

ORIGINAL RESEARCH

Impact of simulated long-term brushing and acid exposure on the surface properties of pit-and-fissure sealants: an *in vitro* study

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Abstract

Background: Pit-and-fissure sealants are widely used to prevent occlusal caries, but daily brushing and repeated acid exposure may compromise their surface integrity. This *in vitro* study evaluated the effects of simulated long-term toothbrushing, with and without intermittent citric acid exposure, on the surface properties of six pit-and-fissure sealants. **Methods:** Disc specimens (n = 84; n = 14 per material; n = 7 per subgroup) prepared from five resin-based sealants and one resin-modified glass-ionomer were subjected to 60,000 brushing strokes under a 2 N load with an abrasive slurry, either alone or combined with intermittent immersion in citric acid (pH 2.5). Surface roughness, surface gloss, mass, and three-dimensional volume were measured before and after aging, and surface topography was assessed by stereomicroscopy and scanning electron microscopy. Data were analyzed using two-way analysis of variance and Tukey's test (alpha = 0.05). **Results:** Acid exposure significantly affected roughness change ($p < 0.001$), volume loss ($p < 0.001$), mass loss ($p < 0.001$), and surface roughness index scores ($p = 0.003$). Surface gloss showed a significant material $\times$ acid interaction ($p < 0.001$) without a significant overall acid main effect ($p = 0.126$). Glass Liner showed the greatest deterioration under acid challenge, whereas Grandio Seal showed the lowest volume loss in both conditions and the highest gloss under acid exposure. **Conclusions:** Within the limitations of this *in vitro* study, sealant performance under erosive-abrasive challenge was material dependent. Materials with lower roughness change and volumetric loss may offer better surface durability in patients with frequent acid exposure.

Keywords

Pit and fissure sealants; Toothbrushing; Citric acid; Dental materials; Surface properties

1. Introduction

Occlusal sealants have been used for caries prevention since the 1970s by sealing susceptible pits and fissures that can trap food debris and promote bacterial biofilm accumulation [1, 2]. Resin-based sealants bond micromechanically to etched enamel and generally show superior wear resistance, particularly when highly filled. Glass-ionomer-based sealants bond chemically and can release fluoride, but their polyacid matrix is more susceptible to acidic dissolution and surface breakdown [3].

In the oral environment, two common challenges threaten the surface integrity of sealants: mechanical abrasion from daily toothbrushing and chemical erosion from dietary acids or gastric reflux. Toothbrushing with abrasive dentifrice can create micro-scratches and material loss, and abrasivity depends on toothpaste abrasives as well as brushing mode and bristle arrangement [4, 5]. Acidic exposure can soften the

superficial matrix and facilitate subsequent abrasive removal ("soften-then-scrub"), potentially accelerating surface degradation under erosion-abrasion cycling [6, 7].

Evidence from *in vitro* aging models suggests that combined erosive/abrasive cycling produces greater wear of fissure sealants than either challenge alone [8]. Similarly, resin-modified glass-ionomer materials can show marked surface changes after acid exposure combined with toothbrushing [9]. Optical properties such as surface gloss are also sensitive to chemical degradation and can provide complementary information to roughness measurements [10]. Because wear pathways depend on the interaction of loading conditions with material composition and filler architecture [11], material ranking may differ depending on the endpoint evaluated. Recent work further suggests that brushing modality can modulate sealant surface texture [12].

In this *in vitro* study, we compared six commercially available pit-and-fissure sealants after simulated long-term tooth-

brushing equivalent to approximately six years of twice-daily brushing, with or without intermittent citric acid exposure. We evaluated surface roughness (Ra), gloss, mass loss, and three-dimensional (3D) volume loss, supported by stereomicroscopy and scanning electron microscopy (SEM). The null hypotheses were that (1) intermittent acid exposure during brushing would not influence the measured surface properties or material loss compared with brushing alone, and (2) there would be no material-dependent differences in these outcomes among the tested sealants, including between resin-based and resin-modified glass-ionomer materials.

2. Materials and methods

2.1 Materials and specimen preparation

Six commercial fissure sealants were evaluated: five resin-based materials—Clinpro™ Sealant (3M, Saint Paul, MN, USA), Fissurit-F (VOCO GmbH, Cuxhaven, Germany), Fissurit-FX (VOCO GmbH, Cuxhaven, Germany), Grandio Seal (VOCO GmbH, Cuxhaven, Germany), and Q-Seal (B.J.M. Laboratories Ltd., Or-Yehuda, Israel)—and one light-cured resin-modified glass-ionomer, Glass Liner (WP Dental, Barmstedt, Germany). A total of 84 disc specimens (5 mm diameter × 2 mm thickness) were fabricated ($n = 14$ per material) using Teflon molds under Mylar strips. Resin-based materials were light-cured according to the manufacturers' instructions.

For each material, specimens were randomly allocated into two subgroups ($n = 7$ each): brushing only (non-acid) and brushing with intermittent acid exposure (acid).

Sample size was calculated a priori using G*Power 3.1.9.7 software (Heinrich Heine University Düsseldorf, Düsseldorf, NRW, Germany) [13]. The selected effect size was informed by a previous fissure-sealant toothbrushing study evaluating surface roughness and weight loss [14]. For the planned analysis-of-variance design, with $\alpha = 0.05$, statistical power = 0.85, effect size $f = 0.52$, and 12 experimental cells (six materials × two acid conditions), the calculation indicated a minimum total sample size of 84 specimens; therefore, 7 specimens were allocated to each material/condition cell.

2.2 Brushing simulation and acid challenge

Specimens were mounted in a custom automatic Press-on Force-Guided brushing simulator developed by Tağtekin and Korkut at Marmara University (Istanbul, Türkiye) and previously described by Meşeli *et al.* [15], so that only the top surface was brushed (Fig. 1, Ref. [15]). The brushing procedure was adapted from previously published *in vitro* toothbrushing and erosion-abrasion protocols used for dental hard tissues, fissure sealants, resin-modified glass-ionomer materials, and the same brushing-simulator platform [5, 7–9, 15]. Protocol-specific adaptations were the use of 60,000 strokes, a 2 N load, staged 10,000-stroke blocks, and intermittent citric acid/water immersion, because the purpose was to impose a standardized long-term erosive-abrasive challenge for material comparison rather than to reproduce a single intraoral exposure pattern. A soft-bristle manual toothbrush was used with a standardized toothpaste slurry (relative dentin abrasivity (RDA) ≈ 85) at approximately 120 strokes/min. The toothpaste was selected to provide a standardized abrasive

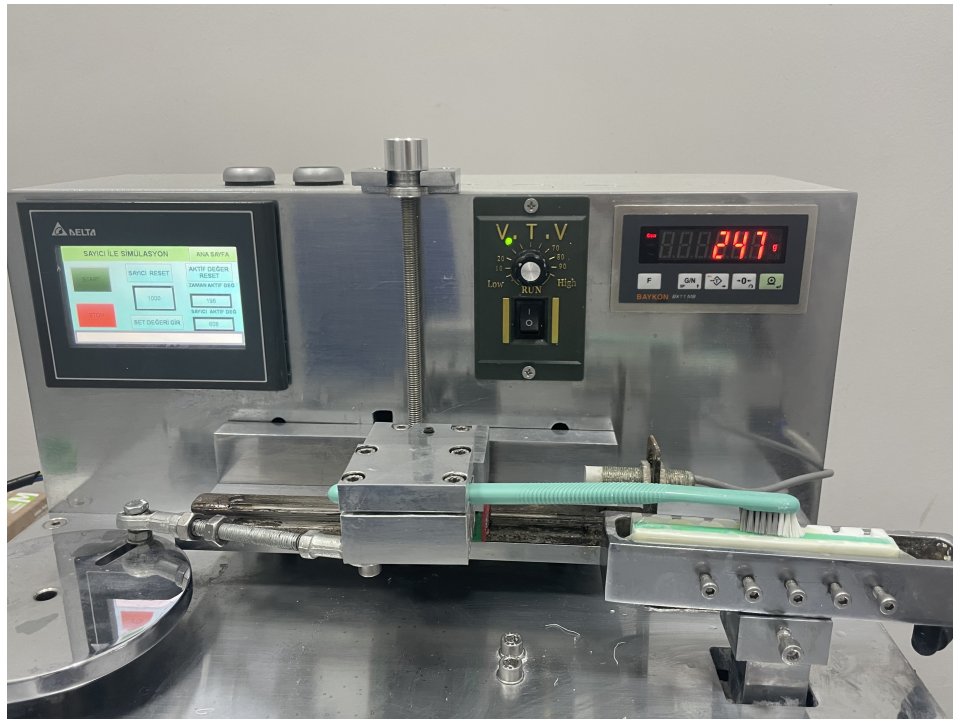


FIGURE 1. Toothbrushing simulator used for mechanical aging. Photograph of the custom Press-on Force-Guided brushing simulator developed by Tağtekin and Korkut at Marmara University (Istanbul, Türkiye) and previously described by Meşeli *et al.* [15]. The simulator was used to deliver 60,000 standardized brushing strokes under controlled load and motion. Specimens were mounted so that only the test surface was exposed to the brushing challenge.

challenge for material comparison; full product details were: Sensodyne Nourish (GlaxoSmithKline Consumer Healthcare, Brentford, UK). Each specimen received 60,000 strokes, corresponding to an estimated six years of twice-daily brushing ($\approx 10,000$ strokes/year). Brushing was performed in staged 10,000-stroke blocks to reduce heat generation and to permit repeated acid/water exposure.

For the acid subgroup, specimens were immersed in stirred citric acid solution (pH 2.5, 37 °C) for 10 min immediately before brushing and after each 10,000 strokes. Control specimens were handled identically, but immersed in distilled water (pH 7.0). During brushing, the lower half of each disc was covered with polyester tape to provide an unbrushed reference surface; the tape was removed after completion of 60,000 strokes.

2.3 Surface roughness and gloss measurements

Surface roughness (R_a , μm) and surface gloss (Gloss Unit (GU)) were measured on the top surface of all specimens before and after the brushing protocol. R_a was measured using a contact profilometer (MarSurf M 300 C, Mahr GmbH, Göttingen, NI, Germany) with a 0.25 mm cut-off length, 4 mm tracing length, and 1 mm/s stylus speed. Three traces were recorded per specimen by the same operator, rotating the specimen 60° clockwise between traces; the mean value was used for analysis. Surface gloss was measured at 60° using a gloss meter (Novo-Curve, Rhopoint Instruments Ltd., St. Leonards-on-Sea, East Sussex, UK) in accordance with International Organization for Standardization (ISO) 2813 [16]. The measurement spot size was 2 mm $\times$ 2 mm, and two readings were averaged per specimen. To minimize ambient light interference and standardize positioning, a custom gray mold was used to ensure that measurements were taken from the same central area of each disc.

2.4 Mass and three-dimensional volume loss

Specimens were dried and weighed before and after aging using an analytical balance (Pioneer, Ohaus Corp., Parsippany, NJ, USA; readability 0.0001 g) to calculate mass loss. Before weighing, specimens were blotted dry with absorbent paper to remove superficial water. Each measurement was repeated three times consecutively, and the mean value was used for analysis. Volumetric loss (mm^3) was assessed by optical scanning (inEos X5, Dentsply Sirona, Bensheim, HE, Germany) to obtain stereolithography (STL) datasets before and after brushing. The reference (pre-brushing) and test (post-brushing) STL files were aligned in 3D inspection software using best-fit registration (Geomagic Control X, version 2022.1.0, 3D Systems, Rock Hill, SC, USA), and volumetric loss was calculated on the brushed half of each specimen as the volume difference between the aligned models [17].

2.5 Stereomicroscopy and surface roughness index scoring

After brushing, surface topography was examined under a stereomicroscope at 30 $\times$ magnification (Leica EZ4 W, Leica Microsystems, Wetzlar, HE, Germany) (Fig. 2). A four-

level surface roughness index was used to categorize visible abrasion and pitting (Table 1), based on Howell and Weekes' criteria [18]. Two blinded examiners scored the images independently and reached consensus in cases of disagreement.

2.6 Scanning electron microscopy

Scanning electron microscopy was conducted as a supplementary qualitative analysis to visualize microstructural surface changes, such as scratches, pitting, crater-like defects, and matrix disruption, and to support interpretation of the quantitative roughness, gloss, mass-loss, and volume-loss findings. Representative specimens from each group were mounted on aluminum stubs and sputter-coated with a platinum-gold alloy before imaging. SEM micrographs were obtained at 10 kV accelerating voltage and 10,000 $\times$ magnification using a scanning electron microscope (EVO MA 10, Carl Zeiss, Oberkochen, BW, Germany). SEM was not used as a primary quantitative outcome.

2.7 Statistical analysis

Statistical analyses were performed using IBM SPSS Statistics version 25.0 (IBM Corp., Armonk, NY, USA) at $\alpha = 0.05$. Change in roughness ($\Delta R_a = \text{post} - \text{baseline}$), volume loss, mass loss, change in gloss ($\Delta \text{GU} = \text{post} - \text{baseline}$), and surface roughness index scores were analyzed using two-way analysis of variance (material $\times$ acid condition) including the interaction term. Partial eta squared (η_p^2) was calculated as an effect size. *Post hoc* comparisons were performed with Tukey's test ($p < 0.05$). Continuous data are reported as mean (standard deviation (SD)).

2.8 Use of artificial intelligence

An artificial intelligence (AI)-assisted language and formatting tool was used during manuscript preparation to improve English language and formatting. The authors reviewed and edited all output and take full responsibility for the final content.

3. Results

3.1 Overall model results

Two-way analysis of variance (material $\times$ acid condition) showed significant main effects of acid exposure on ΔR_a ($p < 0.001$), volume loss ($p < 0.001$), mass loss ($p < 0.001$), and surface roughness index scores ($p = 0.003$), whereas the main effect of acid on ΔGU was not significant ($p = 0.126$). Material type significantly affected ΔR_a ($p = 0.009$), volume loss ($p < 0.001$), surface roughness index scores ($p < 0.001$), and ΔGU ($p < 0.001$). No significant interactions were detected for ΔR_a , volume loss, mass loss, or surface roughness index scores ($p = 0.265$, $p = 0.321$, $p = 0.952$, and $p = 0.904$, respectively), but a significant material $\times$ acid interaction was observed for ΔGU ($p < 0.001$). Acid exposure showed the largest effects on ΔR_a ($\eta_p^2 = 0.573$) and volume loss ($\eta_p^2 = 0.555$) (Table 2).



FIGURE 2. Stereomicroscope setup used for surface evaluation. Photograph of the stereomicroscope setup used to document post-aging surface texture at 30 $\times$ magnification. The setup was used to examine the brushed and protected reference halves of each specimen.

TABLE 1. Howell and Weekes surface roughness index scale.

Score	Description
1	Acceptable surface with fine, scattered scratches
2	Slightly rough surface with more noticeable fine scratches and some coarser scratches
3	Rough surface with many coarse scratches across the entire area
4	Very rough surface with deep, dense, and highly coarse scratches throughout

TABLE 2. Two-way analysis of variance summary (material $\times$ acid condition).

Outcome	Acid condition	Material	Acid $\times$ material
ΔRa (post – baseline)	$p < 0.001$; $\eta_p^2 = 0.573$	$p = 0.009$; $\eta_p^2 = 0.188$	$p = 0.265$; $\eta_p^2 = 0.084$
Volume loss	$p < 0.001$; $\eta_p^2 = 0.555$	$p < 0.001$; $\eta_p^2 = 0.537$	$p = 0.321$; $\eta_p^2 = 0.077$
Mass loss	$p < 0.001$; $\eta_p^2 = 0.156$	$p = 0.284$; $\eta_p^2 = 0.081$	$p = 0.952$; $\eta_p^2 = 0.015$
SRI score	$p = 0.003$; $\eta_p^2 = 0.115$	$p < 0.001$; $\eta_p^2 = 0.296$	$p = 0.904$; $\eta_p^2 = 0.021$
ΔGU (post – baseline)	$p = 0.126$; $\eta_p^2 = 0.032$	$p < 0.001$; $\eta_p^2 = 0.487$	$p < 0.001$; $\eta_p^2 = 0.513$

ΔRa , change in surface roughness; ΔGU , change in gloss units; η_p^2 , partial eta squared; SRI, surface roughness index. Sample sizes were acid condition: $n = 42$ per level; material: $n = 14$ per material; and acid $\times$ material interaction: $n = 7$ per cell.

3.2 Volume loss

Volume loss was significantly higher under acid exposure than under the non-acid condition ($p < 0.001$) and differed significantly among materials ($p < 0.001$), with no significant interaction ($p = 0.321$) (Table 2). The acid-exposed Glass Liner group showed the highest volume loss (1.47 mm^3), whereas the non-acid Grandio Seal group showed the lowest (0.39 mm^3) (Table 3).

3.3 Mass loss

Mass loss was significantly affected by acid exposure ($p < 0.001$), whereas material type and the interaction term were not significant ($p = 0.284$ and $p = 0.952$, respectively) (Table 2). The highest mean loss was observed for acid-exposed Glass Liner (0.00389 g), and the lowest was observed for non-acid Clinpro (0.00219 g) (Table 3).

3.4 Surface roughness and surface roughness index

All materials showed increased Ra after brushing (Table 3). Two-way analysis of variance of ΔRa (post – baseline) indicated significant effects of acid exposure ($p < 0.001$) and material ($p = 0.009$), with no significant interaction ($p = 0.265$) (Table 2). Post-brushing surface roughness index scores were higher under acid exposure ($p = 0.003$) and differed by material ($p < 0.001$), with no interaction ($p = 0.904$). The highest mean surface roughness index was observed for acid-exposed Glass Liner (3.00 ± 0.00), followed by acid-exposed Q-Seal (2.71 ± 0.49) (Table 3).

3.5 Surface gloss

ΔGU differed significantly among materials ($p < 0.001$) and showed a significant material $\times$ acid interaction ($p < 0.001$), whereas the main effect of acid exposure was not significant ($p = 0.126$) (Table 2). The highest gloss change under acid

exposure was recorded for Grandio Seal (76.79 GU), whereas the highest gloss change without acid exposure was recorded for Glass Liner (68.36 GU). The lowest gloss change was observed for non-acid Fissurit-FX (17.09 GU) (Table 3).

3.6 Microscopy

Stereomicroscopy revealed more pronounced surface pitting and irregularity after acid-assisted brushing, particularly for Glass Liner (Figs. 3,4). SEM micrographs (Figs. 5,6) showed that acid-exposed Glass Liner exhibited crater-like defects and disrupted matrix morphology, whereas Grandio Seal and other resin-based materials generally exhibited finer scratches with more preserved surface continuity.

4. Discussion

This study used a high-cycle brushing protocol (60,000 strokes; ≈ 6 -year equivalent) with or without intermittent citric acid exposure to evaluate erosive-abrasive degradation of six pit-and-fissure sealants. Two-way analysis of variance demonstrated that acid exposure significantly increased roughness change (ΔRa), volume loss, mass loss, and surface roughness index scores, whereas gloss change (ΔGU) showed a significant material $\times$ acid interaction and no overall main effect of acid. This design deliberately isolated brushing and citric acid exposure; however, it did not include thermocycling, acquired pellicle formation, salivary buffering, biofilm activity, or dynamic pH cycling. Therefore, the findings should be interpreted as a controlled comparative stress test rather than a full reproduction of intraoral aging.

Regarding the null hypotheses, the first null hypothesis (that intermittent acid exposure during brushing would not influence the measured outcomes) was rejected for ΔRa , volume loss, mass loss, and surface roughness index scores because acid showed significant main effects for these endpoints (Table 2). For ΔGU , we failed to reject the null hypothesis of an overall

TABLE 3. Descriptive statistics for ΔRa , volume loss, mass loss, surface roughness index score, and ΔGU .

Group	Material	ΔRa (Mean $\pm$ SD)	Volume loss (Mean $\pm$ SD)	Mass loss (Mean $\pm$ SD)	SRI score (Mean $\pm$ SD)	ΔGU (Mean $\pm$ SD)
Acid	Clinpro	0.1191 ± 0.0355	0.9500 ± 0.2620	0.0028286 ± 0.0003251	2.29 ± 0.488	26.71 ± 13.38
Acid	Fissurit-F	0.1210 ± 0.0397	1.1754 ± 0.6281	0.0034286 ± 0.0003546	2.43 ± 0.535	55.34 ± 9.74
Acid	Fissurit-FX	0.1079 ± 0.0193	0.8781 ± 0.0799	0.0031286 ± 0.0004645	2.29 ± 0.488	56.29 ± 7.52
Acid	Glass Liner	0.1594 ± 0.0246	1.4704 ± 0.2048	0.0038857 ± 0.0006067	3.00 ± 0.000	57.27 ± 14.66
Acid	Grandio Seal	0.1067 ± 0.0270	0.7127 ± 0.0725	0.0027714 ± 0.0003450	2.14 ± 0.378	76.79 ± 14.12
Acid	Q-seal	0.1316 ± 0.0289	1.2335 ± 0.1461	0.0037571 ± 0.0004577	2.71 ± 0.488	39.96 ± 12.56
Non-acid	Clinpro	0.0794 ± 0.0256	0.5258 ± 0.0770	0.0021857 ± 0.0006568	2.14 ± 0.378	50.00 ± 7.40
Non-acid	Fissurit-F	0.0662 ± 0.0191	0.5301 ± 0.1094	0.0025714 ± 0.0003402	2.29 ± 0.488	44.13 ± 11.51
Non-acid	Fissurit-FX	0.0604 ± 0.0264	0.4652 ± 0.0604	0.0022429 ± 0.0004928	2.00 ± 0.000	17.09 ± 4.95
Non-acid	Glass Liner	0.0745 ± 0.0202	0.8972 ± 0.0743	0.0026286 ± 0.0008077	2.57 ± 0.535	68.36 ± 16.93
Non-acid	Grandio Seal	0.0595 ± 0.0213	0.3918 ± 0.0601	0.0022429 ± 0.0006997	1.86 ± 0.378	52.47 ± 8.15
Non-acid	Q-seal	0.0723 ± 0.0094	0.8854 ± 0.1047	0.0026143 ± 0.0004947	2.29 ± 0.488	56.13 ± 15.50

Acid: brushing with intermittent citric acid exposure; non-acid: brushing without acid exposure. ΔRa , change in surface roughness; ΔGU , change in gloss units; SD, standard deviation; SRI, surface roughness index.

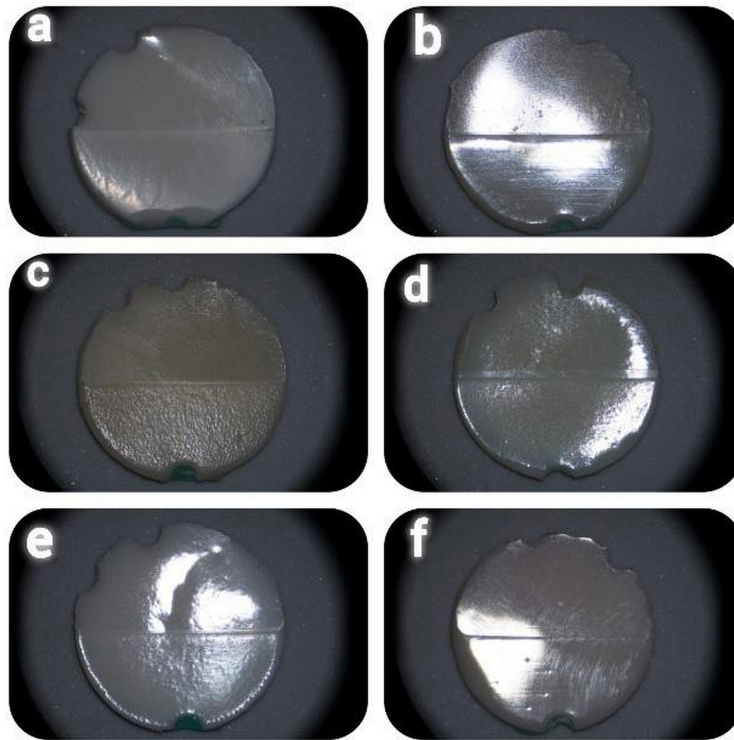


FIGURE 3. Representative stereomicroscope images after brushing without acid exposure. Representative 30 $\times$ stereomicroscope images after 60,000 brushing strokes without acid exposure. In each panel, one half represents the protected, unbrushed reference surface, and the other half represents the brushed test surface. (a) Q-Seal. (b) Grandio Seal. (c) Glass Liner. (d) Fissurit-FX. (e) Fissurit-F. (f) Clinpro.

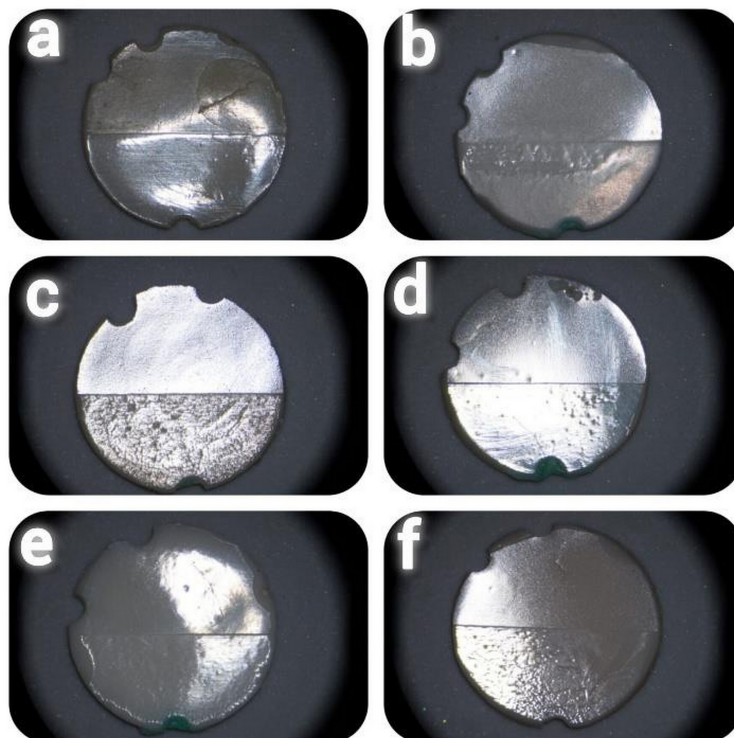


FIGURE 4. Representative stereomicroscope images after brushing with intermittent citric acid exposure. Representative 30 $\times$ stereomicroscope images after 60,000 brushing strokes with intermittent citric acid exposure (pH 2.5). In each panel, one half represents the protected, unbrushed reference surface, and the other half represents the brushed test surface. (a) Q-Seal. (b) Grandio Seal. (c) Glass Liner. (d) Fissurit-FX. (e) Fissurit-F. (f) Clinpro.

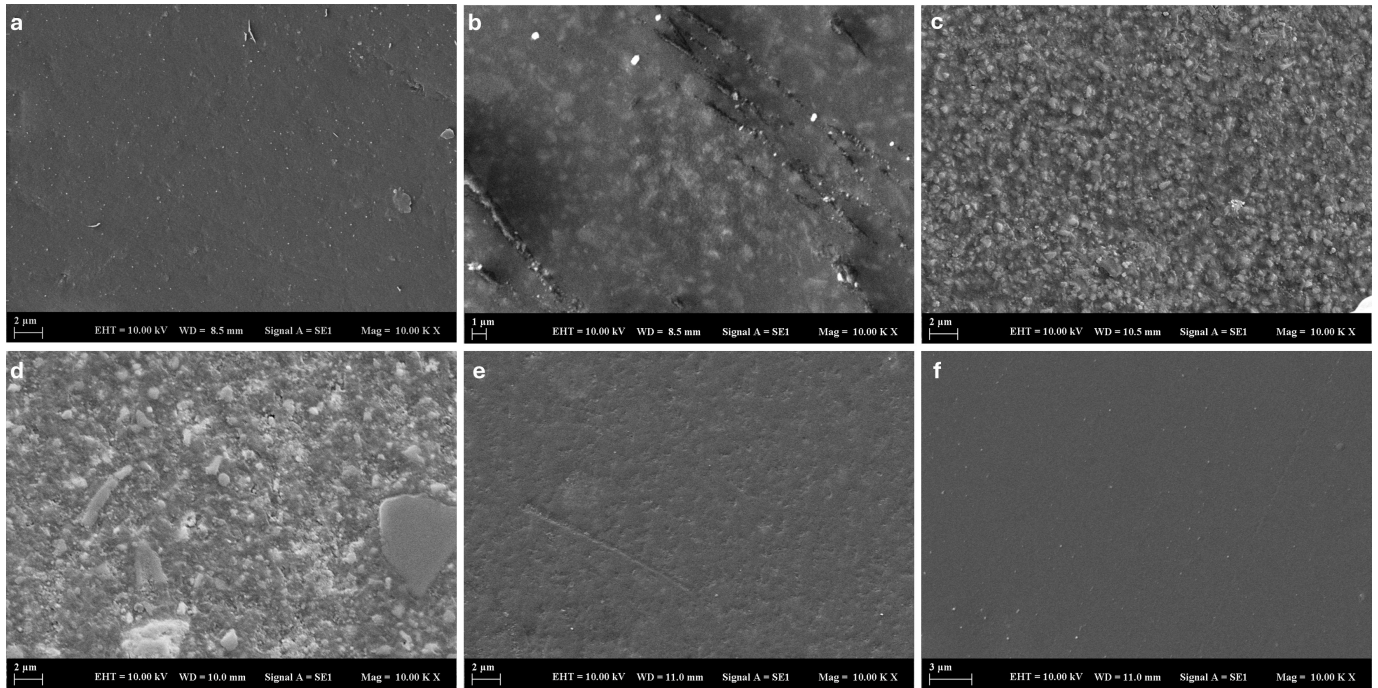


FIGURE 5. Representative scanning electron microscopy (SEM) micrographs after brushing without acid exposure. Representative scanning electron micrographs obtained at 10,000 $\times$ magnification and an accelerating voltage of 10 kilovolts (kV) after 60,000 brushing strokes without acid exposure. (a) Q-Seal. (b) Grandio Seal. (c) Glass Liner. (d) Fissurit-FX. (e) Fissurit-F. (f) Clinpro. Scale bars are shown within the individual panels. EHT, extra high tension (accelerating voltage); WD, working distance; SE, secondary electrons.

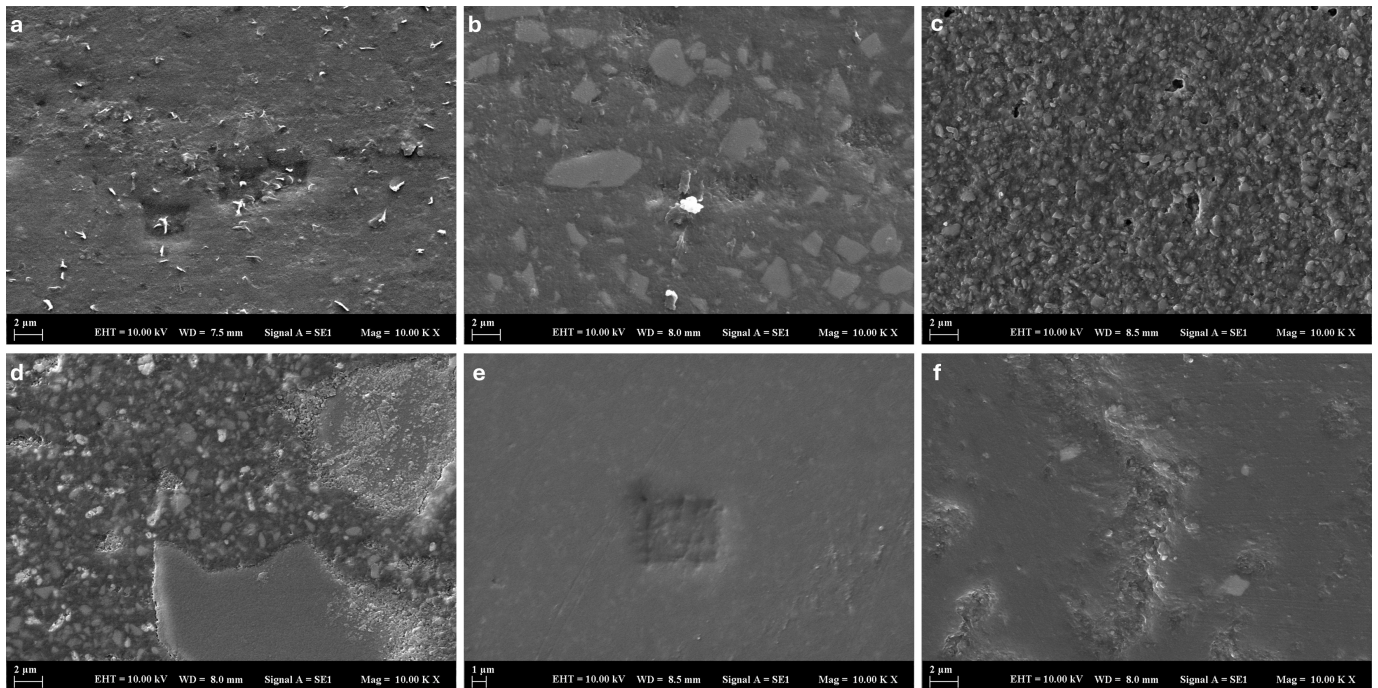


FIGURE 6. Representative scanning electron microscopy (SEM) micrographs after brushing with intermittent citric acid exposure. Representative scanning electron micrographs obtained at 10,000 $\times$ magnification and an accelerating voltage of 10 kilovolts (kV) after 60,000 brushing strokes with intermittent citric acid exposure (pH 2.5). (a) Q-Seal. (b) Grandio Seal. (c) Glass Liner. (d) Fissurit-FX. (e) Fissurit-F. (f) Clinpro. Scale bars are shown within the individual panels. EHT, extra high tension (accelerating voltage); WD, working distance; SE, secondary electrons.

acid main effect ($p = 0.126$), but we rejected the null hypothesis of no material-dependent response because the significant material $\times$ acid interaction indicates that the influence of acid on Δ GU depended on the sealant. The second null hypothesis (that there would be no material-dependent differences among sealants) was rejected for Δ Ra, volume loss, surface roughness index scores, and Δ GU, but not for mass loss (material main effect $p = 0.284$).

Toothbrushing abrasion is influenced by multiple factors, including toothpaste abrasives and brushing mechanics. *In vitro* studies have demonstrated measurable substrate wear from abrasives and detergents [4], and long-term simulations show that abrasivity can vary with brushing mode and bristle arrangement [5]. In our study, all materials showed increased Ra and altered surface topography after brushing, consistent with repeated abrasive contact producing micro-scratches and preferential removal of resin-rich surface layers. Because the toothpaste slurry used here was standardized (RDA ≈ 85), the magnitude of roughening observed can be interpreted within standardized approaches for characterizing dentifrice abrasivity [19, 20]. From a clinical standpoint, surface roughness values above the proposed threshold for plaque retention may increase biofilm accumulation [21].

The greater deterioration observed under acid challenge is consistent with a “soften-then-scrub” mechanism: low-pH exposure weakens the superficial structure and increases susceptibility to abrasive removal. This is particularly relevant for resin-modified glass-ionomer materials, which can be more sensitive to acid-mediated dissolution of the polyacid matrix [3]. Prior work using simulated gastric acid plus toothbrushing has also reported clinically relevant surface changes in resin-modified glass-ionomer materials under combined challenge [9], and fissure sealant-specific aging studies indicate that combined erosive/abrasive conditions produce more pronounced wear than isolated challenges [8]. In the present study, acid exposure significantly increased roughness change and volume loss overall, and the acid-exposed Glass Liner showed the highest post-brushing Ra and largest volume loss. Similar synergy under erosion-abrasion cycling has been demonstrated for tooth-colored restorative materials and enamel [6]. Moreover, the apparent magnitude of combined effects can vary with protocol design (e.g., timing between erosion and abrasion, type of acid, and abrasion load), which should be considered when interpreting acid-related differences [7].

Material ranking depended on the endpoint evaluated. Grandio Seal showed the lowest volume loss in both conditions and the highest gloss under acid exposure, suggesting favorable resistance to combined erosive-abrasive wear. However, the lowest post-brushing Ra in the non-acid condition was observed for Fissurit-FX, and the highest non-acid gloss was observed for Glass Liner. Because Ra is a line-averaged roughness parameter, whereas gloss is governed by specular reflection and light scattering that depend on texture directionality and microstructural features such as filler exposure or pull-out, gloss rankings may diverge from Ra even when surfaces appear similarly worn [10, 11, 22, 23]. The significant material $\times$ acid interaction for gloss suggests that acid-related changes in light scattering were material specific,

which may explain the observed gloss-Ra mismatches.

A strength of the present study is that material loss was evaluated using both gravimetric and volumetric approaches. In resin-based systems, small mass differences can be difficult to interpret because of water sorption and short-term hydration effects. Therefore, we complemented mass loss with 3D volumetric analysis based on pre- and post-scanning and surface-model superimposition, which can provide sensitive discrimination of surface loss patterns [17]. This approach is supported by validation studies indicating that sequential 3D scanning with surface superimposition can provide reproducible wear measurements, but that alignment strategy may influence the measured magnitude of small volume changes [24–26]. Consistent with this, the effect sizes observed here were larger for Δ Ra and volume loss than for mass loss, reinforcing the value of volume-based outcomes when comparing materials with different chemistries and filler architectures.

Qualitative microscopy supported the quantitative findings. Stereomicroscopy and SEM revealed more pronounced surface disruption under acid-assisted brushing, with crater-like defects and matrix breakdown in Glass Liner, whereas resin-based materials generally showed finer scratch patterns and more preserved surface continuity. Such observations align with integrative evidence that sealant wear pathways are multifactorial and depend on the interaction of loading conditions with composition and filler architecture [11]. Although two blinded examiners scored the surface roughness index with consensus, future work should report inter-examiner agreement (e.g., weighted kappa) to strengthen reproducibility.

This study has limitations inherent to *in vitro* aging. Although this design helped isolate erosive-abrasive effects, it does not reproduce salivary buffering, acquired pellicle formation, biofilm activity, thermocycling, or dynamic pH cycling, and therefore should be interpreted as a controlled comparative stress test rather than a direct clinical simulation. The study was also not designed to model additional clinical modifiers or to test different erosive regimens. Although the sample size was calculated a priori and supported by a comparable fissure-sealant brushing study, the subgroup size was still limited ($n = 7$ per cell), so material-specific estimates should be interpreted with caution and confirmed in larger studies. Future work should incorporate thermocycling, pellicle and biofilm models, and diversified erosive protocols to better approximate clinical conditions, and clinical studies are needed to confirm whether the relative performance patterns observed *in vitro* translate to long-term sealant longevity. *In situ* work suggests that intraoral saliva exposure can partially reharder erosively softened enamel and influences subsequent toothbrush abrasion [27]. Because pit-and-fissure sealants are recommended and supported by systematic evidence for preventing occlusal caries, understanding durability under erosive-abrasive challenges may inform material selection and maintenance in high-risk patients [28, 29].

5. Conclusions

Simulated long-term toothbrushing increased surface roughness and altered surface topography for all tested sealants. Intermittent citric acid exposure significantly

increased roughness change and material loss (volume and mass) and worsened surface roughness index scores overall, whereas the change in gloss response depended on material type/composition. Grandio Seal showed the most favorable volumetric stability and maintained the highest gloss change under acid exposure; however, endpoint-specific rankings differed across roughness and gloss outcomes. Material selection for patients with elevated erosive risk should consider both material chemistry and resistance to erosive-abrasive wear.

AVAILABILITY OF DATA AND MATERIALS

The datasets generated and analyzed during the current study are available from the corresponding author on reasonable request.

AUTHOR CONTRIBUTIONS

EA—performed the main experimental work and drafted the manuscript. MCA, İHO, VÖE, TÜK and AB—contributed to the methodology. DT—supervised the study. AB and EA—interpreted the results and performed the SEM analyses. BK, TÜK and DT—critically revised the manuscript. All authors reviewed and approved the final version.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This *in vitro* study did not involve human participants, human tissue, personal data, or animals; therefore, ethics approval and informed consent were not required.

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Not applicable.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

REFERENCES

- [1] Simonsen RJ. Pit and fissure sealants. In Simonsen RJ (ed.) Clinical applications of the acid etch technique (pp. 19–42). 1st edn. Quintessence Publishing Co.: Chicago. 1978.
- [2] Cvikl B, Moritz A, Bekes K. Pit and fissure sealants—a comprehensive review. *Dentistry Journal*. 2018; 6: 18.
- [3] Nicholson JW. Chemistry of glass-ionomer cements: a review. *Biomaterials*. 1998; 19: 485–494.
- [4] Moore C, Addy M. Wear of dentine *in vitro* by toothpaste abrasives and detergents alone and combined. *Journal of Clinical Periodontology*. 2005; 32: 1242–1246.
- [5] Bizhang M, Schmidt I, Chun YP, Arnold WH, Zimmer S. Toothbrush abrasivity in a long-term simulation on human dentin depends on brushing mode and bristle arrangement. *PLOS ONE*. 2017; 12: e0172060.
- [6] Yu H, Wegehaupt FJ, Wiegand A, Roos M, Attin T, Buchalla W. Erosion and abrasion of tooth-colored restorative materials and human enamel. *Journal of Dentistry*. 2009; 37: 913–922.
- [7] Wiegand A, Attin T. Design of erosion/abrasion studies—insights and rational concepts. *Caries Research*. 2011; 45: 53–59.
- [8] Hamza B, Sekularac M, Attin T, Wegehaupt FJ. *In vitro* evaluation of fissure sealants' wear under erosive, abrasive and erosive/abrasive conditions. *European Archives of Paediatric Dentistry*. 2023; 24: 55–61.
- [9] Tărăboanță I, Buhățel D, Nica I, Stoleriu S, Ghiorghe AC, Pancu G, *et al*. The impact of simulated gastric acid and toothbrushing on surface characteristics of resin-modified glass-ionomer cements. *Medicina*. 2022; 58: 1149.
- [10] Zovko R, Cvitanović S, Mabić M, Šarac Z, Čorić A, Glavina D, *et al*. The effect of chemical degradation and polishing on the gloss of composite dental materials. *Materials*. 2023; 16: 3727.
- [11] Faria M, Guedes A, Rompante P, Carvalho O, Silva F, Henriques B, *et al*. Wear pathways of tooth occlusal fissure sealants: an integrative review. *Biotribology*. 2021; 27: 100190.
- [12] Ludovichetti FS, Rizzato A, Galante V, Tinazzo G, Magrin GL, Comitale E, *et al*. Effect of manual, electric, and sonic toothbrushing on retention and surface integrity of resin-based sealants. *International Journal of Dentistry*. 2025; 2025: 5740355.
- [13] Faul F, Erdfelder E, Lang AG, Buchner A. G*Power 3: a flexible statistical power analysis program for the social, behavioral, and biomedical sciences. *Behavior Research Methods*. 2007; 39: 175–191.
- [14] Bodrumlu EH. Effects of tooth brushing on surface roughness and weight loss in fissure sealants with different filler ratios. *Annals of Abbasi Shaheed Hospital and Karachi Medical & Dental College*. 2025; 30: 25–32.
- [15] Meseli S, Alkan E, Korkut B, Kanar O, Tagtekin D. Abrasiveness and bleaching level of toothpastes on composite resins: a quantitative analysis using a novel brushing simulator. *Applied Sciences*. 2025; 15: 2314.
- [16] International Organization for Standardization. ISO 2813:2014. Paints and varnishes—determination of gloss value at 20°, 60° and 85°. 2014. Available at: <https://www.iso.org/standard/56807.html> (Accessed: 01 September 2023).
- [17] Korkut B, Uzun KE, Hacıali C, Unal T, Tagtekin D. Evaluation of enamel surface roughness and volumetric change after resin remnant removal following orthodontic bracket debonding. *Oral Health and Preventive Dentistry*. 2025; 23: 355–364.
- [18] Howell S, Weekes WT. An electron microscopic evaluation of the enamel surface subsequent to various debonding procedures. *Australian Dental Journal*. 1990; 35: 245–252.
- [19] Hefferen JJ. A laboratory method for assessment of dentifrice abrasivity. *Journal of Dental Research*. 1976; 55: 563–573.
- [20] International Organization for Standardization. ISO 11609:2026. Dentistry—dentifrices—requirements, test methods and marking. 2026. Available at: <https://www.iso.org/standard/11609> (Accessed: 04 August 2026).
- [21] Bollen CML, Lambrechts P, Quirynen M. Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. *Dental Materials*. 1997; 13: 258–269.
- [22] O'Brien WJ, Johnston WM, Fanian F, Lambert S. The surface roughness and gloss of composites. *Journal of Dental Research*. 1984; 63: 685–688.
- [23] Heintze SD, Forjanic M, Rousson V. Surface roughness and gloss of dental materials as a function of force and polishing time *in vitro*. *Dental Materials*. 2006; 22: 146–165.
- [24] O'Toole S, Osnes C, Bartlett D, Keeling A. Investigation into the accuracy and measurement methods of sequential 3D dental scan alignment. *Dental Materials*. 2019; 35: 495–500.
- [25] Stober T, Heuschmid N, Zellweger G, Rousson V, Rues S, Heintze SD. Comparability of clinical wear measurements by optical 3D laser scanning in two different centers. *Dental Materials*. 2014; 30: 499–506.
- [26] Gkantidis N, Dritsas K, Ren Y, Halazonetis D, Katsaros C. An accurate

- and efficient method for occlusal tooth wear assessment using 3D digital dental models. *Scientific Reports*. 2020; 10: 10103.
- [27] Jaeggi T, Lussi A. Toothbrush abrasion of erosively altered enamel after intraoral exposure to saliva: an *in situ* study. *Caries Research*. 1999; 33: 455–461.
- [28] Wright JT, Tampi MP, Graham L, Estrich C, Crall JJ, Fontana M, *et al.* Sealants for preventing and arresting pit-and-fissure occlusal caries in primary and permanent molars: a systematic review of randomized controlled trials—a report of the American Dental Association and the American Academy of Pediatric Dentistry. *Journal of the American Dental Association*. 2016; 147: 631–645.e18.
- [29] Ahovuo-Saloranta A, Forss H, Walsh T, Nordblad A, Mäkelä M,

Worthington HV. Pit and fissure sealants for preventing dental decay in permanent teeth. *Cochrane Database of Systematic Reviews*. 2017; 7: CD001830.

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