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Fluoride bioactive glass paste improves bond durability and remineralizes tooth structure prior to adhesive restoration

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ABSTRACT

Objective. The current study aimed at examining a fluoride containing bioactive glass (BiominF®) paste as a temporary filling material capable of remineralizing the demineralized enamel or dentin, and its ability to decrease a simulated dentinal fluids pressure on the resin/dentin interface, without affecting the shear bond strength of a universal bonding agent to enamel and dentin.

Methods. 60 premolars were utilized for the acid resistance, trans-microradiography (TMR) and shear bond strength (SBS) experiments. Enamel and dentin discs were demineralized for 4 days to create a subsurface demineralized zone followed by applying BiominF® paste, 1.23% acidulated phosphate fluoride, or a temporary filling material for 24 h.

30 extracted human non-carious third molars were utilized for the pulpal pressure experiment in which direct communication to the pulp chamber was created by cutting at a level approximately 1 mm below the cemento-enamel junction while the coronal enamel was ground to expose mid coronal dentin. The dentin surface was exposed to a simulated pulpal pressure. The dentin surfaces had BiominF® paste, an oxalate desensitizing agent, or temporary filling material followed by application of a universal adhesive system.

Results. One way ANOVA showed that BiominF® paste remineralized effectively the demineralized enamel or dentin, did not affect the bond strength of the enamel and dentin surfaces to the tested adhesive system $p < 0.05$, and improved the acid resistance of the demineralized enamel and dentin against a secondary erosive challenge. Moreover, BiominF® paste decreased the nanoleakage expression in the dentin/adhesive interface exposed to a simulated pulpal pressure.

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Significance. BiominF® paste may serve as a temporary filling material that may improve the longevity of adhesive restorations and help to conserve tooth structures by preserving the demineralized enamel and dentin form cutting during cavity preparation.

1. Introduction

Modern adhesive dentistry provided patients with many optimal solutions for treating their carious teeth however, the durability of adhesive restorations and increasing their longevity inside patients' oral cavities are among the main aims of development in dental field [1].

One of the limitations of adhesive resins is its questionable ability to be bonded to demineralized enamel and dentin [2]. Although, many adhesive systems nowadays involve the phosphoric acid monomer 10-MDP to provide protection to the tooth cavities' enamel and dentin cavosurface margins [3–5], previous research indicated that 10-MDP monomer has limited protective effect on phosphoric acid etched dentin [6]. Moreover, the prevalence of Molar Incisor Hypomineralization (MIH) recently increased reaching 4–20% incidence [7]. This increases the demands of using effective remineralization strategies capable of restoring the teeth calcium and phosphate levels to their original levels within a short duration of time prior to bonding these hypomineralized enamel surfaces to adhesive restorative materials [8].

Other challenges faced by adhesive restorations is its limited ability to remineralize the enamel and dentin walls due to the resinous matrix of these materials that limit the transfer of any inorganic ions from the restorative material to the bonded enamel [9] or dentin walls. Moreover, the nanoleakage expression at the dentin resin interface is an indication for decreased durability of resin-dentin durability and possible short term degradation of the hybrid layer [10] especially after etching the dentin surface exposed to pulpal pressure [11]. The use of bioactive glasses for the remineralization of enamel and dentin surfaces through forming a calcium phosphate rich layer showed promising results during the past ten years [11–21].

This study tested the potential of using a fluoride bioactive glass (FBG) as a temporary filling material prior to bonding procedures. The null hypotheses in the current study are that FBG paste will not (a) Remineralize the demineralized enamel or dentin. (b) Improve the acid resistant of enamel and dentin resin interface at the cavosurface margin. (c) Decrease the nanoleakage expression due to pulpal pressure at the resin-dentin interface.

2. Materials and methods

The experimental procedures and materials tested are summarized in Fig. 1, and Table 1. 120 teeth were utilized in the current experiment.

2.1. TMR (trans-microradiograph), acid resistance, and shear bond strength specimens' preparation

90 premolars indicated for extraction due to orthodontic reasons were obtained according to the guidelines of the university. The extracted sound premolar teeth were collected from the oral surgery department after obtaining the permission of the ethical committee of the faculty of Dentistry under registration number (114-10-19). The teeth were hand scaled from any calculus or soft tissues. The teeth were stored in 0.1% thymol till the start of the experiment according to the guidelines approved by the University and in accordance with the principles of the Declaration of Helsinki and its later amendments or comparable ethical standards. The number of specimens assigned to each group was adopted according to previous literature and according to 80% power of test. Randomization of the specimens was done using a computer program (Excel 2007, Microsoft, Redmond, WA, USA). All teeth were examined by light microscope to exclude any teeth having cracks, restorations, demineralization or any defects. Intra and inter examiners calibrations were conducted before actual recording of the obtained results.

The premolars were sectioned vertically where the buccal sections were slightly ground to obtain 90 flat enamel specimens, while the lingual sections were ground to expose 90 mid-coronal dentin specimens. All specimens were embedded in acrylic blocks and were challenged with buffered demineralization solution (2.2 mM $CaCl_2$, 10 mM NaH_2PO_4, 50 mM acetic acid, 100 mM NaCl, 1 ppm NaF, 0.02% NaN_3; pH 4.5), [13]. The demineralized enamel and dentin disc specimens were assigned into 3 groups according to the treatment method into: (Temporary) group had a temporary filling material (Cavit; 3M ESPE, St. Paul, MN, USA) applied onto its enamel surfaces for 24 h, (Fluoride 24 h) group had a fluoride gel (Ionite Acidulated Fluoride Gel, 1.23%, Dharma research, Miami, USA) applied followed by placing a temporary filling material (Cavit) for 24 h; and (Biomin) group specimens had Fluoride bioactive glass (BioMinF®, BioMin Technologies Limited, Queen Mary University of London, Mile End, London) applied on its surface. After 24 h of storage in distilled water all remnants of temporary filling material and fluoride bioactive glass (FBG) were removed and washed by deionized water [18–20].

2.2. FBG (fluoride bioactive glass) application

One tenth of a gram of FBG powder composed of (22–24 mol % Na_2O, 28–30 mol % CaO, 4–6 mol % P_2O_5, 36–40 mol % SiO_2, and 1.5–3.0 mol % CaF_2) was mixed on a glass slab with 2 drops of 50 wt% phosphoric acid, which was prepared by the diluting 85 wt% phosphoric acid (Wako, Osaka, Japan). The resulting paste had a pH 2.5. The FBG paste was applied onto the enamel

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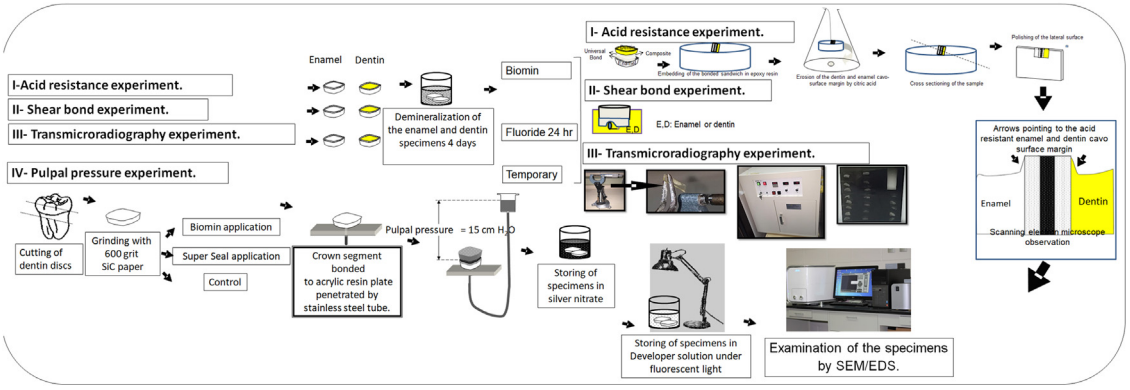

Fig. 1 – Summary of the experimental procedures.

Table 1 – Materials used in this study.

Materials	Composition	Procedures
BioMinF®	36–40 mol % SiO2, 22–24 mol % Na2O, 28–30 mol % CaO, 4–6 mol % P2O5 1.5–3.0 mol % CaF2	Mix 0.1 g of 45S5 Biomin F to 0.2 ml of phosphoric acid
Clearfil universal bond quick (Kuraray,Osaka,Japan)	HEMA, (BIS-GMA), (MDP), hydrophilic amide monomers, colloidal silica, silane, sodium fluoride, CQ in ethanol and water	Apply adhesive system light cure (10 s)
Cavit (3M ESPE, St. Paul, MN, USA)	Zinc Oxide (30–50%) Calcium Sulfate (1–30%) Barium Sulfate (0–20%) Ethylene Bis (Oxyethylene) Diacetate (10–20%) Talc (0–20%) Zinc Sulfate (5–10%) Poly (Vinyl Acetate) (1–5%)	
Tetric N-Ceram (IvoclarVivadent, Schaan, Liechtenstein)	Bis-GMA, Bis-EMA, TEGDMA Barium glass, ytterbium difluoride 56	Light cure 30 s
Super Seal (Phoenix Dental, Fenton, MI, USA.)	Oxalic acid, Potassium salt, water	Apply directly

HEMA = 2-Hydroxyethyl methacrylate; (BIS-GMA) bisphenol A diglycidylmethacrylate, MDP = 10-methacryloxydecyl. TEGDMA (Triethylene glycol dimethacrylate). CQ: Camphor quinone.

or dentin surfaces of the (FBG group) by microbrush. The FBG paste was covered temporarily by a thin layer of adhesive resin (Clearfil Universal Bond Quick). After 24 h the adhesive layer together with remnants of the FBG paste group were washed by an air water jet followed by cleaning the surface by a brush installed on a low speed hand-piece [18–20].

2.3. *Trans-microradiograph (TMR) examination*

30 enamel and 30 dentin specimens that were treated by the aforementioned methods were embedded in resin material to maintain the stability of the enamel and dentin specimens that were cross sectioned using a low-speed diamond saw (Isomet 5000; Buehler, Lake Bluff, Illinois, USA). The specimens n = 10 were then manually thinned to obtain enamel and dentin slabs having a maximum thickness of 100 μm for the enamel specimens and 150 μm for the dentin specimens [14,22,23]. All specimens were exposed to high dosage of X-ray generated from an X-ray generator (CMR 2; Softex, Tokyo, Japan) at 25 kV voltage and 4 mA. The distance between the X-ray tube and the specimen was 15 cm. The TMR images, together with 15 aluminum step wedges (each 15 μm in thickness), were captured on an X-ray glass plate film (High Precision Photo Plate PXHW, Konica Minolta Photo, Tokyo, Japan).

The specimens were mounted on X-ray glass plate sensitive films (High Precision Photo Plate PXHW; Konica Minolta Photo, Tokyo, Japan) with a 15-step Aluminum step-wedge and a Ni filter in a dark room and were exposed to high dosage of X-rays produced by an X-ray generator (CMR-2, SOFTEX, Kanagawa, Japan) at 20 kV, 2.5 mA for 10 min. The TMR glass films were processed using standard developer and fixer solutions (Anatomix RTU Developer- Replenisher, Fuji, Tokyo, Japan). Digital images were obtained for the tested samples from the glass films using a digital camera attached to a microscope (ML 8500, Meiji, Techno, Japan). The images were analyzed by two computer applications

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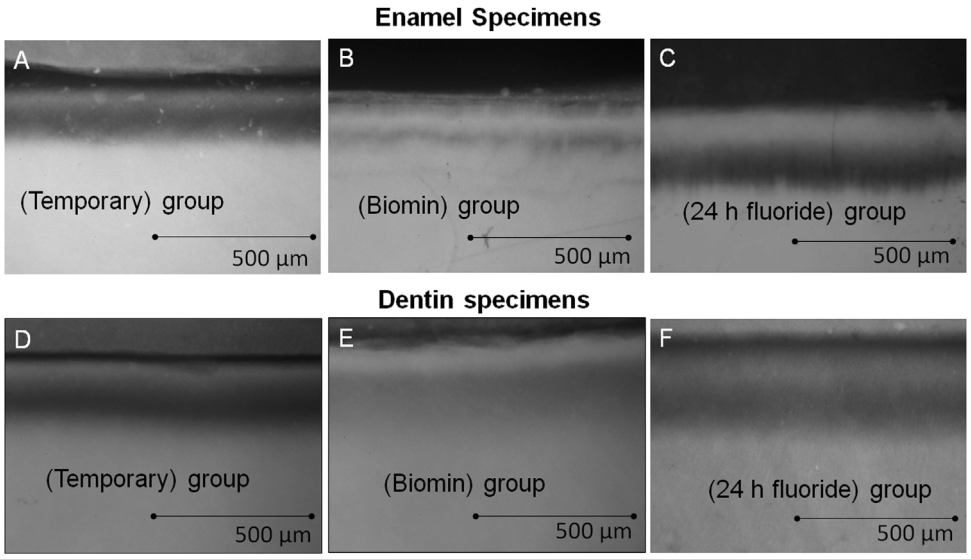

Fig. 2 – Representative Trans-microradiograph images for specimens after 4 days demineralization challenge and application of; (A) Temporary filling material on enamel (Temporary) group. (B) FBG paste on enamel (Biomin) group. (C) Fluoride on enamel (Fluoride) group. (D) Temporary filling material on dentin (Temporary) group. (E) FBG paste on dentin (Biomin) group. (F) Fluoride on dentin (Fluoride) group.

(Image J, USA) and a customized application in Microsoft Excel [5,14,15,22].

The areas of analysis for all samples were confined to the central portion of the artificial caries lesions produced in either enamel or dentin. The mineral density (vol%) values were determined in reference to the calibration curve considering that sound dentine contains 48 vol% [24,25] and enamel contains 87 vol% minerals. Values of ΔZ (Mineral density loss) and LD (lesion depth.) were calculated utilizing the aforementioned computer applications. LD measurement in the current study was detected at a distance from the lesion surface where the mineral density was 5% less than that in the sound area, while ΔZ was defined by the integrated mineral loss from the surface of the lesion to the lesion depth [26].

2.4. *Acid resistance experiment*

30 treated enamel and 30 treated dentin were used in this experiment. A light curing bonding system (Clearfil Universal Bond Quick, Kuraray Medical; Tokyo, Japan) was applied onto the treated enamel and dentin surfaces in all groups according to the manufacturer's instructions (using the etch and rinse mode.). After photo-curing the bonding resin, a restorative composite resin (Tetric N Ceram composite; Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied on the bonded enamel surfaces that was further embedded in a self-curing epoxy resin. The resin/enamel or dentin interfaces were ground with abrasive papers to ensure having flat surfaces as was described previously [3,17,27–29]. The specimens were protected by nail varnish leaving a treatment window of 2 mm surrounding the enamel or dentin resin interface. The specimens were exposed to an erosive challenge from a buffered demineralizing solution of 1% Citric acid for 18 min [5] which was kept under continuous stirring at room temperature. The specimens were washed thoroughly and embedded in resin material (Fig. 1) as was previously described [5]. The specimens were then cross-cut, finished, polished, dried and gold coated to be ready for the SEM observation.

2.5. *Shear bond strength*

45 treated enamel and 45 treated dentin samples were ground using 600 grit SiC paper under wet condition to create a standardized smear layer followed by etching with phosphoric acid gel (37%, Ultradent, UT, USA) and bonded using a bonding system (Clearfil Universal Bond Quick, Kuraray Medical; Tokyo, Japan.). Building up of composite resin (Tetric N Ceram composite; Ivoclar Vivadent AG, Schaan, Liechtenstein) was carried out onto the enamel or the dentin bonded surfaces by condensing the composite resin into teflon tubes having a height of 3 mm and an internal diameter of 3 mm. The adhesive system and the composite resin were light cured by a light curing unit for 10 s and 30 s respectively. The specimens were mounted on the universal testing machine (ElectroPlus E1000, Instron, Canton, MA, USA) and subjected to a shear force at the interface between the composite resin and enamel/dentin at a crosshead speed of 0.5 mm/min.

2.6. *Specimens preparation for the pulpal pressure and nanoleakage expression experiment*

30 freshly extracted third molar teeth were utilized in which the occlusal surfaces of the teeth were ground to expose the mid coronal dentin using a water-cooled low speed diamond saw (Isomet; Buehler, IL, USA) followed by grinding the surface by 600 grit SiC paper. Direct communication to the pulp chamber was created by cutting at a level approximately 1 mm below the cemento-enamel junction, and parallel to the occlusal surface using a water-cooled diamond saw (Isomet; Buehler, IL, USA). The apparatus for conducting the exper-

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Table 2 – Results of TMR mineral loss (ΔZ, vol. %) μm).

			Temporary	Biomin	24h Fluoride
Enamel	Mineral loss (ΔZ, vol. % μm)	Mean	4823.5[a]	1752.194[b]	4700[a]
		SD	245.90	384.10	324.90
Dentin	Mineral loss (ΔZ, vol. % μm)	Mean	4830[a]	1391.28[b]	4138.91[a]
		SD	489.24	207.98	793.73

Horizontal lines having different superscript are statistically significant $p \leq 0.05$.

Table 3 – Results of lesion depth in microns.

			Temporary	Biomin	24h Fluoride
Enamel	Lesion depth	Mean	230[a]	200[a]	235[a]
		SD	37.7	40	32
Dentin	Lesion depth	Mean	259.6[a]	231.6[a]	234.4[a]
		SD	21.12	33.8	23.78

Horizontal lines having different superscript are statistically significant $p \leq 0.05$.

iment is illustrated in Fig. 1 and was described previously [11,30].

The teeth were divided into three groups; (Temporary) group in which the temporary filling material was applied onto the exposed occlusal dentin; (Oxalic) group in which oxalic acid (Super seal, Phoenix Dental, Fenton, MI, USA) was applied onto the dentin surface; and (Biomin) group in which fluoride bioactive glass was applied. All teeth treated by three methods were exposed to a simulated pulpal pressure of 15 cm H_2O (Fig. 1) as was described previously [11,30].

2.7. *Effect of pulpal pressure and nanoleakage expression experiment*

30 freshly extracted third molar teeth were collected according to the criteria previously described. The teeth had their dentin surfaces exposed to the pulpal pressure and were bonded to Clearfil Universal Bond Quick (Kuraray Medical; Tokyo, Japan.) utilized in the etch and rinse mode followed by building up of composite resin (Tetric N Ceram composite; Ivoclar Vivadent AG, Schaan, Liechtenstein). The adhesive system and the composite resin were light cured by a light curing unit for 10 s and 30 s respectively. The specimens were kept under pulpal pressure for 24 h then were vertically sectioned with a diamond saw under water cooling, through the composite buildups and the dentin. Two central slabs were chosen from each tooth, forming a total of 20 specimens per group n = 20. Bonded slabs were ground and polished using wet #1000 silicone-carbide paper, then coated with two layers of fast-drying nail varnish applied up to 1 mm of the bonded interfaces. Ammoniacal silver nitrate was prepared according to the protocol previously reported [31–33] and the specimens were stored in the ammoniacal silver nitrate in total darkness for 18 h, rinsed thoroughly and immersed in photo developing solution (Kodak, NY, USA) for 6 h under a fluorescent light to reduce silver ions into metallic silver. The silver stained resin-bonded specimens were lightly polished to remove the superficial silver remnants [11,31–33], followed by drying the specimens and gold-sputter coating.

The specimens were observed using SEM/EDS (JCM-6000 NeoScope, JEOL, Tokyo, Japan). Line scans across the resin composite–adhesive–superficial dentin interface were then performed. Evaluation of nanoleakage locations was carried out through analysis of the obtained images.

Penetration of silver nitrate into the interface was evaluated as follows; Hybrid layer, adhesive–hybrid interface and adhesive layer were evaluated and graded as (No) (no leakage, score 0), (Slight) (slight leakage, score 1) and (Distinct) (distinct leakage, score 2) [10].

2.8. *Statistical analysis*

One way ANOVA was used to analyze the shear bond strength, Δz, and lesion depth, whenever, there was a significant difference so the post hoc test was utilized for further analysis. The nanoleakage expression results were analyzed by Kruskal–Wallis test [10]. The level of significance for all tests were set at $\alpha = 0.05$. The software utilized for analysis was SPSS v24, IBM, Armonk, US.

3. Results

3.1. *Trans-microradiography (TMR) experiment*

Statistical analysis for ΔZ (mineral loss), lesion depth, in addition to representative TMR images are presented in (Fig. 2, Tables 2 and 3). (Biomin) group showed significant low ΔZ values when compared to the Fluoride 24 h and the Temporary groups ($p \geq 0.05$). There was no statistical significance in ΔZ values among the fluoride 24 h and the temporary groups for enamel and dentin specimens $p \geq 0.05$. All of the examined groups in the current study did not show any significant difference regarding the subsurface lesion depths observed in either the enamel or dentin specimens $p \geq 0.05$.

3.2. *Acid resistance experiment*

Figs. 3 and 4 show a summary for SEM-EDS images examining the acid resistance of enamel and dentin to the citric acid erosion challenge adopted in the current study. An outer lesion (OL) was observed in all groups. Acid resistant area was observed in all groups except in the (Temporary) enamel and dentin groups which showed the resistance of the hybrid layer

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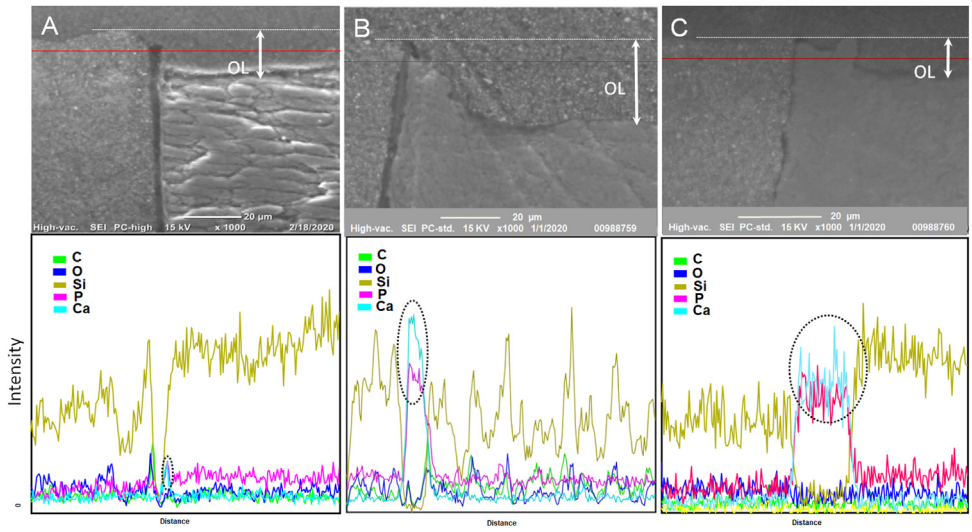

Fig. 3 – SEM-EDS examination for enamel specimens challenged by citric acid 1%. (A) (Temporary), the acid resistant hybrid layer (Dotted circle) was evident however, no acid resistant layer beneath the hybrid layer was not detected. (B) (Biomin) a significant acid resistant zone was detected beneath the hybrid layer (Dotted circle). (C) (Fluoride 24 h) a significant acid resistant zone was detected beneath the hybrid layer (Dotted circle). Outer lesion (OL) was formed in all specimens.

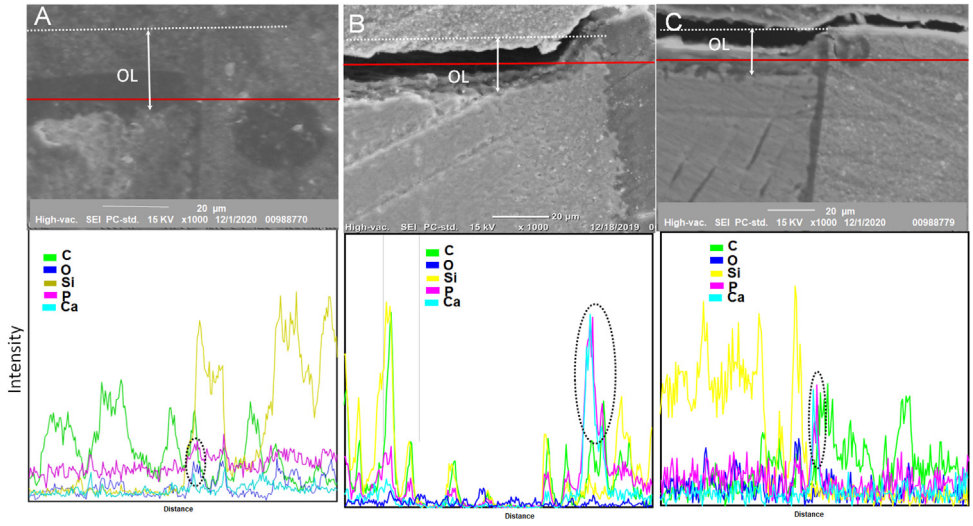

Fig. 4 – SEM-EDS examination for dentin specimens challenged by citric acid 1%. (A) (Temporary), the acid resistant hybrid layer (Dotted circle) was evident however; no acid resistant layer beneath the hybrid layer was detected. (B) (Biomin) a significant acid resistant zone was detected beneath the hybrid layer (Dotted circle). (C) (Fluoride 24 h) an acid resistant zone was detected beneath the hybrid layer (Dotted circle). Outer lesion (OL) was formed in all specimens.

for demineralization with no formation for any acid resistant layer beneath the hybrid layer.

3.3. *Shear bond strength*

Fig. 5 presents the means and standard deviations for the shear bond strength of enamel and dentin specimens. The results show that specimens treated by fluoride for 24 h showed significant deterioration of the shear bond strength compared to the shear bond strength values of enamel or dentin specimens treated by temporary filling material or FBG paste $p \geq 0.05$. Statistical analysis showed that the bond strength values of enamel or dentin specimens treated by either temporary filling material or FBG did not show significant differences.

3.4. *Nanoleakage expression experiment*

Slight leakage was observed in the hybrid layer of all Biomin specimens (Fig. 6, Table 4) while there was distinct nanoleakage in the hybrid layer associated with the (oxalic acid) group. The (Temporary) group showed distinct nanoleakage in the

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Table 4 – Evaluation of nanoleakage location.

	Adhesive			Adhesive-hybrid layer interface			Hybrid layer		
	No	Slight	Distinct	No	Slight	Distinct	No	Slight	Distinct
Temporary	0	0	10	0	0	10	0	0	10
Biomin	10	0	0	9	1	0	3	7	0
Oxalic acid	10	0	0	1	1	8	0	0	10

n = 10 (No) No nanoleakage, (Slight) Slight nanoleakage, (Distinct) Distinct nanoleakage.

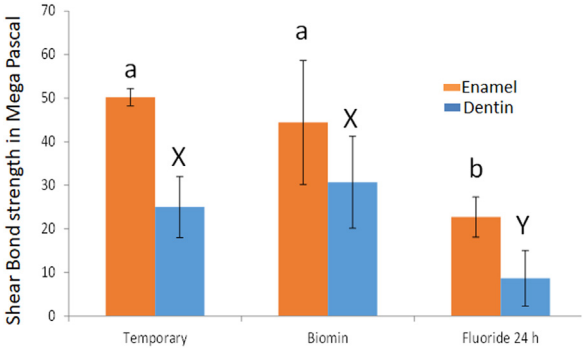

Fig. 5 – Shear bond strength mean and standard deviation values in mega pascal. Different letters in the same category are significantly different P < 0.05.

hybrid layer, adhesive-hybrid layer interface and the adhesive layer $p \geq 0.05$.

4. Discussion

The null hypotheses in the current study were totally rejected; FBG paste improved the acid resistance of the demineralized enamel and dentin cavosurface margin. Moreover, FBG paste application did not affect the shear bond strength of the Clearfil universal bond to enamel or dentin. Additionally, FBG paste decreased the deteriorating effect of the pulpal pressure on the bond-dentin interface.

Previous research showed that 10-MDP monomer can form insoluble salts with the inorganic components of the phosphoric acid etched enamel [3,28]. In the current study 10-MDP monomer contained in the clearfil universal bond quick did not protect the cavosurface margin of the bond-demineralized enamel/dentin interfaces especially when it was used in the total etch mode recommended by the manufacturer. This might be attributed to the decrease of the inorganic components available for bonding to the 10-MDP [27] causing failure of the cavosurface margin and its demineralization when exposed to a secondary caries attack. However, application of the FBG paste on enamel and dentin regained the capability of the 10-MDP monomer to form a protective acid resistant area at the cavosurface margin. This might be attributed to the high release of calcium and phosphate ions that were released from the FBG paste [15] which regained most of the lost minerals in the tested enamel and dentin surfaces. These minerals might have combined with the 10-MDP monomer and improved its ability to form an acid resistant layer at the cavosurface margin. It worth mentioning here that the current experiment adopted the etch and rinse mode on both enamel and dentin as recommended by the manufacturer.

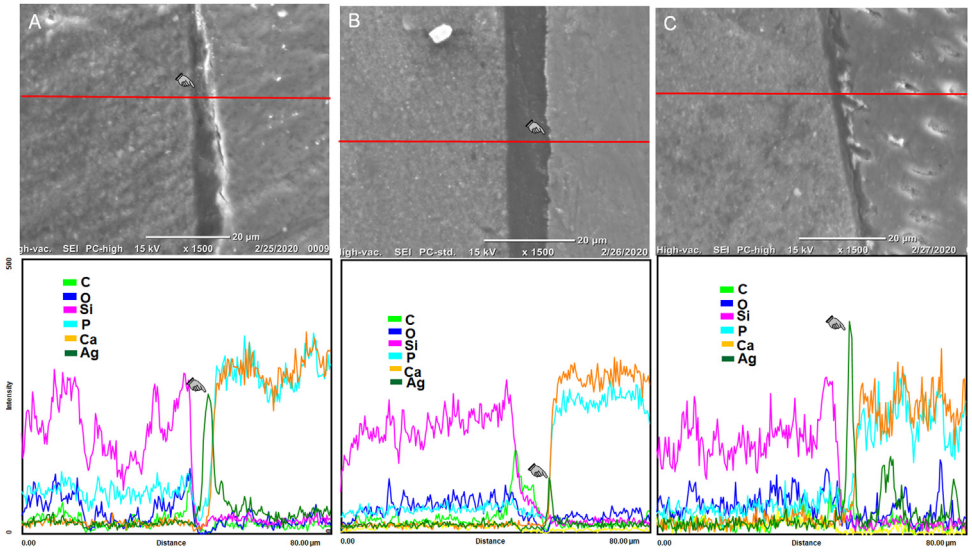

Fig. 6 – SEM/EDS analysis for EDS line scans across the interface between dentin and the adhesive system. (A) Heavy infiltration of silver nitrate deposits within the hybrid layer, and the bonding layer itself. Finger pointer showing diagnostic peak of silver in the bonding layer. (B) Scattered silver nitrate particles detected in the hybrid layer. Finger pointer showing a weak peak of silver in the hybrid layer. (C) Heavy infiltration of silver nitrate deposits within the hybrid layer. Finger pointer showing diagnostic peak of silver in the Hybrid layer.

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A similar effect was observed upon applying fluoride for 24 h on the surface of enamel and dentin. There was an improvement for the acid resistance of the enamel/dentin cavosurface margin suggesting that fluoride ions combined with the enamel and dentin hydroxyapatite and formed a more acid resistant crystals of fluoroapatite [34] which resisted the secondary erosive challenge [34] adopted in the current experiment.

However, the application of fluoride in the this experiment caused serious deterioration in the shear bond strength of the universal bond quick to enamel and dentin which might be due to the capability of the fluoride ions to form CaF_2 globules onto the enamel and dentin surfaces. This might hinder the proper etching of enamel and conditioning of dentin resulting in compromising the formation of a proper hybrid layer between the bonding agent and the coronal hard tooth structures [35]. It is worth mentioning here that the recorded standard deviation for the shear bond strength values of the (Biomin) group was high although all attempts were done to adjust the weight of the powder/ratio of the FBG paste, however the sensitivity of the powder for atmospheric humidity sorption [36] slightly affected the bioactivity of the paste causing slight difference in the calcium and phosphate ion released from the paste which in turn may have affected the remineralization capacity of the powder. This slight variation in the bioactive reaction of the paste was reflected and might have increased the standard deviation observed in the shear bond strength values of the (Biomin) group. This limitation can be amended if the powder and liquid are loaded in a sealed capsule that will be only mixed directly before its application onto tooth structure.

Moreover, fluoride application for 24 h did not cause a significant remineralization for the artificial subsurface lesions induced neither in enamel nor dentin, which may be attributed to the high affinity of the fluoride ions to combine with calcium ions abundant in the enamel and dentin structures [37]. This affinity prevented the fluoride ions from penetrating the demineralized enamel and dentin surfaces and remineralizing the demineralized subsurface lesions [16,37].

On the other hand, FBG paste was capable of remineralizing effectively the subsurface enamel and dentin lesions within the short duration adopted in the current study. This might be due to the nature of the bioactive cycle of this paste as it releases unstable calcium and phosphate ions [38] without forming any stable hydroxyapatite crystals except after 24 h giving the chance for the calcium and phosphate ions to penetrate the enamel or dentin surfaces and remineralize the body of the lesions [15].

The current experiment attempted to test the obliteration capability of the fluoride bioactive glass for fluid filtration across the dentin under a pulpal pressure simulating the natural pulpal pressure. The fluoride bioactive glass could produce an interaction layer rich in calcium and phosphate compounds that was deposited within the lumen of dentin thus decreasing the flow of fluids through the dentin and hindered the flow of the pressurized water to reach the dentin-bond interface. This obliteration capability aided in decreasing the deteriorating effects of the fluid dentinal filtration (under pulpal pressure) on the dentin-adhesive interface after conducting the adhesive restoration of the dental cavities. Previous quantitative analysis [19] for the decrease of the fluid filtration was previously conducted utilizing a fluoride free bioactive glass [19] and showed the significant decrease in fluid filtration values after the bioactive glass application. On the other hand the use of the Clearfil universal bond quick in the total etch mode showed nanoleakage expression in the bonding layer itself suggesting that the phosphoric acid application removed the smear layer and the smear plugs and opened the dentinal tubules orifices [10,11]. This allowed significant amount of moisture to penetrate the interface from the pulpal side and contaminate the cured resin with water droplets that were occupied by the silver nitrate and observed by the FE-SEM.

The aforementioned results of the current experiments suggested that FBG paste mechanism of action might be as follows; The FBG powder that was mixed with 50% phosphoric acid released calcium, phosphate, fluoride, and sodium ions onto the demineralized enamel and dentin surfaces [18–21]. This caused the mobilization of some calcium and phosphate ions from the enamel and dentin surfaces [18–21]. The phosphate ions released from FBG powder and the diluted phosphoric acid solution, in addition to the calcium ions released from FBG and the phosphate ions penetrated the outer teeth surfaces to reach the sub-surface enamel and dentin lesions, rendering this sub-surface lesion saturated with calcium and phosphate ions [15]. Moreover, some calcium and phosphate ions were catalyzed by the presence of the fluoride ions to form a layer of acidic calcium-phosphate salts [39] which might have obliterated the dentinal tubule orifices and decreased the deteriorating effects of the pulpal pressure on the dentin-resin interface. Failure to observe any significant peaks of silica in the (Biomin) group by SEM/EDS (although it is one of the main components of the FBG) might be attributed to the breaking down of the silica network of the FBG. This occurred due to the action of the water content of the aqueous part of the acidic solution used to mix the FBG powder forming the highly soluble Si−OH groups (Silanol groups) [40], upon being mixed with water that was washed out upon being rinsed with water spray after 24 h. The difficulty in detecting the fluoride peaks may be attributed to the low photon energy of the fluoride, which may render it difficult to be detected by SEM-EDS [15]. The currently reported results suggest that the fluoride bioactive paste may be used as a temporary filling in patients at high risk to develop caries. The fluoride bioactive paste may remineralize the enamel and dentin walls efficiently irrelevant of the saliva condition (regarding its deficient flow and/or low mineral content) which may aid in decreasing the risk of demineralization of the cavosurface margins of restorations and possibility of developing recurrent caries. Moreover, it is speculated that in patients of normal salivary condition the suggested paste may remineralize already demineralized enamel and/or dentin margins allowing the conservation of the prepared cavities. Additionally, the suggested paste may increase the durability of the resin-dentin interface of the restoration because it can obliterate the dentinal tubule lumens causing a decrease in the fluid filtration to the resin-dentin interface.

The currently suggested technique has the limitation of utilizing the phosphoric acid to boost the bioactivity of the flu-

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oride bioactive powder which should be supplied in accurate quantities to achieve the reported results. As was previously mentioned, the powder/liquid ratio and sensitivity of the powder to moisture [36] are limitations for the currently reported technique however, premeasuring the required amounts and supplying the powder and liquid in capsule form may aid in solving this problem.

5. Conclusions

Within the limitations of the current in-vitro study, it may be suggested that the application of the FBG as a temporary filling material may provide the following advantages: (a) Remineralize the demineralized enamel and dentin surfaces allowing for the possibility of preparing more conservative teeth cavities. (b) Does not decrease the shear bond strength of enamel and dentin to a one bottle universal bonding system applied in etch and rinse mode. (c) Provide sealing for the resin-dentin interface against the deteriorating effects of the pulpal pressure.

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