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The Potential of a Bioactive, Pre-reacted, Glass-Ionomer Filler Resin Composite to Inhibit the Demineralization of Enamel *in Vitro*

IF Leão • N Araújo • CK Scotti • RFL Mondelli • MM de Amoêdo Campos Velo • JFS Bombonatti

Clinical Relevance

A prereacted, glass-ionomer filler fluoride-containing resin composite had lower remineralization potential than glass-ionomer cements but was able to inhibit enamel demineralization; thus, it may be an option for restoring dental surfaces for patients at high risk of caries.

SUMMARY

Evidence is lacking on the use of surface prereacted glass-ionomer filler resin composites to inhibit demineralization and that simulate real clinical conditions. The present laboratory study evaluated the potential of such composites to prevent demineralization and quantified fluoride (F) and other ions released from restorative materials after a dynamic pH-cycling regimen applied to the tooth material interface in vitro. The pH-cycling regimen was assessed by measuring surface hardness (SH) along with energy dispersive X-ray spectroscopy (EDX).

Methods and Materials: Ninety blocks of bovine enamel were subjected to composition analysis with EDX, and were further categorized based on SH. The blocks were randomly divided into 6 treatment groups (n=15 each): F IX (Fuji IX

Isabela Furlaneto Lesão, MSc, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Nayara Araújo, MSc, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Cassiana Koch Scotti, MSc, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Rafael Francisco Lia Mondelli, DDS, MS, PhD, Full Professor, Department of Operative Dentistry, Endodontic and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Marilia Mattar de Amoêdo Campos Velo, DDS, PhD, Post-doctoral researcher, Professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

*Juliana Fraga Soares Bombonatti, DDS, MS, PhD, Associate Professor, Department of Operative Dentistry, Endodontic and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

*Corresponding author: Alameda Octávio Pinheiro Brisolla 9-75, Bauru - SP - 17012-901, Brazil; e-mail: julianafraga@usp.br

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Extra; GC Corporation); IZ (Ion Z, FGM); F II (Fuji II LC, GC Corporation); B II (Beautifil II, Shofu); F250 (Filtek Z250 XT, 3M ESPE); and NT (control, no treatment). The blocks were subjected to a dynamic pH-cycling regimen at 37°C for 7 days concurrently with daily alternations of immersion in demineralizing/remineralizing solutions. EDX was conducted and a final SH was determined at standard distances from the restorative materials (150, 300, and 400 μm).

Results: The EDX findings revealed a significant increase in F concentration and a decrease in Ca^{2+} in the enamel blocks of group B II after the pH-cycling regimen ($p<0.05$). SH values for groups F IX, IZ, and F II were greater than those for groups B II, F250, and NT at all distances from the materials.

Conclusions: The results suggest that each of 3 restorative materials, F IX, IZ, and F II, partially inhibited enamel demineralization under a dynamic pH-cycling regimen.

INTRODUCTION

Owing to their bonding capacity and good physical properties, composite resins have become the most commonly used restorative materials in clinical practice.[1,2] Acid conditioning of enamel as proposed by Buonocore[3] and new adhesive systems have significantly improved the long-term stability of composite resin restorations, mainly of those restricted to the enamel or with enamel-located margins. Although the adhesion of restorative materials to the enamel results in a stable, disease-resistant, and long-lived restoration, the lack of marginal integrity and sealing increases the risk of caries at the margin of the restoration. The main challenges to achieving a long-lived marginal seal include the existence of extensive cavities, the clinician's knowledge of adhesive-system techniques and composition, the type of enamel substrate (ie, sound or carious), the presence of fluorosis or anomalies, and whether the tooth is primary or permanent.[4] Although these variables are known to not affect marginal sealing, it is essential to develop new restorative materials that can boost the long-term performance of restorations.

Composite resins are used for restoration, but several clinical studies have shown higher failure rates for resin composites than for amalgam restorations,[5,6] mainly attributed to secondary caries that develop adjacent to the filling.[7] Secondary caries are responsible for 60% of all replacement restorations in dental practice;[8] for this reason, a Cochrane systematic review has reinforced the benefit of amalgam restorations to restore posterior teeth because the incidence of secondary caries is higher with composites than with amalgam or other restorative materials.[9,10] However, dental amalgam contains mercury, and reducing the environmental burden of metals through improved environmental practices is a concern, as highlighted by the Treaty of Minamata.[11] Therefore, the use of amalgam is being gradually reduced in clinical use, focusing rather on alternative materials that also are based on minimally invasive dentistry.[12]

Owing to the limitations of composite materials, the principle of restorative dentistry in recent decades has prompted new technologies that improve restorative materials while aligning aesthetics as well as the function/integrity of the dental structure with the challenges inherent in the oral environment. Conventional glass-ionomer cements have interesting properties such as biocompatibility, fluoride (F) release, modulus of elasticity similar to tooth structure, and the ability to chemically bond to the tooth structure,[13] but poor mechanical properties. To overcome these poor properties, resin-modified glass-ionomer cements were developed. These are considered to be a significant advancement, as they improved the physical properties of the cements and also enabled an ion-exchange–based adhesive surface to form and concomitantly release F, which can inhibit dental caries from forming adjacent to the restoration.[14-16] However, the hydrophilic nature of polyhydroxyethylmethacrylate hydrogels results in increased water uptake and solubility, which negatively influences the mechanical properties and clinical performance of these materials in areas that bear stress, such as the posterior teeth.[17,18]

Based on the F-releasing mechanism of glass-ionomer cements during the acid-base reaction phase, in 1999, Roberts and others synthesized a prereacted glass-ionomer (PRG) filler that could be incorporated with polyalkenoic acids into resinous materials from the complete or partial reaction of ion-leachable glasses.[19] PRG fillers consist of fluoroaluminosilicate glass that forms a water-rich siliceous hydrogel in the presence of water. The result is a stable PRG filler with a trilaminar structure that allows the release and recharge of F via a ligand-exchange mechanism within the prereacted hydrogel.[20,21]

The giomer (glass-ionomer + polymer), a novel group of hybrid composite restorative materials based on surface-PRG (S-PRG) fillers, is a technology of interest because it provides biofunctions to restorative materials. Such hybrids have been used in various dental materials such as composite resins, bonding agents, cements, and resin sealants.[22,23] Previous studies have reported that materials based on S-PRG fillers have the potential to prevent the demineralization of enamel and dentin, as shown by the use of coatings, solutions, or sealing agents;[23,24] therefore, this new generation of F-releasing materials deserves further investigation. Evidence is lacking on restorations carried out with S-PRG fillers to prevent the development of caries and that simulate real clinical conditions, and a dynamic pH-cycling regimen could help clarify this aspect. Moreover, it is important to evaluate the ions released from this class of restorative material that could act as a mineral reservoir to combat caries developing around restorations; these ions can be detected by quantifying the inorganic components in caries-like lesions adjacent to materials.

We aimed to evaluate the potential of S-PRG fillers and F-releasing restorative materials to inhibit the demineralization of enamel compared with conventional and resin-modified glass-ionomer cements. We quantified the ions released from these materials in areas adjacent to the tooth–restoration interface using the combined analyses of surface hardness (SH) and energy dispersive X-ray spectroscopy (EDX). The null hypotheses tested were: (1) restorations using S-PRG fillers would not prevent enamel demineralization inhibition; and (2) that there would be no difference in the quantification of chemical elements in the enamel mineral elements around restorative materials submitted for compositional analysis via EDX.

METHODS AND MATERIALS

Experimental Design

The current laboratory study involved 1 factor restorative material in 6 levels: NT (no treatment [control group]); conventional glass-ionomer cement F IX (Fuji IX Extra; GC Corporation, Tokyo, Japan); conventional glass-ionomer cement IZ (Ion Z; FGM, Pembroke Pines, FL, USA); resin-modified glass-ionomer cement F II (Fuji II LC; GC Corporation); S-PRG fillers, F-releasing restorative material B II (Beautifil II; Shofu, San Marcos, CA, USA); and composite resin F250 (Filtek Z250 XT; 3M ESPE, St. Paul, MN, USA). The experimental units were enamel blocks obtained from bovine incisors and selected by SH. The response variables were based on the SH and EDX analyses. Table 1 lists the specifications of all materials used.

Table 1: *Materials, Classification, and Composition of Materials Evaluated in this Study*

Materials	Manufacturer	Classification	Composition
Beautifil II (B II)	Shofu (Kyoto, Japan)	Composite resin: fluoride-containing resin composite (bioactive prereacted glass-ionomer filler; giomer system)	Glass particle S-PRG, glass fluoride, aluminum, borosilicate particles, TEGDMA, Bis-GMA, particle size 20–40 nm
Filtek Z250 (F250)	3M ESPE (St. Paul, USA)	Composite resin (negative control)	Bis-GMA, UDMA, Bis-EMA (zirconia/silica), particle size 0.01–3.5μm
Fuji IX Extra (F IX)	GC Corporation (Tokyo, Japan)	Conventional glass ionomer cement (positive control)	Fluoroaluminosilicate glass, potassium persulphate, ascorbic acid
Fuji II LC (F II)	GC Corporation (Tokyo, Japan)	Modified glass ionomer cement (positive control)	Fluoroaluminosilicate glass particles, composite monomers, photo initiators
Ion Z (IZ)	FGM (Joinville, Brazil)	Conventional glass ionomer cement (positive control)	Glass of calcium, aluminum, zinc, fluoride, silicate, polycarboxilic acid, deionized water, titanium dioxide, iron oxide

Abbreviations: Bis-EMA, bisphenol A diglycidyl methacrylate ethoxylated; Bis-GMA, bisphenol A-glycidyl methacrylate; S-PRG, surface prereacted glass; TEGDMA, triethyleneglycoldimethacrylate; UDMA, urethanedimethacrylate.

Preparation and Selection of Samples from Enamel Blocks

Enamel specimens (4×4×2 mm) were obtained from bovine incisors, which were cut using an ISOMET low-speed saw (Buehler Ltd, Lake Bluff, IL, USA). The blocks were polished sequentially using #600 and #1200 grit sandpaper discs (CarbiMet paper discs; Buehler Ltd). For the final polishing a felt disc with a 1-μm diamond suspension (Buehler Ltd) was used at high speed under a weight of 172 g. During each change of grit, as well as at the end of the polishing process, the specimens were ultrasonicated in deionized water for 2 minutes using an ultrasonic device (USC 750; Unique Group, Indaiatuba, São Paulo, Brazil). Baseline Knoop SH (KHN) was determined by making 5 indentations (spaced 100-μm apart) using a microhardness tester (Model HMV-2000 OR HMV-2; Shimadzu Corporation, Kiyamachi-Nijo, Kyoto, Japan) under a 25-g load for 10 seconds. Assessments were made under a 25-g load for 10 seconds. To establish the homogeneity of the samples, specimens with an average SH >20% or <350 KHN were excluded.

Treatment of the Enamel Blocks

The selected enamel blocks were randomized according to baseline SH and randomly divided into 6 groups (n=15 each): NT (no treatment [control]), F IX, IZ, F II (positive control), B II (evaluated material), and F250 (negative control). The 4 × 4 mm surface of each of the 90 randomized blocks was divided into 2 regions, delimiting the area where standardized cavities were prepared (3×1.5 mm), with diamond tip N° 1093/1093F (Figure 1A) for posterior restorative treatment.

For restorative procedures, resin composites were inserted incrementally, covered with a polyester strip, pressed with a glass slide to delimit the thickness of the material by digital pressure, and then light-cured with a light-emitting diode–curing device (Dabi Atlante, São Paulo, Brazil) operating at 961 mW/cm^2 for 20 seconds.

Before initiating the pH-cycling regimen, half of each specimen was secured with tape to treat the enamel with the various resin composites, limiting the experimental area. After restorative treatment, the protective tape was removed, and the specimens were stored for 24 hours in a chamber with relative humidity of 100% at 37°C for 24 hours before testing. Specimens were protected using a base, which was then removed by acetone.[25] No cavities were prepared in the blocks used for the control group, and half of each control block was coated with an acid-resistant varnish to protect that surface for subsequent pH cycling (Figure 1B).

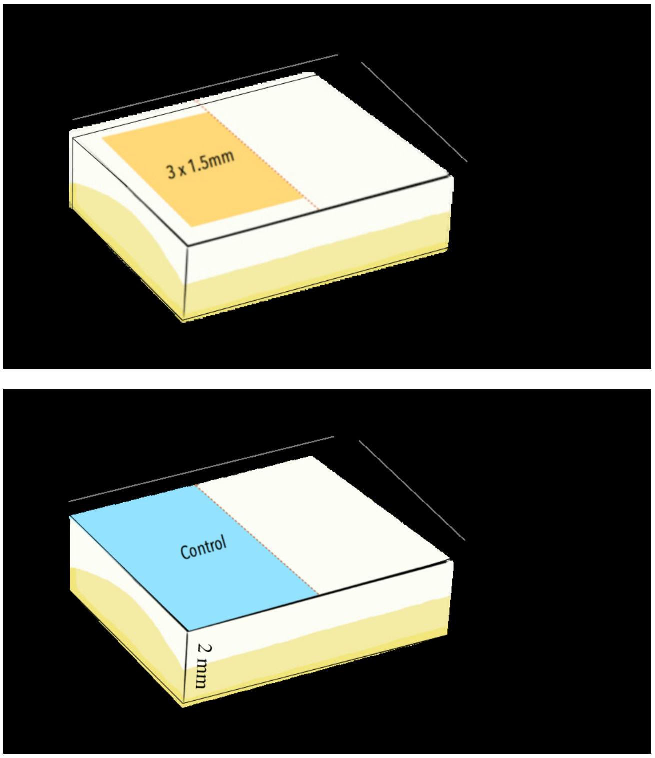

Figure 1. ***A:*** *Delimited area that was used to prepare a standardized cavity (3×1.5 mm).* ***B:*** *Control area of the block coated with varnish protecting this region for posterior pH cycling.*

The pH-cycling Regimen

After the treatments, the specimens were subjected to a dynamic pH-cycling regimen for 7 days at 37°C.[26] Each day, the specimens were subjected to alternating immersion in 30 mL of demineralizing solution (2.0 mM $Ca(NO_3)_2 \cdot 4H_2O$, 2.0 mM $NaH_2PO_4 \cdot 2H_2O$, 0.077 mM acetate buffer, 0.02 ppm F, pH 4.7) for 6 hours and in a remineralizing solution (1.5 mM $Ca(NO_3)_2 \cdot 4H_2O$, 0.9 mM $NaH_2PO_4 \cdot 2H_2O$, 150 mM KCl, 0.1 mM sodium acetate, 0.03 ppm F, pH 7.0) for 18 hours for 5 days.[26] In the last 2 days, the blocks were immersed in the remineralization solution according to Vieira and others.[26] Each specimen was stored in a plastic container to avoid any possible effects of the F released by the materials. When each solution was exchanged, the blocks were washed under deionized water and then dried with blotting paper before being transferred to the next solution.

Surface Hardness Analysis

The SH of the blocks (n=15) was again determined at the end of the pH-cycling regimen. Five indentations were made at 3 standard distances (150, 300, and 450 μm) from the treatment; the indentations were separated by 100 μm (Figure 2). For each block, the mean value of

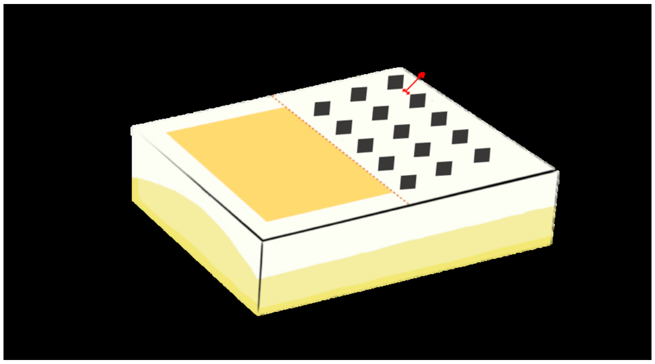

Figure 2. *Representative figure of the cavities at three standard distances from the treatment area (150, 300, and 450 µm, with a separation of 100 µm between indentations).*

the 5 indentations were calculated and compared with the baseline mean.

Energy Dispersive X-ray Spectroscopy Analysis

The amount of each enamel component was assessed by EDX, as described by Velo and others.[27] The blocks were mounted onto aluminum stubs with acrylic resin (Palavit M, Heraeus, Germany) without contaminating the treated enamel surfaces. Only specimens restored with glass-ionomer cement were protected in the restoration area by a base acid during analysis. All specimens were examined by scanning electron microscopy (Personal SEM EeXpress; Aspex Corporation, Delmont, PA, USA) at an accelerating voltage of 15–20 kV before and after the pH-cycling regimen *in vacuo*. Elemental analysis by EDX was conducted over the surface area of each block around restorations to determine the relative amounts of calcium (Ca), phosphorus (P), and F by the weight percentage. Parameters for sound enamel were the chemical formula of hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$, with a density of 3.021 g/cm^3.[28]

Statistical Analyses

Data were subjected to statistical analysis using the statistics program SPSS-17 (SPSS; IBM, Chicago, IL, USA). Normal distribution and equality of variances were checked for all the variables using the Kolmogorov–Smirnov test. SH was analyzed using two-way repeated-measures analysis of variance followed by Tukey's test. For EDX data, the amounts of Ca, P, and F were compared between samples using paired *t*-tests. The level of statistical significance was set at $p<0.05$.

RESULTS

Surface Hardness Measurements

Table 2 presents all the mean values and corresponding standard deviation values for SH (KHN). All groups had essentially the same initial SH ($p>0.05$), implying all the blocks had a uniform hardness to conduct the treatments. For all treatment groups, the pH-cycling regimen caused demineralization of the enamel proximal to the restorative material. The glass-ionomer cements F IX, IZ, and F II had the highest postcycling values for hardness at all 3 distances evaluated (150, 300, and 450 µm), and these values were significantly different from those of the other groups ($p<0.001$). The Z250 composite resin had the lowest SH value, which did not differ significantly from that of the NT group at all distances evaluated ($p>0.05$).

The S-PRG filler F-releasing restorative material B II group (giomer system) yielded a postcycle SH value that was intermediary among the other glass-ionomer cements we evaluated, and it was significantly higher than the SH values for the Z250 resin and NT groups at the 3 distances evaluated ($p<0.05$). For all groups, the SH value decreased as the distance from the restoration increased (Table 2).

Table 2: *Mean ± Standard Deviation of the Initial and Final Surface Hardness (SH, KHN) at Three Distances from the Treatment Area (150, 300, and 450 µm) That Were Evaluated for All Groups*[a]

Groups	Initial SH (Kg/mm^2)	SH (Kg/mm^2) (final 150 µm)	SH (Kg/mm^2) (final 300 µm)	SH (Kg/mm^2) (final 450 µm)
F IX	345.53+36.74 Aa	287.40+57.78 Ba	267.07+58.29B Ca	242.93+63.32 Ca
I Z	346.00+35.59 Aa	284.00+39.08 Ba	261.53+47.93B Ca	244.80+46.22 Ca
F II	352.93+31.04 Aa	309.13+29.67 Ba	267.80+23.02 Ca	231.73+35.85 Da
B II	345.93+33.70 Aa	216.87+45.63 Bb	175.67+31.41 Cb	151.73+32.32 Cb
F 250	379.60+24.55 Aa	122.67+22.21 Bc	89.67+11.69 Cc	86.67+20.28 Cc
NT	374.80+32.23 Aa	88.13+6.48 Bc	81.80+6.28 Bc	77.07+9.31 Bc

Abbreviation: SH, surface hardness.

[a] *Different capital letters in the same row indicate statistically significant differences (p<0.05; repeated measures analysis of variance and Tukey´s tests); different lower case letters in same column indicate statistically significant differences (p<0.05; analysis of variance and Tukey´s tests).*

Energy Dispersive X-ray Spectroscopy

Table 3 presents the atomic percentages of Ca, P, F, and the Ca/P weight ratio on the enamel substrates that were determined by EDX. The postcycling P content did not differ significantly among the groups ($p>0.05$). After pH cycling, the content of Ca, P, and F did not differ among the groups (including NT), with the exception of the B II group, which had a significantly lower amount of Ca ($p=0.003$) but a significantly higher amount of F ($p=0.003$) in the enamel immediately adjacent to the restoration.

DISCUSSION

Hybrid composite restorative materials based on S-PRG fillers have been touted in the dental materials field to provide biofunctionality to restorative materials. In the current study, we evaluated the potential of restorations with an S-PRG filler F-released composite to inhibit enamel demineralization against glass-ionomer cements and conventional composite resins to prevent caries developing adjacent to restorations, simulating real clinical conditions. In addition, we evaluated the elemental inorganic content of enamel around restorations before and after treatments. The null hypothesis that the S-PRG restorative materials would not have the potential to prevent demineralization was rejected, as demonstrated by the SH and EDX data.

In this study, the pH-cycling regimen was used according to Vieira and others[26] to simulate the demineralizing and remineralizing episodes that occur in the oral cavity and create caries-like lesions similar to those occurring *in vivo*.[24] Dental caries is a biofilm-sugar dependent disease[29] and, therefore, caries lesions will develop on intact or restored dental surfaces on which a biofilm forms. The acidic pH environment produced from the fermentation of dietary sugars promotes the dissolution of the underlying dental minerals.[30] This is the first step in the demineralization process, when acid reaches the site on a crystal surface and Ca/P are dissolved into the surrounding aqueous phase between the crystals.[31] Thus, pH is a driving force that regulates the loss or gain of Ca and P from the mineral structure of teeth.[32] If F ions are present at the crystal surface during demineralization, these ions can adsorb onto the surface of the crystals and inhibit demineralization by acids.[31]

The difference between caries progression on tooth substrates adjacent to restorations and the sound tooth surface is the possibility of a biofilm accumulating at the interface. This problem associated with the shrinkage stress of restorative materials could boost secondary caries development.[33,34] Therefore, for restorative materials that release F, besides restoring function and esthetics, they can also control the development of caries adjacent to the filling,[32] as F can reduce demineralization and promote remineralization of dental hard tissues. Based on our EDX results, the S-PRG material (group B II) retained the largest amount of F ($p=0.003$) levels in the enamel adjacent to the restoration (Table 3). Naoum and others demonstrated that Beautifil II released more F than other resin-based

Table 3: *Element Content in Atomic Percentage (At%; mean ± SD) at the Initial Condition and After Undergoing the pH-cycling Regimen According to Different Groups*

Groups	Initial Ca	Final Ca	Initial P	Final P	Initial F	Final F	Ca/P Ratio	Ca/P Ratio
F IX	54.05 ±0.95	53.60 ±0.83	37.09 ±0.21	37.16 ±0.15	1.55 ±0.15	1.60 ±0.15	1.45 ±0.03	1.44 ±0.02
I Z	53.80 ±0.95	53.89 ±0.93	37.12 ±0.13	37.20 ±0.26	1.58 ±0.17	1.55 ±0.14	1.44 ±0.02	1.44 ±0.03
F II	54.01 ±0.77	53.29 ±1.04	37.11 ±0.15	37.2 ±0.32	1.52 ±0.13	1.62 ±0.14	1.45 ±0.02	1.43 ±0.03
B II	54.05 ±0.69[a]	53.13 ±0.75[a]	37.16 ±0.19	37.11 ±0.27	1.51 ±0.10*	1.67 ±0.12[a]	1.45 ±0.02[a]	1.43 ±0.02[a]
F 250	53.65 ±0.92	52.89 ±1.52	37.35 ±0.17	37.39 ±0.34	1.52 ±0.15	1.54 ±0.16	1.43 ±0.02	1.41 ±0.05
NT	53.69 ±1.02	53.82 ±1.51	37.37 ±0.25	37.41 ±0.26	1.55 ±0.15	1.52 ±0.24	1.43 ±0.03	1.43 ±0.04

Abbreviations: Ca, calcium; F, fluoride; P, phosphorous.
[a] *Statistically significant difference (p<0.05)*

materials.[35] In this study, which simulates real clinical challenges, the higher F levels presented are explained as follows: S-PRG fillers promote a rapid F release via ligand exchange between F and cations within the prereacted hydrogel.[36] This ability to release F implies that B II is the most capable of providing F to the surrounding tooth structure at times when the adjacent enamel is most susceptible to demineralization.[35]

On the other hand, the pH-cycling regimen significantly decreased the Ca level for B II, which confirms the dissolution of hydroxyapatite. Such results were unexpected because when F is present, the amount of mineral dissolved is reduced because a certain proportion of Ca and P ions are incorporated into enamel as fluorapatite, thus reducing demineralization.[32] Therefore, based on these results, we can state that although the S-PRG material is able to release F in the enamel adjacent to the restoration, the bioavailable F is not enough to develop a calcium fluoride (CaF_2)-like particles reservoir and increase mineral resistance to acid through the formation of fluorapatite (ie, remineralization process).[37]

Despite F-enhanced remineralization incorporated Ca and P ions into the surface, in low concentrations it only partially inhibited the net dissolution of enamel, while remineralization requires the presence of Ca and P and an F reservoir preventing the oral environment from becoming unsaturated.[38] In addition, F release in B II is accompanied by other ions, such as aluminum, which present a strong affinity to fluoride-forming Al-F complex and reduces the levels of bioavailable F ions.[39] This fact can interfere with the dynamic caries process since the presence of free ions is important to ensure F bioavailability. These results were confirmed by the SH analysis, because the B II group had a lower mean value for hardness than the glass-ionomer cements, for which the Ca concentration remained unchanged after pH cycling.

A previous study has shown that an S-PRG filler-containing tooth-coating material inhibited demineralization around the coating.[40] However, it used a static model (not a dynamic pH-cycling model) to induce demineralization, which may have resulted in overestimating the effect of the coating.[40] In the present study, we speculate that although the F release was not able to form a CaF_2-like particles reservoir, F ions might be bioavailable in the environment, but more studies are necessary to confirm this. In this study, the higher F release values occur by exposure to an acid pH (4.7), which enhances hydrolysis of the F component in the material.[41] However, although EDX analysis evaluated the atomic percentage composition of the blocks, it is recommended to ensure the bioavailable ions to determine the potential protective benefits of this class of material to tooth structure.

At the same time, the B II group presented lower values of hardness than glass-ionomer cements, which were able to maintain a constant Ca concentration after the pH-cycling regimen. The Ca/P weight ratio and Ca/P molar ratio determine the rate of hydroxyapatite mineralization, and it is important to evaluate them as the mechanical properties of the tooth substrate, as its rate of biodegradation strongly depends on it. This ratio was calculated for stoichiometric hydroxyapatite (HA; Ca/P weight ratio = 253/2.151) and varies accompanying tissue mineralization.[42] The lower values of Ca and the Ca/P ratio presented by B II confirm that despite F release being able to prevent demineralization, it is not enough to improve the remineralization process because of the uptake of lower levels of Ca ions. Therefore, F released from a material should not be the only factor that determines the potential protective benefits of different bioactive materials to the tooth structure.

Besides that, B II presents less controlled F-release than glass-ionomer cement, as the F glass within B II presents little or no glass-ionomer matrix phase due to the lack of a significant acid-base reaction. Glass-ionomer cements are also more porous, which may influence the amounts of F released. In addition, when compared with glass-ionomers, giomer composites have more resin contents added and the barrier through which water and F diffuse also increases.[43,44]

Glass-ionomer cements release F into the oral environment via 2 processes: (1) a short-term reaction involving a fast transfer of F to the oral environment, and (2) a gradual diffusion of F through the developing matrix, which undergoes a gradual increase in crosslink.[35,36] The amount of F released in this second process depends on the nature of the matrix formed.[45] In the present study, the release of F shown in the F IX, IZ, and F II groups (conventional and resin-modified glass-ionomer cements) probably occurred by the second process, and for this reason we did not observe an initial increase in the F level for these materials when evaluated by EDX (Table 3). In this same context F diffused through the cement, because the enamel around the F IX, IZ, and F II glass-ionomer cements had the highest values for hardness at the 3 distances evaluated (150, 300, and 450 μm), and these values differed significantly from those of the B II and Z250 groups ($p<0.001$; Table 2). The SH results for our glass ionomers agree with the results of Okada and others: we observed an uptake of Ca and P ions with a consequent increase in hardness.[46] The delayed release of F enhanced their potential to inhibit the recurrence of caries. The outcomes presented here reflect the ability of certain resins to reduce demineralization based on F availability as a consequence of the dissemination of F, since the continuous availability of F in the oral environment can slow demineralization.[32] This slow release of F presented by glass-ionomer cements has clinical implications, as F-released from an ionomer follows a continuous uptake process and increases F concentration in the oral environment.

Among the F-releasing materials we evaluated, the results showed that hardness values decrease with distance from the restorative material (300 and 450 μm), although the F IX, IZ, and F II groups maintained consistently high hardness values (Table 2) in comparison with the other groups. The loss of hardness in the NT group was important for validating the pH cycling used in our study;[24] it demonstrated that the bovine enamel had demineralized, providing the proposed demineralizing challenge. Our results show that EDX is an effective method for detecting minor alterations in Ca, P, and F mineral content. The glass-ionomer cements of the F IX, IZ, and F II groups yielded similar results, without significant differences in the percentages of Ca, P, and F between the initial and final conditions after the pH-cycling regimen. These results agree with previous findings.[37,47] For the Z250 and NT (negative control groups), there was no significant shift in Ca, P, or F content values after the pH-cycling experiment, as expected.

A limitation of the current study must be highlighted. Although the percentages of F did not differ between the initial and final conditions for the glass-ionomer cements, they are known to have better capability to act as an F reservoir than composite resin-based materials,[48] implying that over long-term continuous episodes of demineralization/remineralization, the amount of F released by the glass-ionomer cements differed from that of the other cements, as shown by the EDX results. Therefore, further studies are necessary to evaluate the effect of F release into the adjacent enamel in real time over a long period.

Within the limitations of this laboratory study, based in the current findings, the glass-ionomer–based materials we evaluated were able to release F at sufficient doses to slow the rate of demineralization. The bioactive PRG filler F-containing resin composite can be considered an effective option for restorations in patients at high risk of dental caries, especially in stress-bearing areas such as in posterior tooth restorations or when the aesthetic factor is essential, as this class of material has the potential to prevent new carious lesions developing around restorations. However, further long-term analysis and *in vivo* studies are required to determine the efficacy of these materials for controlling caries lesions.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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Departments

Errata

Operative Dentistry apologizes for the errors in the following manuscripts.

IF Leão, N Araújo, CK Scotti, RFL Mondelli, MM de Amoêdo Campos Velo, JFS Bombonatti; The Potential of a Bioactive, Pre-reacted, Glass-Ionomer Filler Resin Composite to Inhibit the Demineralization of Enamel *in Vitro*. *Oper Dent* 1 January 2021 **46** (**1**): E11-E20. doi: https://doi.org/10.2341/19-151-L

There are errors in the Author contact list. The correct author and author affiliations list should read (corrections are underlined):

IF Leão; NA Oliveira; CK Scotti; RFL Mondelli; MMAC Velo; JFS Bombonatti

Isabela Furlaneto Leão, MSc, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Naiara Araújo de Oliveira, MSc, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Cassiana Koch Scotti, MSc, PhD student, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

Rafael Francisco Lia Mondelli, DDS, MS, PhD, Full Professor, Department of Operative Dentistry, Endodontic and Dental Materials, Bauru School of Dentistry, University of São Paulo,São Paulo, Brazil

Marilia Mattar de Amoêdo Campos Velo, DDS, PhD, Postdoctoral researcher, Professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

*Juliana Fraga Soares Bombonatti, DDS, MS, PhD, Associate Professor, Department of Operative Dentistry, Endodontic and Dental Materials, Bauru School of Dentistry, University of São Paulo, São Paulo, Brazil

*Corresponding author: Alameda Octávio Pinheiro Brisolla 9-75, Bauru-SP-17012-901, Brazil; e-mail: julianafraga@usp.br

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DM De Paula, AD Loguercio, A Reis, S Sauro, AH Alves, PR Picanço, K Yoshihara, VP Feitosa; Lack of Neutralization of 10-MDP Primers by Zirconia May Affect the Degree of Conversion of Dual-cure Resin Cement. *Oper Dent* 1 January 2021 **46** (**1**) 107-115. doi: https://doi.org/10.2341/18-189-L

There are errors in the author affiliation list. The correct the author affiliation should read (corrections are underlined):

Diego M De Paula, DDS, MSc, Post-graduate program in dentistry, Department of Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil; Paulo Picanço School of Dentistry, Fortaleza, Brazil

Alessandro Loguercio, DDS, PhD, State University of Ponta Grossa, Ponta Grossa, Paraná, Brazil

Alessandra Reis, DDS, PhD, State University of Ponta Grossa, Ponta Grossa, Paraná, Brazil

Salvatore Sauro, DDS, University of CEU-Cardenal Herrera, Dental Biomaterials and Minimally Invasive Dentistry, Department of Dentistry, Faculty of Sciences and Health, Valencia, Spain; & Department of Therapeutic Dentistry, I.M. Sechenov First Moscow State Medical University, Moscow, Russia

Adyson H Alves, DDS, MSc student, Paulo Picanço School of Dentistry, Fortaleza, Brazil

Paulo R Picanço, DDS, MSc, PhD, Paulo Picanço School of Dentistry, Fortaleza, Brazil

Kumiko Yoshihara, National Institute of Advanced Industrial Science and Technology (AIST), Health and Medical Research Institute, Kagawa, Japan; & Okayama University, Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Department of Pathology & Experimental Medicine, Okayama, Japan

*Victor P Feitosa, DDS, MsC, PhD, professor, Post-graduate program in dentistry, Department of Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil; Paulo Picanço School of Dentistry, Fortaleza, Brazil

*Corresponding author: Department of Restorative Dentistry, Dental School - Federal University of Ceará, Rua Monsenhor Furtado S/N, Bairro Rodolfo Teofilo, 60430-325 Fortaleza, CE, Brazil; e-mail: victorpfeitosa@hotmail.com or victor.feitosa@facpp.edu.br

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F Ozer, O Irmak, O Yakymiv, A Mohammed, R Pande, N Saleh, M Blatz; Three-year Clinical Performance of Two Giomer Restorative Materials in Restorations. *Oper Dent* 1 January 2021 **46** (**1**) E60-E67. doi: https://doi.org/10.2341/17-353-C

There are errors in the corresponding author information. The corresponding author information should read (corrections are underlined):

*Ozgur Irmak, DDS, PhD, Department of Restorative Dentistry, Faculty of Dentistry, Yakın Doğu University, Lefkoşa, Turkey
*Corresponding author: Yakın Doğu Üniversitesi, Diş Hekimliği Fakültesi, Restoratif Diş Tedavisi Anabilim Dalı Kliniği, Yakın Doğu Bulvarı, 99138 Lefkoşa, KKTC Mersin-10, Turkey

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IBL Soares-Rusu, CA Villavicencio-Espinoza, NA de Oliveira, L Wang, HM Honório, JH Rubo, PAS Francisconi, AFS Borges; Clinical Evaluation of Lithium Disilicate Veneers Manufactured by CAD/CAM Compared with Heat-pressed Methods: Randomized Controlled Clinical Trial. *Oper Dent* 1 January 2021 **46** (**1**) 4-14. doi: https://doi.org/10.2341/19-233-C

There are errors in the corresponding author information and in paragraph 1 under Treatment Planning and Tooth Preparation in the Methods and Materials section (p6), and in the legend for Figure 10 (p11). They should read (corrections are underlined):

Idiane Bianca Lima Soares-Rusu, DDS, MSc, PhD, Bauru School of Dentistry, University of São Paulo, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, São Paulo, Brazil

Carlos Andres Villavicencio-Espinoza, DDS, MSc, PhD, Bauru School of Dentistry, University of São Paulo, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, São Paulo, Brazil

Naiara Araújo de Oliveira, DDS, MSc, PhD, Bauru School of Dentistry, University of São Paulo, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, São Paulo, Brazil

Linda Wang, DDS, MSc, PhD, professor, Bauru School of Dentistry, University of São Paulo, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, São Paulo, Brazil

Heitor Marques Honório, DDS, MSc, PhD, associate professor, Bauru School of Dentistry, University of São Paulo, Pediatric Dentistry, Orthodontics and Public Health, Bauru, São Paulo, Brazil

Jose Henrique Rubo, DDS, MSc, PhD, professor, Bauru School of Dentistry, University of São Paulo, Department of Periodontics and Prosthesis, Bauru, São Paulo, Brazil

Paulo Afonso Silveira Francisconi, DDS, MSc, PhD, professor, University of São Paulo, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, São Paulo, Brazil

*Ana Flávia S Borges, DDS, MSc, PhD, associate professor, University of São Paulo, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, São Paulo, Brazil

Digital smile design was performed using Apple's Keynote Software to obtain veneer proportions and to enable communication and increase predictability. Standardized extraoral photographs were taken with a Nikon D5300 digital reflex camera using the following parameters: manual mode 1/125, f22, and ISO 125, coupled with Sigma Macro 105mm DX lens; twin manual 1/1 flash (Nikon R1C1 Wireless Close-UP Speedlight System) equipped with Watson CR123A Rechargeable Lithium Battery (3V, 400mAh); and a flash holder (Agnòs, Italy). The analysis of the face thirds was made according to the participant's smile. Initial impressions were taken using a heavy and light condensation silicone (Xantopren/Optosil, Heraeus Kulzer, Germany) onestep technique. The study casts were obtained, and the wax-up was performed according to digital planning.

Figure 10. *Means and standard deviation of the manufacturing process at the study periods (baseline, 6 months, and 12 months of follow-up) for the topics marginal adaptation (MARA), marginal discoloration (MARD), restoration fracture (RESF), and postoperative sensitivity (POSTS).*