







Erosive and abrasive challenge effects on superficial roughness of resin infiltrants and sealant

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Editor: Dr. Altair A. Del Bel Cury

Received: April 24, 2024

Accepted: December 11, 2024

Aim: This study aimed to evaluate changes in surface roughness of an experimental resin infiltrant, in comparison with the commercial infiltrant Icon® and the resin sealant Prevent®, following different erosive challenges and abrasive simulations with brushing cycles. **Methods:** A total of 20 samples per group were prepared: (I) Commercial Infiltrant Icon® (IC), (II) Experimental Infiltrant (EI), and (III) Commercial Resin Sealant Prevent® (SR). Surface roughness was assessed using a rugosimeter at three distinct time points: prior to the erosive challenge (T0), post-erosive challenge (T1), and following the abrasive simulation (T2). Each group was subdivided into two subgroups (n=10) to undergo different erosive challenges: intrinsic acid and extrinsic acid simulations. The intrinsic acid challenge was conducted by immersing the samples in a hydrochloric acid demineralizing solution (pH=2.3), while the extrinsic acid challenge involved immersion in a soft drink demineralizing solution (pH=2.9). Following the erosive challenges, the specimens were maintained in relative humidity and surface roughness was reassessed (T1). Subsequently, the same groups underwent brushing simulation (10.000 cycles), after which surface roughness was measured again. Data were analyzed by three-way ANOVA with repeated measures, followed by Tukey's post-hoc test for multiple comparisons, with a significance level set at 5%. **Results:** All groups demonstrated an increase on surface roughness, regardless of demineralizing method used (p<0.001). The resin sealant exhibited the highest surface roughness changes under both erosive conditions when compared to the resin infiltrants. There was no statistical difference between the two erosive challenges, regardless of the material and time. **Conclusion:** In conclusion, all three tested materials showed an increase in surface roughness following erosive and abrasive challenges.

Keywords: Tooth erosion. Composite resins. Surface properties. Pit and fissure sealants.



Introduction

Dental erosion is characterized by enamel loss resulting from chemical process in the absence of bacterial involvement¹⁻⁵, and can be caused by intrinsic or extrinsic acids, or a combination of both⁶. Intrinsic factors include gastroesophageal disorders, such as reflux, and the use of medicines that cause hyposalivation, leading to an acid saliva². Extrinsic factors are primarily associated with the consumption of acidic foods and beverages, such as soft drinks, citric fruits and natural juices, but can also be caused by habits like regular swimming (in pool treated with chloride) and drug use^{2,4,7}.

Additionally, to dental intervention and preventive measures, multidisciplinary treatment with nutritional and gastroesophageal follow-up is necessary for early detection and management of habits that contribute to erosion process^{6,8}. In this regard, studies have shown that fluoride solutions can serve as protective agents, however their efficacy relies on frequent applications⁵. Other fluoride-based materials, such as resin sealants and infiltrants, have also been investigated for their protective properties^{5,9,10}. Nevertheless, once erosion extends into the dentin, restorative procedures become necessary to reduce hypersensitivity and restore both esthetics and function⁶.

Icon®, a commercially available resin infiltrant, tends to occlude enamel pores in initial caries lesion, stabilizing them by filling the pores, thereby preventing bacterial infiltration and halting further progression¹¹. Recent studies have explored the use of this material as a potential protector for dental surfaces exhibiting erosive wear resulting from acid challenges without bacterial involvement^{5,10}. The resin infiltrant is regarded as an effective material as it increases microhardness in demineralized areas, presents higher resistance to demineralization on infiltrated surfaces and can be applied in one session, saving clinical time¹¹.

However, resin infiltrants primarily contain triethylene glycol dimethacrylate (TEGDMA), a base monomer that enhances penetration capability in demineralized areas. Due to its low molecular weight, TEGDMA has a hydrophilic profile, which can lead to increased solubility in the high-humidity oral environment compared to other monomers such as BisGMA and BisEMA¹¹.

High hydrophilicity can reduce resin infiltrant mechanical properties and compromise their clinical behavior in medium and long terms¹¹. Given this scenario, some researchers have been investigating other formulations to reduce or eliminate the current limitations¹¹. An experimental resin infiltrant associating BisEMA (a monomer of high molecular weight) with TEGDMA was developed as an attempt to promote high hydrophilicity and improve mechanical properties¹¹.

In vitro² and in situ¹⁰ studies have demonstrated that resin sealants can protect enamel and dentin from erosive processes by creating a barrier on the surface of non-carious lesions⁵. Resin infiltrants are commonly used to treat early-stage white-spot lesions¹¹⁻¹³ but have also been considered for initial erosion lesions due to their enamel penetration capability, contributing to mechanical protection against acidic challenges⁵. A previous in vitro study by Oliveira et al observed that resin

infiltrants have a protective effect on enamel surfaces against dental erosion caused by hydrochloric acid, similar to that of resin sealants¹⁴.

As an oral environment, patients are the ones responsible for habits such as diet and oral hygiene⁶. In addition to being considered indispensable for maintaining good oral health, brushing can accelerate or aggravate dental erosion, since it can cause abrasive wear^{6,15}. Applying a material layer that can protect dental tissue from erosive challenges capable of resisting this abrasive effect is important to minimize this process¹⁶.

Another factor to consider is the behavior of resin infiltrants under erosive challenges in the oral environment, specifically investigating whether resin materials exhibit differences in response to abrasive and erosive challenges. Given the need of research on the superficial alterations of resin infiltrants used as protective materials in erosive and abrasive conditions, this study aimed to evaluate and compare the surface roughness of three materials: an experimental resin infiltrant (EI), the commercial resin infiltrant Icon® (IC), and the commercial resin sealant Prevent® (SR). These materials were subjected to different erosive challenges and abrasive wear from brushing. The null hypotheses were as follows: (I) no significant difference in surface roughness exists between the materials, (II) no significant difference in surface roughness exists across the evaluation timepoints, and (III) no significant difference in surface roughness exists between the erosive solutions.

Materials and Methods

Materials

Three different materials as surface protectors against erosive challenges were selected: (I) commercial infiltrant Icon® (DMG, Hamburg, Germany), (II) commercial resin sealant Prevent® (FGM, São Paulo, Brazil), and (III) an experimental resin infiltrant.

Table 1. Distribution and classification of experimental groups

Group	Composition
IC	TEGDMA, initiators – additives*
SR	BisGMA, TEGDMA, methacrylic acids, stabilizer, CQ, co-initiators and load of fluorine-aluminum-silicate glass
EI	25% Bis-EMA + 75% TEGDMA + 0.5mol% CQ + 1mol% EDAB

Bis-EMA: ethoxybisphenol A glycidyl dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; CQ: camphorquinine; EDAB: dimethylaminoethylbenzoate. * According to manufacturer DMG, Hamburg, Germany

Specimens preparation

20 disc-shaped specimens (5mm x 1mm) of each group was made using a silicone matrix, totaling 60 specimens. After light-curing for 40s (IC and EI) and 20s (SR), as per manufacture instructions, and excess removal with a scalpel and #1200-grit sandpaper, all samples were stored at 37°C during 24h. As standard protocol, all

surfaces were polished with #2000 and #2500 grit water sandpaper (Norton Ltda., Guarulhos – SP) for 30s with water on a polisher (Arotec S/A Indústria e Comércio, Cotia – SP) and washed in ultrasonic bath (Marconi - 1450A, Piracicaba – SP) for 5 minutes between polishings.

Erosive simulation

Each group was subdivided (n=20) into two subgroups (n=10) and submitted to (I) intrinsic (gastric acid) or (II) extrinsic (soft drink) erosive simulation, according to the following protocols:

- I. Intrinsic (gastric acid simulation): the specimens (n=10) were individually immersed in 15ml of hydrochloric acid demineralizing solution (pH=2.3) for 2 minutes, then washed with deionized water and immersed for 60 minutes in stabilizer Tris Buffered Saline (TBS). The cycle was repeated four times a day for 5 days. At the end of each cycle, the specimens were stored in relative humidity with deionized water^{10,17}.
- II. Extrinsic (soft drink simulation): the specimens (n=10) were individually immersed in 15ml of soft drink demineralizing solution (pH=2.9) for 5 minutes^{18,19}, washed with deionized water and then immersed for 60 minutes in stabilizer Tris Buffered Saline (TBS)¹⁹. This cycle was repeated four times a day for 5 days. At the end of each cycle, the specimens were stored in relative humidity with deionized water.

Surface roughness evaluation

Specimens of each subgroup (n=10) underwent surface roughness evaluation before (T0) and after (T1) erosive simulation. Initial means were obtained from three different points (rotating 120° after each analysis) using a rugosimeter (SurfecorderSE 1700 – Kosaka Laboratory, Sotokonda Chiyoda-KuTokyo, Japan). Subsequently, the specimens underwent erosive simulation according to item 2.3 and the surface roughness was once again evaluated following the same parameters as the first analysis. Values and means were obtained for each group.

Surface abrasion by mechanical brushing

After exposure to intrinsic or extrinsic acidic challenge and roughness evaluation, specimens of each subgroup underwent abrasive simulation by mechanical brushing for 10.000 cycles (ODEME Dental research - Toothbrushing simulator (MEV4T 10X) São Francisco, Luzerna – SC). Dental brushes with soft bristles (Colgate Classic Clean, Colgate-Palmolive Company, São Paulo, SP, Brasil) were inserted into the equipment, staying parallel to the specimen surface.

A slurry was prepared by diluting 90g of dentifrice (Colgate total 12 - Clean mint 90g; Colgate-Palmolive Company, São Paulo, SP, Brazil) in 270ml of deionized water (1:3 proportion) and mixed on a vibration plaque to obtain a homogenous mixture.

The 10.000 cycles were performed in a frequency of 6Hz at $37^{\circ} \pm 0.5^{\circ}\text{C}$, at 25mm with a 200g load to simulate force during brushing under normal conditions. After the cycles, the specimens were washed with deionized water, dried with absorbent paper and the surface was then reevaluated with the rugosimeter (T2).

Qualitative surface analysis by scanning electron microscopy (SEM)

Two specimens from each group were randomly selected and placed into metallic stubs with carbon tape and gold coated. Then, the samples were evaluated by scanning electronic microscopy (SEM) at 15kV (JSM 5600LV – Jeol Inc., Peabody, MA, EUA) at 1000x magnification.

Statistical analysis

Data were tabulated and investigated according to parametric analysis using IBM SPSS Statistic version 21 (R Core Team, 2020, Vienna, Austria). Repeated measures (material, time and erosive method) were analyzed by three-way ANOVA and multiple comparisons (material X time, time X treatment, treatment X material) by Tukey's test, with a 5% significance level.

Results

Surface roughness

Table 2 summarizes the findings. The results indicate a significant statistical difference among the evaluation timepoints (T0, T1, T2), with the surface roughness of all three tested materials increasing after the erosive simulation (T1) and further increasing following the combined erosive and abrasive simulation (T2), regardless of the solution (intrinsic or extrinsic). In terms of materials, the resin sealant (SR) exhibited a significant increase in surface roughness at both T1 and T2, showing statistical differences compared to the resin infiltrant Icon® (IC) and the experimental resin infiltrant (EI), regardless of the erosive solution. Both resin infiltrants demonstrated similar results across all timepoints and abrasive method. No significant differences were observed between the extrinsic and intrinsic simulations, irrespective of the timepoint or material analyzed.

Table 2. Mean (standard deviation) roughness as a function of the material, erosive solution and time

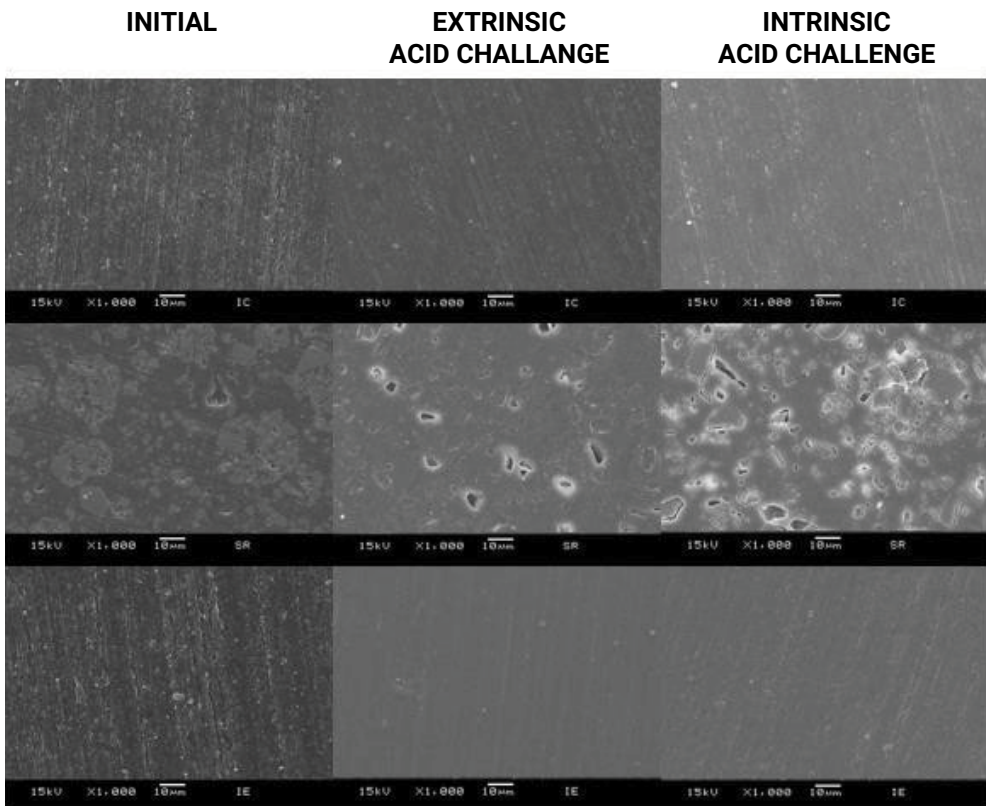
Erosive solution	Material	Time		
		Initial (T0)	After erosion (T1)	After erosion+abrasion (T2)
Intrinsic	IC	0.279 (0.054) Ca*	0.598 (0.164) Bb*	0.865 (0.159) Ab*
	RS	0.335 (0.072) Ca*	0.909 (0.252) Ba*	1.681 (0.213) Aa*
	EI	0.291 (0.038) Ca*	0.624 (0.067) Bb*	0.757 (0.070) Ab*
Extrinsic	IC	0.274 (0.066) Ca*	0.551 (0.121) Bb*	0.785 (0.164) Ab*
	RS	0.337 (0.077) Ca*	0.957 (0.204) Ba*	1.601 (0.122) Aa*
	EI	0.281 (0.041) Ca*	0.557 (0.070) Bb*	0.688 (0.077) Ab*

Means followed by distinct letters (uppercase horizontally and lowercase vertically) differ from each other ($p \leq 0.05$), p (material) < 0.0001 ; p (solution) = 0.1221; p (material x solution) = 0.7356; p (time) < 0.0001 ; p (time x solution) = 0.2363; p (material x time) < 0.0001 ; p (material x solution x time) = 0.7885. (*) Represents absence of difference between intrinsic and extrinsic solutions.

Scanning Electron Microscopy (SEM)

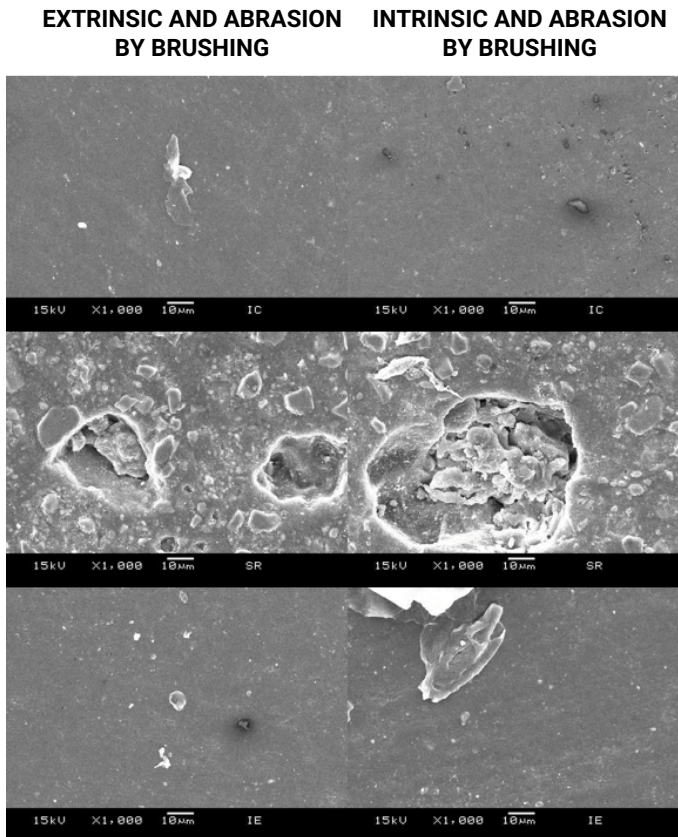
Figure 1 illustrates the surface morphology patterns of the three tested materials – Icon® (IC), Resin Sealant (RS), and the experimental infiltrant (EI) – before and after both extrinsic and intrinsic erosive challenges. The images suggest surface alterations across all three groups, irrespective of the erosive challenge. The resin sealant exhibited the most pronounced surface changes, particularly after the intrinsic simulation. However, it remains unclear which erosive method had the greatest impact on the material's surface.

Figure 2 presents the surface morphology of each material following abrasive simulation by mechanical brushing, combined with extrinsic and intrinsic erosive challenges. These images indicate surface changes in all three materials, regardless of the erosive challenge. The resin sealant again appeared to show the greatest surface alteration, though it is still inconclusive as to which method most significantly affected the material's surface.



Left column initial condition, middle column extrinsic challenge, right column intrinsic challenge. IC: Icon; SR: Resinous Sealant; IE: Experimental Infiltrant

Figure 1. Representative images of the three materials evaluated after erosive challenge with 1000x magnification.



Left column: extrinsic and abrasion by brushing. Right column: intrinsic and abrasion by brushing. IC: Icon; SR: Resinous Sealant; IE: Experimental Infiltrant

Figure 2. Representative images of the three materials evaluated after abrasive simulation associated with erosive challenges with 1000x magnification.

Discussion

Methods to control dental erosive wear aim to prevent the progression of lesions by minimizing surface dissolution. This study evaluated the surface roughness of three materials – the commercial resin infiltrant Icon® (IC), the commercial resin sealant Prevent® (RS), and an experimental resin infiltrant (EI) – subjected to different erosive challenges and abrasive simulation via mechanical brushing. All tested materials demonstrated an increase in surface roughness, irrespective of the demineralization method. The RS group exhibited a statistically significant difference compared to the resin infiltrants, thus rejecting the first null hypothesis.

Changes in the surface roughness of restorative materials, particularly resin-based materials, can have a direct impact on their clinical longevity²⁰. The increased roughness observed in the resin sealant may be attributed to the exposure of inorganic particles within its composition. Borges et al.²¹ argue that this exposure occurs because of the wear of the organic matrix, which, when degraded, leads to particle exposure and consequently a rougher surface. The chemical composition of the

material and the characteristics of the inorganic particles can influence the extent of surface wear in resin-based materials^{22,23}. SEM analysis (Figure 1 and 2) shows a difference in surface material among the resin infiltrants and the resin sealant, which presented an irregular surface at all evaluation times and especially after mechanical brushing.

For effective enamel protection, the resin material must be capable of sealing the surface, ensuring that any damage occurs to the resin layer rather than the dental tissue¹⁶. The use of resin infiltrants as a protective coating in eroded areas remains a controversial topic. Zhao et al.²⁴ observed that the penetration capability of resin infiltrants was insufficient, as the entire superficial layer of the infiltrant was removed following erosive challenges, leaving the enamel exposed. In contrast, Oliveira et al.¹⁴ demonstrated that resin infiltrants effectively protected dental tissue against erosive challenges.

The second null hypothesis, that suggested no variation in surface roughness over time was also rejected. In terms of material comparison, our results showed statistically significant difference at all evaluation timepoints (Table 1). The increase in surface roughness at T1 can be attributed to the erosive cycle (intrinsic or extrinsic), which typically causes alterations in the material's surface¹⁷⁻¹⁹. Erosion increases surface roughness by the action of acids on the dental surface, leading to the loss of mineral content a few micrometers deep. This demineralization softens the outer layer, making it more vulnerable to abrasive wear²⁵.

Another important point to consider is the composition of the resin material's organic matrix, as it can influence water sorption and solubility²⁶. Monomers with lower molecular weight such as TEGDMA are often used in combination with higher molecular weight monomers (e.g., BisGMA), to increase their hydrophilic properties. However, they may become more susceptible to acid degradation²³.

Surface roughness also increased after mechanical brushing (T2), consistent with previous studies^{27,28}. This increase may be attributed to the material's composition and the post-erosive wear mechanism, as acid attacks (extrinsic or intrinsic) soften the outermost layer, making it more prone to removal and subsequent wear by mechanical brushing²⁵.

Roughness analysis of resin materials in an *in vitro* study allow for the simulation of clinical conditions related to patient habits, providing insights into the material's behavior over the medium and long term. High hydrophilicity, which leads greater exposure of the inorganic matrix, can result in undesirable clinical outcomes such as susceptibility to degradation, increase in surface roughness, discoloration and reduced mechanical properties²⁹⁻³².

The third null hypothesis, however, was not rejected. Despite involving different erosion mechanisms, no statistically significant difference were observed between the two erosive challenges. This is likely due to their similar pH and immersion times (intrinsic: pH=2.3; extrinsic pH=2.9). SEM analysis (Figure 1) revealed similar surface roughness for all materials following immersion in both extrinsic and intrinsic solutions, supporting the statistical analysis.

Our findings suggest that resin infiltrants may be a viable alternative for treating initial erosive lesions, as they exhibited less surface roughness compared to resin sealant. However, the limitations of the in vitro design did not prevent a full assessment of real oral conditions. Future studies should focus on the performance of resin infiltrants on dental surfaces, incorporating in situ and in vivo analysis for more comprehensive data.

In conclusion, all three tested materials demonstrated an increase in surface roughness at both T1 and T2 evaluation times, with the commercial sealant showing a significantly greater increase when compared to the infiltrants. No significant differences were observed between extrinsic and intrinsic erosion simulations.

Acknowledgments

The authors acknowledge the contribution of Adriano Luis Martins from the Microscopy and Image Center at Piracicaba Dental School (University of Campinas, Piracicaba, São Paulo, Brazil) for the assistance in scanning electron microscopy analyses.

This study was financed in part by the Fundação de Amparo à Pesquisa de São Paulo (FAPESP) – Process Code 2019/11396-9.

Conflict of Interest

The authors have no conflict of interest to disclose.

Data availability

Datasets related to this article will be available to the corresponding author upon request.

Author Contribution

Janaína Cardoso Santos Couto: Performed all methodology, obtained data and wrote initial draft. **Janaína Emanuela Damasceno:** Performed all methodology, obtained data, described the results and reviewed initial draft. **Gabriela Alves de Cerqueira:** Performed brushing methodology, reviewed initial draft and wrote final manuscript. **Priscila Régis Pedreira:** Performed all methodology and obtained data. **Jade Laísa Gordilio Zago:** reviewed initial draft, wrote final manuscript and manuscript submission. **Flávio Henrique Baggio Aguiar:** conceptualization, reviewed methodology. **Giselle Maria Marchi:** conceptualization, reviewed methodology and wrote final manuscript. All authors actively revised and approved the final version of the manuscript.

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