® Plus software (Manugistics) was used to perform the statistical calculations.

RESULTS

The median scores per restorative materials and per surface treatment, the average rank values, and the pairwise comparisons are listed in Table 4. The Kruskal-Wallis test showed no statistically significant differences between treatments for Fuji II LC Improved (H= 3.70, with p = 0.1570) and for Dyract (H= 5.29, with p = 0.0710). As can be seen from Figures 1 and 2, moderate degradation occurred for all groups. Statistically significant differences between treatments for Photac-Fil Aplicap (H= 20.31, with p = 0.0000) and F2000 (H= 19.80, with p = 0.0000) were detected. Comparisons of the average ranks for Photac-Fil Aplicap showed no statistically significant difference between the NNaF and APF groups (Fig 3B and C), while the DW group showed significantly lower micromorphology change (Fig 3A). For F2000, the highest surface degradation was caused by the APF gel (Fig 4C), while DW and NNaF treatments showed no significant difference from each other (Fig 4A and B).

DISCUSSION

The ability to carry out experiments under highly controlled conditions represents the major advantage of *in vitro* experimentation.[28] However, it is important that these studies simulate the clinical situation as closely as possible to obtain realistic results.[29] Despite this concern, some investigations regarding the effect of fluoride gels on esthetic materials have been performed under conditions that may limit their clinical relevance, since specimens have been stored in these agents during extended and uninterrupted periods of time to simulate repeated exposure to fluorides.[25,27]

Considering that fluoride gels are to be used in patients with a moderate or a high caries risk,[11] the de- and remineralization model employed in this study, which shows correlation with high cariogenic challenges *in vivo*,[26] provides an attempt to reduce the limited predictive value of an *in vitro* protocol.

The effect of fluoride solutions or gels on the surface of dental materials has been evaluated through scanning electron microscopy (SEM),[27,30-34] surface profile tracings,[35] mass monitoring,[36] or microhardness evaluations.[25] SEM studies are extremely useful because surface changes can be qualitatively investigated.[29] However, traditional scanning electron microscopy involves the use of a high vacuum chamber and thus requires drying procedures and a conducting coating. The potential for artifact occurrence with this method may account for erroneous interpretation of photomicrographs.[37] In this study, an attempt was made to minimize this by using an environmental scanning electron microscopy (ESEM), which differs from the conventional, since the sample chamber allows the presence

of gas.[38] Therefore, specimens can be evaluated without any preparation,[39] which is desirable with resin-modified glass ionomer cements, considering their susceptibility to dehydration.[40]

The materials under study revealed erratic patterns concerning their micromorphology as a result of fluoride gel treatments. The characteristics of filler particles such as their composition,[27] shape and size,[16,30] as well as the entanglement of the resinous and inorganic matrices[16,34] play an important role in the behavior of resin-based materials subjected to fluoride gels or solutions. In this context, the absence of significant difference among treatments for Fuji II LC and Dyract may be attributed to the lower size of their particles, since eventual degradations would be less evident, as showed by Figures 1 and 2. On the other hand, for Photac-Fil and F2000, whose particles are larger (Fig 3 and 4), the potential effects of treatments can be more accurately evaluated.[31]

Concerning the behavior of Photac-Fil, a substantial degradation was detected for specimens treated by both neutral sodium and acidulated phosphate fluoride. The filler particles were eroded and partially or completely exposed due to the absence of the surrounding matrix, and the matrix also appeared to be severely degraded. It is likely that the lack of significant difference between the neutral fluoride group and the acidulated group was a result of the fragility of this material due to insufficient coherence between the ionic matrix of the acid-base reaction and the polymerization matrix of the radical reaction,[16,34] which could increase its susceptibility to erosion. The degradation provided by the APF gel was probably due to leaching of the matrix-forming cations, Na, Ca, Al, and La, and the formation of complexes with the acid anions.[27] The surface erosion observed on the NNaF group can be attributed to an increase in alkalinity of the NNaF gel, as speculated by Billington et al.[41] for glass ionomer cements. However, this would be unlikely to occur in

vivo.[27] Thus, according to this study both acidity and alkalinity can account for Photac-Fil micromorphological damage.

Unlike the results observed with Photac-Fil, F2000 revealed significantly more severe degradation after APF applications in comparison with DW and NNaF treatments, which did not differ from each other. This may be attributed to the different nature of these two materials, since for polyacid-modified composite resins, glass particles are partially silanized providing a direct bond with the resin matrix.[42] In this way F2000 behave more like composite resins than glass ionomers [16], which may explain the fact that the NNaF gel has no significant effect on this material. Based on the erratic behavior pattern shown by Photac-Fil and F2000 as a result of NNaF and APF treatments, degradation depends not only on the pH of the gel, but probably also on the gel's ability to form a complex with the metal ions of the restorative material.

Although additional information could be obtained from the comparison of the behavior of restorative materials after NNaF and APF treatments, the dissimilar size of the filler particles does not make this comparison by photomicrographs possible.

Controversial findings have been reported on the susceptibility to degradation of resin-modified glass ionomer cements and polyacid-modified composite resins treated with fluoride gels.[25,34,35] Due to the diversity in methodologies used, it is difficult to compare the present observations with those of other studies. Moreover, scanning electron microscopy may not be a sensitive tool for detecting changes in surface micromorphology. Therefore, further investigations are needed to elucidate the behavior of dental materials under *in vivo* conditions, since *in vitro* tests are not capable of reproducing the inherent complexity of the oral environment.[43]

CONCLUSIONS

Under the experimental conditions used in this study, the following conclusion can be drawn:

1. The surface micromorphology of both Fuji II LC Improved and Dyract was not significantly affected by applications of any of the fluoride gels, neutral and acidulated.
2. The resin-modified glass ionomer Photac-Fil showed a substantial degradation due to the applications of both neutral and acidulated fluoride gels.
3. The surface of the polyacid-modified composite resin F2000 was significantly degraded by the acidulated fluoride gel.

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Table 1. Restorative materials tested.

Brand Name	Code	Type	Basic Composition*	Particle size (μm)	Batch #	Manufacturer
Fuji II LC Improved	Fj	Resin-modified glass ionomer	Al-fluorosilicate glass, tartaric acid, polyacrylic acid, HEMA	1.8 *	031271	GC Corp
Photac-Fil Aplicap	Po	Resin-modified glass ionomer	Na-Ca-Al-La-fluorosilicate glass, polyacrylic acid, maleic acid, HEMA	7 – 40 *	FW0042436	Espe
Dyract	Dy	Polyacid-modified resin composite	Sr-Al-fluorosilicate glass, UDMA, TCB resin	2.37 ∀	9706000436	LD Caulk/Dentsply
F2000	F2	Polyacid-modified resin composite	Al-fluorosilicate glass, colloidal silica, CDMA-oligomer, GDMA, hydrophilic polymer	3 - 10 *	7AD	3M Dental Products

* According to the manufacturers
∀ Information taken from Gladys et al. (1997)

Table 2. Restorative materials used according to manufacturers' recommendation.

Restorative Material	Shade	Dispensing / Mixing	Insertion	Light Curing
Fuji II LC Improved	A3	Powder/liquid (ratio 3.2g:1.0g) / Manually	Centrix syringe	20 s
Photac-Fil Aplicap	C4	Predosed capsule / Capmix	Aplicap system	20 s
Dyract	C4	Capsule / No mixing	Capsule tip and syringe	40 s
F2000	CY	Syringe / No mixing	Metal spatula	40 s

Table 3. Description of fluoride gels used.

Brand	Composition*	Code	pH*	Batch #	Manufacturer
Nupro - Neutral	Sodium fluoride	NNaF	6.5 – 7.5	8E014	Dentsply Ltda.
Nupro - Acidulated	Sodium fluoride, fluoridric and phosphoric acids	APF	3.6 – 3.9	8C257	Dentsply Ltda.

*Information taken from the manufacturer

Table 4. Results of Kruskal-Wallis test and of pairwise comparisons, to compare the effect of surface treatments on the micromorphology of studied materials.

Restorative Material	Treatment	n	Median	Average Ranks
Fuji II LC Improved	DW	10	1	11.9 ┐
	NNaF	10	1	17.3 ┤
	APF	10	1	17.3 ┘
Photac-Fil Aplicap	DW	10	1	7.0
	NNaF	10	2	18.6 ┐
	APF	9	2	20.0 ┘
Dyract	DW	10	1	12.0 ┐
	NNaF	10	1	16.0 ┤
	APF	10	1	18.7 ┘
F2000	DW	9	1	8.5 ┐
	NNaF	10	1	12.9 ┘
	APF	10	2	23.0

Values connected by brackets did not differ from each other
lsd = 7.67: for n_1=10 and n_2=9
lsd = 7.46: for n_1=10 and n_2=10

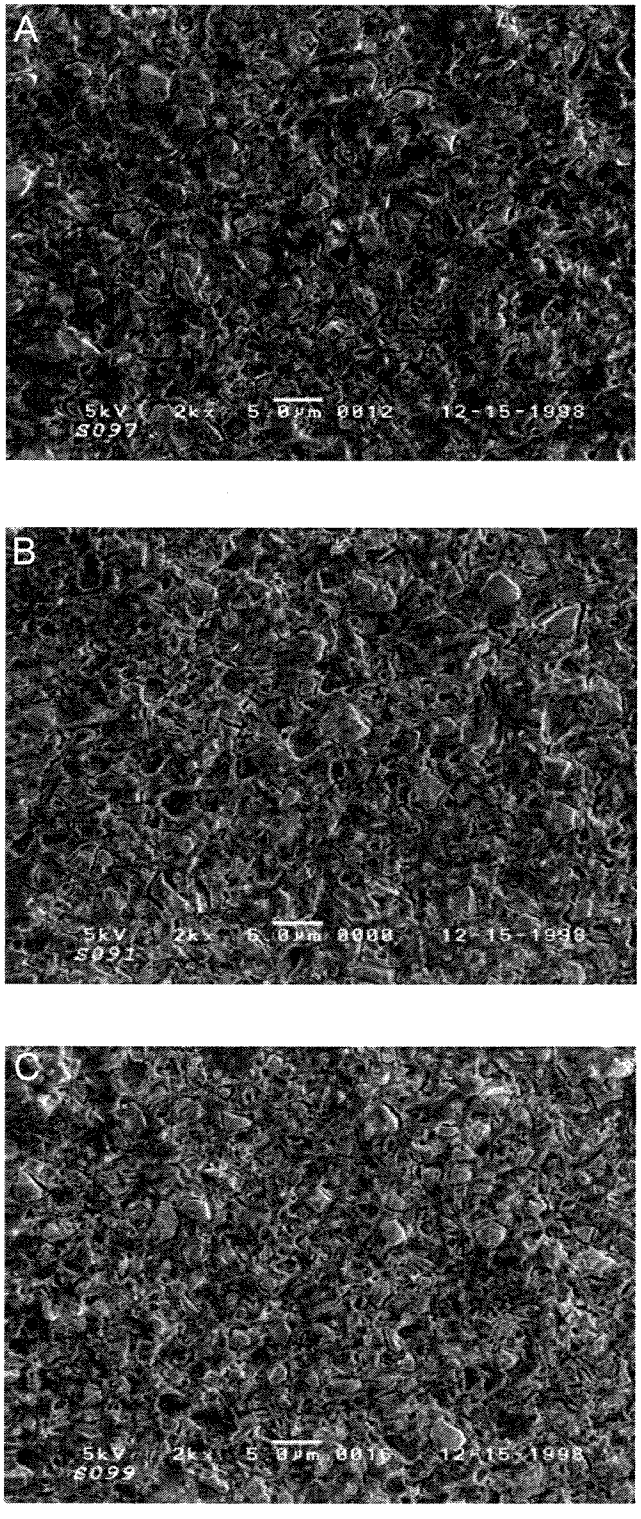

Fig 1. Micromorphological pattern of Fuji II LC Improved treated with DW (A), NNaF (B), and APF (C), revealing no differences as a result of these treatments.

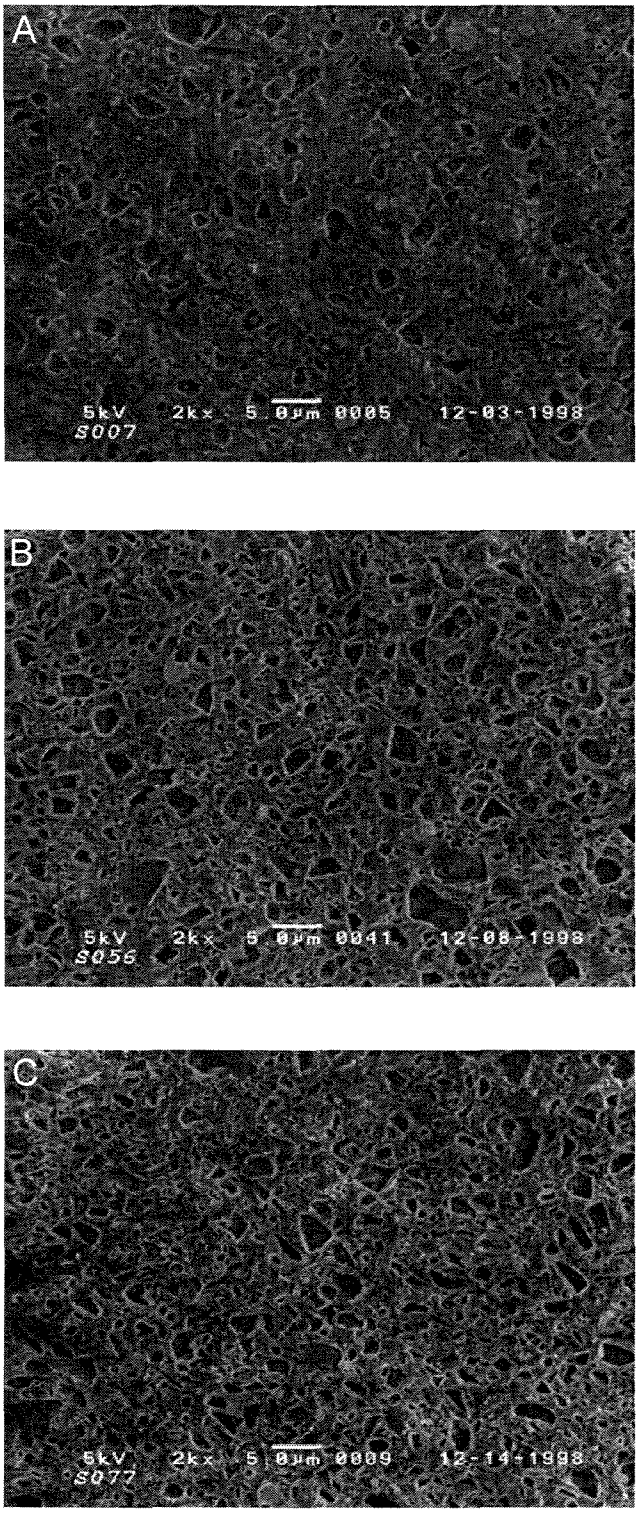

Fig 2. Similarity of the Dyract surface appearance after treatment with DW (A), NNaF (B), and APF (C).

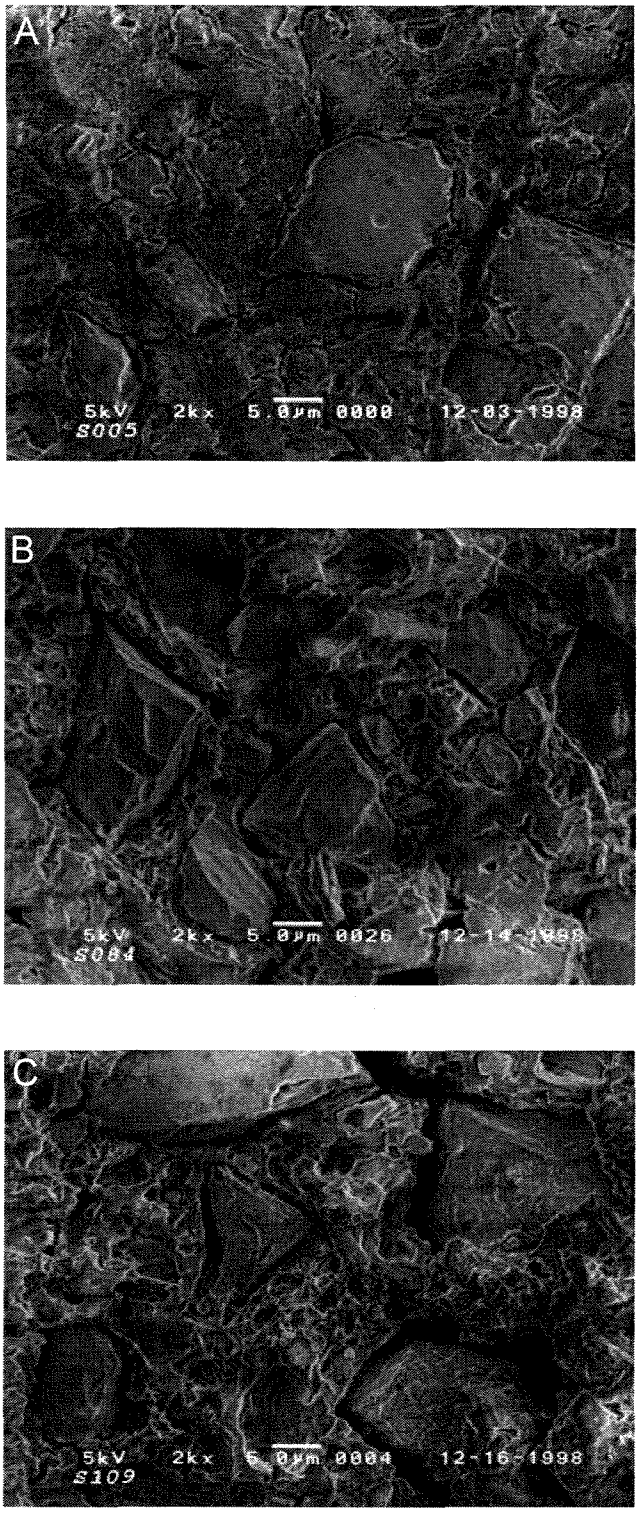

Fig 3. Photomicrographs of Photac-Fil Aplicap after application of DW (A), NNaF (B), and APF (C), showing degradation of both filler particles and the resinous matrix after NNaF and APF applications.

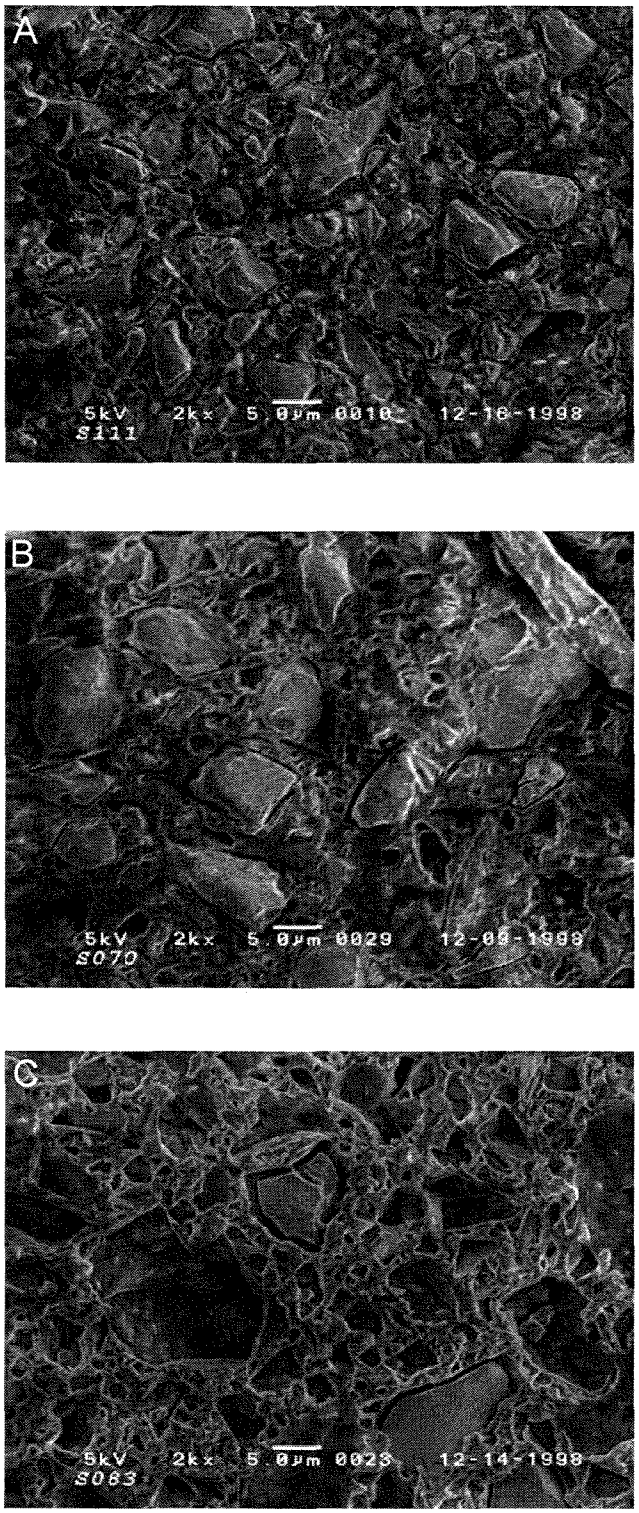

Fig 4. F2000 surface micromorphology following DW (A), NNaF (B), and APF (C) treatments, exhibiting the highest degradation with the APF agent.

CONTINUING EDUCATION

1. Which of the following is false about fluoride gels?
   A. They can inhibit tooth demineralization.
   B. They can enhance tooth remineralizing potential.
   C. They are not indicated for patients who have teeth restored with resin-modified glass ionomer cements or polyacid-modified composite resins.
   D. They can promote fluoride re-uptake by glass ionomers.
2. Which of the following can play a part in the degradation by fluoride gels of resin-modified glass ionomer cements and polyacid-modified composite resins?
   A. Composition of filler particles.
   B. Entanglement of the resinous and inorganic matrixes.
   C. Size of filler particles.
   D. All of the above.
3. The degradation rate of resin-modified glass ionomer cements and polyacid-modified composite resins were found to be:
   A. Dependent on the particular characteristics of each material.
   B. Higher after APF applications.
   C. Lower as a result of NNaF gel applications.
   D. More evident for those with low particle sizes.
4. APF applications can provide:
   A. Severe erosion in all hybrid materials.
   B. An increase in alkalinity.
   C. A formation of a complex with the metal ions of the restorative materials.
   D. All of the above.

Answers

	A	B	C	D
1.	☐	☐	☒	☐
2.	☐	☐	☐	☒
3.	☒	☐	☐	☐
5.	☐	☐	☒	☐

4. CONCLUSÃO

Nas condições em que estes quatro estudos foram conduzidos, pôde-se concluir que diferentes processos podem proporcionar alterações micromorfológicas aos materiais restauradores estéticos e, especificamente, que:

1) os materiais híbridos e as resinas compostas apresentaram alterações micromorfológicas quando submetidos a uma condição de alto desafio ácido;

2) a degradação proporcionada pela simulação de uma condição de alto desafio ácido, quando seguida pela escovação, desencadeou aos materiais híbridos superfícies menos rugosas em comparação àqueles armazenados em água destilada e deionizada ou saliva artificial. A textura dos compósitos, conseqüente à escovação, não foi influenciada pelo meio de armazenamento;

3) ao longo dos ensaios de escovação, precedidos por ciclos de pH, a rugosidade superficial de resinas compostas e de resinas compostas modificadas por poliácidos apresentou-se inalterada. Aumento progressivo, intercalado por períodos de estabilização, foi observado na rugosidade superficial de um ionômero de vidro modificado por resina;

4) os ionômeros de vidro modificados por resina e as resinas modificadas por poliácidos apresentaram comportamentos peculiares frente à aplicação de géis fluoretados.

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18th January 2001

Dear Dr Serra

Re " Effect of storage media upon the surface micro-morphology of resin based restorative materials "

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a) Title page - please add " Running title " and " Key Words " (6)
b) Page 9 - 1st line- van Groeningen et al
22nd line- Nicholson et al 1999 a & b
c) Page 11 3rd line - El-Badrawy & McComb 1998
d) Figures - It would be preferable to have glossy prints available.

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January 29, 2001

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I have received our referees' comments regarding your article **"Surface Roughness Assessment of Resin-Based Materials Throughout Brushing Preceded by pH-Cycling Simulations"**. Copies of these comments are included for your review. On the basis of these reviews I am pleased to inform you that your paper is conditionally accepted for publication in *Operative Dentistry*. Full acceptance is predicated on addressing the areas of concern expressed by the referees. The concerns expressed by the referees appear to be addressable areas, thereby making it possible to do the corrections without changing your research design. Please make the suggested changes to your paper and return the altered manuscript and diskette within 30 days. If you feel some changes are not justified, please defend your position without making the recommended changes.

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Congratulations on the acceptance of your article, **"Effect of fluoride gels on micromorphology of resin-modified glass ionomer cements and polyacid-modified composite resins."** The editorial board has recommended your manuscript for publication in *Quintessence International* and we look forward to sharing your work with our readers. The manuscript will be forwarded to our editorial office where it will be edited and typeset. All further communication should be directed to **Ms. Sally Schmid, Assistant Editor, Quintessence Publishing Company, 551 Kimberly Drive, Carol Stream, Illinois 60188.**

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