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Bulk-fill restorative composites under simulated carious and erosive conditions

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ABSTRACT

Acidic conditions can cause hydrolysis and accelerate degradation of resin composites (RCs). Since there are limited and controversial data on the effect of acids on bulk-fill RCs, this study assessed the surface roughness (SR) and flexural strength (FS) of these RCs under simulated carious and erosion conditions. Bars of Filtek Bulk Fill (FBF, 3M/ESPE), X-tra fil (XTF, Voco), Tetric N-Ceram Bulk Fill (TBF, Ivoclar/Vivadent), and Aura Bulk Fill (ABF, SDI) and a conventional RC [Filtek Z350XT (FZ, 3M/ESPE)] were allocated (n=15) to undergo caries or erosion conditions. The control group was kept in artificial saliva (AS). The bars were evaluated for SR change (final-baseline) and for three-point FS. Data were analyzed using ANOVA and Tukey's test. At the baseline (p < 0.001), the SR of RCs ranked as follows: (TBF = XTF) < FBF (none differed from FZ) < ABF. The interplay between RCs and conditions affected SR change (p = 0.025). While after storage in AS, there was no difference among RCs, following carious and erosive conditions, ABF showed higher SR change. For FS (p < 0.001), XTF > (FBF = FZ) > (TBF = FZ) > ABF, with no difference among control, carious and erosive conditions (p = 0.148). Depending on the restorative bulk-fill RCs, carious and erosive conditions roughen the surface but do not affect the FS of these materials.

Keywords: *composite resins - acids - dental caries - tooth erosion.*

Resinas compostas restauradoras bulk-fill sob condições simuladas de cárie e erosão

RESUMO

Condições acídicas podem causar hidrólise e acelerar a degradação de resinas compostas (RCs). Como há dados limitados e controversos sobre os efeitos de ácidos sobre RCs bulk-fill, este estudo avaliou a rugosidade de superfície (RS) e a resistência flexural (RF) dessas RCs sob condições simuladas de cárie e erosão. Barras de Filtek Bulk Fill (FBF, 3M/ESPE), X-tra fil (XTF, Voco), Tetric N-Ceram Bulk Fill (TBF, Ivoclar/Vivadent) e Aura Bulk Fill (ABF, SDI) e de uma RC convencional [Filtek Z350XT (FZ, 3M/ESPE)] foram alocadas (n=15) para estarem sob condições cariogênicas ou erosivas. O grupo controle foi mantido em saliva artificial saliva (SA). As barras foram avaliadas quanto à alteração de RS (final-inicial) e à RF de três pontos. Os dados foram analisados utilizando ANOVA e teste de Tukey. Inicialmente (p < 0,001) a RS das RCs foi a seguinte: (TBF = XTF) < FBF (nenhuma diferiu de FZ) < ABF. A interação entre as RCs e as condições acídicas influenciou a alteração de RS (p = 0,025). Após armazenamento na SA, não houve diferença entre as RCs, enquanto após condições cariogênicas e erosivas a ABF mostrou a maior alteração de RS. Para FS (p < 0,001), XTF > (FBF = FZ) > (TBF = FZ) >ABF, sem diferença entre controle e condições cariogênicas e erosivas (p = 0,148). Dependendo da RC bulk-fill restauradora, condições cariogênicas e erosivas aumentam a RS, mas não alteram a RF desses materiais.

Palavras-chave: *resinas compostas - ácidos - cárie dentária - erosão dentária.*

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INTRODUCTION

Resin composites are widely used due to their direct filling capability, minimally invasive nature, esthetics, and clinical performance[1]. A significant concern when placing a resin restoration is reducing polymerization shrinkage stress. One method to do so is to layer the resin composite incrementally[2]. However, since its efficiency in mitigating deleterious effects at the adhesive interface has been questioned[3], the bulk-filling technique has become more widely used following the development of materials with less shrinkage, polymerization stress and cusp deflection[4]. Such benefits have been attributed mainly to the increased translucency and the modification of the resin matrix, or photo-initiator dynamics[5].

Although previous meta-analyses have reported that bulk-fill restorations present survival rates and clinical performance similar to those of conventional composites[6,7] in contact with erosive drinks, there is evidence showing that physical and mechanical properties of bulk-fill resin composites are more negatively influenced than a conventional counterpart[8]. On the other hand, when the acid is of cariogenic origin, bulk-fill and conventional resin composites do not seem differ in degradation[9,10].

One reason for the acid-dependent behavior of bulk-fill resin composites may be the lower pH of erosive acids in comparison to cariogenic acids. This explanation is supported by the fact that hydrolysis can speed up under more acidic conditions, as pH affects reaction rates through catalysis[11,12]. Low pH solutions can act on the polymeric matrix of composites through catalysis of ester groups from dimethacrylate monomers present in their compositions (Bis-GMA, Bis-EMA, UDMA and TEGDMA)[13]. The hydrolysis of these ester groups can form alcohol and carboxylic acid molecules that may accelerate degradation of the resin composites due to a lowering of the pH within the resin matrix[13]. In addition, low pH solutions may also cause erosion of inorganic fillers and thereby their debonding[13].

Despite these possibilities, to the best of the authors' knowledge, no previous study has compared the effect of erosive and cariogenic acids on the physical and mechanical properties of bulk-fill resin composites. Thus, the aim of this study was to compare the surface roughness and the flexural strength of various bulk-fill restorative composites under simulated carious and erosive conditions.

MATERIALS AND METHODS

Experimental design and sample size calculation

This study followed a 5x3 factorial design, using five resin composites (one conventional: Filtek Z350 XT and four bulk-fill: Filtek Bulk Fill, X-tra fil, Tetric N-Ceram Bulk Fill and Aura Bulk fill, as shown in Table 1), and three storage conditions (carious, erosive, control), comprising 15 groups.

Sample size was calculated (G*Power 3.1.9.4, Heinrich-Heine Düsseldorf University, Düsseldorf, Germany) based on preliminary data collected from three samples per group, from which an effect size of 0.27 was obtained. At $\alpha = 0.05$ and a power of 0.80, a total sample size of 215 resin composite bars would be needed. Based on the 15 groups of this study, 15 specimens per group were required.

The dependent variables were: 1) average surface roughness (Ra, in μm), measured at baseline and after storage in the allocated conditions; and 2) three-point flexural strength (in MPa). Scanning electron microscopy (SEM) images were obtained to illustrate the changes that occurred.

Specimen preparation

For each resin composite (Table 1), 45 bars were prepared in a PTFE split mold (length: 12 mm; height and width: 2 mm). The mold was filled with the resin composite and covered with a polyester strip. The resin composite was made flush with the mold by use of glass slide and a 500-g axial load applied for 60 s. Specimens were light-cured for the time recommended by each manufacturer at three different locations along the top bar length with a LED curing unit (Radii-cal, Victoria, Australia, light power density: 950 mW/cm^2). A LED curing unit was used based on a previous paper that showed that it improved the mechanical properties of Filtek Bulk Fill[14]. After removal of the polyester strip, the bar was retrieved from the mold and stored (24 h, 37 °C, 100% relative humidity).

Storage of the bars under carious and erosive conditions

The bars of each resin composite were randomly allocated into three groups (n = 15), to be stored under carious, erosive or control conditions, as follows:

- *Acidic condition simulating caries:* bars were stored in demineralizing solution (pH 4.3)[15]. The solution contained 2.0 mM calcium, 2.0

Table 1. Characterization of the resin composites tested

Resin composite	Composition and filler loading	Shade	Manufacturer/ Batch number
Filtek Z350 XT *(conventional resin composite)*	Bis-GMA, UDMA, TEGDMA, Bis-EMA, Polyethylene glycol dimethacrylate, silane treated ceramic, silane treated silica, and silane treated zirconia 78.5% (by weight) / 58.5% (by volume)	A2	3M ESPE, St. Paul, MN, USA Batch number: 424453
Filtek Bulk Fill *(bulk-fill resin composite)*	Bis-GMA, Bis-EMA, UDMA, TEGDMA, polyacrilic resin, silica, zirconia, and zirconia/silica agglomerates 76.5% (by weight) / 58.4% (by volume)	A2	3M ESPE, St. Paul, MN, USA Batch number: 685666
X-tra Fil *(bulk-fill resin composite)*	Bis-GMA, UDMA, TEGDMA, barium aluminum silicate vitreous particles 86.0% (by weight) / 70.1% (by volume)	U	Voco Cuxhaven, Germany Batch number: 1514217
Tetric N-Ceram Bulk Fill *(bulk-fill resin composite)*	Bis-GMA, UDMA, Bis-EMA, mixed oxides, barium glass, isofillers, and ytterbium trifluoride 77.0% (by weight) / 55.0% (by volume)	IVA	Ivoclar Vivadent Schaan, Liechtenstein Batch number: 03089/03
Aura Bulk Fill *(bulk-fill resin composite)*	Bis-GMA, UDMA, silica, barium glass. 65.0% (by weight) / 81.0% (by volume)	BKF	SDI, Bayswater, Victoria, Australia Batch number: 150931

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

mM phosphate, 0.03 ppm F, and 75 mM acetate buffer. Storage time was based on a report that resin-based materials soften after 14 days of 10 daily cariogenic challenges[16]. Since the decrease of saliva pH is below 5.5 for approximately 45 minutes, in order to simulate a total 14 days, the resin composite bars were stored individually in 1.0 mL of the demineralizing solution for 6,300 minutes (45 minutes for each one of the 10 cariogenic episodes over 14 days). During the storage time, the bars were kept in an oven at 37 °C. The demineralizing solution was renewed daily.

- *Acidic condition simulating erosion:* bars allocated to this storage condition were immersed in a 0.05 M citric acid solution (pH 2.3), commonly used in erosion models as a source of exogenous acid. To make the conditions of carious and erosive conditions equivalent, the bars in the erosion group were also exposed to 140 erosive episodes. Since salivary pH remains below the baseline value for 90 s after the intake of a citric-containing beverage[17], the bars were stored individually in 1.0 mL of the citric acid solution for 210 min (90 s for each one of the 10 erosive episodes over 14 days). Bars remained stored in an oven at 37 °C and erosive solution was renewed on a daily basis.
- *Control condition:* control group bars were stored in 1.0 mL of artificial saliva containing $MgCl_2$, NaCl, KCl, and $CaCl_2$ at 37 °C, for the same time used in the carious condition. As for the other two groups, the bars were stored in an oven at 37 °C and the immersion medium was renewed daily.

Measurement of baseline and final surface roughness

The top surface of each resin composite bar was evaluated using a surface roughness tester (Surftest SJ-210, Mitutoyo, Japan) at three different random locations, before and after storing the bars in their respective media. The cutoff value was set at 0.25 mm. Surface roughness was measured using the mean arithmetic deviation of the profile (Ra, in µm). The three values obtained at each time point were averaged and recorded as baseline and final Ra values, which were used to calculate the Ra change (ΔRa = final - baseline). Positive values indicate increase in surface roughness.

Flexural strength

After the storage period, each bar was positioned on a three-point bending apparatus with a span length of 20.0 mm. Each bar then underwent three-point bending test using a universal testing machine (Emic DL2000, São José dos Pinhais, Brazil) at a crosshead speed of 1 mm/min. The flexural strength was determined according to the following equation: $\sigma=3PL/[(2wb)]^2$, where "P" is the maximum load (in Newtons); "L" is the distance between the two supports (in mm); "w" is the bar width (mm) and "b" is the bar height (mm).

Scanning electron microscopy (SEM)

Bars from each group were sputter-coated and imaged at 1,000x- magnification using a scanning electron microscope (TM3030, Hitachi Ltd, Japan) to illustrate the surface micromorphology of each resin composite after being stored in carious or erosive conditions and artificial saliva.

Statistical analysis

Bulk-fill resin composites were compared for their baseline surface roughness using one-way analysis of variance (ANOVA) and Tukey's test. The interplay between the resin composites and storage conditions on the surface roughness change (ΔRa) and flexural strength was investigated using two-way ANOVA and Tukey's tests. IBM SPSS software (version 23, SPSS Inc., Chicago, IL, USA) was used for all statistical calculations. The significance level was set at 5%.

RESULTS

One-way ANOVA indicated that prior to storing the bars under different conditions, there were statistically significant differences among the resin composites ($p < 0.001$). Tetric N-Ceram and X-tra fil were significantly smoother than Filtek Bulk Fill and Aura Bulk Fill. Except for Aura Bulk Fill, which presented the highest surface roughness, none of the bulk-fill composites differed significantly from the conventional counterpart (Table 2).

Two-way ANOVA showed a significant interaction between the resin composites and storage conditions ($p = 0.025$) for surface roughness change (ΔRa). As found by Tukey's test, there was no statistically significant difference among resin composites when they were stored in artificial saliva (control). Under either carious or erosive conditions, the composite Aura Bulk Fill showed higher ΔRa than the other resin composites, which did not differ from each other (Table 2). Tukey's test also indicated that for the composites Filtek Bulk Fill, Tetric N-Ceram Bulk Fill and Filtek Z350 XT, the carious and erosive conditions did not pose increased roughness changes in relation to those caused by artificial saliva. For Aura Bulk Fill, however, both acidic conditions (carious and erosive) resulted in higher surface roughness change.

For the flexural strength data (Table 3), there was a statistically significant difference among resin composites ($p < 0.001$), with X-tra fil providing higher values than Filtek Bulk Fill, which presented higher flexural strength than Tetric N-Ceram Bulk Fill. The latter two bulk-fill resin composites did not differ from the conventional resin composite Filtek Z350 XT. Aura Bulk Fill had the lowest flexural strength values. The storage conditions did not significantly influence the flexural strength of the tested resin composites ($p = 0.148$).

The photomicrographs in Fig. 1 show a smooth surface and similar micromorphology for Filtek Z350 XT and Filtek Bulk Fill when comparing the bars stored in artificial saliva (1A and 1D), carious (1B and 1E) and erosive conditions (1C and 1F). X-tra fil (1G, 1H and 1I) presented detachment of some filler particles (line arrows), and bars stored under carious and erosive conditions (1H and 1I) had a rougher surface than the control group (artificial saliva). The Tetric N-Ceram Bulk Fill composite resin subjected to erosive condition (1L) presented filler particles exposed on the surface with irregularities (right-pointing double arrow). The Aura Bulk Fill composite resin displayed marked surface damage as a consequence of carious and erosive conditions (right-pointing thick arrow, 1N and 1O).

Table 2. Mean (SD) of baseline and increase in surface roughness (ΔRa = final – baseline) according to the resin composite and storage condition

Resin composite	Baseline Ra	ΔRa (roughness increase)		
		Artificial saliva	Cariogenic condition	Erosive condition
Filtek Z350 XT	0.129 AB (0.114)	0.008 Aa (0.013)	0.014 Aa (0.020)	0.013 Aa (0.009)
Filtek Bulk Fill	0.170 B (0.109)	0.012 Aa (0.008)	0.012 Aa (0.013)	0.017 Aa (0.010)
X-tra fil	0.088 A (0.098)	0.004 Aa (0.007)	0.016 Aa (0.020)	0.011 Aa (0.007)
Tetric N-Ceram Bulk Fill	0.083 A (0.068)	0.005 Aa (0.010)	0.008 Aa (0.006)	0.009 Aa (0.008)
Aura Bulk Fill	0.375 C (0.180)	0.007 Aa (0.011)	0.028 Bb (0.021)	0.032 Bb (0.022)
Grand mean	—	—	—	—

At the baseline, composite resins indicated by different uppercase letters differ from each other. For ΔRa, means followed by different uppercase letters indicate difference between resin composites (comparisons within each column), while lowercase letters indicate difference between storage conditions (comparisons within each row).

DISCUSSION

The main clinical benefits associated with bulk-fill resin composites are reduction in polymerization stress[18], shorter working time, and polymerization of increments up to 4-5-mm thick[19]. Notwithstanding these advantages over conventional resin composites, some bulk-fill composites degrade in the oral cavity as much as[9,10] or more[8] than conventional composites. This performance of bulk-fill composites seems to depend on the conditions to which they are exposed and/or the composition/brand of these restorative materials. Such speculations are supported by 1) studies that used acids associated with caries episodes, which did not affect physical and mechanical properties of some bulk-fill composites[9,10] and 2) a study that found that erosive beverages harmed the surface of bulk-fill composites[8]. However, to date, no research has been published on whether erosive and cariogenic acids, whose strengths are different, would affect bulk-fill resin composites differently. Thus, the current paper compares the effect of erosive and cariogenic acids on physical and mechanical properties of varying bulk-fill composites.

The findings of our study showed that both acidic solutions similarly increased the surface roughness of all composites, but the damage caused by cariogenic and erosive acids surpassed that caused by artificial saliva only for Aura Bulk Fill. This suggests that the behavior of bulk-fill composites may be more likely attributable to the differing chemistry of the monomeric resin formulations and filler characteristics (type, volume fraction, density and particle size and distribution)[20] as well as the filler-resin interface of the bulk-fill resin composite itself, than to the pH of the storage solution. Thus, even though the erosive solution had a substantially lower pH (2.3) than the cariogenic solution (4.3) and could potentially speed up hydrolysis and degradation of resin-based materials, such pH discrepancy did not cause detectable differences in the composite's surface roughness measured through the Ra parameter. However, as can be seem in Fig. 1 F, I, L and O, there was a trend toward the presence of increased irregularities, voids and cracks when the bulk-fill composites were under erosive conditions. Such qualitative evidence may be due not only to potentially accelerated hydrolysis by the low pH erosive solution, but also by erosion and debonding of inorganic fillers[13].

It is worth mentioning that among the resin composites tested in this study, Aura Bulk Fill had the lowest filler content by weight and thereby the highest resin matrix content. This may have accounted not

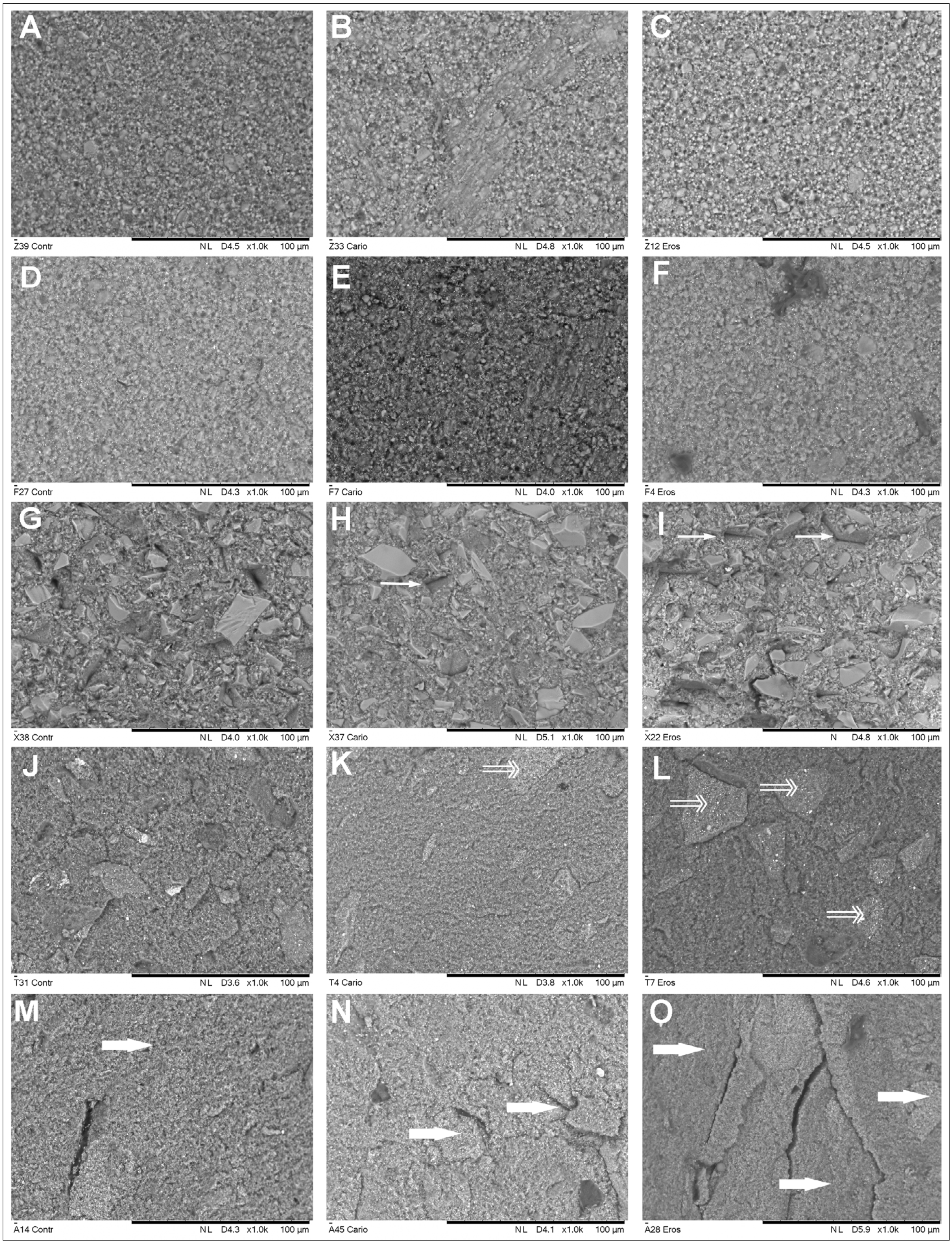

Fig. 1: Photomicrographs of restorative bulk-fill and conventional composites after storage in conditions of carious or erosive challenges or in artificial saliva.
A, B, and C) Filtek Z350 XT; D, E, and F) Filtek Bulk Fill; G, H, and I) X-tra fil; J, K, and L) Tetric N-Ceram Bulk Fill; M, N, and O) Aura Bulk Fill. The first column presents resin composites stored in artificial saliva (A, D, G, J, and M). The second column presents resin composites submitted to the in vitro caries model (B, E, H, K, and N). The third column presents resin composites submitted to the in vitro erosion model (C, F, I, L, and O). Note: The arrows indicate surface alterations: the line arrows (→) indicate a detachment of filler particles (H and I); the right-pointing double arrow (⇛) represents an exposure of filler particles (L); and the right-pointing thick arrow (⇨) indicates degradation associated with damage and irregular surface (N and O).

only for Aura Bulk Fill's higher susceptibility to the acidic solutions but also for its higher baseline surface roughness and lower flexural strength in comparison to the other materials tested. Indeed, when weight percentage of fillers decrease, the sorption increases[21,22], causing swelling and thereby softening and plasticization of composites[23] as well as compromising their mechanical properties[20]. This statement is supported by previous observations that low inorganic filler contents (<75 wt%) have been associated with low flexural strength[24] and Aura Bulk Fill was the only composite having low filler loading (65.0%) by weight. This is probably a result of the inclusion of pre-polymerized fillers[25].

In contrast to Aura Bulk Fill, the other resin composites (Filtek Z350 XT, Filtek Bulk Fill, X-tra fil and Tetric N-Ceram Bulk Fill) did not differ from each other regarding surface roughness changes, and were not affected by the storage conditions. This finding corroborates a previous paper that found that under biofilm accumulation and cariogenic challenges, a resin composite did not degrade faster[15], but disagrees with another study in which the lowest pH erosive solution caused the highest increase in surface roughness[26]. This may be ascribed to the fact that our solution had 10% of citric acid (0.05 M), whereas the other study used plain passion fruit juice containing 55% citric acid, in addition to other acids.

It is worth noting that although there is no clear information on the surface roughness threshold and the ideal morphological features of a resin composite to reduce biofilm accumulation, surface staining and wear, and to increase gloss retention, the smoother the surface, the better. In this regard, although Aura Bulk Fill became only 7% and 8% rougher after immersion in the carious and erosive acids, respectively, the roughness attained was the highest among the materials tested. Considering that the restoration surface should have a maximum roughness 0.50 µm if it is not to be detected by the patient[27], Aura Bulk Fill may not seem a good choice of restorative material as, on average, such threshold is exceeded when this composite is exposed to cariogenic and erosive acids. On the other hand, despite showing the lowest flexural strength values among the investigated materials, they are above the minimum 80 MPa stated in ISO standard 4049/2019[28].

The present findings on flexural strength agree with published data showing that the effects of lactic acid, a caries-associated acid[29] and citric acid solutions[30] did not differ from that caused by distilled or deionized water or artificial saliva[31]. Flexural strength was, however, composite-dependent, with the highest value measured for X-tra Fil, which contained the highest percentage of filler by weight. X-tra Fil, Filtek Bulk Fill and Tetric N-Ceram Bulk Fill presented flexural strength equivalent to the conventional composite.

Whether the current results hold up in clinical practice remains to be elucidated. It may be assumed that in the clinical scenario, while polishing procedures may reduce the resin matrix exposed, they may also cause filler particle dislodgement[32] and may aggravate sorption, swelling, softening and plasticization and compromise the mechanical properties of composites. On the other hand, such effects may be counteracted by saliva, which plays important roles in forming acquired pellicle, buffering, clearing, and diluting acids[33], which may reduce their effects on resin composites in the oral cavity.

Depending on the restorative bulk-fill composite, carious and erosive conditions roughen the surface, and therefore, not all materials would remain undetectable by patients. However, acidic conditions resembling caries and erosion do not affect the flexural strength of restorative bulk-fill composites, which still meet the minimal value recommended for load-bearing areas.

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DECLARATION OF CONFLICTING INTERESTS

The authors declare no potential conflicts of interest with regarding the research, authorship, and/or publication of this article.

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