

UNIVERSIDADE ESTADUAL DE CAMPINAS FACULDADE DE ODONTOLOGIA DE PIRACICABA

VINICIUS DE OLIVEIRA LOIOLA

AVALIAÇÃO DA MICRODUREZA E DA RUGOSIDADE DE DIFERENTES TIPOS DE RESINA FRENTE À ESCOVAÇÃO MECÂNICA SIMULADA E AO CICLO EROSIVO

EVALUATION OF MICROHARDNESS AND ROUGHNESS OF DIFFERENT TYPES OF RESIN IN SIMULATED TOOTH BRUSHING AND EROSIVE CYCLE

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Trabalho de Conclusão de Curso apresentado à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Cirurgião Dentista.

Undergraduate final work presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Dental Surgeon

Orientadora: Marcela Alvarez Ferretti

Coorientador: Prof. Dr. Flávio Henrique Baggio Aguiar

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RESUMO

O obietivo deste trabalho foi avaliar a rugosidade superficial (Ra) e a microdureza Knoop (KNH) das resinas compostas convencionais e do tipo bulk-fill. Oitenta amostras foram feitas e dividas em 8 grupos (n=10): G1 - Filtek Z350 XT (3M CONV E), G2 - Filtek Z350 XT (3M CONV E+C), G3 - Filtek Bulk-fill (3M BULK E), G4 - Filtek Bulk-fill (3M BULK E+C), G5 -Tetric N-Ceram (IVO CONV E), G6 - Tetric N-Ceram (IVO CONV E+C), G7 - Tetric N-Ceram Bulk-fill (IVO BULK E) e G8 - Tetric N-Ceram Bulk-fill (IVO BULK E+C). Ø 4x2 mm para convencionais e 4x4 mm para bulk-fill. Todos os grupos foram polidos e receberam o desafio abrasivo (E), com simulação de escovação mecânica com 20.000 ciclos escovatórios por 15 dias. Quatro grupos receberam o desafio abrasivo somado ao erosivo (E+C), com a imersão em 15 mL de ácido cítrico 0.02 M por 1 minuto por 4x/dia por 15 dias. Foram avaliados Ra e KNH antes e após os ciclos abrasivos e somados ou não aos erosivos. Os dados foram analisados pela ANOVA de medidas repetidas de três vias com post hoc de Bonferroni (a =0.05). IVO CONV E, IVO BULK E, 3M CONV E+C, 3M BULK E+C e IVO CONV E+C aumentaram a Ra. 3M BULK E e 3M BULK E+C aumentaram a KNH. Portanto, a Ra é influenciada pelo tipo de composição química do compósito, eventos abrasivos e erosivos possibilitam o aumento da Ra e o ciclo erosivo não depreciou a KNH.

Palavras-chave: Resinas compostas, escovação dentária, erosão dentária.

ABSTRACT

The aim of this study was to evaluate surface roughness (Ra) and Knoop microhardness (KHN) of conventional and bulk-fill resin composites. Eighty cylindrical specimens were made and were divided into 8 experimental groups (n=10): G1 - Filtek Z350 XT (3M CONV E), G2 - Filtek Z350 XT (3M CONV E+C), G3 - Filtek Bulk-fill (3M BULK E), G4 - Filtek Bulk-fill (3M BULK E+C), G5 - Tetric N-Ceram (IVO CONV E), G6 - Tetric N-Ceram (IVO CONV E+C), G7 - Tetric N-Ceram Bulk-fill (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill (IVO BULK E+C). Ø 4x2 mm for conventional and 4x4 mm for bulk-fill. All the groups were polished and submitted to simulated toothbrushing (E) with 20,000 reciprocal strokes for 15 days. Four groups were submitted to erosive cycle and associated with toothbrushing (E+C), through immersion in citric acid solution (0,02 M) for 1 min and repeated 4x/day during 15 days. Ra and the KHN were evaluated before and after the treatments. Data were analyzed by three-way ANOVA with Bonferroni *post-hoc* test ($\alpha = 0.05$). IVO CONV E, IVO BULK E, 3M CONV E+C, 3M BULK E+C and IVO CONV E+C increased the surface roughness. 3M BULK E and 3M BULK E+C increased the KHN. Thus, Ra is influenced by the chemical resin composition, abrasive and erosive cycles increase the roughness and the erosive cycle did not decrease the Knoop microhardness of the resin composites.

Key words: Composite Resins, Toothbrushing, Tooth Erosion.

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1 INTRODUÇÃO

A ampla utilização de resinas compostas como material restaurador é justificada pelos fatores relacionados à coloração dental, às propriedades mecânicas e à substituição das restaurações de amálgama (Ferracane, 2011). Desde o seu desenvolvimento, ocorreram avanços em sua composição, como a redução do tamanho da partícula de carga, visando aprimorar a estética, o polimento e a resistência ao desgaste (Lu et al., 2006).

Algumas técnicas restauradoras foram desenvolvidas, a fim de reduzir a quantidade de falhas entre o compósito e a estrutura dental adjacente. Procedimentos restauradores com resinas compostas convencionais requerem a realização da técnica de inserção incremental, a qual define a inserção de incrementos em aproximadamente 2 mm de espessura (Bicalho et al., 2014b). Essa técnica é indicada por promover uma polimerização eficiente e uma menor contração de polimerização do compósito, aumentando a taxa de sucesso desta restauração ao longo do tempo. Contudo, a inserção de vários incrementos no procedimento aumenta a chance de erro e a contaminação entre eles, além de ocasionar maior tempo clínico para a sua execução (Bicalho et al., 2014a).

Com o intuito de contrapor esses fatores, foram desenvolvidas as resinas compostas do tipo bulk-fill, cuja alteração em sua composição proporcionou a inserção de incrementos de 4 a 6 mm (Sampaio et al., 2017). Diferentes abordagens foram utilizadas para aumentar a profundidade de polimerização do compósito, como a utilização de fotoiniciadores adicionais, o aumento da translucidez do material através da diminuição da quantidade e do aumento dimensional das partículas de carga do compósito (Chesterman et al., 2017). A possibilidade de trabalhar com incrementos maiores propicia rapidez, facilidade no processo de restauração e diminuição do risco de contaminação em razão da redução de etapas (Reis et al., 2017).

A presença do compósito resinoso na cavidade oral está suscetível aos possíveis desafios químicos e mecânicos, que podem influenciar na depreciação de suas propriedades mecânicas e, como resultado, alterar a sua longevidade. Os ácidos podem ser endógenos, advindos dos ácidos gástricos, e exógenos, proveniente dos alimentos e bebidas da dieta (Shellis e Addy, 2014). O hábito de higiene oral com uma escovação vigorosa pode ocasionar a abrasão, que é o desgaste da estrutura dental pela força mecânica aplicada em sua superfície, podendo causar aumento na rugosidade dos materiais restauradores, a qual se liga às suas propriedades ópticas e propicia o acúmulo de biofilme (Roque et al., 2015; Reis et al., 2017).

Com o aumento do consumo de alimentos ácidos somado à escovação mecânica com desgaste abrasivo, tornam-se necessários estudos acerca de sua influência nas resinas compostas convencionais e do tipo bulk-fill em sua rugosidade superficial e microdureza Knoop, com intuito de fornecer conhecimento e auxiliar na escolha dos materiais restauradores (Attin et al., 2003). As hipóteses testadas foram: 1 - escovação altera a rugosidade superficial das resinas compostas convencionais e bulk-fill, 2 - escovação altera a microdureza Knoop das resinas compostas convencionais e bulk-fill, 3 - ciclo erosivo altera a rugosidade superficial de resinas compostas convencionais e bulk-fill e 4 - ciclo erosivo altera a microdureza Knoop de resinas compostas convencionais e bulk-fill.

2 ARTIGO: SUPERFICIAL BEHAVIOR OF ROUGHNESS AND KNOOP MICROHARDNESS OF DIFFERENT TYPES OF RESIN IN SIMULATED TOOTH BRUSHING AND EROSIVE CYCLE

Submetido no periódico Journal of Esthetic and Restorative Dentistry (Anexo 3)

ABSTRACT

Objective: Evaluate surface roughness and Knoop microhardness of conventional and bulk-fill resin composites. Materials and Methods: Four resin composites were evaluated: Filtek Z350 XT 3M ESPE, Filtek Bulk-fill 3M ESPE, Tetric N-Ceram Ivoclar – Vivadent and Tetric N-Ceram Bulk-fill Ivoclar – Vivadent. Twenty cylindrical specimens of each material were divided into 8 experimental groups (n=10). All the groups were polished and submitted to simulated toothbrushing (E) with 20,000 reciprocal strokes, 200 g load and slurry. Four groups were submitted to erosion cycle (E+C), through immersion in citric acid solution (0,02 M) for 1 min and repeated 4x/day. The surface roughness and the Knoop microhardness were evaluated before and after 15 days of both treatments. Data were analyzed by three-way ANOVA with Bonferroni *post-hoc* test. The level of significance was 5%. Results: IVO CONV E, IVO BULK E, 3M CONV E+C, 3M BULK E+C and IVO CONV E+C increased the surface roughness. 3M BULK E and 3M BULK E+C increased the Knoop microhardness. Conclusion: Surface roughness is influenced by the type of composition of the restorative material, abrasive and erosive cycles increase the roughness and the erosive cycle did not decrease the Knoop microhardness of the resin composites.

Clinical relevance: The surface roughness and Knoop microhardness changes according to the composition of the resin composite and its related to the longevity of the restorative material.

Key words: Composite Resins, Toothbrushing, Tooth Erosion, Surface Roughness, Knoop Microhardness.

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INTRODUCTION

The wide use of resin composite as a restorative material is justified by the factors related to capability of mimic natural teeth, as well as its similarity in their mechanical properties, as its color that simulates the aesthetic of a natural tooth (1). Since its development, there was several advances in its composition, such as the reduction of the size of filler particles, which aims to improve the aesthetics, the polishing and the wear resistance, change in resin matrix and shape (2).

Some restorative techniques were developed to reduce the failure between the composite and the adjacent tooth structure. Restorative procedures with conventional composite resins require the use of the incremental filling technique, which defines the insertion of increments in approximately 2 mm thick (3). This technique is indicated for promoting an efficient polymerization, reducing polymerization shrinkage and increasing the success rate of restoration over time. However, the insertion of several increments increases the probability of failure, contamination among the composite increments and requiring an increasing chairside period (4).

In order to counteract these factors, there were developed bulk-fill resin composites, which alterations in its composition provided inserting increments of 4 mm up to 6 mm thick (5). Different strategies were utilized to increase the curing depth of the composite, such as the increase of translucency of the material through the decrease of the quantity, the dimensional increase of filer particles of the composite and a higher incorporation of photoinitiator reagents (6). The possibility to work with the increase of thickness reduces the clinical chair-side time, facilitates the restoration process and reduce the risk of failure because of the decrease of restoration steps (7).

Moreover, the presence of resin composite in the oral cavity is susceptible to chemical and mechanical challenges, which can influence the depreciation of its mechanical properties and, as a result, reducing its longevity. The acid can be endogenous, from gastric acid, and exogenous, from food and drinks and its presence may causes dental erosion, which the presence of acid causes demineralization of the tooth structure (8). The habit of daily brushing promotes an abrasion on tooth surface and is related to the load, type of toothbrush and toothpaste with abrasive applied. These events may increase the surface roughness and hardness of the restorative material, which is correlated with its optical properties and may cause accumulation of biofilm (7,8).

The increase of the consumption of acidic beverages associated with toothbrushing demonstrates the importance of studies about their influence on the surface of conventional

and bulk-fill resin composites. Within, roughness surface and Knoop microhardness were evaluated, in order to provide knowledge and facilitate the ideal choice of restorative materials (10). The hypotheses tested were: 1 - simulated toothbrushing change the surface roughness of conventional and bulk-fill resin composites, 2 - simulated toothbrushing change the Knoop microhardness of conventional and bulk-fill resin composites, 3 - erosive cycle change the surface roughness of conventional and bulk-fill resin composites and 4 - erosive cycle change the Knoop microhardness of conventional and bulk-fill resin composites and 4 - erosive cycle change the Knoop microhardness of conventional and bulk-fill resin composites.

MATERIAL AND METHODS

Eighty light-curing specimens were made to form 8 groups (n=10): G1 - Filtek Z350 XT 3M ESPE (3M CONV E), G2 - Filtek Z350 XT 3M ESPE (3M CONV E+C), G3 - Filtek Bulk-fill 3M ESPE (3M BULK E), G4 - Filtek Bulk-fill 3M ESPE (3M BULK E+C), G5 - Tetric N-Ceram Ivoclar – Vivadent (IVO CONV E), G6 - Tetric N-Ceram Ivoclar – Vivadent (IVO CONV E+C), G7 - Tetric N-Ceram Bulk-fill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulkfill Ivoclar – Vivadent (IVO BULK E) and G8 - Tetric N-Ceram Bulk-

Resin composite	Туре	Manufacturer	Organic matrix	Inorganic matrix	Shade
Filtek Z350 XT	Nanofilled	3M ESPE, St Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Silica, zirconia and aggregated zirconia/silica cluster filler	A2E
Filtek Bulk-fill	Nanofilled	3M ESPE, St Paul, MN, USA	Bis-GMA, Bis- EMA, UDMA, TEGDMA, resin Procrylat	Silica, zirconia, an aggregated zirconia/silica cluster filler and a ytterbium trifluoride filler	A2
Tetric N-Ceram	Nanohybrid	lvoclar ivadent, AG, Schaan, Listeinstaine	TEDGMA, Bis- GMA, UDMA, Bis- EMA	Barium glass, ytterbium, triflouride, mixed oxid and prepolymer	A2
Tetric N-Ceram Bulk-fill	Nanohybrid	lvoclar ivadent, AG, Schaan, Listeinstaine	Bis-GMA, UDMA	Barium glass, ytterbium, Trifluoride, prepolymer and mixed oxide	IVA

Table 1: Technical specifications of materials according to the manufacturer.

Bis-GMA: bisphenol A diglycidyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated.

The cavity of the matrix was filled with single increments of light-curing resin composite using composite spatula (Suprafil SSWhithe Duflex - Juiz de Fora, MG, Brazil). After the insertion of the composite inside the matrix, it was placed on the surface a polyester strip, a glass coverslip and 500 load were put on the increment for 3 min, promoting a flat and regular surface, without the presence of voids. After the removal of the weight, the resin composites

were light-cured (VALO, Ultradent Products Inc; S. Jordan, UT, EUA - 1400mW/cm²) for a period of time indicated by the instructions of the manufactures, and the excess was removed by scalpel blade number 15 (Free-Bac, Zhangjing Town, XC, China). The specimens were stored in microtubes in relative humidity for 24 hours in an incubator at 37°C, in order to guarantee the full polymerization of the material.

After 24 hours, the specimens were subjected to a polishing process in a polishing machine (modelAPL-4; Arotec, Cotia, SP, Brazil). The polishing was performed with silicon carbide sandpaper in the followed grains side (#1200, #2000 and #4000) (CARBIMET Paper Discs; Buehler, IL, EUA) under water-cooling. The last silicon carbide sandpaper was standardized for 1 minute and a half. After each silicon carbide sandpaper utilized, aiming to remove any debris that may exist on the surface between one sandpaper and another, the specimens received a wash in ultrasonic baths (Ultra Clearer USC-1450A/Frequency 25kHz, UNIQUE, Indaiatuba, SP, Brazil). At the end of the process of polishing, the specimens were again immersed in ultrasonic bath with distilled water for 15 minutes. Subsequently, there were measured the initial values of surface roughness and Knoop microhardness of the 80 specimens. Through the results of the average surface roughness, it was randomization the samples and were divided in the following groups (Table 2).

Groups (n=10)	Description of groups
3M CONV E	Resin composite Filtek Z350 XT + simulated toothbrushing
3M CONV E+C	Resin composite Filtek Z350 XT + simulated toothbrushing and erosive cycle
3M BULK E	Resin composite Filtek Bulk-fill + simulated toothbrushing
3M BULK E+C	Resin composite Filtek Bulk-fill + simulated toothbrushing and erosive cycle
IVO CONV E	Resin composite Tetric N-Ceram + simulated toothbrushing
IVO CONV E+C	Resin composite Tetric N-Ceram + simulated toothbrushing and erosive cycle
IVO BULK E	Resin composite Tetric N-Ceram Bulk-fill + simulated toothbrushing
IVO BULK E+C	Resin composite Tetric N-Ceram Bulk-fill + simulated toothbrushing and erosive cycle

EROSIVE CYCLE

The groups 3M CONV E+C, 3M BULK E+C, IVO CONV E+C and IVO BULK E+C were submitted to erosive cycles. The demineralizing solution was made with 19,2130 g of citric acid 0,02 M (ANIDRO P.A.-A.C.S., Synth, Diadema, SP, Brazil), the powder was placed in a volumetric flask and 5 L and the distilled water was slowly added. As the mixture between acid and distilled water occurred, circular movements were performed with the volumetric flask in

order to homogenize the solution. The solution was made one day before the start of the erosive cycle and was properly sealed. The specimens were immersed in 15 mL of citric acid pH 2.0 for 1 minute, 4 times a day for 15 days. This process was realized at 8h, 14h, 18h and 20h, according to a modification of the protocol proposed by Roque et al. 2015 (9). Each specimen were washed with distilled water between the immersions and dried-stored in an incubator at 37°C.

SIMULATED TOOTHBRUSHING

The simulated toothbrushing was executed in all groups using 80 toothbrushes (Oral B Indicator Plus 30 – Procter & Gamble, Cincinnati, USA), one for each specimen. A doublesided diamond disk (KG Sorensen, Barueri, Brazil) was used to separate the brush heads, and their handles. Thus, they were fixed in a brush holder device of the brushing machine MSet (Marcelo Nucci ME, São Carlos, Brazil) through thermal glue (Brascola, São Bernardo do Campo, SP, Brazil). The toothbrush heads were positioned parallel of of the specimens' surface. Furthermore, the toothbrushing utilized a slurry, which was made with 8 g of dentifrice, utilizing an analytical balance, and with 24 ml of distilled water with a precision pipette forming a ratio of 1:3 to perform the dilution of the dentifrice through its mass. The specimens received 40,000 movements (20,000 cycles) linear brushing movements, corresponding to 2 years of brushing (10). The frequency was 4 Hz with a 200 g load to simulate the force applied on the specimens'surface. The 40,000 movements were divided and applied equally over 15 days. The specimens that were abraded and associated with the erosive cycle, started the toothbrushing after the first day of erosive cycle.

After 15 days, the specimens were removed from the machine, washed with distilled water and dried with absorbent paper (Kleenex - KimberlyClark, São Paulo, SP, Brazil). At the end of this treatment, the surface roughness and Knoop microhardness were evaluated.

ANALYSIS OF SURFACE ROUGHNESS

The reading of the surface roughness was made before (baseline) and after the simulated toothbrushing associated or not to the erosion cycle (final) by the profilometer (Surftest 211; Mitutoyo Corp.,Tokyo, Japan). All the specimens were put on an acrylic base and above the surface of the specimen was positioned the measuring tip. The values were measured utilizing cut-off 0,25 mm and 0,05 mm/s speed. Three readings were made on the surface of the specimens on different positions and the mean of these values was obtained. The three readings occurred with an equally rotation in manner that the last reading was rotated a total of 120°. The mean values of the baseline were used for randomization of the specimens from the same group of the same resin composite (11).

ANALYSIS OF THE KNOOP MICROHARDNESS (KHN)

After the initial measurement of the surface roughness, the specimens were taken to the microhardness tester (HMV-2000, Shimadzu Corporation, Tokyo, Japan), which contains a Knoop diamond indenter to realize the reading with 25 gf load for 10 s. It was realized five Knoop indentations on the surface of the specimen, one at the center with a distance of 100 μ m from the center indentation. The mean values of the indentations were calculated as KNH for each specimen (12).

ANALYSIS OF RESULTS

The statistical analysis was made using SPSS 21.0 (SPSS, Chicago, IL, USA) program. The values of surface roughness and Knoop microhardness were analyzed its normality on Kolmogorov-Smirnov. It was performed three-way repeated measures ANOVA, due to three factors: comparison between the resin composites resins (3M CONV, 3M BULK, IVO CONV and IVO BULK at the same time), the time (before and after) and the treatment (simulated toothbrushing and erosion cycle) with Bonferroni *post-hoc* test. The significance level was set at 5%.

RESULTS

SURFACE ROUGHNESS

The mean and standard deviation values of the surface roughness are shown in Table 3. Before the simulated toothbrushing, all the groups were statistically similar. After the toothbrushing, there was a statistical difference between the groups. IVO E differed statistically from all composites and showed the highest surface roughness, followed by IVO BULK E, 3M CONV E and 3M BULK E. Comparing the time, before and after, IVO CONV E and IVO BULK E showed statistical difference on the simulated toothbrushing.

	Simulated toothbrushing		Simulated toothbrushing + Erosive cycle	
	Before	After	Before	After
3M CONV	0.083 (1.5·10⁻²) Aa	0.093 (1.5·10 ⁻²) Abc	0.079 (0.9·10 ⁻²) Ab*	0.097 (1.1·10 ⁻²) Aa*
3M BULK	0.085 (1.1·10 ⁻ 2) Aa	0.089 (1.6·10 ⁻²) Ac	0.085 (1.4·10 ⁻²) Aab*	0.100 (2.3·10 ⁻²) Aa*
IVO CONV	0.089 (1.0·10 ⁻²) Aa*	0.127 (1.2·10 ⁻²) Aa*	0.091 (1.0·10 ⁻²) Aa*	0.110 (1.9·10 ⁻²) Aa*
IVO BULK	0.090 (1.1·10 ⁻²) Aa*	0.110 (1.7·10 ⁻²) Ab*	0.096 (1.2·10 ⁻²) Aa	0.093 (1.7·10 ⁻²) Aa

Tabel 3. Mean and standard deviation of surface roughness.

Different lowercase letters refer to the statistical differences between the composite resins at each time of each treatment (column). Different capital letters refer to the statistical differences between treatments at

each evaluated time of each composite resin. * refer to statistical differences between the evaluated times of each composite resin for each treatment performed (line).

After the simulated toothbrushing and erosive cycle, all the resin composites were statistically similar. IVO CONV E+C presented the highest surface roughness, followed by 3M BULK E+C, 3M CONV E+C and IVO BULK E+C. Comparing the time, before and after, 3M CONV E+C, 3M BULK E+C and IVO CONV E+C differed statically with the simulated toothbrushing and erosive cycle.

The surface roughness values isolating times, before the treatments on simulated toothbrushing and added by the erosive cycle, there was no statistical difference between each other. After both treatments, there was also no statistical difference isolating times.

KNOOP MICROHARDNESS

The mean and standard deviation values of the Knoop microhardness are shown in Table 4. Before the simulated toothbrushing, there was a statistical difference between all the resin composites. After the simulated toothbrushing, the 3M composites were statistically similar to each other, as the IVO composites were statistically similar to each other. 3M BULK E presented the highest Knoop microhardness, followed by 3M CONV E, IVO CONV E and IVO BULK E. Comparing the time, before and after, only 3M BULK E showed a statistical difference with the simulated toothbrushing, increasing its value.

	Simulated	toothbrughing	Simulated toothbrushing + Erosive		
	Simulated toothbrushing		cycle		
	Before	After	Before	After	
3M CONV	81.38 (2.8) Aa	81.15 (8.6) Aa	80.64 (6.8) Aa	83.44 (5.4) Aa	
3M BULK	73.01 (3.9) Ab*	84.89 (5.2) Aa*	72.38 (4.8) Ab*	80.78 (9.4) Aa*	
IVO CONV	64.15 (6.7) Ac	59.51 (9.3) Ab	65.00 (6.1) Ac	59.76 (6.8) Ab	
IVO BULK	59.06 (4.0) Ad	59.18 (7.9) Ab	59.13 (6.8) Ad	57.69 (3.8) Ab	

Tabel 4. Mean and standard deviation of Knoop microhardness.

Different lowercase letters refer to the statistical differences between the composite resins at each time of each treatment (column). Different capital letters refer to the statistical differences between treatments at each evaluated time of each composite resin. * refer to statistical differences between the evaluated times of each composite resin for each treatment performed (line).

Before the simulated toothbrushing and erosive cycle, all the groups differed statistically from each other. After the toothbrushing and erosive cycle, the 3M groups were statistically similar when compared to the IVO composites.

The 3M groups were statistically different from the IVO groups. 3M CONV E+C presented the highest Knoop microhardness, followed by 3M BULK E+C, IVO CONV E+C and IVO BULK E+C. The Knoop microhardness values isolating times, before the treatments on simulated toothbrushing and added by the erosive cycle, there was no statistical difference between each other. After both treatments, there was also no statistical difference isolating times.

DISCUSSION

Resin composites are used to restore the function and the aesthetic of the tooth, they can have varied compositions and mechanical properties. The presence of these materials in the oral cavity is susceptible to routine acts as toothbrushing and acid beverage consumption, which may influence surface alterations of restorative materials. Thus, negative effects on the surface can change its roughness and microhardness, which are related to gingival inflammation and biofilm accumulation (13). In this way, this study evaluated the influence of abrasive associated or not to erosive cycle on the surface properties of conventional and bulk-fill resin composites.

The toothbrushing is related to the abrasive process, which happens through gradual degradation of the organic matrix, which is softer, resulting in elevation and removal of filler particles that cause the formation of protuberance and depressions on the composite surface (14). When the filler particles are not well located its elimination occurs easily with abrasive challenges and the absence of this particle on the surface of the resin composite favors its wear in a faster and uninterrupted way. As a consequence, it can have an alteration of the surface roughness of the restorative material, which is influenced by applied force, duration of the toothbrushing and abrasives present in the dentifrice (15). Furthermore, particularities of the restorative material can impact the abrasive challenge, as the size of the filler particle, type of monomer of the organic matrix and the photopolymerization (16). After 20.000 cycles of simulated toothbrushing, which corresponds approximately to 2 years of brushing, resulted in an increase of the surface roughness for IVO CONV E and IVO BULK E, while 3M CONV E and 3M BULK E did not alter its surface roughness. The dimensional size of the filler particles can impact the result. Once 3M conventional and bulk-fill have a maximum size of 20 nm, except for ytterbium trifluoride particles that agglomerated can reach 100 nm on 3M bulk-fill and the IVO conventional and bulk-fill can reach 3000 nm. The increase of the surface roughness of IVO restorative materials may be influenced by its composition of prepolymer fillers, which act reducing the polymerization shrinkage still on abrasion cycle impacts on larger size fillers and the removal of this particle creates space on the surface of the resin compos affecting the longevity of the polish of these materials (17). Furthermore, the presence of prepolimerized fillers is associated with a limitation of the quantity of fillers that can be incorporated on the resin composite and can be related to a lower mechanical property (18). Consequently, the first hypothesis that simulated toothbrushing would change the surface roughness of conventional and bulk-fill composites was accepted.

Resin composites can be classified according to the size of the filler particles. The dimensional size of filler particles on nanohybrid composites 40 to 3000 nm, while for nanocomposites 5 to 20 nm (19). An important mechanical property that is related to dimensional size of filler particles concerns wear resistance is hardness, which is also responsible for the resin composite behavior on brushing (20). Smaller filler particles of composites show a better resistance to processes that cause wear since they present a greater homogeneity and a smaller amount of protruding particles. On the other hand, bigger filler particles have less resistance to wear when it comes to nanofilled. Therefore, it is expected that nanofilled resin composites present a better wear resistance since it has a greater amount of particles than nanohybrid (21, 22). The abrasive process caused by brushing, the 3M CONV E, IVO CONV E and IVO BULK E did not change its Knoop microhardness, while 3M BULK E increased the Knoop microhardness, rejecting the second hypothesis that brushing would change the Knoop microhardness of conventional and bulk-fill resin composites.

The ingestion of acid beverages increases the chances of occurrence of dental erosion and denotes the importance of studies aiming to produce knowledge about its effect on restorative materials (23). Chemical substances can increase the surface roughness and wear of these materials, increasing their process of degradation (24). Studies have shown that beverages with a pH of 5,5 or less are capable to cause enamel wear (25). In the case of composite resins, low pH beverages causes dissolution of the organic matrix due to the erosive wear, leaving it softened and favoring the leaching of the inorganic portion of the resin composite. Consequently, increases the possibility of filler particles being removed. There is also interference on filler particles, which makes them unstable and reduces the strength of these materials. The citric acid, utilized on the erosive cycle, has a derogatory action on both hard dental tissues and resin restorative materials. The dissolution of the organic matrix is intensified with the abrasion caused by toothbrushing, increasing the wear and the removal of filler particles promoting an irregularities on the surface roughness of resin composite (26). Through the analysis of surface roughness values, it was observed alteration of this property with simulated toothbrushing and erosive cycle. 3M CONV E+C, 3M BULK E+C and IVO CONV E+C increased the surface roughness, except IVO BULK E+C, which did not alter this property after the simulated toothbrushing and erosive cycle. Accepting the third hypothesis that erosive cycle would change the surface roughness of conventional and bulk-fill resin composites.

Resin composites can absorb water through the organic portion, which is expanded and favors leaching, resulting in hydrolysis and the breaking of chemical bonds between the polymer matrix and filler particles present in the composite composition. Hence, facilitating the removal of the filler particles on the surface, making the region rougher and decreasing its microhardness (27). Even though the effects of chemical substances promote changes in the organic matrix developing an important role in the properties of the restorative material, the composition of these materials influence directly the behavior of the resin composites on wear (28). It is possible to notice this through the study of Tantanuch et al. 2016, in which the erosion was proportionated by wines and it was verified that nanofilled composites presented less wear on their surface roughness when compared to the nanohybrid composites. this demostrates that nanofilled composites have better filler homogeneity and present a better wear resistance, due to increasing its inorganic portion incorporated to the organic matrix and a greater amount of filler particles that are related to better physical and mechanic properties than nanohybrid.

Tanthanuch et al. 2018 (29) exposed different types of composite resins to cycle with different beverages, such as pineapple juice and passion fruit juice, all the resin composites presented a decrease in their microhardness. Nonetheless, in our study only 3M BULK E+C presented alteration in Knoop microhardness with the increase of this property. The 3M BULK have on its composition zirconia and denotes higher resistance to wear than the organic matrix, a possible explanation for the increase in Knoop microhardness is through the exposure of these filler particles as a result of the abrasive and erosive process (30). Although the IVO COV E, IVO CONV E+C, IVO BULK E and IVO BULK E+C composites have in their composition barium glass, these resin composites did not alter its Knoop microhardness. The presence of barium glass makes the restorative material more susceptible to attacks an aqueous environment. This is explained due to the material having contact with water result in exchange of hydrogen ions from barium with water causing an increase in pH and promotes glass leaching. Furthermore, the alkaline medium accelerates the hydrolysis of the silanol bond between the inorganic particle and the silanizing agent (31). Torres et al. 2015 (32) performed the immersion of composites in 2 ml of citric acid and did not verify alteration in Knoop microhardness of the resin composites that were cycled. This result corroborates with the findings of this study with the absence of alteration in Knoop microhardness on 3M CONV E+C, IVO CONV E+C and IVO BULK E+C resin composites. Possibly this result could be different if the time of the cycle was longer. The abrasive process added by the erosive cycle altered only 3M BULK E+C Knoop microhardness, rejecting the fourth hypothesis.

Thus, observing the values of the surface roughness, the simulated toothbrushing altered the properties of IVO CONV E and IVO BULK E composites. The simulated toothbrushing and erosive cycle changed the Knoop microhardness of 3M CONV E+C, 3M BUL E+C and IVO CONV E+C. When it comes to the Knoop microhardness, the simulated toothbrushing isolated and added by the erosive cycle only 3M BULK E and 3M BULK E+C altered this property increasing its value. Therefore, occurred a difference between the resin composites with different treatments.

A limitation of this study was the period of time that was conducted the erosive and abrasion cycle, probably a longer duration of treatment would alter the results and the absence of the effect remineralizer of the saliva.

CONCLUSION

Within the limitations of this current study, it was concluded that:

- The surface roughness is influenced by the type of chemical composition of the restorative material.
- The abrasive and erosive cycles can increase the roughness.
- The erosive cycle associated with the toothbrushing did not decreased the Knoop microhardness after 15 days.

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3 CONCLUSÃO

De acordo com os resultados deste estudo, podemos concluir que a rugosidade superficial é influenciada pelo tipo de composição do material restaurador utilizado. Eventos abrasivos e erosivos conseguem aumentar a rugosidade. O processo erosivo proporcionado pelo ácido cítrico não depreciou a microdureza Knoop dos compósitos com a ciclagem erosiva durante os 15 dias.

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^{*} De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors - Vancouver Group. Abreviatura dos periódicos em conformidade com o PubMed.

ANEXOS

Anexo 1 – Verificação de originalidade e prevenção de plágio

EVALUATION OF MICROHARDNESS AND ROUGHNESS OF DIFFERENT TYPES OF RESIN IN SIMULATED TOOTH BRUSHING AND EROSIVE CYCLE

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Anexo 2 – Iniciação Científica



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Bolsista: Vinicius De Oliveira Loiola- RA 206751

Orientador(a): Prof.(a) Dr.(a) FLÁVIO HENRIQUE BAGGIO AGUIAR

Projeto: "Avaliação da microdureza e rugosidade da resina bulk fill frente à escovação mecânica simulada e ciclo erosivo"

Bolsa: SAE/Unicamp Vigência: 01/08/2019 a 30/06/2020 Processo: 01-P-175/2019 e 01.P.96/2020

PARECER

Conclusão do Parecer:

Diante da pandemia de Covid-19, os esclarecimentos incluídos no relatório são importantes, visto que a pesquisa foi bastante afetada. O aluno focou em revisão de literatura sobre o tema da pesquisa e adquiriu bastante conhecimento teórico. Ele pretende confeccionar novas amostras e trabalhar até as leituras finais assim que o retorno das atividades presenciais for possível.

Aprovado

Pró-Reitoria de Pesquisa, 06 de outubro de 2021.

Samuelson Alex Nanini Pereira

PR ASSADMINISTRATIVOS / TÉCNICO EM ADMINISTRAÇÃO

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Atenciosamente, Luiz Eugênio A. M. Mello Diretor Científico

Frases para o Beneficiário

Não há frases associadas.

Transcrição de Parecer para o Beneficiário Não há transcrição associada.

Frases para Termo de Outorga Não há frases associadas.

Relatório Científico 2 (Aprovado)

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Anexo 3 – Comprovante de submissão do Artigo

Fwd: Manuscript submitted to Journal of Esthetic and Restorative Dentistry

De: Journal of Esthetic and Restorative Dentistry <<u>no-reply@atyponrex.com</u>> Assunto: Manuscript submitted to Journal of Esthetic and Restorative Dentistry Data: 14 de outubro de 2021 17:20:21 BRT Para: Marcela Ferretti <<u>marcela.a.ferretti@gmail.com</u>>

Dear Marcela Ferretti,

Your manuscript entitled "SUPERFICIAL BEHAVIOR OF ROUGHNESS AND KNOOP MICROHARDNESS OF DIFFERENT TYPES OF RESIN IN SIMULATED TOOTH BRUSHING AND EROSIVE CYCLE" has been successfully submitted online and is being delivered to the Editorial Office of *Journal of Esthetic and Restorative Dentistry* for consideration.

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