Brazilian Dental Science
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UNIVERSIDADE ESTADUAL PAULISTA "JÚLIO DE MESQUITA FILHO"
Instituto de Ciência e Tecnologia
Campus de São José dos Campos



ORIGINAL ARTICLE

doi: 10.14295/bds.2014.v17i3.1014

Effect of mouthrinses on the surface roughness of nanofilled composite

Efeito de enxaguantes bucais sobre a rugosidade de superfície de um compósito nanoparticulado

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ABSTRACT

Objective: The aim of this study was to evaluate the effect of mouthrinses on the surface roughness of nanofilled restorative composite. **Material and Methods:** Twenty Filtek Z350 resin composite discs (4 mm in diameter and 1 mm in height) were made and roughness (μm) was measured by using profilometer with diamond measuring needle tip of 0.5 μm and accuracy of 0.01 μm. The discs were stored in distilled water at 37 °C for 24 h and randomly divided into four groups (n=5): Control - distilled water; Group 1- Plax alcohol free mouthrinse ; Group 2 –Plax Whiteningmouthrinse; Group 3 –Listerine mouthrinseand individually stored in Eppendorf tubes with distilled water at 37 °C. The specimens were maintained in distilled water during 30 days and immersed in mouthrinses every 12 h for 1 min. Elapsed the 30 days, the specimens were cleaned and storedin distilled water, inside anincubatorat 37 °C during 24 h and the surface roughness test was repeated with the same parameters previously described. The data were submitted to Two-way ANOVA. **Results:** No significant differences among groups were detectedfor the variables mouthrinse($p = 0.9038$) and time ($p = 0.2056$), R1: Control (0.12 ± 0.04); G1 (0.13 ± 0.05); G2 (0.11 ± 0.03); G3 (0.11 ± 0.02); and R2: Control (0.16 ± 0.04); G1 (0.15 ± 0.02); G2 (0.13 ± 0.01) and G3 (0.15 ± 0.02). **Conclusions:** In this study the mouthrinses solutions did not promote significant changes in the surface roughness of Filtek Z350 after 30 days.

KEYWORDS

Composites; Polymer; Dental materials

RESUMO

Objetivo: O objetivo nesse estudo foi avaliar o efeito de enxaguantes bucais sobre a rugosidade superfície de um compósito restaurador nanoparticulado. **Material e Métodos:** Vinte discos (4 mm de diâmetro e 1 mm de altura) do compósito restaurador Filtek Z350 foram confeccionados e sua rugosidade (μm) foi mensurada utilizando um rugosímetro de contato com ponta de diamante de 0,5 μm e precisão de 0,01 μm. Os discos foram armazenados em água destilada a 37 °C por 24 h, aleatoriamente divididos em quarto grupos (n=5): Controle– água destilada; Grupo 1 –EnxaguantePlaxálcool free; Grupo 2 – EnxaguantePlaxWhitening; Grupo3 – EnxaguanteListerinee armazenados individualmenteem Frascos Eppendorfs com água destilada a 37°C. Durante os 30 dias de armazenamento os corpos-de-prova foram imersos nos enxaguantes bucais a cada 12 h por 1 min. Após os 30 diasos corpos-de-provaforam limpos e armazenados em água destilada a 37 °C por 24 h e as medidas de rugosidade refeitas com os mesmos parâmetros descritos anteriormente. Os dados de rugosidade foram submetidos à Análise de Variância de dois fatores. **Resultados:** Não foram identificadas diferenças significativas entre os grupos para as variáveis enxaguante ($p=0,9038$) e tempo ($p=0,2056$), R1: Controle (0,12 ± 0,04); Grupo1 (0,13 ± 0,05); Grupo2 (0,11 ± 0,03); Grupo3 (0,11 ± 0,02); e R2: Controle (0,16 ± 0,04); Grupo1 (0,15 ± 0,02); Grupo2 (0,13 ± 0,01) e Grupo3 (0,15 ± 0,02). **Conclusões:** No presente estudo, os enxaguantes bucais não promoveram alterações significativas na rugosidade de superfície do compósito Filtek Z350 após 30 dias.

PALAVRAS-CHAVE

Compósitos; Polímeros; Materiais dentários.

INTRODUCTION

The use of restorative composites has been consolidated as the material of choice for direct dental restorations [1,2]. According to a previousstudy [3],the clinical performance of restorative composites depends on the ability to resist to both masticatory stress and degradation by organic solvents. The restorative composites are composed by polymeric matrix, filler particles, and coupling agents [4], these components directly influenceon the properties of composites [5,6]. The resin matrix consists of ablend of organic dimethacrylatesmonomers, such as:Bisphenol A-Glycidyl methacrylate (Bis-GMA), Urethane Dimethacrylate (UDMA) and Triethylene glycol dimethacrylate (TEGDMA) that will determine thepolymer properties [7,8]. In a previous study, it was observed that organic solvents as ethanol and water may degradethe polymer network over time [9].

Filler particles play an important role on the mechanical properties of composites, such as flexural strength, fracture toughness, microhardness, and surface roughness[10]. The composites can be classified according to the size of the filler particles, for example: traditional micro-hybrids (0.01 – 3.5 μm) and nanofilled (particles smaller than 100 nm) [11,12]. According to manufacturers, nanofilled composites are the most indicated for anterior and posterior restorations, because the nano-sized fillers decreasethe polymerization shrinkage and improve their mechanical properties [13].

Mouthrinses are widely used to help in controlling and preventing caries lesions and periodontal disease, even without dentist's recommendation [14], and may be incorrectly overused. The mouthrinse composition varies according to the manufacturer and isbasically water, antimicrobial agents, salts, preservatives and, in some cases, alcohol [4]. The concentration variation of these substances affects oral pH [15]. Previous studies have shown that mouthrinses with low pH and high alcohol content may affect some physical-mechanical properties of the composites [16,17]. Accordingly, a previous study reported that Listerine produced major reduction of hardness, while another in vitro study [17] showed that resin has significantly gained weight while immersed in alcohol-containing mouthrinses in comparison with alcohol-free ones. In another study, the action of mouthrinses had promoted a significant modification on the surface of resin composite and enhanced its solubility [4].

Due to the consolidation of resin composites as restorative material and the widespread use of mouthrinses, it is important to evaluate the effect of these solutions with different pH and different concentrations of ethyl-alcohol on the surface of restorative composite.Thus, the aim of this study was to investigate the effects of three mouthrinses available in the market (one alcohol free, onecontaining alcohol, and one containing alcohol and whitening agent), on the surface roughness of a nanofilled restorative composite.The null-hypothesis tested was that there would be no difference in surface roughness of composite resin after the use of the mouthrinses.

MATERIAL AND METHODS

pHevaluation of the solutions

The mouthrinses used in this study are shown in Table 1. The solutions were agitated during 1 min in a magnetic agitator (model 752, Fisatom Scientific equipment Ltd., São Paulo - SP, Brazil.) and the assessments were made in a pH meter (Orion 3-star, pH portable meter, Thermo Electron Corporation, Beverly, MA, USA).

Preparation of specimens

The nanofilled composite resinshade A1 (Filtek Z350, 3M ESPE, St Paul, MN, USA.) was inserted into a metallic mold with 4 mm of internal diameter and 1 mm of height. Two polyester strips were positioned above and below the matrix, the composite resin was inserted into a single increment, and then a constant pressure of 1kgF were applied for 1 min using a device for standardization. The specimenswere photo-activated with a LED source unit (Bluephase, Ivoclar-Vivadent, Schaan, Liechtenstein) with 946 mW/cm^2 of output power measured according to a calibrated power meter (OphirOptronics Ltd, Jerusalem, Israel) for 20 s (18,9 J/cm^2) in contact with the glass slab on the topof the specimen.After the matrix removal,the bottom surface of the resin discs were marked with a diamond burand then the specimens were stored for 24 h in a light-free environment at 37 ºC.

Surface Roughness

The surface roughness initial evaluation of the specimens wasobtained through a rugosimeter (Surfcorder SE 1700, Kosaka Lab, Tokyo, Japan) witha diamond needle with tip measuring of 0.5 μm, accuracy of 0.01 μm, and cut-off value was set at 0.08 mm. The stylus was traversed across the diameter at the central portion onthe top-face of each sample three times, and the mean roughness parameter for each specimen (Ra, μm) was recorded as the mean of the three readings.

Use of mouthrinses

After Ra, the specimens were randomly divided into 4 groups: Control - distilled water (no mouthrinse was used); Group 1- Plax alcohol free; Group 2 – Plax Whitening; Group 3 – Listerine, and then, stored into individual vials containing 1 ml of distilled water at 37ºC. Atevery 12 h, the specimens were removed from the vials, dried in absorbing paper and immersed in 1 ml of the solution for1 min. After the immersion, each resin composite disc was washed for 60 s in distilled water and stored again in their respective vials with 1 ml of distilled water at 37 ºC. The distilled water in the storage vials was replaced atevery 12 h, including the control group, which has not been exposed to any solution. The mouthrinses used in this study are shown in Table 1.

The exposure of the specimens to the rinsing solutions was maintained for 30 days. The composite discs were removed from the distilled water, kept in dry environment at 37 ºC for 24 h, and then the surface roughness test was repeated through using the same parameters and positionspreviously described.The results were submitted totwo-way ANOVAtest ($p < 0.05$).

SEM preparation

Fifteen additional specimens were constructed as previously described and randomly divided into five groups for SEM examination (n = 3). Three specimens were observed asphoto-activated, without any

Table 1 - Mouthrinse solutions and their composition

Solution	Manufacturer	Batch number	Composition
Listerine	Johnson & Johnson Industrial Ltd., São José dos Campos, SP, Brazil	L 2700803	Water, ethyl alcohol, sorbitol, PEG-40, aroma, sodium saccharin, cetylpyridinium chloride, sodium, phosphate, eucalyptol, methyl salicylate, fluorinated sodium, sodium fluoride, 110 ppm of fluorine, menthol and CI42090*
Plax alcohol free	Colgate-Palmolive Industry e Commerce Ltd., S.B Campo, SP, Brazil	BR122A	Water, glycerin, propylene glycol, sorbitol, PEG-40, sodium benzoate, aroma, phosphoric acid, fluorinated sodium, cetylpyridinium chloride, sodium saccharin, sodium fluoride, 225 ppm of fluorine*
Plax Whitening	Colgate-Palmolive Industry e Commerce Ltd., S.B Campo, SP, Brazil	BR122A	Water, sorbitol, ethyl alcohol, hydrogen peroxide (1.5%), polymer 338, polysorbate 20, methyl salicylate, menthol, sodium saccharin, CI 42090*

**Information provided by the manufacturer*

treatment. The remaining resin discs were treated as aforementioned described for each experimental group. After the treatments all the specimens were sputtering-coated with gold and examined by SEM.

RESULTS

Two-way ANOVA revealed no significant difference between the values of surface roughness of the nanofilled composite at the baseline measurements and after 30 days of exposure to mouthrinse solutions (Table 2). Values ofsurface roughnessare described in Table 3. No differences were detectedin the composite surface before and after the exposure to the mouthrinses (Figure 1.) All the specimens presented the same feature: a smooth surface with the presence of filler clusters on the subsurface. Table 3 shows the pH values found for each solution, which turned out to be slightly acid even without alcohol.

Table 2 - Two-way ANOVA

Source of Variation	Df	Sum-of-squares	Mean square	F	P value
Interaction	3	0.005080	0.001693	0.6359	0.6027
Mouthrinses	3	0.001192	0.0003975	0.1869	0.9038
Time	1	0.004635	0.004635	1.741	0.2056
Subjects (matching)	16	0.03403	0.002127	0.7988	0.6707
Residual	16	0.04261	0.002663		

Table 3 - Evaluation of pH of mouthrinses and surface roughness values expressed in micrometer (µm) followed by standard deviation in parenthesis.

Mouthrinse	pH	Surface roughness (µm)	
		24 h	30 days
Control	6.21	0.1219 (0.04) Aa	0.1667 (0.035) Aa
Group 1 (Plax alcohol free)	5.10	0.12622 (0.05) Aa	0.14616 (0.024) Aa
Group 2 (Plax Whitening)	3.52	0.10844 (0.03) Aa	0.1341 (0.007) Aa
Group 3 (Listerine)	5.43	0.11204 (0.02) Aa	0.14776 (0.024) Aa

Equal capital letters in line and equal lower case letter in column represents statistics similarityforTwo-way ANOVA.

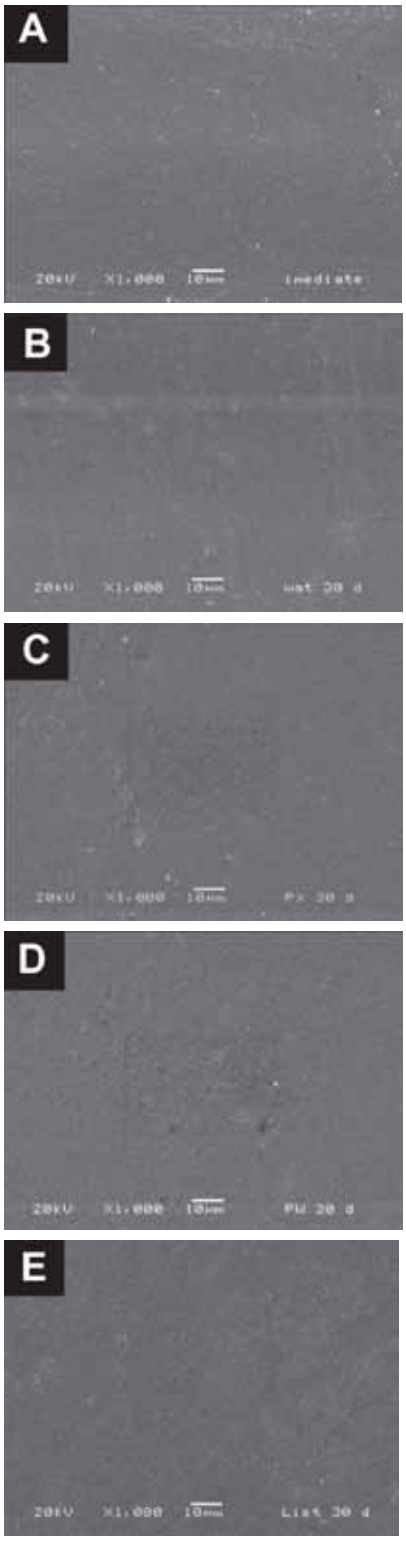


Figure 1 - SEM images of resin composite surface.(A) Immediately after the polymerization. (B) Control – after 30 d storage in water; (C) after 30 d storage in water and exposed to Plax; (D) after 30 d storage in water and exposed to Plax Whitening and (E) after 30 d storage in water and exposed to Listerine.

DISCUSSION

The composition of mouthrinses commercially available variesaccording to the manufacturer.In some cases, the alcohol may be present, such as ethanol. The resins undergodegradation inside the mouth [18-20], which changethe properties and can also interfere in clinical performance. Previous studies have shown that alcohol interferes in the physical-mechanical properties of composite resins [1,21].However, in this present study, the null-hypothesis was accepted.

Some studies have observed a reduction in the resin properties when higher ethyl-alcohol concentration mouthrinses were employed [4,22]. In contrast, the current study revealedthat the resin surface roughness was not altered by the mouthwashes tested. These findings maybe explained by the time (1 min in every 12 h) to which the mouthrinses were exposed, because itcould have been insufficient to cause alterations in compositesurface. Notwithstanding,we used the time period recommended by the manufacturer in order to inhibit biofilm formation. In a previous study [4], the mouthrinse exposure time was 2 min and promotedboth the surface degradationand the sorption and solubility increase.

Although alcohols and substances with low pH may degrade restorative composites [16,17], in thispresent study, the alcohol concentration and pH of the tested solutions did not influence the composite surface roughness. The alcohol concentration ranged from0% (Plax alcohol free) to21% (Listerine), according to the Material Safety Data Sheet (MSDS) of each material, and mayhave not been enough to break significantly the cross-linking of the polymeric matrix,to promotevisible modifications on the material surface (Figure 1),and toincrease the surface roughness. Previous studies have showed the action of acid substances onthe restorative materials [16-18]and dental tissues [23-25], nevertheless, the pH presented by the solutions used in this study(between 3.42 and 5.43) were not able to produce significant degradation on the surface, probably because of the short time of contact (1 min) and the interval between the applications (12 h) which enabled returning the pH to neutral conditions due to the storage in distilled water. This storage mediumwas chosen because this study's objective was to evaluate only the influence of the mouthrinses on the surface roughness, not the storage. However, it is important to emphasize that the buffer capacity of saliva in vivocan further reduce the effects of these substances on the restorative composite.

In this present study, the specimens were exposed to the mouthrinses atevery 12 h for 30 days, situation that mostly approaches to that recommended by the manufacturers. This exposure time was insufficient to cause composite surface alterations, although it was longer than the time employed in a previous study [4]. Additionally, the specimens were not polished, procedure that enhances the surface area and creates surface irregularities when compared to the polishing left by the polyester strip, allowing solution infiltration in the polymeric matrix and resulting in the resin degradation [26-30]. Therefore, these facts could explain the different results reported by previous investigations [4,22].

Although nanofilled composite resin surface roughness has not been altered by the mouthrinses tested, other studies are necessary to evaluate longer exposure times using artificial saliva as storage solution in order to mimic the clinical condition. Also, other properties such as hardness, color stability and tensile strength may be assessed to provide more precise data about the effects of pH and alcohol concentration of the solutions on the restorative materials.

CONCLUSION

Based on the results of this study and within the limitations of the applied methodology, it was possible to conclude that regardless the formulation, the mouthrinses did not affect the composite surface roughness during the study period (30 days).

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Date submitted: 2014 Jun 18
Accept submission: 2014 Sep 18