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EVALUATING THE EFFICACY OF RESISTANCE TO ENAMEL DEMINERALIZATION OF A LOW-SHRINKAGE SURFACE PRE-REACTED GLASS FILLED RESTORATIVE MATERIAL VERSUS CONVENTIONAL RESIN COMPOSITE: AN IN VITRO STUDY

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ABSTRACT

Aim of the study: To evaluate the efficacy of Beautifil-II LS giomer restorative material to inhibit enamel demineralization, as compared to conventional resin composite and glass ionomer restorative materials.

Materials and methods: Nine premolar teeth were randomly divided into three groups (n=3): group A (Beautifil-II LS), group B (3M Filtek Z-250), and group C (GI FX-Ultra), Nine premolar teeth were restored with each material, subjected to pH-cycling, and analyzed via environmental scanning electron microscope coupled with energy dispersive X-ray (ESEM-EDX) analysis. The calcium to phosphate ratio (Ca/P) of the enamel surface was calculated according to the atom% of elements in EDX analysis in each stage.

Results: According to the Ca/P recovery ratio, group A (Beautifil-II LS) showed higher results as compared to other tested restorative materials.

Conclusion: Beautifil-II LS might represent a promising restorative material to decrease the incidence of recurrent caries around existing restorations.

KEYWORDS: Giomer, Glass ionomer, Enamel demineralization, Secondary caries, Fluoride.

INTRODUCTION

Recurrent caries is considered one of the clinical issues that face the dental practitioners and may risk the durability of the existing restoration. Therefore, the marginal integrity and physical properties of the restorative material are important parameters to avoid microleakage and achieve durable restorations [1,2].

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Dental researchers have focused on the development of bioactive restorative materials that are characterized by their smart behavior in response to surrounding stimuli (e.g. pH) and their anti-cariogenic effect. Thereby, fluoride (F)-releasing materials have attracted the attention of researchers and practitioners because F has an anti-cariogenic effect and has the ability for reversal of demineralization process and enhancement of remineralization by replacing hydroxyl groups of the hydroxyapatite (HAP) crystals, forming fluorapatite, which results in a hard dental tissue with less enamel solubility [3–6].

It is well-known that glass ionomer cements (GICs) are characterized by their F^- ion release and recharge ability in response to changes in pH, showing the highest levels of F ion release among various F-releasing restorative materials. Besides this important property, GICs are biocompatible materials and can bond chemically to the tooth structure. Hence, GICs can be successfully used as a filling material in pediatric dentistry, as a base or lining material and as atraumatic restorative treatment (ART) material [7,8].

Another category of F-releasing materials is giomer which has similar composition of resin composites with pre-reacted glass (PRG) fillers in its structure. These fillers provide giomer restorations with the property of F ion release and recharge in addition to other beneficial ions such as strontium (Sr^{2+}), silicate (SiO_3^{2-}), etc. [9,10].

Beautifil-II Low-shrinkage (Beautifil-II LS) resin-based restorative material exhibits low polymerization shrinkage as claimed by the manufacturer which helps to reduce marginal leakage and secondary caries. In addition, the bioactive surface pre-reacted glass (S-PRG) fillers incorporated in Beautifil-II LS offer an acid-neutralizing capacity and provide a F-rich environment [10–12].

Thus, the aim of this research was to evaluate the efficacy of low-shrinkage surface pre-reacted glass filled resin-based restorative material (Beautifil-II LS, Shofu Inc., Japan) on the resistance to enamel demineralization as compared to 3M Filtek Z-250 resin composite and GI FX-ultra.

MATERIALS AND METHODS:

Restorative materials:

1. Group A: Beautifil-II LS resin composite (giomer, SHOFU Inc., Kyoto, Japan).
2. Group B: Filtek Z-250 resin composite (micro hybrid composite, 3M ESPE, USA).
3. Group C: GI FX ultra (conventional glass ionomer, SHOFU Inc., Kyoto, Japan).

Selection of teeth and sample preparation:

A total of 9 prepared premolars extracted for orthodontic purposes, were randomly divided into three groups (n=3). The extracted teeth were cleaned using hand scaler (413/414 universal curette, Hu-Friedy, USA) to remove all calculus and deposits. The teeth were then cleaned with F free paste (NADA™, Preventech, USA) and low speed handpiece (W&H Alegra, WE-56T, Austria).

The teeth were sectioned into two halves in a mesio-distal direction using a diamond-coated band saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) fixed into straight hand piece (W&H Alegra, HE-43T, Austria) under continuous water spray into two halves. All samples were stored in deionized water in sequential numbered vials [13].

Preparation of the artificial cavities:

An artificial Class V cavity was prepared in the center of the enamel of the sectioned samples with the following dimensions: 3 mm (mesiodistal), 2 mm (occlusogingival) and 1.5 mm depth with the preparation extending 1 mm above the cementoenamel junction). The bur was marked after 1.5 mm with nail varnish and the cavity was measured with a periodontal probe to standardize the dimensions of the cavity.

Placement of restorative material:

In groups A and B, selective acid etching using 37% phosphoric acid (Meta Biomed, Korea) was applied on the boundaries of the cavities on teeth samples for 15 seconds, according to the manufacturers' instructions, then rinsed, and air dried gently for two seconds to remove the excess water.

The bonding agent (BISCO All-Bond Universal, Bisco, USA) was applied using a micro brush then air dried for two seconds and light cured for 20 seconds, according to manufacturers' instructions, using LED F light curing unit (Woodpecker, China).

The artificial cavities in the extracted teeth were then ready to be filled with the designated restorative material (group A: Beautifil-II LS and group B: 3M Filtek Z-250); The restorative material was applied using a composite applicator, finished using fine needle stone (010, Diaswiss, Nyon, Swiss) and polished using composite polishing discs (Super-Snap X-treme, Shofu, Japan) to obtain the final finish.

While for group C, the capsules were placed into amalgamator (FOMOS Amalgamator I MIX, China) and mixed for 10 seconds, the capsule content of mixed GIC was extruded into the cavity with a capsule applicator gun (Generic, China), adapted into the cavity with an applicator, finished using fine needle stone and left for 2.5 mins to set according to manufacturers' instructions [14].

pH-cycling:

The prepared teeth samples were subjected to pH-cycling to simulate the cariogenic challenge (demineralization and remineralization). The samples were immersed individually in 50 ml of the remineralizing solution (0.4g NaCl, 0.4g KCl, 0.6g CaCl2, 0.6g NaH_2PO_4, 4g Urea, 4g Mucin, 0.0016g Na_2S, 0.0016g $Mg_2P_2O_7$, at pH 7.2) for 10 minutes, then in 50 ml of the demineralizing solution (2.2mM Calcium chloride ($CaCl_2$), 2.2mM Potassium phosphate (KH_2PO_4), 0.05M Acetic acid, and 1M Potassium hydroxide (KOH), at pH 4.6) for 30 minutes and finally in 50 ml of the buffered solution (20 mM HEPES, 2.25 mM $CaCl_2$2H2O, 1.35 mM KH_2PO_4; 130 mm KCl, at pH 7.0) for 10 minutes. These cycles were performed three times a day for 28 days [15–17].

The prepared teeth samples were washed by deionized water between each solution during the pH-cycling. Between the daily cycling processes, the samples were stored in 50 ml buffered solution overnight at 37°C using a constant temperature incubator (BTC, Egypt) [15–17].

Surface morphology evaluation and elemental analysis:

After 28 days, the samples were removed from the solution, washed with deionized water, dried with a cotton gauze and the teeth-restorations interfaces were analyzed using high resolution environmental scanning electron microscope coupled with energy dispersive X-ray (ESEM-EDX) analysis (FEI Quanta FEG 250 instrument, FEI Company, Eindhoven, Netherlands) operated at 20 KV under magnification 1000X. EDX was used in conjunction with ESEM analysis to provide surface elemental identification and quantitative compositional information. Samples were imaged without any coating. [15–17].

The average Ca/P ratio was calculated according to the atom% of Ca and P elements obtained from the EDX analysis. Also, comparing the Ca/P ratio of different groups was performed, where the percentage of change (Ca/P recovery) was calculated for each group by dividing the values of Ca/P ratio after pH-cycling and the values of Ca/P ratio in the baseline phase.

RESULTS

SE micrographs

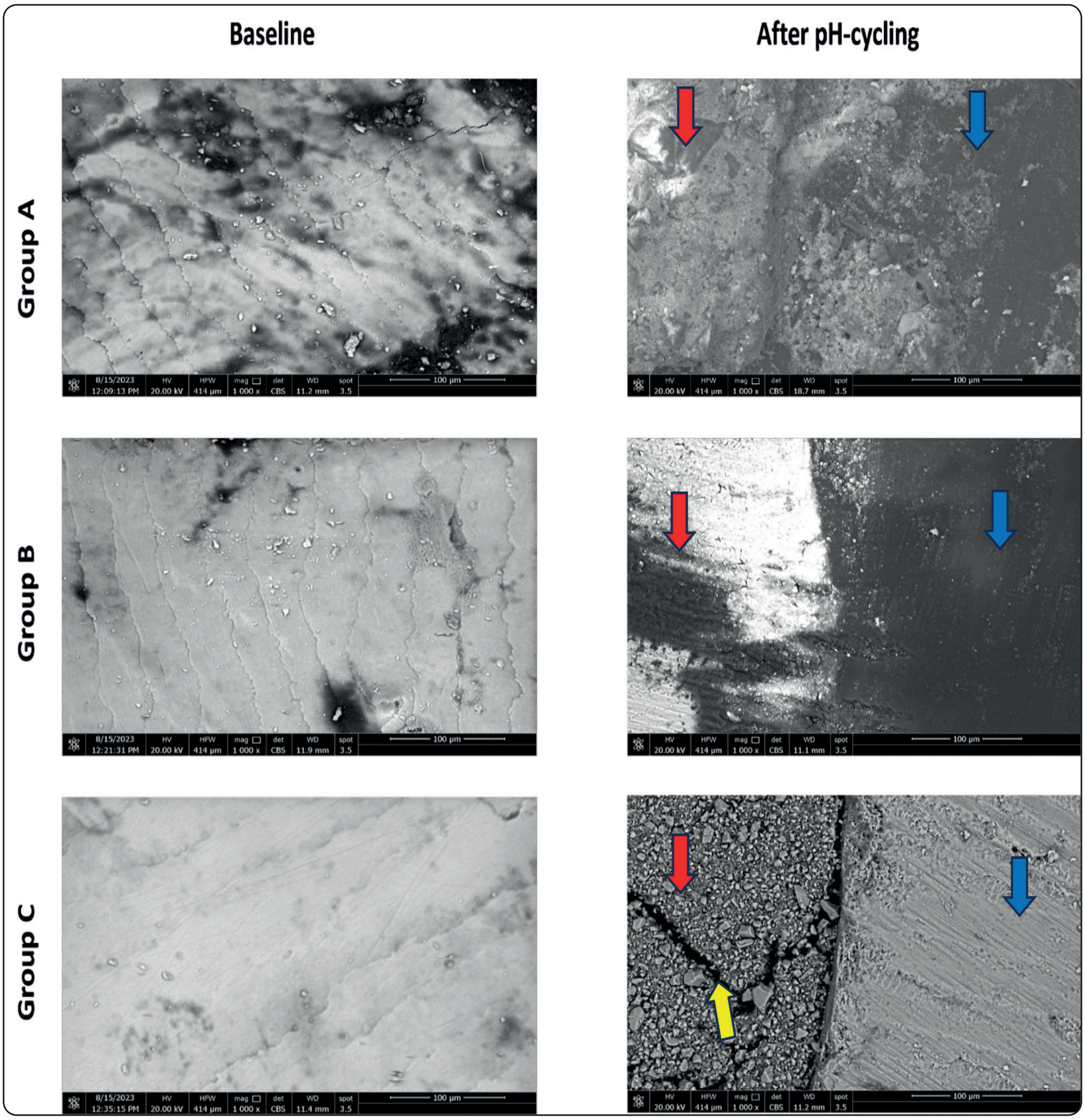

Fig. (1) The ESEM micrographs of the enamel surface (group A, B, and C) under magnification 1000 X at different stages: baseline (before restoration) and after restoration followed by pH-cycling for 28 days. Red arrows indicate the restoration, blue arrows indicate the enamel surface beside the restoration, and yellow arrow indicates microcrack within the glass ionomer restoration

EDX elemental analysis:

Group A (Beautifil-II LS):

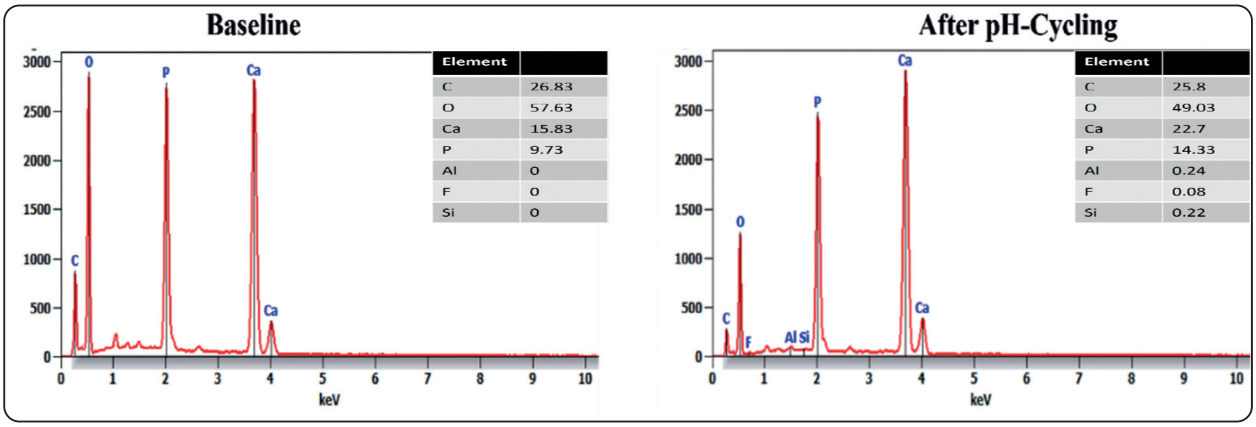

Fig. (2) The EDX spectrum and the average elemental analysis of the enamel surface of group A at different stages; Baseline and after restoration with Beautifil-II LS followed by pH-cycling for 28 days.

Group B (3M Filtek Z-250):

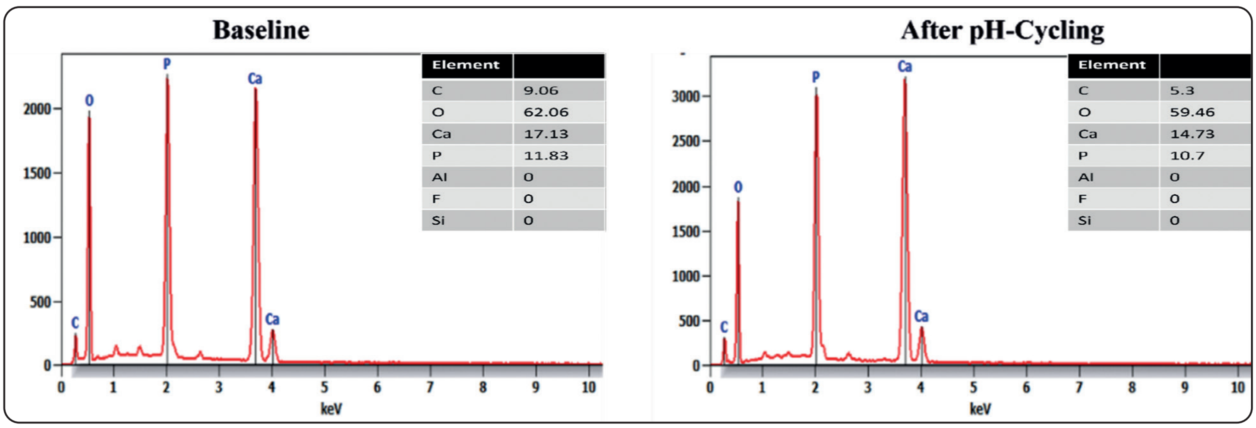

Fig. (3)The EDX spectrum and the average elemental analysis of the enamel surface of group B at different stages; Baseline and after restoration with 3M Filtek Z-250 followed by pH-cycling for 28 days.

Group C (GI FX-Ultra):

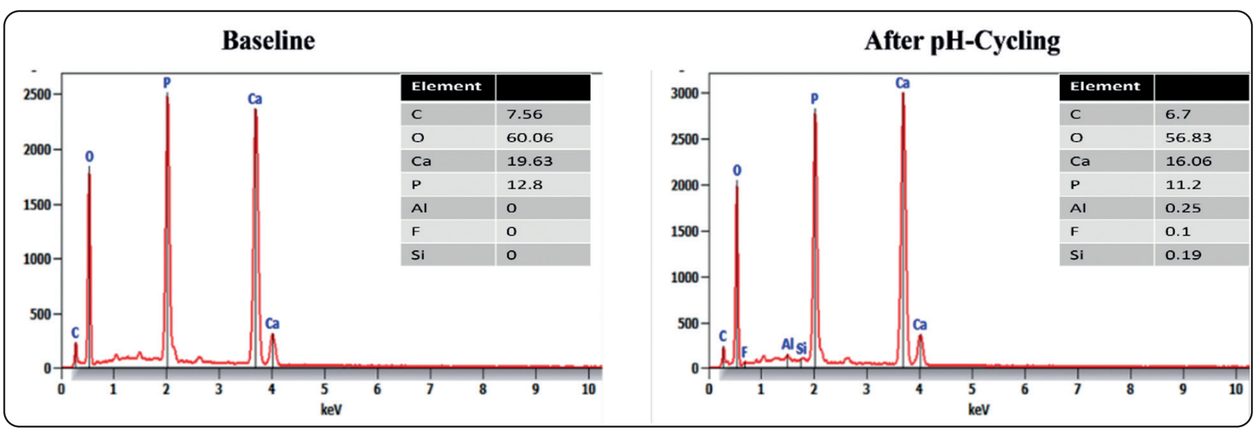

Fig. (4) The EDX spectrum and the average elemental analysis of the enamel surface of group C at different stages; Baseline and after restoration with GI FX-ultra followed by pH-cycling for 28 days.

Ca/P ratio after pH-cycling:

The average Ca/P molar ratio of the enamel surface, as shown in table 1, was calculated according to the atom% of elements in EDX analysis in each stage (Baseline and after pH-cycling for 28 days) at three distances: At the tooth-restoration interface, 0.5 mm and 1 mm from the tooth-restoration interface.

TABLE (1) The average Ca/P molar ratio of different groups in each stage at three distances on the enamel surface.

Stage	Group A (Beautifil-II LS)			Group B (3M Filtek Z-250)			Group C (GI FX-ultra)		
	At interface	0.5 mm	1 mm	At interface	0.5 mm	1 mm	At interface	0.5 mm	1 mm
Baseline	1.61	1.6	1.65	1.45	1.44	1.43	1.52	1.56	1.51
After pH-cycling	1.56	1.65	1.52	1.23	1.33	1.25	1.43	1.44	1.42
Percentage of change in Ca/P ratio	96.8%	103%	92.1%	84.8%	92.3%	87.4%	94%	92.3%	94%

DISCUSSION

The current study was performed to evaluate the efficacy of low-shrinkage Beautifil-II giomer to resist further enamel demineralization. The resistance to enamel demineralization was evaluated through surface morphological evaluation and elemental analysis of the enamel surface after pH-cycling. A pH-cycling regimen was selected in the current test to induce demineralization-remineralization processes simulating those occurring in the oral cavity and to evaluate the potential of the tested restorative material in the prevention of secondary carious lesions [16–18].

Surface morphological evaluation and elemental analysis were performed through ESEM coupled with EDX analysis. EDX analysis is a micro-analytical technique at the ultra-structural level used to identify the elemental composition of materials and to evaluate the enamel remineralization capacity of the tested restorative materials after demineralization through artificial caries development in the tested teeth samples. [19].

The results of the Ca/P ratio of the enamel surface (table 1), after pH-cycling according to EDX analysis, showed different remineralization levels between the tested groups. This might be attributed to the deposition of crystals into the demineralized enamel due to the potential of artificial saliva to deposit minerals. In addition to the ability of group A and group C to release beneficial ions (e.g., F, Si, Ca, and Sr) which could play an important role in the process of remineralization. This might also explain the appearance of new F peaks in the EDX analysis of groups A and C (Figs. 2 and 4). This explanation comes in accordance with the studies of Fujimoto et al. and Nicholson et al. [12,20].

The ESE micrograph of group C (Fig. 1) revealed the appearance of microcracks within the restoration and a slight interfacial gap between the restoration and the tooth after pH-cycling. This might be due to the erosive effect of dynamic pH-cycling which might result in the matrix dissolution peripheral to glass particles and the dissolution of the siliceous hydrogel. This was in agreement with the studies of Honorio et al and Culina et al [21,22]. Another possible explanation is that the setting contraction of the GIC restorative material during initial time of setting might not be compensated by water absorption during the limited time of the study which might affect the bonding of GIC with the tooth structure and lead to the development of marginal gap. Such findings were also in accordance with the studies of Alsari et al. and Hamed et al. [23,24].

The percentage of change in Ca/P ratio of group A was higher as compared to other groups, which could give an indication about the potential of the Beautifil-II LS restorative material to resist the development of further secondary caries around the old restoration. This could be attributed to the availability of beneficial ions, particularly F ions, released from the restoration, in the enamel sites adjacent to the existing filling which are more susceptible to demineralization. This was in accordance with the findings of previous researchers [6,10,17].

Although Beautifil-II LS restorative material was able to release beneficial ions, especially F, in the susceptible sites for demineralization, the available F ions might not be sufficient at some sites of the prepared tooth to produce a reservoir of CaF particles and promote the resistance to secondary caries. The F ion release from Beautifil-II LS is also associated with Al ion release which has a strong affinity to F which could lead to the formation of Al-F complexes and could decrease the bioavailability of F ions, this might decrease the potential of F to enhance the remineralization process because this dynamic process requires the presence of free ions which act as a F reservoir [17].

These findings (table 1) are consistent with the study of Leão IF et al. who concluded that the potential effects of F-releasing materials could not be only evaluated through their ability to release F ions. In addition, the remineralization process necessitates the availability of Ca, P and a reservoir of F preventing the supersaturation of the oral environment [17].

The F-releasing restorative materials could provide a reservoir of F to ensure the bioavailability of F ions around the existing restorations, which could inhibit the incidence of recurrence of carious lesions. The potential of the F-releasing materials to release and recharge of F ions depends on the chemical composition of the material and the frequency of F exposure from the surrounding environment [12,17,25].

Beautifil-II resin-based restorative material is a second-generation giomer which has incorporated S-PRG fillers that are found to be a reservoir for F that can release and recharge F ions in addition to other beneficial ions such as Si, Al, and Sr. These ions could offer a solution for prevention of secondary caries through counteracting the demineralization process and favoring the remineralization process [10,26].

The S-PRG filler modulates the oral pH of the surrounding medium and shifts it toward neutral and weak alkaline values. Additionally, the release of F and Sr from S-PRG filler strengthens the tooth substrate through the formation of fluorapatite which is much harder and more resistant to acid attacks comparable to HAP. These effects could be important in the prevention of further carious lesions [10].

The F ions released from Beautifil-II LS might be attributed to the ability of incorporated S-PRG fillers to release F ions through continual dissolution of the F-containing glass core and this is facilitated by the acidified water within the hydrogel surrounding the inner glass of S-PRG particles. Additionally, the hydrogel of S-PRG particles exhibits a higher permeability and porosity than conventional resin matrices. This hydrogel enhances the ability of Beautifil-II for F uptake through areas within its porous hydrogel structure [6].

CONCLUSION

Within the limitations of the current study, it can be concluded that:

Beautifil-II LS may be able to decrease the incidence of recurrent caries around existing restorations, further *in vivo* investigations are suggested to confirm the findings of the study.

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Conflict of interest

The authors declare no conflict of interest.

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