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Changes in the properties of bulk-fill resins under conditions of gastroesophageal reflux and bulimia

Mayara Manfrin Arnez[1], Tatiane Cristina Dotta[1,*], Leonardo de Pádua Andrade Almeida[1], Raisa Castelo[1], David Emanuel Ugarte[2], Andréa Cândido dos Reis[1], Alma Blasida Concepcion Elizaur Benitez Catirse[1]

[1] Department of Dental Materials and Prosthodontics, Ribeirão Preto School of Dentistry, University of São Paulo, Ribeirão Preto, São Paulo, Brazil.

[2] DDS, MS, PhD, Department of Oral Rehabilitation, School of Dentistry, National University of Concepción, Concepción, Paraguay.

Corresponding author:
Tatiane Cristina Dotta
Department of Dental Materials and Prosthodontics
School of Dentistry of Ribeirão Preto, University of São Paulo
Av. do Café, s/n, 14040-904,
Ribeirão Preto – SP, Brazil
Telephone: +55(16) 3315-4790
e-mail: tatianedotta@usp.br

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Aim: Evaluate the roughness, microhardness and color change of different Bulk Fill resins when submitted to the condition of gastroesophageal reflux and bulimia. **Methods:** 60 specimens (n = 10) of Bulk-Fill composite resins were made: M_1 – Filtek™; M_2 – Tetric N-Ceram and M_3 – OPUS, through a matrix 2x6 mm and light cured by the VALO light source. After polishing, initial analyzes (48 hours - T_0) of surface roughness (Ra), microhardness (VHN) and color change (ΔE) were performed. To simulate the oral condition of severe gastroesophageal reflux and bulimia, the specimens were immersed in hydrochloric acid (S_1) (pH 1.7) 4 minutes a day, for 7 days. Control group specimens were immersed in artificial saliva (S_2). Subsequently to immersions, mechanical brushing was performed for 3 minutes, three times a day, simulating 7 days of brushing. And again, the analyzes of Ra, VHN and ΔE were performed (7 days - T_1). Thus, hydrochloric acid immersion, mechanical brushing and Ra analysis were repeated at 14 days (T_2) and 21 days (T_3); and T_2, T_3 and T_4 (3 years) for VHN and ΔE. **Results:** After Shapiro-Wilk statistical test, ANOVA and Tukey test with Bonferroni adjustment ($p>0.05$), M_3 showed the lowest Ra at all times compared to the other resins, while the highest Ra was at T_0. M_1 and T_1 showed higher VHN. And M_2 and T_4 showed higher ΔE. **Conclusion:** Bulk Fill resins can be indicated for patients with Gastroesophageal Reflux and Bulimia, nonetheless, Tetric N-Ceram resin showed the worst results.

Keywords: Composite resins. Color. Hardness. Hydrochloric acid.

Introduction

Dental erosion and consequent loss and demineralization of mineralized tissues may be present in individuals with eating disorders, bulimia nervosa or gastroesophageal reflux disease, due to exposure to gastric acids[1-4]. Acids of extrinsic or intrinsic origins in contact with the dental surface can promote the irreversible loss of this substrate, whose increase in incidence and prevalence has been documented[5].

The pH of pure hydrochloric acid varies between 0.9 to 1.5 and after episodes of vomiting in the oral cavity, this pH does not fall below 1.5 due to esophageal buffering and dilution of saliva, leading to a serious and high-risk condition for the formation and progression of erosive lesions[6,7].

The durability of restorations depends on some factors, such as the choice of a suitable restorative material. Composite resins provide excellent restoration properties such as increased wear resistance[8]. However, acidic conditions can damage the physical and mechanical properties of these materials, leading to the degradation of the organic matrix and the exposure of inorganic filaments, changing the properties of resins reducing the durability of restorations[9].

With advances in the development of dental materials and clinical techniques, composite resins have become more widely used as direct restorative materials to satisfy patients with esthetic demands[10]. Many changes in its composition have been carried out since its inception, as well as the use of low-shrink, high-molecular-weight monomers to overcome the effects of polymerization shrinkage, one of the main deficiencies in the mechanical and chemical properties of these materials[11].

Bulk-Fill resins were developed to simplify the time-consuming incremental technique, with 4-5mm depth increments, and featuring bisGMA, UDMA, bisEMA and Procrylate monomer composition, plus a combination of ytterbium trifluoride and zirconia/ silica, giving the material a lower polymerization shrinkage[11-13].

Surface roughness, color stability and microhardness can affect the survival of restorations as well as the dentist's decision to replace them[14]. Faced with the development of materials with new chemical formulations, there is a need for new experimental studies that evaluate the physical and mechanical properties of these new composite resins, in the conditions of oral challenges of patients with gastroesophageal reflux and/or bulimia, in order to to propose a more specific observation in the manufacture of materials, so that the longevity of these resins can be guaranteed during aggressive and extreme oral situations.

Thus, this study aims to evaluate the effect of hydrochloric acid on the roughness, microhardness and color of different Bulk resins over 48 hours, 7 days, 14 days, 21 days and 3 years.

Material and Methods

Experimental Design

For the evaluation of the effect of the acid challenge associated with the mechanic on the variables surface roughness, microhardness and color change, the factors

for this study were: Restorative material (3 levels: M_1 – Composite Resin Filtek™ Bulk Fill (3M, Ribeirão Preto, São Paulo, Brazil); M_2 – Composite Resin Tetric N-Ceram Bulk Fill (Ivoclar Vivavent, São Paulo, Brazil) and M_3 – Composite Resin OPUS Bulk Fill (FGM, Santa Catarina, Brazil)), Solution (2 levels: S_1 - Hydrochloric Acid, S_2 - Artificial saliva) and Time (4 levels: T_0 - 48 hours; T_1 - 7 days; T_2 - 14 days; T_3 - 21 days; T_4 - 3 years) (Table 1).

Table 1. Division of groups according to response variables, variation factors and different levels

	SURFACE ROUGHNESS	MICROHARDNESS	COLOR CHANGE
Restorative material	M_1 – Composite Resin FiltekTM Bulk Fill;	M_1	M_1
	M_2 – Composite Resin Tetric N-Ceram Bulk Fill;	M_2	M_2
	M_3 – Composite Resin OPUS Bulk Fil	M_3	M_3
Solution	S_1 – Hydrochloric Acid;	S_1	S_1
	S_2 – Artificial saliva	S_2	S_2
Time	T_0 – 48 hours;	T_0	T_0
	T_1 – 7 days;	T_1	T_1
	T_2 – 14 days;	T_2	T_2
	T_3 – 21 days;	T_3	T_3
		T_4 – 3 years	T_4

Fabrication of test specimes

60 specimens (n=10) were made according to the manufacturer's instructions using a teflox matrix measuring 6 mm in diameter and 2 mm in depth.

With the aid of a resin spatula (Duflex, São Paulo, Brazil), the material was inserted into the matrix in a single increment. A polyester matrix and a glass plate were put on top of the filled cavity. Additionally, a weight of 1 kg was put on top to guarantee the complete filling of the matrix and to produce the overflowing of any excess material. Next, the light activation was carried out on the specimens light-curable using a curing light VALO (Ultradent – São Paulo, Brazil), in accordance with instructions from the manufacturer.

After the polymerization, samples were taken out of the matrix and kept in relative humidity for 24 hours, in the oven at 37±1 °C. Afterwards, the specimens were polished with Sof Lex discs (3M, São Paulo, Brazil) in a decreasing sequence of granulation, and one of the faces was marked to serve as a positioning guide, to be used with the Confocal Laser Microscope, Microhardness and spectrophotometer.

Storage of Specimens

All specimens were kept in relative humidity of artificial saliva in an oven at 37 ±1°C throughout the experiment period, they were only removed from the oven to be submitted to the action of hydrochloric acid and to the tests at the proposed times.

Gastroesophageal reflux condition and Bulimia

For the specimens of each material that were subjected to the acid challenge, each specimen was individually immersed in 15 mL of hydrochloric acid (pH = 1.7) for 4 min, once a day, for 21 days, under vibration. And for the time of 3 years, the specimens were immersed for 3 uninterrupted days[15].

Mechanical challenge

The brushing of the specimens was performed using the Pepsodent brushing machine. This test was performed before the readings of the times of 7 days, 14 days, 21 days and 3 years. Colgate Total 12 toothpaste was used. The volume of 10 g suspended in 10 mL of distilled water (1:1 proportion) in the appliance vats on the specimens. To perform the brushing, the time of 3 minutes was used, corresponding to 1025 cycles of the machine to simulate 7 days of brushing, three times a day. And for the 3-year brushing time, 2 hours and 5 minutes of brushing was used. After brushing, the specimens were washed in running water for 30 seconds and inserted again in relative humidity with artificial saliva in the oven at 37±1 °C.

Surface Roughness Readouts

Surface roughness readouts were performed after polishing the specimens at 48 hours, 7 days, 14 days and 21 days using a confocal laser microscope (LEXT OLS4000, Olympus, Japan). The device was calibrated to focus an image at 1500 µm through the 5x objective lens. The average roughness of the area (Sa, µm) of the polished surface of the specimens was measured. Data were obtained using OLS4000 software version 2.0 (LEXT OLS4000, Olympus Corporation, Tokyo, Japan).

Microhardness Readouts

The microhardness readouts were performed in the experimental time intervals of 48 hours, 7 days, 14 days, 21 days and 3 years. For this analysis, the microdurometer (HMV-2000 Shimadzu Corporation, Japan) was used, with a pyramid-shaped diamond coated penetrator of the Vickers type, with a load of 100 g, applied for 10 seconds. 3 readings were made in the upper surface region of each specimen at points equidistant from each other, and the average of the measurements was obtained.

Color Change Readouts

The color change readouts were performed after polishing the specimens at 48 hours, 7 days, 14 days, 21 days and 3 years using the SP62S spectrophotometer with Model QA Master I Software (X-RiteIncorporated - Neu-Isenburg Germany. Each specimen was carefully manipulated using clinical forceps (Millennium, Golgran, SP, Brazil), dried with absorbent paper, and kept in a device duly prepared with niches for placement of the specimens and standardization of the readouts against an opaque white background.

Color measurements were performed using the CIE L* a* b* color system. The ΔE* value is the total difference between two color stimuli and was calculated using following formula:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}.$$

Statistical Analysis

The results obtained were submitted to the Shapiro-Wilk normality test and data were analyzed using ANOVA test ($p \leq 0.05$) and Tukey test with Bonferroni adjustment, using the Assistat (7.7 beta) software package.

Results

In the interaction of the Material x Time of surface roughness, it was found that T_0 (48 hours) had higher averages than the other times and M_3 had means statistically lower than M_1 and M_2 ($p< 0,001$) (Table 2).

Table 2. Mean values for Roughness by interaction Material (M) x Time (T)

	M_1	M_2	M_3
T_0	2,39 ± 0,56 aA	2,80 ± 0,37 bA	2,48 ± 0,30 abA
T_1	2,43 ± 0,62 aA	2,63 ± 0,50 aA	1,78 ± 0,27 bB
T_2	2,37 ± 0,59 aA	2,36 ± 0,55 aB	1,89 ± 0,38 bB
T_3	2,28 ± 0,56 aA	2,77 ± 0,68 bA	1,77 ± 0,23 cB

Lowercase letter line sense
Capital letter column sense

For microhardness, in the means for the interaction of the Time x Material ($p< 0,001$) and Time x Solution ($p< 0,001$), it was found that the T_1 time presented averages statistically higher than the other times. The acid solution (S_2) showed statistically higher averages than artificial saliva (S_1). It is possible to verify that the material M_2 showed statistically lower averages than M_1 and M_3 (Table 3).

Table 3. Mean values for Microhardness by interactions Time (T) x Material (M) and Time (T) x Solution (S)

	T_0	T_1	T_2	T_3	T_4
M_1	49,15 ± 6,47 aA	52,53 ± 3,94 cA	53,03 ± 5,60 bA	47,23 ± 2,76 dA	47,72 ± 6,27 dA
M_2	40,09 ± 6,35 aB	44,32 ± 7,23 bB	38,99 ± 5,31 aB	40,60 ± 5,34 aB	41,72 ± 7,04 aB
M_3	43,74 ± 5,51 bB	51,26 ± 7,11 aA	46,31 ± 6,56 bC	47,42 ± 6,26 cA	52,58 ± 6,60 aC
S_1	45,46 ± 7,78 aA	48,06 ± 7,78 bA	42,88 ± 6,51 aA	43,54 ± 5,59 aA	44,75 ± 7,80 aA
S_2	43,20 ± 6,26 aA	50,67 ± 6,35 bA	49,33 ± 8,44 bB	46,63 ± 5,83 cB	49,96 ± 7,24 bB

Lowercase letter line sense
Capital letter column sense

For color change, in the means for the interaction of the Time x Material ($p< 0,001$), it was found that M_2 had higher averages than M_1 and M_3 and T_4 had means statistically higher than the other times (Table 4).

Table 4. Mean values for Color Change by Time (T) x Material (M)

	M_1	M_2	M_3
T_1	2,72 ± 0,93 aA	2,84 ± 1,42 aA	2,63 ± 1,33 aA
T_2	1,87± 0,87 aB	3,16 ± 1,59 bA	2,19 ± 1,76 aA
T_3	2,92 ± 0,99 aC	3,74 ± 1,81 bB	2,34 ± 1,45 aB
T_4	2,85 ± 1,48 aC	4,20 ± 1,65 bB	2,83 ± 1,19 aA

Lowercase letter line sense
Capital letter column sense

Discussion

The null hypothesis is that the acid does not change the properties of Bulk Fill resins, although in this work, OPUS Bulk Fill resin presented less roughness than Filtek Bulk Fill and Tetric N Ceram. This result may be related, mainly to the amount of inorganic components of Bulk Fill resins. The OPUS Bulk fill resin (79% by weight) has more inorganic component than Filtek Bulk Fill (76% by weight) and Tetric N Ceram (75% by weight), this composition probably favored its lower roughness and greater hardness.

The roughness was higher in the 48 hours when compared to the times of 7 days, 14 days, 21 days and 3 years. These results are not in accordance with several studies[16] where they stated that, over time, there is a degradation of the organic matrix of resins, which provides an increase in surface roughness as a function of the time of restorative materials. According to Ishii et al.[17] (2020), the polishing technique can cause the release of the particles of charge, which generate voids on the resin surface and thus collaborate to increase the roughness. On the other hand, the effect of daily brushing, may favor the smoothing of the surfaces of the specimens over time, due to the abrasion process, which is in accordance with Somacal et al.[18] (2020).

Somacal and collaborators[18] (2020) evaluated the effect of pH cycling and simulated brushing on the surface roughness of Bulk Fill resins, and although the pH cycle caused changes in the surface of the studied resins, it was not enough to generate changes in surface roughness. This result corroborates with the present study, since the low pH acid solution did not negatively influence the roughness of Bulk Fill resins.

In the literature, acidic solutions can result in damage to the surface and reduce the microhardness of restorative materials, as they cause the material to dissolve, soften the polymeric matrices and detach the filler particles(11,14). Tanthanuch et al.[16] (2018) also reported that the immersion of Bulk Fill resins in liquids and acid-simulating food drinks can negatively influence the surface properties of restorative materials.

The lower microhardness of the Tetric N-Ceram Bulk Fill (M_2) resin is probably related to the type of photoinitiator that this material presents in its composition, Ivocerin. This germanium-based initiator system has a high light-curing activity and an absorption spectrum that extends below 380 nm to 460 nm, with an absorption peak close to 408 nm[19]. When materials with this photoinitiator are photopoly-

merized with polywave light sources, they may present polymerization impairment, since the light source with this characteristic may present a problem of homogeneous light emission and thus interfere with the material's microhardness[20,21].

In addition, the lower microhardness of the Tetric N-Ceram Bulk Fill (M_2) resin may be related to the reduction in the percentage of inorganic components when compared to the other resins. This lower inorganic amount in the composition could have influenced the lower microhardness of the restorative material[16].

Alencar and collaborators[22] (2020), reported that after 7 days of immersion, the restorative materials used in the study (Filtek Z350XT, GrandioSO, Filtek Bulk Fill, X-tra fil) showed less microhardness in different solutions (deionized water, acid citric 5% and hydrochloric acid 0.1%). These previous results corroborate with the data of the present study, where the microhardness was reduced over the different analyzed times.

Acidic solutions can degrade the monomeric matrix of restorative materials, impairing hardness, roughness and increasing water sorption. Thus, the importance of the correct choice of restorative material in patients with severe dental erosion is evidenced[23].

It was found that the Tetric N-Ceram Bulk resin (M_2) under the action of acid (S_1) was more sensitive, as it presented a greater color change than the M_1 and M_2 resins after 14 days, a result that showed that there was time addiction.

When the color change occurs, this change may be related to the composition of the material, such as the type of photoinitiator system, type of monomer, percentage by weight and volume; and size of the charge particles, which can influence the stain susceptibility[24-26].

The color stability of composite resins can be mainly caused by water absorption and the hydrophilicity of the matrix. Most resin matrix compositions such as bisphenolglycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA) present hydrophilic molecules in their formulation, that is, with the ability to attract water with Bis-GMA, a slightly more hydrophilic component when compared to UDMA. Such a situation will have a direct impact on the detection of stains found when resins with this monomeric composition are immersed in solutions. In the present study, Tetric N-ceram resin was the compound that showed the greatest color change, and this fact is probably due to the presence of Bis-GMA in its monomeric composition[14,27,28].

According to Rüttermann et al.[29] (2010), color stability is related to the conversion of the photoinitiator system. This system can form by-products that fade thermally or under ultraviolet light and shift the color of the resin to a more red or yellow color. Tetric N-Ceram Bulk Fill resin has its own photoinitiator, Ivocerin. This type of photoinitiator can influence the susceptibility of material stains[30]. In addition, a hydrophilic matrix contributes to the discoloration of the material. However, even if the matrix structure is not hydrophilic, water and coloring fluids can diffuse in the composite resin and cause susceptibility to discoloration. Diffusion and discoloration occur when the inorganic and organic contents are not silanized correctly or when the integration in the resin matrix is not sufficient[31]. Moreover, the Tetric

N-Ceram Bulk Fill resin compared to the other study resins is the material that has a lower percentage of charge, an amount that could contribute to a greater color change in this material.

Smaller particles affect the pigment adsorption on the material surface, affecting the overall color saturation after staining[32]. According to Gönülol and Yilmaz[33] (2012), the monomer content and the surface roughness affect the color change of composite resins, more than the size of the filler particles. In the absence of pigments, the degree of conversion (proportion of remaining unreacted carbon-carbon bonds) and greater translucency of composite resins may be one of the other factors of color change, that is, color change is a multifactorial problem.

Acid solutions can promote the degeneration of resins leaving the surface rougher, which could allow greater pigment retention and thus influence the color change, justifying the results found when observing the significant averages according to the respective solution[16,30].

Clinical experiments are necessary for the validation of the methods used in this study, since the evaluation made in the present research was an in vitro analysis. The results of this study show that the choice of material should be considered when planning restorations in patients with gastroesophageal reflux. Furthermore, it can be observed that the composition of the restorative material (monomer, photoinitiator, particle size, and inorganic filler) and the presence of acid can have considerable effects on the properties of the different resins tested.

In conclusion and according to the methodology used, it is possible to conclude that the roughness was higher at 48 hours and the composite resin OPUS Bulk Fill always showed lower roughness when compared to the other resins. In addition, it was possible to observe that the acid did not negatively influence this property. The composite resin Filtek™ Bulk Fill showed the highest microhardness in 7 days. The acid negatively influenced the microhardness of the resins, however, Tetric N-Ceram Bulk Fill behaved better. Also, color change has increased over time. At 3 years, composite resin Tetric N-Ceram Bulk Fill showed greater color change when associated with hydrochloric acid.

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Declaration of Interests

The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

Author Contribution

Substantial contributions to the conception and design of the work: MMA; TCD; LPAA; RC. Substantial contributions to the analysis; MMA; TCD; LPAA, RC, Substan-

tial contributions to the interpretation of data for the work: MMA; ABCEBC. Drafting the work: MMA; ABCEBC. Reviewing it critically for important intellectual content: MMA; ACR; ABCEBC. Manuscript findings: MMA; TCD; LPAA; RC; DEU; ACR; ABCEBC. Final review and approval of the version to be published: MMA; TCD; LPAA; RC; DEU; ACR; ABCEBC. Agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved: MMA; TCD; ACR; ABCEBC. All authors actively participated in the manuscript's findings, revised and approved the final version of the manuscript.

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