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Effect of gastric acids on the mechanical properties of conventional and CAD/CAM resin composites - An in-vitro study

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ABSTRACT

Objectives: Dental erosion in patients with gastroesophageal reflux disease (GERD) is a current and frequent condition that may compromise the mechanical properties and clinical durability of resin-based composites (RBCs). This study assessed the mechanical properties of conventional and computer-aided design/computer-aided manufacturing (CAD/CAM) RBCs subsequent to simulated gastric acid aging.
Materials and method: Three conventional and three CAD/CAM composites were assessed. They were divided into an experimental group (exposed to simulated gastric acid aging) and a control group (no aging). Both groups were analyzed for Vickers microhardness (VHN), wear and flexural strength over a period of six months. The failure rate probability for each RBC was calculated through the Weibull cumulative distribution function (*m*). Statistical analysis was conducted using repeated measures ANOVA, 3-way ANOVA, a non-parametric Kruskal-Wallis and U Mann-Whitney tests ($\alpha = 0.05$).
Results: The mechanical properties of all the RBCs dropped significantly after aging ($p < 0.05$). Lower VHN and flexural strength values, along with greater wear values were evident in the experimental groups, though the effects of the treatment varied between RBCs. The Weibull *m* of all the RBCs decreased over time.
Conclusion: Conventional RBCs might show greater reduction in mechanical properties compared to CAD/CAM RBCs when exposed to gastric acid attack. Thus, CAD/CAM composites may represent a suitable choice for the treatment of patients presenting erosive issues.

1. Introduction

The etiology, diagnosis, and treatment of dental erosion have become a subject of great interest in general dentistry (Young and Tenuta, 2009; Shellis et al., 2011; Cruz et al., 2019; Sulaiman et al., 2015; Alencar et al., 2019). The European Federation of Conservative Dentistry (EFCD) defined erosion as "a chemical-mechanical process resulting in a cumulative loss of hard dental tissues caused by any cause not related to bacteria activity" (Carvalho et al., 2016). For instance, one of the main etiological factors involved in dental erosion is related to exposure of the teeth to gastric acid (intrinsic erosion). Such a scenario is principally evident in patients with gastroesophageal reflux disease (GERD) and eating disorders (Hermont et al., 2014; Loke et al., 2016; Ranjitkar et al., 2012; Orr, 2003).

Despite the heterogeneous designs employed by epidemiological studies on dental erosion, it was agreed that dental wear is increasing, in particular in young people due to the cumulative and irreversible effects of dental erosion (Vailati et al., 2013; Salas et al., 2015; Schlueter and Luka, 2018; Pini et al., 2018). In general, erosive/wear lesions are characterized by loss of the natural surface morphology and contour of the tooth. Consequently, such lesions have ceased to be categorized only as smooth concave surfaces, typically wider than deeper (Carvalho et al.,

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2016), and they are now considered as a pathological condition whose uncontrolled evolution can lead to severe tooth wear requiring full restorative management (Pini et al., 2018; Mehta et al., 2021).

The constant improvement in adhesive restorative materials (Ferracane, 2011; Alzraikat et al., 2018; Pires et al., 2024) has made it easier to treat such lesions in a less invasively manner via using resin-based materials that can reinstate the lost function and esthetics of teeth (Pini et al., 2018; Tsujimoto et al., 2018; Örtengren et al., 2001; Rahim et al., 2012). Indeed, there are specific clinical techniques based on conservative approaches that allow the restoration of the remaining dental tissues (Vailati et al., 2013) using resin composites with similar resistance to wear to that of natural teeth (Osiewicz et al., 2019; D Arcangelo et al., 2014).

The oral cavity is a challenging environment in which resin-based composites (RBCs) are typically exposed to saliva, as well as to a wide variety of enzymes (e.g. esterase) and several extrinsic chemicals, such as acids, alkalis, salts, alcohols, and free radicals; exposure time and the type of materials employed are decisive factors for the longevity of such dental restorations (Ferracane, 2006; Alshali et al., 2015; Krüger et al., 2018; Randolph et al., 2016; Ferracane et al., 1998; Wendler et al., 2021). RBCs are methacrylate-based materials characterized by a certain water absorption that is determined by the chemistry and structure of the polymer network, the size and distribution of the filler and the properties of the matrix/filler bond (Rahim et al., 2012; Ferracane, 2006; Curtis et al., 2008). RBCs exposed for a long time to a humid medium, may swell and plasticize, causing a loss of mechanical properties (Ferracane, 2006; Sideridou et al., 2003; Calais and Söderholm, 1988), with consequent reduction of the longevity of restorations (Örtengren et al., 2001; Alshali et al., 2015; Krüger et al., 2018; Moreira da Silva et al., 2011).

Gastric juice is an intrinsic solution mainly constituted of hydrochloric acid (HCl) with a strong pH of 0.9–1.5 (Young and Tenuta, 2009); it can be harmful both to dental structures and to restorative materials. Gastric acid is commonly found in the oral cavity of those people affected by GERD and/or eating disorders, such as anorexia and bulimia nervosa. Several studies have assessed the erosion resistance of conventional (Alencar et al., 2019; Cabral et al., 2015; Albuquerque et al., 2018; Briso et al., 2011; Cilli et al., 2012) and/or computer-aided design/computer-aided manufacturing (CAD/CAM) RBCs (Cruz et al., 2019; Backer et al., 2017; Egilmez et al., 2018; Cengiz et al., 2014), but no standardized protocol (exposure time and/or HCl concentration) was employed to simulate intrinsic erosion situations. However, the catalyzing effect of HCl in accelerating the hydrolytic degradation of the resin composites is well known (Moreira da Silva et al., 2011; Göpferich, 1996). Most of the available information in literature focus on the erosive effects of HCl on the surface of the materials' substrate (Cruz et al., 2019; Alencar et al., 2019; Cabral et al., 2015; Albuquerque et al., 2018; Briso et al., 2011; Cilli et al., 2012; Backer et al., 2017; Egilmez et al., 2018; Cengiz et al., 2014; Borges et al., 2019), but little attention has been paid (Egilmez et al., 2018) on the consequences that gastric acids can have on the microstructure (e.g. flexural strength) and on the clinical performance of the RBCs.

The resistance of the such materials does not only depend on their composition, but also on the distribution of the internal defects within the bulk materials (internal porosity and/or surface defects) (Quinn and Quinn, 2009). Mechanical tests such as hardness, flexural strength, and wear resistance, statistically complemented by a Weibull analysis result very useful for the calculation of the clinical reliability of resin-based restorative materials (Quinn and Quinn, 2009; Kumar, 2012; McCabe and Carrick, 1986).

Therefore, as dental erosion is a current and frequent condition that may compromise the mechanical properties and clinical lifespan of RBCs, this study aimed at assessing the mechanical properties of conventional and CAD/CAM RBCs exposed to simulated erosion induced by gastric acid for six months. Such an aim was accomplished by measuring the microhardness, flexural strength of the tested materials, as well as assessing the surface wear after simulated chewing performed in presence of HCl (60,000 cycles). Moreover, the reliability of each RBC in the experimental groups and in the control group over time, was also estimated using the Weibull cumulative distribution function.

2. Materials and methods

This experimental study employed resin-based composites (RBCs), three were conventional composites — FILTEK Supreme XTE (FS), BRILLIANT EverGlow (BE) and GrandioSo (GS) — and three were CAD/CAM — Lava Ultimate (LU), BRILLIANT Crios (BC) and Grandio Blocs (GB). Table 1 describes the characteristics of the RBCs employed in this study.

2.1. Specimen preparation

The specimens for the Vickers microhardness and wear/profilometry tests were obtained by applying a conventional composite resin into

Table 1
Materials characteristics used and their composition.

Resin composites	Code	Composition	Manufacturer
Conventional resin composites			
Filtek Supreme XTE (Nanofilled)	FS	Bis-GMA, Bis-EMA, TEGDMA, UDMA. Non-agglomerated or non-aggregated 20 nm silica, non-agglomerated or non-aggregated 4–11 nm zirconia filler, aggregated zirconia/silica cluster filler 0.6–10 μm. *78.5 wt% 63.3 vol% inorganic fillers.*	3 M ESPE™ (St Paul, Minnesota, USA) N895712
Brilliant EverGlow (Nanohybrid)	BE	Bis-GMA, Bis-EMA, TEGDMA. Barium glass powder, milled to below 1 μm. Pyrogenic silica, SiO_2 nanoparticles non-aggregated and ZnO nanoparticles (0.02–1.5 μm). No clustered nanoparticles *74 wt% - 56 vol% inorganic fillers.*	Coltene/Whaledent AG (Altstätten, Switzerland) I45464
GrandioSo (Nano-hybrid)	GS	Bis-GMA, Bis-EMA, TEGDMA. Glass ceramic filler (0.5–3 μm), SiO_2 nanoparticles (20–40 nm). *89 wt% -73 vol% inorganic fillers.*	Voco GmbH (Cuxhaven, Germany) 1821537
CAD/CAM resin composites			
Lava Ultimate (Nano-ceramic)	LU	Bis-GMA, Bis-EMA, TEGDMA, UDMA. SiO_2: 20 nm; ZnO_2: 4–11 nm; silica-zirconia nanoclusters: 0.6–10 μm. *80 wt% - 65 vol% inorganic fillers.*	3 M ESPE™ (St Paul, Minnesota, USA) N389824
Brilliant Crios (Reinforced composite)	BC	Cross-linked methacrylates. Bis-GMA, Bis-EMA, TEGDMA. Barium glass: milled <1 μm, SiO_2 nanoparticles: < 20 nm. *70.7 wt% - 51.5 vol% inorganic fillers.*	Coltene/Whaledent AG (Altstätten, Switzerland) I40697
Grandio Blocs (Nano-ceramic)	GB	UDMA + DMA Glass ceramic particles: 0.5–3 μm, SiO2 nanoparticles: 0–40 nm. 86 wt% - 71 vol% inorganic fillers.	Voco GmbH (Cuxhaven, Germany) 1809299

BisGMA: Bisphenol A diglycidylether dimethacrylate; UDMA: Urethane dimethacrylate; BisEMA: Ethoxylated bisphenol A dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate. Most of the data were collected from the manufacturer's technical or information sheets.

stainless-steel molds (Smile Line USA Inc., Colorado, USA) and applying pressure with a glass for 180 s (ISO 4049:2009) (International Organization for Standardization, 2009). The size of the specimens was 10 mm × 7 mm × 1.5 mm (±0.05 mm). For the flexural strength test, a custom mold was used to prepare the specimens with dimensions of 12 mm × 2 mm × 2 mm (±0.01 mm) (Yap and Teoh, 2003; Yap et al., 2018; Beun et al., 2007). The specimens were light-cured for 40 s on each side (>1000 mW/cm^2) using a light-curing system (Valo, Ultradent Products Inc., South Jordan, UT, USA) and then placed in a polymerization chamber (Visio Beta Vario, 3 M/ESPE, Seefeld, Germany) for 7 min. The CAD/CAM composite resin block samples were obtained by cutting the blocks using a automatized cutting device (Minitom, Struers, Rodovre, Denmark) equipped with a diamond disc at a speed of 400 rpm under continuous irrigation. All the specimens were wet-polished using P500, P1200, P2400 and P4000 silicon carbide abrasive discs (LaboPol-1, Struers, Willich, Germany). The thickness of each specimen was measured using a digital caliper (Coolant-proof IP65 Micrometer, Mitutoyo Corporation, Kanagawa, Japan) and then examined radiographically (RXDC Extend, MyRay, Bicocca, Italy) to make sure of the absence of any internal defects created during the preparation of the specimens. Finally, they were cleaned in an ultrasound bath for 10 min and stored for 24 h in distilled water at 37 °C before obtaining the baseline measurement.

2.2. Study design

A total of 222 specimens for each tested RBCs were fabricated. A specific number of specimens (n = 60) were used for the microhardness test, while 150 specimens were employed for the flexural strength test and 12 specimens for the wear/profilometry test. For each test, random sampling was used to assign the specimens to the control group or to the experimental group (gastric acid) (www.random.org). The mechanical properties were assessed at 24 h (T1), 3 months (T2) and 6 months (T3). The experimental design of the study is described in Fig. 1.

2.3. Simulated erosion protocol

An acidic solution was prepared by mixing 0.2% (w/v) sodium chloride with 0.7% (v/v) hydrochloric acid, with a pH of 1.5 ± 0.2 (Backer et al., 2017; Kulkarni et al., 2018) to simulate the gastric acid juice. The erosion protocol for this study was used in accordance with the parameters established by Shellis et al. (2011). The specimens in the experimental group were exposed to the simulated gastric acid for a total of 4 min per day, performed into 2 cycles of 2 min daily at 37 °C and 70 rpm for a period up to 6 months. Distilled water was used for the specimens in the control group. The aging media was refreshed daily (24h). Before the microhardness, flexural strength, and wear tests, the specimens in the control group were immersed in distilled water for 180 days. Between erosion cycles and at the end, the specimens were rinsed for 2 min with distilled water and stored in distilled water until the next day (37 °C and 70 rpm). Both fluids were replaced daily (37 °C and 70 rpm) during the 6-month study. After completing the erosion protocol, the specimens were submitted to the different tests. The wear test was carried out with the specimens immersed in distilled water.

2.4. Microhardness evaluation

The microhardness (VHN) assessment was performed by using a Vickers diamond indenter in the HMV-2 Micro Hardness Tester (Shimadzu corp, Kyoto, Japan). A load of 980.7 mN was applied for 15s as specified by the ISO 6507–1:2018 standard for Vickers microhardness testing (International Organization for Standardization, 2018).

The following equation was used:

$$\mathrm{VHN} = 1854\frac{P}{d^2}$$

The mean value for each specimen was the average of five measurements performed at a distance of at least 1 mm.

2.5. Flexural strength evaluation

Flexural strength was measured in an universal testing machine (Model 4502, Instron Corp., Massachusetts, USA) using a three-point bending test device. A 5 kN load cell with a crosshead speed of 0.75 ± 0.25 mm/min was used, in accordance with ISO 4049/2009 (International Organization for Standardization, 2009). The span distance was set at 10 mm for mini flexural strength testing (Yap and Teoh, 2003; Yap et al., 2018; Wang et al., 2005). The T1 specimens were previously stored in water at 37 °C for 24 h.

The uniaxial flexural strength was calculated from the maximum load recorded as follows:

$$\sigma = \frac{\mathbf{3Pl}}{\mathbf{2bh^2}}.$$

Where P is the maximum load (in Newtons) applied to the sample, I is the span (10 mm) and b is the width and h the height of the sample in millimeters.

2.6. Wear evaluation

The test was performed in a dual-axis chewing simulator (CS-4.2, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany). Each specimen was submitted to an effective load of 49 N. To simulate chewing, two types of movements were adopted: a 3 mm vertical movement and a 0.7 mm horizontal movement. The lowering and sliding speed of the antagonist axes was set at a frequency of 1.6 Hz. A total number of 60,000 cycles was performed to simulate approximately 6 months of clinical service (Lazaridou et al., 2015). The conical stylus antagonists (Ø = 2.36 mm, h = 0.6 mm) were made of ceramic reinforced with leucite (IPS Empress, Ivoclar Vivadent, Schaan, Liechtenstein) and were glazed twice at 870 °C (Heintze et al., 2005). Wear was defined as the greatest vertical loss of material observed in the contact area. The wear region was measured using a contact profilometer (Dektak 150 Veeco, CA, USA). The tip path was placed perpendicular to the track path in an

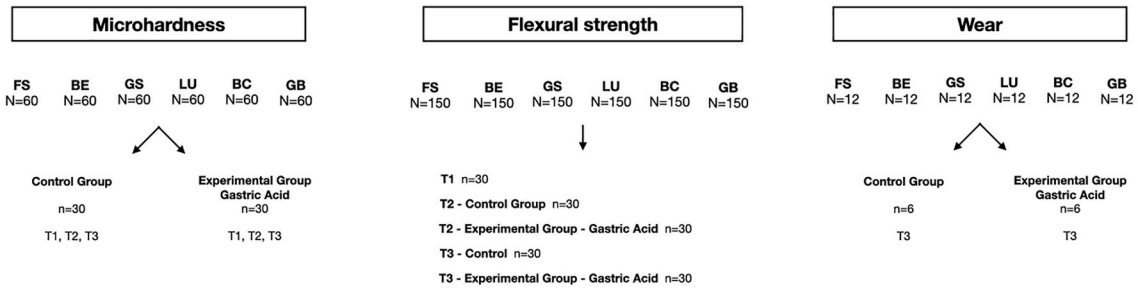

Fig. 1. Distribution of resin composites within each mechanical property under the different study conditions. T1 (24 h h), T2 (3 months) and T3 (6 months).

area of 4.5 mm × 4.5 mm for each specimen. A 10 μm-diameter tip was used with a resolution of 0.20 μm/sample and a speed of 0.06 mm/s. The central (deep) area of the footprint was scanned three times, following the direction of wear. Data analysis software (MountainsMap Premium 8.0.9139-USA) was used to plot the wear profile from the exported coordinates of each scan, and thus measure the maximum vertical loss located in the deepest area of the center of each specimen. During the wear test, all specimens were exposed to simulated gastric acid (experimental group) or distilled water (control group).

2.7. Statistical analysis

Statistical analysis software (G*Power, Düsseldorf University) was used to calculate the sample size for microhardness. Backer et al. (2017) was taken as the reference for estimating the standard deviation, the statistical power was set at 90% and the confidence interval at 95%. The sample size recommended in the literature was 30 samples for flexural strength (Quinn and Quinn, 2009) and 6 for wear (Heintze, 2006).

A further statistical analysis software package (SPSS Inc. Version 20, SPSS Inc., Illinois, USA) was employed. The Kolmogorov-Smirnov test was used to check for normal distributions for the VHN and flexural strength tests, but not for the wear test. Parametric tests were employed for the former two and non-parametric tests for the wear test. A repeated measures ANOVA model with material and treatment as the "within-subject" factors were estimated for VHN. Because flexural strength is a destructive test and therefore the samples assessed were different each time, a multifactor (3-way) ANOVA model was estimated. When the ANOVA tests detected significant differences, the Bonferroni test was performed to check for type 1 statistical error. Non-parametric Kruskal-Wallis and Mann-Whitney U tests for independent samples were used to assess the effect of the treatment on wear (vertical loss). Throughout the study, the confidence level was set at 95% and a significance level set at 5% ($\alpha = 0.05$).

The average values were considered according to the material and treatment at 6 months (T3) to evaluate the correlation between microhardness and wear using the Pearson's correlation method.

Table 2
Changes in microhardness and flexural strength over the experimental period by material and treatment: F-test of the repeated measures ANOVA and the 3-way ANOVA model respectively.

	Microhardness		Flexural strength	
	F	p-valor	F	p-valor
Time	1431.42	**<0.001**[c]	335.29	**<0.001**[c]
Material	24,883.41	**<0.001**[c]	2407.35	**<0.001**[c]
Treatment	443.84	**<0.001**[c]	104.97	**<0.001**[c]
Material[a]Treatment	4.41	**0.001**[b]	2.28	**0.044**[a]
Material[a]Time	34.31	**<0.001**[c]	4.09	**<0.001**[c]
Treatment[a]Time	123.66	**<0.001**[c]	28.41	**<0.001**[c]
Material[a]Treatment[a]Time	5.13	**<0.001**[c]	1.03	0.420

[a] p<0.05.
[b] p<0.01.
[c] p<0.001.

In the flexural strength tests, reliability was assessed by a 2-parameter Weibull cumulative distribution function, using the following equation:

$$\boldsymbol{P}(\sigma) = 1 - exp\left[- \left(\frac{\sigma}{\sigma 0} \right)^{m} \right]$$

where P(σ) is the cumulative probability of failure, σ is the flexural strength, σ0 is the characteristic force and m is the Weibull modulus. By plotting ln (1/[1-P]) versus lnσc, a straight line results, with the upward gradient m, whereas the intersection with the x-axes gives the logarithm of the characteristic strength. (Bütikofer et al., 2015; Roos et al., 2016).

3. Results

3.1. Microhardness evaluation

The repeated measures ANOVA detected that the triple interaction (material/treatment/time) was significant (F = 5.13, p = <0.01). This means that the loss of hardness over time depended on the treatment applied and that the effect of the treatment varied between materials (Table 2).

The mean VHN values and standard deviations (SDs) under the different study conditions are depicted in Table 3. The interaction between the variables was significant. The RBC with the highest VHN values was GB ($p < 0.001$), followed by GS, LU, FS, BC and BE. This order remained the same throughout the study. The reduction in VHN was progressive over time and greater in the experimental groups. At T2, the only specimens to show no significant reduction compared to the initial VHN values were those in the GB control group ($p = 1.00$). At T3, all the RBCs showed considerable reductions in comparison, not only with their initial (T1) values, but also with their T2 measurements. With regard to the treatment parameter, significant differences between the control and experimental groups were observed in the mean VHN values at T2 and T3.

In Fig. 2 are depicted the average microhardness relative values of all RBCs in order to visualize the percentage reduction of VHN in the control and gastric acid groups over time. While, in Fig. 3 it is possible to see the Pearson's correlation between microhardness and wear.

3.2. Flexural strength evaluation

The 3-way ANOVA statistical model for flexural strength values detected that the triple interaction between material/treatment/time was not significant (F = 1.03; p = 0.420), but the drop in flexural strength over time was significant ($p < 0.001$). While this reduction depended on the material ($p < 0.001$) and treatment ($p = 0.001$), the differences between materials were similar in both the control and the experimental group ($p = 0.420$) (Table 2).

The mean flexural strengths (±SD) of all the resin composites under the different studied conditions are shown in Table 4. The GB CAD/CAM composite showed greater flexural strength than the other RBCs at all the evaluation times, in both the control and the experimental group. The BE conventional composite exhibited the lowest flexural strength at

Table 3
Microhardness in Vickers Numbers (VHN): Mean (SD) for tested resins by length of exposure to the different media.

	Control Group			Experimental Group Gastric Acid		
	T1	T2	T3	T1	T2	T3
FS	76.48 (2.08) [aD]	70.90 (2.90) [bD]	67.44 (2.94) [cD]	75.47 (2.62) [aD]	68.17 (2.61) [cD]	59.95 (3.31) [dD]
LU	98.93 (2.01) [aC]	95.76 (2.15) [bC]	90.88 (2.33) [dC]	98.72 (1.96) [aC]	92.5 (2.33) [eC]	86.75 (2.95) [eC]
BE	55.50 (3.25) [aF]	50.29 (2.12) [bF]	47.22 (2.18) [cF]	56.07 (3.62) [aF]	46.08 (2.65) [cF]	38.02 (3.06) [dE]
BC	69.68 (3.04) [aE]	67.31 (2.71) [bE]	65.55 (3.22) [cE]	69.01 (3.05) [aE]	64.68 (3.39) [cE]	60.88 (1.85) [dD]
GS	107.96 (1.92) [aB]	103.37 (2.06) [bB]	101.10 (1.37) [cB]	107.70 (2.09) [aB]	98.88 (3.43) [cB]	95.91 (3.16) [dB]
GB	128.99 (2.28) [aA]	128.66 (2.11) [aA]	126.25 (1.94) [bA]	128.85 (2.54) [aA]	125.22 (1.51) [bA]	122.69 (2.80) [cA]

Different capital letters in columns and lowercase letters in rows indicate significant differences ($p < 0.05$).

Fig. 2. Average microhardness relative values of all RBCs to visualize the percentage reduction of VHN in the control and gastric acid groups over time.

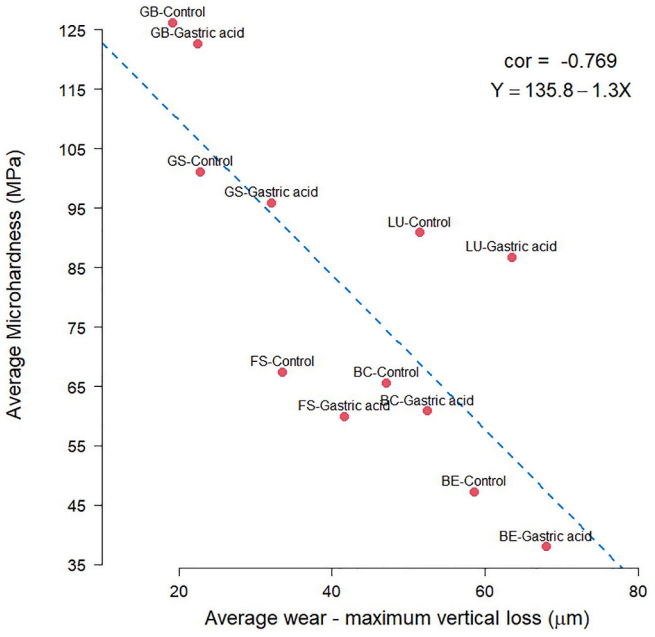

Fig. 3. Pearson's correlation between microhardness and wear.

all the evaluation times, in both the control and the experimental groups. Over time, all the RBCs presented significant differences compared to the initial (T1) values, but there was no significant difference between the values at 3 months (T2) and 6 months (T3) in the experimental groups were GB ($p = 0.506$) and BC ($p = 0.207$). The "r" Average flexural strength relative values of all RBCs are presented in Fig. 4 in order to highlight the percentage reduction of flexural strength in the control and gastric acid groups over time.

3.3. Weibull distribution

The maps of the cumulative probability of failure curves, which follow a Weibull distribution for each RBC are reported in Figs. 5–7. The probability of failure of all the RBCs increased over time and was accentuated in the experimental group. The highest Weibull modulus values (m) were found in the GB control group at T1 ($m = 27.11$) and in the BC control group at T2 ($m = 23.63$), and the lowest in the BE at T1 ($m = 11.70$) and BE at experimental group T2 ($m = 7,67$). At the 6-month evaluation (T3), the highest Weibull modulus values (m) were found in the CAD/CAM resin composites, in the GB control group ($m = 20.19$) and the GB experimental group ($m = 18.29$). The lowest Weibull modulus values (m) were observed in the BE conventional composite resin, in both the control group ($m = 7.21$) and the experimental group ($m = 4.78$).

3.4. Wear evaluation

The mean maximum vertical losses (SDs) of all the RBCs under the different study conditions are depicted in Table 5 and Fig. 8. The greatest vertical loss (μm) was recorded in BE (58.53 μm control and 67.95 μm experimental) and the lowest in GB (19.13 μm control and 22.49 μm experimental). Of the CAD/CAM resin composites, BC ($p = 0.191$) and GB ($p = 0.051$) showed no significant differences between the control and experimental groups. In the control group, no differences were found between LU and BC ($p = 0.327$); in the experimental group, no differences were observed between LU and BE ($p = 0.448$) after six months.

Table 4
Mean (SD) values for flexural strength (MPa) by length of exposure to the different treatments.

	Baseline	Control Group		Experimental Group Gastric Acid	
	T1	T2	T3	T2	T3
FS	109.09 (8.27) ^{aD}	99.53 (7.64) ^{bD}	93.73 (13.57) ^{bcD}	92.14 (10.15) ^{cD}	77.81 (15.25) ^{dD}
LU	148.97 (10.84) ^{aC}	139.58 (10.55) ^{bC}	137.06 (11.65) ^{bcC}	133.18 (10.64) ^{cC}	125.27 (13.23) ^{dC}
BE	96.25 (10.01) ^{aE}	83.78 (10.81) ^{bE}	77.25 (12.92) ^{bcE}	71.30 (11.07) ^{cE}	59.70 (15.47) ^{dE}
BC	166.31 (7.50) ^{aB}	158.93 (11.40) ^{bB}	153.60 (7.77) ^{bcB}	150.76 (9.08) ^{cB}	145.80 (11.58) ^{cB}
GS	105.17 (10.58) ^{aD}	97.59 (10.07) ^{bD}	88.94 (9.54) ^{cdD}	87.38 (9.68) ^{dD}	77.18 (11.06) ^{eD}
GB	182.41 (8.09) ^{aA}	175.93 (9.88) ^{abA}	171.55 (10.13) ^{bcA}	171.08 (9.16) ^{bcA}	167.33 (11.02) ^{cA}

Different capital letters in columns and lowercase letters in rows indicate significant differences ($p < 0.05$).

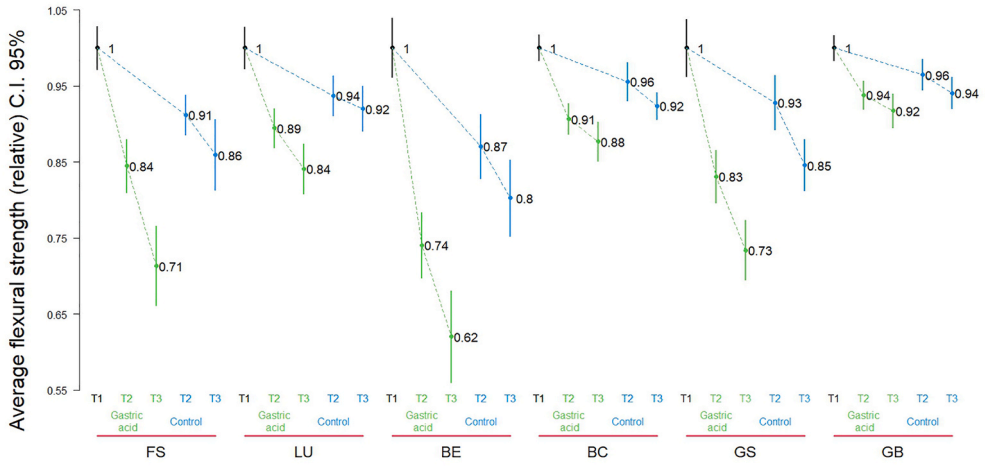

Fig. 4. The "r" Average flexural strength relative values of all RBCs to visualize the percentage reduction of flexural strength in the control and gastric acid groups over time.

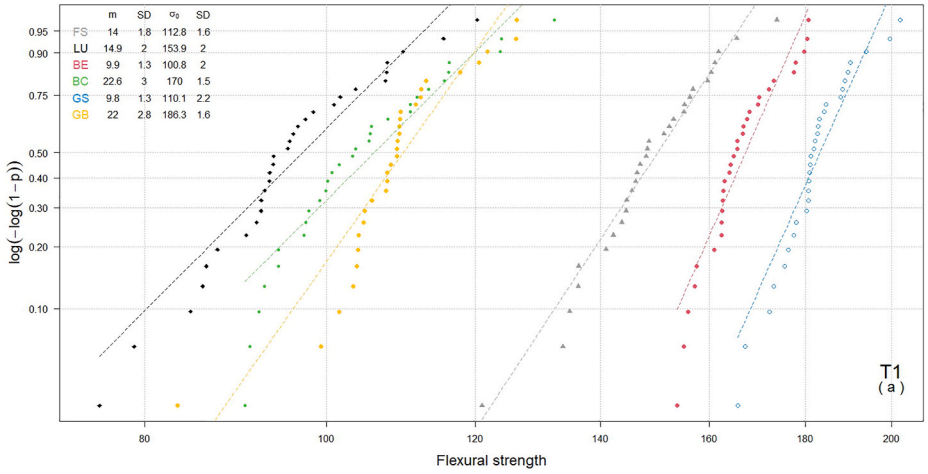

Fig. 5. Weibull plots of composites by period of exposure T1 to the different media. Two-parameter Weibull cumulative distribution function: characteristic strength σ 0 and Weibull modulus m.

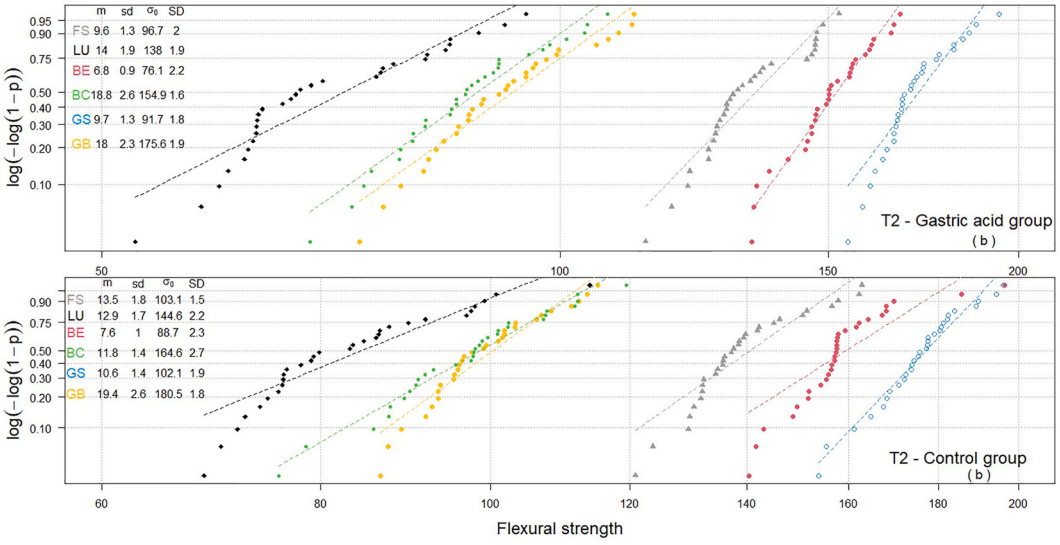

Fig. 6. Weibull plots of composites by period of exposure T2 to the different media (control and gastric acid group). Two-parameter Weibull cumulative distribution function: characteristic strength σ 0 and Weibull modulus m.

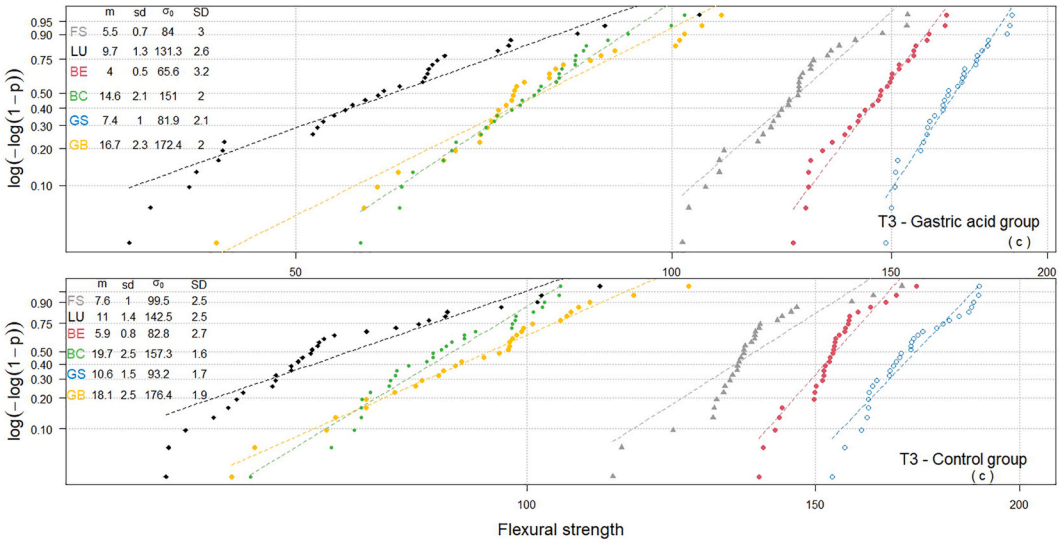

Fig. 7. Weibull plots of composites by period of exposure T3 to the different media (control and gastric acid group). Two-parameter Weibull cumulative distribution function: characteristic strength σ 0 and Weibull modulus m.

Table 5
Mean (SD) values for maximum vertical loss (μm) by treatment.

	Control Group	Experimental Group Gastric Acid
FS	33.48 (4.09) ^{bC}	41.57 (7.74) ^{aC}
LU	51.50 (6.85) ^{bB}	63.46 (5.90) ^{aA}
BE	58.53 (9.41) ^{bA}	67.96 (14.33) ^{aA}
BC	47.10 (11.22) ^{aB}	52.50 (13.01) ^{aB}
GS	22.78 (5.60) ^{bD}	32.07 (7.10) ^{aD}
GB	19.13 (5.74) ^{aE}	22.49 (4.04) ^{aE}

Different capital letters in columns and lowercase letters in rows indicate significant differences ($p < 0.05$).

4. Discussion

The first result to highlight in this study is that the RBCs exposed to simulated gastric acid attack presented greater reduction of mechanical properties compared to the control group. Rahim et al. (2012) revealed that acid solutions act as strong plasticizers that can accelerate the diffusion coefficient, water absorption and organic matrix solubility, particularly in methacrylate-based polymers. This is because protons (H^+) liberated by acids can hydrolyze the ester groups in di-methacrylate resin monomers (Chadwick et al., 1990; Nishiyama et al., 2004), forming alcohol and carboxylic acid. This reaction accelerates breakdown of monomers and promotes their elution (Moreira da Silva et al., 2011; Göpferich, 1996), so affecting the mechanical performance of the resin composites (Cruz et al., 2019; Ferracane, 2006; Badra et al., 2005). Water acts as a plasticizer within the matrix, where a process of oxidation degrades the polymer chains and break them into smaller molecules. This makes the matrix weaker and more plastic (Ferracane, 2006; Alshali et al., 2015) so resulting in a reduction in VHN (Ionescu et al., 2022).

The higher the solvent diffusion rate within the matrix, the faster the degradation of the material. As previously stated, solutions with acidic pH may accelerate the diffusion rate (Rahim et al., 2012; Moreira da Silva et al., 2011); this was observed in the present study, where most of the RBCs showed reductions in VHN. Degradation of the tested composites occurred more rapidly in the group exposed to gastric acid, and such resuls were observed after three months (T2) with no significant difference when compared to those of the control group after six months (T3). This latter results in terms of reductions in VHN (Alencar et al., 2019; Albuquerque et al., 2018; Briso et al., 2011) and changes in surface roughness (Alencar et al., 2019; Albuquerque et al., 2018; Briso et al., 2011; Egilmez et al., 2018) are in accordance with previous studies where resin composites with similar compositions to those of FS and LU were used.

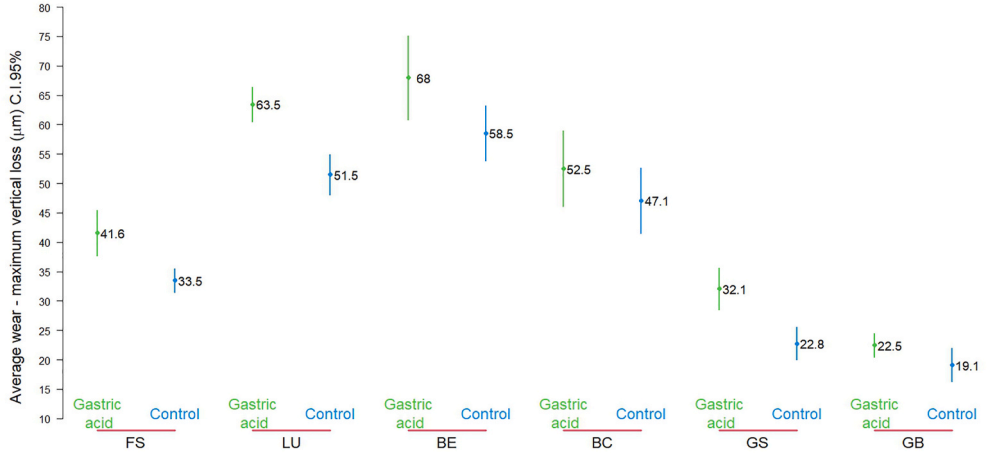

Fig. 8. Vertical loss (μm) for each composite after wear test at 60,000 cycles in the chewing simulator.

In a real clinical scenario, the synergy of mechanical forces and chemical changes can contribute to the wear of teeth and restorative materials. Indeed, the experimental group of RBCs, which was exposed to gastric acid, presented greater vertical loss than the control group after chewing simulation (60,000 cycles). The GB and BC CAD/CAM composites presented no significant differences in maximum vertical loss between the gastric acid exposure group and the control group. It is important to consider that CAD/CAM composites are polymerized at high pressure and temperature, resulting in improved mechanical properties and less hydrolytic degradation (Nguyen et al., 2012). Although LU is a CAD/CAM composite, it showed low wear resistance. We hypothesize that the dimensions and volume of the filler particles present in LU may have influenced its wear resistance. Indeed, large fillers can cause a high friction coefficient, leading to greater internal stress within the polymer matrix and a greater possibility of detachment (Heintze et al., 2019); this was observed in FS and LU, which exhibited lower wear resistance despite their high percentage of filler. Matzinger et al. (2018) reported that the mechanical properties of LU were a consequence of the large filler clusters, which are not strongly compacted within the matrix. Larger-sized fillers can be exposed and dislodged more easily; as a result, a greater quantity of exposed matrix would be easily removed by mechanical means.

A variety of systems can be used to assess wear. Mechanical systems (such as stylus profilometry and atomic force microscopy) rely on physical contact with the surface where contours are mapped. Optical systems (laser scanning microscopy and white light optical profilometry) depend on the interactions of light with the surface being captured. The quantification of wear can be through volume (3D) or maximum vertical loss (2D), although a quantitative assessment of wear with 3D methods will provide greater information on topography, roughness, and fractal dimension such as loss of wear—material, etc. Heintze et al. (2005) found that regardless of the quantification method, both volume and vertical loss were highly correlated with each other, so it was not necessary to measure both variables to evaluate the wear resistance of materials. Therefore, it is reasonable to accomplish the objective of this study by considering the value of the maximum vertical loss.

An interesting finding observed in the present study was that, although the materials in both groups showed a drop in the flexural strength and VHN over time, this reduction depended on the specific composition of each material. Ferracane et al. (1998) stated that water diffusion within the polymer network requires time. It occurs quickly initially, but it continues slowly and in a controlled manner until the hydrophilic resin matrix and the silane interface layer become saturated and stabilized (Curtis et al., 2008). The degree of water absorption by the organic matrix depends not only on the physical and chemical affinity to water of the different groups and links in the polymer network (Ferracane et al., 1998), but also on the inorganic filler content of the material, as a higher proportion of inorganic particles reduces the total quantity of polymer available for water absorption (Wendler et al., 2021). The GS and GB resin composites showed the least changes in their mechanical properties, possibly because of their composition and their high inorganic filler content. Wendler et al. reported that GS and GB (73% and 71% filler by volume respectively) absorb 40%–50% less water than the other resin composites studied (Wendler et al., 2021). The exception was LU, which despite its relatively high filler content (65% by volume), it was the CAD/CAM composite resin with the greatest changes in mechanical properties. This issue was probably due to the fact that LU contains a large quantity of zirconia nanoparticles, which may favor water absorption (Wendler et al., 2021; Ergun et al., 2018).

The moderate negative correlation found between the averages of microhardness and wear suggests that, as the microhardness of a material increases, its tendency to wear tends to decrease. This finding supports the idea that surface microhardness can be a critical indicator of wear resistance in various materials. However, it is essential to consider other factors that may have influenced this correlation, such as the microstructure of the tested materials, the presence and distribution of defects, as well as the loading conditions and wear environment.

Immersion time is also a decisive factor in lowering the mechanical properties of RBCs; these can reduce continuously until the polymer network is completely saturated. (Ferracane et al., 1998; Calais and Söderholm, 1988). A period between 7 and 60 days seems to be adequate for most resin composites to reach a saturation state (Ferracane, 2006). In the present study, both the experimental and control groups of FS, LU, BE, BC, and GS showed a significant reduction in flexural strength and VHN values after the first three months (T1–T2). In the case of GB, no significant changes in mechanical properties were observed during the first three months (T1–T2). This may be attributed to two factors already mentioned in this section: (i) lower percentage of organic matrix (ii) high degree of polymer conversion (Wendler et al., 2021; Nguyen et al., 2012). Either factors may influence the level of absorption of the solvent to which resin composites are exposed. Some studies on intrinsic erosion (Cruz et al., 2019; Sulaiman et al., 2015; Briso et al., 2011; Backer et al., 2017) applied accelerated protocols between 12 h and 4 days continuous immersion in different HCl solutions, with the aim of simulating various years of erosion. Despite a HCL solution with a stronger acidic pH was used in the present study, there was no significant changes in the VHN (Cruz et al., 2019; Backer et al., 2017) or surface roughness (Sulaiman et al., 2015; Briso et al., 2011; Backer et al., 2017) of FS and LU composites compared to the initial values. Such a scenario was probably due to the lack of time to achieve total saturation of the polymer network.

Although the limited information offered by manufacturers, conventional and CAD/CAM composites from the same manufacturer present similar compositions (Table 1), but their mechanical properties differ considerably. The greatest resistance to erosion was recorded for the CAD/CAM resin composites (GB, BC, and LU). The reason for such high values in the mechanical tests and Weibull modulus may be related to their level of polymerization, which is performed under controlled conditions at high temperature and pressure (HT/HP) (Mainjot et al., 2016; Wendler et al., 2021; Nguyen et al., 2012). The main effects of pressure on a mixture of monomers are to reduce intermolecular distances and the volume and free movement of the monomers. This slows kinetic polymerization (Nguyen et al., 2012), favor the formation of a more homogeneous structure with fewer internal defects (Tsujimoto et al., 2018). The present study showed that m values of the tested CAD/CAD RBCs were higher compared to those of the tested conventional RBCs. In contrast, the use of conventional composites, which are light-cured during direct clinical procedures, can be characterized by internal defects that jeopardize the longevity of the restoration, as they constitute stress zones from which cracks can propagate (Curtis et al., 2008).

The reliability of the flexural strength data was assessed through a Weibull analysis to achieve a more realistic approach to the clinical performance of the tested materials (Quinn and Quinn, 2009). Indeed, the Weibull modulus (m) was used to describe the variations in distribution of strength due to defects and microcracks that can develop both on the surface and within the material's microstructure. The larger the "m" value, the smaller the range of stresses which can probability cause a fracture; thus, there is a smaller range of error and potentially a greater clinical reliability. (Quinn and Quinn, 2009; McCabe and Carrick, 1986; Bütikofer et al., 2015; Takeshige et al., 2007). Micro-fissures behave in accordance with Griffith's law, whereby the presence of any internal or surface defect can act as a weak zone and, consequently, as a critical defect that reduces the flexural strength of materials and accelerates their failure over time (Curtis et al., 2008).

In the present study, the GB CAD/CAM composite resin showed higher flexural strength and "m" values than all the other resin composites after being subjected to the erosion protocol for six months. Egilmez et al. (2018) reported no significant decrease in flexural strength in LU; they also found increased values after exposure to a HCl solution at 24 h. The authors stated that such results were probably due

to a possible delay in the propagation of cracks in the resin composites in aqueous media, as described by Takeshige et al. (2007). However, the current literature contains no information on the effect of HCl on the flexural strength of CAD/CAM resin composites. Indeed, the evidence available at the moment on these materials concerns on the combined effect of hydrolytic degradation in an aqueous medium and mechanical degradation of the material (Porto et al., 2018; Lauvahutanon et al., 2014; Niem et al., 2020; Hibino et al., 2020). This is probably the main reason for the reduction in flexural strength, as well as the drop in *m* values observed in the present study.

Because of the absence of standardized protocols for the study of erosion, the published data are very disparate. Despite the lack of consensus, it is known that acid in the oral cavity, whether the source be gastric or dietary, only retains its original acidic pH for a few minutes (Shellis et al., 2011; Ranjitkar et al., 2012). The studies of intrinsic erosion are heterogeneous as regards the number of cycles and length of exposure to gastric acid: 4 cycles/1 min (Cabral et al., 2015), 3 cycles/10 min (Albuquerque et al., 2018), 6 cycles/2 min (Kulkarni et al., 2018), or continuous exposure for several hours (Cruz et al., 2019; Sulaiman et al., 2015; Alencar et al., 2019; Briso et al., 2011; Backer et al., 2017; Egilmez et al., 2018; Kulkarni et al., 2018). The present study applied 2 cycles of 2 min per cycle, contemplating post-prandial erosion episodes, as most gastric reflux events or vomiting episodes take place after ingestion of the main meals (Orr, 2003).

The changes observed in the mechanical properties of the tested composites show that no RBCs may be inert when immersed for a long period in an aqueous medium, as well as in circumstances of extreme pH values, such as gastric acid (pH 1.5–3.0) (Ranjitkar et al., 2012; Willumsen et al., 2004; Quigley and Turnberg, 1987).

In conclusion, reductions over time in microhardness, flexural strength and wear resistance are common issues in dental RBCs. In addition, exposure to gastric acid accelerates the deterioration of the mechanical properties in particular in the conventional RBCs, rather than CAD/CAM ones. Thus, considering the limitations of this study, which include the short time period (6 months) for the mechanical properties to evolve and the use of only six RBCs, it is possible to conclude that the choice of a suitable restoration material plays an important part in the durability of dental restorations.

CRediT authorship contribution statement

Alexandra Gil-Pozo: Writing – original draft, Visualization, Methodology, Investigation, Conceptualization. **Daniela Astudillo-Rubio:** Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Álvaro Ferrando Cascales:** Writing – original draft, Methodology, Data curation. **Francesco Inchingolo:** Writing – review & editing, Formal analysis. **Ronaldo Hirata:** Visualization, Conceptualization. **Salvatore Sauro:** Writing – review & editing, Visualization, Validation, Conceptualization. **Andrés Delgado-Gaete:** Writing – original draft, Validation, Supervision, Resources, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

References

Albuquerque, A.P., Oliveira-Reis, B., Catelan, A., Suzuki, T.Y.U., Briso, A.L.F., Santos, PH Dos, 2018. Mechanical and surface properties analysis of restorative materials submitted to erosive challenges in situ. Eur. J. Dermatol. 12, 559–565. https://doi.org/10.4103/ejd.ejd_188_18.

Alencar, M.F., Pereira, M.T., De-Moraes, M.D.R., Santiago, S.L., Passos, V.F., 2019. The effects of intrinsic and extrinsic acids on nanofilled and bulk fill resin composites: roughness, surface hardness, and scanning electron microscopy analysis. Microsc. Res. Tech. 83, 202–207. https://doi.org/10.1002/jemt.23403.

Alshali, R.Z., Salim, N.A., Satterthwaite, J.D., Silikas, N., 2015. Long-term sorption and solubility of bulk-fill and conventional resin-composites in water and artificial saliva. J. Dent. 43, 1511–1518. https://doi.org/10.1016/j.jdent.2015.10.001.

Alzraikat, H., Burrow, M., Maghaireh, G., Taha, N., 2018. Nanofilled resin composite properties and clinical performance: a review. Oper Dent 43, 173–190. https://doi.org/10.2341/17-208-t.

Backer, A.D., Münchow, E.A., Eckert, G.J., Hara, A.T., Platt, J.A., Bottino, M.C., 2017. Effects of simulated gastric juice on CAD/CAM resin composites-morphological and mechanical evaluations. J. Prosthodont. 26, 424–431. https://doi.org/10.1111/jopr.12420.

Badra, V., Faraoni, J., Ramos, R., Palma-Dibb, R., 2005. Influence of different Beverages on the microhardness and surface roughness of resin composites. Oper Dent 30, 213–219.

Beun, S., Glorieux, T., Devaux, J., Vreven, J., Leloup, G., 2007. Characterization of nanofilled compared to universal and microfilled composites. Dent. Mater. 23, 51–59. https://doi.org/10.1016/j.dental.2005.12.003.

Borges, M.G., Soares, C.J., Maia, T.S., Bicalho, A.A., Barbosa, T.P., Costa, H.L., et al., 2019. Effect of acidic drinks on shade matching, surface topography, and mechanical properties of conventional and bulk-fill resin composites. J. Prosthet. Dent 121, 868–876. https://doi.org/10.1016/j.prosdent.2019.02.006.

Briso, A.L.F., Caruzo, L.P., Guedes, A.P.A., Santos, P.H., 2011. In vitro evaluation of surface roughness and microhardness of restorative materials submitted to erosive challenges. Oper Dent 36, 397–402. https://doi.org/10.2341/10-356-L.

Bütikofer, L., Stawarczyk, B., Roos, M., 2015. Two regression methods for estimation of a two-parameter Weibull distribution for reliability of dental materials. Dent. Mater. 31, 33–50. https://doi.org/10.1016/j.dental.2014.11.014.

Cabral, A.C., Lima, L.O., Terossi de Godoi, A.P., Colucci, V., Milori Corona, S.A., Benitez, Catirse ABCE., 2015. Surface roughness of resin composites subjected to hydrochloric acid. Braz. Dent. J. 26, 268–271. https://doi.org/10.1590/0103-6440201300271.

Calais, J.G., Söderholm, K.J.M., 1988. Influence of filler type and water exposure on flexural strength of experimental resin composites. J. Dent. Res. 67, 836–840. https://doi.org/10.1177/00220345880670050801.

Carvalho, T.S., Colon, P., Ganss, C., Huysmans, M.C., Lussi, A., Schlueter, N., et al., 2016. Consensus report of the European Federation of Conservative Dentistry: erosive tooth wear—diagnosis and management. Clin. Oral Invest. 126, 342–346.

Cengiz, S., Sarac, S., Özcan, M., 2014. Effects of simulated gastric juice on color stability , surface roughness and microhardness of laboratory-processed composites. Dent. Mater. J. 33, 343–348. https://doi.org/10.4012/dmj.2013-265.

Chadwick, R.G., McCabe, J.F., Walls, A.W.G., Storer, R., 1990. The effect of storage media upon the surface microhardness and abrasion resistance of three composites. Dent. Mater. 6, 123–128. https://doi.org/10.1016/S0109-5641(05)80042-9.

Cilli, R., Pereira, J.C., Prakki, A., 2012. Properties of dental resins submitted to pH catalysed hydrolysis. J. Dent. 40, 1144–1150. https://doi.org/10.1016/j.jdent.2012.09.012.

Cruz, M.E., Simões, R., Martins, S.B., Trindade, F.Z., Dovigo, L.N., Fonseca, R.G., 2019. Influence of simulated gastric juice on surface characteristics of CAD-CAM monolithic materials. J. Prosthet. Dent 78, 1–8. https://doi.org/10.1016/j.prosdent.2019.04.018.

Curtis, A.R., Shortall, A.C., Marquis, P.M., Palin, W.M., 2008. Water uptake and strength characteristics of a nanofilled resin-based composite. J. Dent. 36, 186–193. https://doi.org/10.1016/j.jdent.2007.11.015.

D Arcangelo, C., Vanini, L., Rondoni, G.D., Pirani, M., Vadini, M., Gattone, M., et al., 2014. Wear properties of a novel resin composite compared to human enamel and other restorative materials. Oper Dent 39, 612–618. https://doi.org/10.2341/13-108-L.

Egilmez, F., Ergun, G., Cekic-Nagas, I., Vallittu, P.K., Lassila, L.V.J., 2018. Does artificial aging affect mechanical properties of CAD/CAM composite materials. J Prosthodont Res 62, 65–74. https://doi.org/10.1016/j.jpor.2017.06.001.

Ergun, G., Sahin, Z., Ataol, A.S., 2018. The effects of adding various ratios of zirconium oxide nanoparticles to poly(methyl methacrylate) on physical and mechanical properties. J. Oral Sci. 60, 304–315. https://doi.org/10.2334/josnusd.17-0206.

Ferracane, J.L., 2006. Hygroscopic and hydrolytic effects in dental polymer networks. Dent. Mater. 22, 211–222. https://doi.org/10.1016/j.dental.2005.05.005.

Ferracane, J.L., 2011. Resin composite - state of the art. Dent. Mater. 27, 29–38. https://doi.org/10.1016/j.dental.2010.10.020.

Ferracane, J.L., Berge, H.X., Condon, J.R., 1998. In vitro aging of dental composites in water - effect of degree of conversion, filler volume, and filler/matrix coupling. J. Biomed. Mater. Res. 42, 465–472. https://doi.org/10.1002/(SICI)1097-4636(19981205)42:3<465::AID-JBM17>3.0.CO;2-F.

Göpferich, A., 1996. Mechanisms of polymer degradation and erosion. Biomater Silver Jubil Compend 17, 117–128. https://doi.org/10.1016/B978-008045154-1.50016-2.

Heintze, S., 2006. How to qualify and validate wear simulation devices and methods. Dent. Mater. 2, 712–734. https://doi.org/10.1016/j.dental.2006.02.002.

Heintze, S.D., Zappini, G., Rousson, V., 2005. Wear of ten dental restorative materials in five wear simulators — results of a round robin test. Dent. Mater. 21, 304–317. https://doi.org/10.1016/j.dental.2004.05.003.

Heintze, S.D., Reichl, F.-X., Hickel, R., 2019. Wear of dental materials: clinical significance and laboratory wear simulation methods —a review. Dent. Mater. J. 38 (3), 343–353. https://doi.org/10.4012/dmj.2018-140, 1.

Hermont, A.P., Oliveira, P.A.D., Martins, C.C., Paiva, S.M., Pordeus, I.A., Auad, S.M., 2014. Tooth erosion and eating disorders: a systematic review and meta-analysis. PLoS One 11, 1–15. https://doi.org/10.1371/journal.pone.0111123.

Hibino, Y., Nagasawa, Y., Eda, Y., Shigeta, H., Nakajima, H., 2020. Effect of storage conditions on mechanical properties of resin composite blanks for cad/cam crowns. Dent. Mater. J. 39, 742–751. https://doi.org/10.4012/dmj.2019-202.

International Organization for Standardization, 2009. ISO 4049:2009.Dentistry — Polymer-Based Restorative Materials, fifth ed. Geneva, Suiza.

International Organization for Standardization, 2018. ISO-6507-1:2018. Metallic Materials -Vickers Hardness Test - Part 1: Test Method. Geneva, Suiza.

Ionescu, AC, Brambilla, E, Pires, PM, López-Castellano, A, Alambiaga-Caravaca, AM, Lenardi, C, Sauro, S, 2022 Sep. Physical-chemical and microbiological performances of graphene-doped PMMA for CAD/CAM applications before and after accelerated aging protocols. Dent Mater 38 (9), 1470–1481. https://doi.org/10.1016/j.dental.2022.06.032. Epub 2022 Jul 7. PMID: 35810033.

Krüger, J., Maletz, R., Ottl, P., Warkentin, M., 2018. In vitro aging behavior of dental composites considering the influence of filler content , storage media and incubation time. PLoS One 13, 1–19. https://doi.org/10.1371/journal.pone.0195160.

Kulkarni, A., Rothrock, J., Thompson, J., 2018. Impact of gastric acid induced surface changes on mechanical behavior and optical characteristics of dental ceramics. J. Prosthodont. 29, 207–218. https://doi.org/10.1111/jopr.12716.

Kumar, N., 2012. Inconsistency in the strength testing of dental resin-based composites among researchers. Pakistan J. Med. Sci. 29, 205–210. https://doi.org/10.12669/pjms.291.2922.

Lauvahutanon, S., Takahashi, H., Shiozawa, M., Iwasaki, N., Asakawa, Y., Oki, M., et al., 2014. Mechanical properties of composite resin blocks for CAD/CAM. Dent. Mater. J. 33, 705–710. https://doi.org/10.4012/dmj.2014-208.

Lazaridou, D., Belli, R., Petschelt, A., Lohbauer, U., 2015. Are resin composites suitable replacements for amalgam? A study of two-body wear. Clin. Oral Invest. 19, 1485–1492. https://doi.org/10.1007/s00784-014-1373-4.

Loke, C., Lee, J., Sander, S., Mei, L., Farella, M., 2016. Factors affecting intra-oral pH – a review. J. Oral Rehabil. 43, 778–785. https://doi.org/10.1111/joor.12429.

Mainjot, A.K., Dupont, N.M., Oudkerk, J.C., Dewael, T.Y., Sadoun, M.J., 2016. From Artisanal to CAD-CAM blocks: state of the art of Indirect composites. J. Dent. Res. 95, 487–495. https://doi.org/10.1177/0022034516634286.

Matzinger, M., Hahnel, S., Preis, V., Rosentritt, M., 2018. Polishing effects and wear performance of chairside CAD/CAM materials. Clin. Oral Invest. 23, 725–737. https://doi.org/10.1007/s00784-018-2473-3.

McCabe, J.F., Carrick, T.E., 1986. A statistical approach to the mechanical testing of dental materials. Dent. Mater. 2, 139–142. https://doi.org/10.1016/s0109-5641(86)80021-5.

Mehta, S.B., Bronkhorst, E.M., Lima, V.P., Crins, L., Bronkhorst, H., Opdam, N.J.M., et al., 2021. The effect of pre-treatment levels of tooth wear and the applied increase in the vertical dimension of occlusion (VDO) on the survival of direct resin composite restorations. J. Dent. 111, 103712 https://doi.org/10.1016/j.jdent.2021.103712.

Moreira da Silva, E., Gonçalves, L., Antunes, J.G., Poskus, L.T., Fellows, C.E., 2011. The diffusion kinetics of a nanofilled and a midifilled resin composite immersed in distilled water, artificial saliva, and lactic acid. Clin. Oral Invest. 15, 393–401. https://doi.org/10.1007/s00784-010-0392-z.

Nguyen, J.F., Migonney, V., Ruse, N.D., Sadoun, M., 2012. Resin composite blocks via high-pressure high-temperature polymerization. Dent. Mater. 28, 529–534. https://doi.org/10.1016/j.dental.2011.12.003.

Niem, T., Youssef, N., Wöstmann, B., 2020. Influence of accelerated ageing on the physical properties of CAD/CAM restorative materials. Clin. Oral Invest. 24, 2415–2425. https://doi.org/10.1007/s00784-019-03101-w.

Nishiyama, N., Suzuki, K., Yoshida, H., Teshima, H., Nemoto, K., 2004. Hydrolytic stability of methacrylamide in acidic aqueous solution. Biomaterials 25, 965–969. https://doi.org/10.1016/S0142-9612(03)00616-1.

Orr, W.C., 2003. Sleep and gastroesophageal reflux: what are the risks? Am. J. Med. 115, 109–113. https://doi.org/10.1016/S0002-9343(03)00207-9.

Örtengren, U., Wellendorf, H., Karlsson, S., Ruyter, I.E., 2001. Water sorption and solubility of dental composites and identification of monomers released in an aqueous environment. J. Oral Rehabil. 28, 1106–1115. https://doi.org/10.1046/j.1365-2842.2001.00802.x.

Osiewicz, A., Werner, A., Roeters, F.J.M., Kleverlaan, C.J., 2019. Wear of direct resin composites and teeth: considerations for oral rehabilitation. Eur. J. Oral Sci. 127, 156–161. https://doi.org/10.1111/eos.12600.

Pini, N.P., De Marchi, L., Ramos, A., Pascotto, R., 2018. Minimally invasive adhesive rehabilitation for a patient with tooth erosion: seven-year follow-up. Oper Dent 44, 45–57. https://doi.org/10.2341/17-181-t.

Pires, PM, de Almeida Neves, A, Lukomska-Szymanska, M, Farrar, P, Cascales, ÁF, Sauro, S, 2024 Feb 2. Bonding performance and interfacial adaptation of modern bulk-fill restorative composites after aging in artificial saliva: an in vitro study. Clin. Oral Investig. 28 (2), 132. https://doi.org/10.1007/s00784-024-05525-5. PMID: 38308668.

Porto, T.S., Roperto, R.C., Akkus, A., Akkus, O., Teich, S., 2018. Effect of storage and aging conditions on the flexural strength and flexural modulus of CAD/CAM materials. Dent. Mater. J. 38, 1–7. https://doi.org/10.4012/dmj.2018-111.

Quigley, E.M.M., Turnberg, L.A., 1987. pH of the microclimate lining human gastric and duodenal mucosa in vivo. Studies in control subjects and in duodenal ulcer patients. Gastroenterology 92, 1876–1884. https://doi.org/10.1016/0016-5085(87)90619-6.

Quinn, J.B., Quinn, G.D., 2009. A practical and systematic review of Weibull statistics for reporting strengths of dental materials. Dent. Mater. 6, 135–147. https://doi.org/10.1016/j.dental.2009.09.006.

Rahim, T.N.A.T., Mohamad, D., Md Akil, H., Ab Rahman, I., 2012. Water sorption characteristics of restorative dental composites immersed in acidic drinks. Dent. Mater. 28, 63–70. https://doi.org/10.1016/j.dental.2012.03.011.

Randolph, L.D., Palin, W.M., Leloup, G., Leprince, J.G., 2016. Filler characteristics of modern dental resin composites and their influence on physico-mechanical properties. Dent. Mater. 32, 1586–1599. https://doi.org/10.1016/j.dental.2016.09.034.

Ranjitkar, S., Kaidonis, J.A., Smales, R.J., 2012. Gastroesophageal reflux disease and tooth erosion. Int J Dent 15, 1–10. https://doi.org/10.1155/2012/479850.

Roos, M., Schatz, C., Stawarczyk, B., 2016. Two independent prospectively planned blinded weibull statistical analyses of flexural strength data of zirconia materials. Materials 9, 1–17. https://doi.org/10.3390/ma9070512.

Salas, M.M.S., Nascimento, G.G., Huysmans, M.C., Demarco, F.F., 2015. Estimated prevalence of erosive tooth wear in permanent teeth of children and adolescents: an epidemiological systematic review and meta-regression analysis. J. Dent. 43, 42–50. https://doi.org/10.1016/j.jdent.2014.10.012.

Schlueter, N., Luka, B., 2018. Erosive tooth wear - a review on global prevalence and on its prevalence in risk groups. Br. Dent. J. 224, 1–7. https://doi.org/10.1038/sj.bdj.2018.167.

Shellis, R.P., Ganss, C., Ren, Y., Zero, D.T., Lussi, A., 2011. Methodology and models in erosion research: Discussion and conclusions. Caries Res. 45, 69–77. https://doi.org/10.1159/000325971.

Sideridou, I., Tserki, V., Papanastasiou, G., 2003. Study of water sorption, solubility and modulus of elasticity of light-cured dimethacrylate-based dental resins. Biomaterials 24, 655–665. https://doi.org/10.1016/S0142-9612(02)00380-0.

Sulaiman, T.A., Abdulmajeed, A.A., Shahramian, K., Hupa, L., Donovan, T.E., Vallittu, P., et al., 2015. Impact of gastric acidic challenge on surface topography and optical properties of monolithic zirconia. Dent. Mater. 31, 1445–1452. https://doi.org/10.1016/j.dental.2015.09.010.

Takeshige, F., Kawakami, Y., Hayashi, M., Ebisu, S., 2007. Fatigue behavior of resin composites in aqueous environments. Dent. Mater. 23, 893–899. https://doi.org/10.1016/j.dental.2006.06.031.

Tsujimoto, A., Barkmeier, W., Fischer, N., Nojiri, K., Nagura, Y., Takamizawa, T., et al., 2018. Wear of resin composites: current insights into underlying mechanisms, evaluation methods and influential factors. Jpn Dent Sci Rev 54, 76–87. https://doi.org/10.1016/j.jdsr.2017.11.002.

Vailati, F., Gruetter, L., Belser, U.C., 2013. Adhesively restored anterior maxillary dentitions affected by severe erosion: up to 6-year results of a prospective clinical study. Eur J Esthet Dent 8, 506–530.

Wang, L., D'Alpino, P.H.P., Lopes, L.G., Pereira, J.C., 2005. Mechanical properties of dental restorative materials: relative contribution of laboratory tests. J. Appl. Oral Sci. 11, 162–167. https://doi.org/10.1590/s1678-77572003000300002.

Wendler, M., Stenger, A., Ripper, J., Priewich, E., Belli, R., Lohbauer, U., 2021. Mechanical degradation of contemporary CAD/CAM resin composite materials after water ageing. Dent. Mater. 37, 1156–1167. https://doi.org/10.1016/j.dental.2021.04.002.

Willumsen, T., Øgaard, B., Hansen, B.F., Rølla, G., 2004. Effects from pretreatment of stannous fluoride versus sodium fluoride on enamel exposed to 0.1 M or 0.01 M hydrochloric acid. Acta Odontol. Scand. 62, 278–281. https://doi.org/10.1080/00016350410000174.

Yap, A.U.J., Teoh, S.H., 2003. Comparison of flexural properties of composite restoratives using the ISO and mini-flexural tests. J. Oral Rehabil. 30, 171–177.

Yap, A., Eweis, A., Yahya, N., 2018. Dynamic and static flexural appraisal of resin-based composites: comparison of the ISO and Mini-flexural tests. Oper Dent 43, 223–231. https://doi.org/10.2341/17-224-l.

Young, A., Tenuta, L.M.A., 2009. Initial erosion models. Caries Res. 45, 33–42. https://doi.org/10.1159/000325943.