



# The efficacy of a bioglass (45S5) paste temporary filling used to remineralize enamel surfaces prior to bonding procedures

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## ABSTRACT

**Objectives:** The efficacy of using a 45S5 Bioglass paste as a remineralizing temporary filling material was compared to fluoride gel (1.23% acidulated-phosphate-fluoride, Gelato Gel, NJ, USA) applied for 24 h and for 4 min and to a temporary filling material (Cavinton; GC, Tokyo, Japan).

**Methods:** 66 extracted human premolars were used. All specimens were sectioned mesio-distally. All specimens were challenged by a demineralization solution (pH4.5) for 4 days. The specimens were divided into 4 groups: (Bioglass), (Fluoride 4minutes), (Fluoride 24Hours), and (Temp) (n = 33). 10 specimens were examined by TMR (Transversal Microradiography) to test remineralization capacity of each agent. 8 specimens were assigned to determine the acid resistance of a bonded resin-enamel interface after the various treatment methods. 15 samples were assigned to determine the effect of each agent on the shear bond strength of a single bottle self-etch adhesive system. One way ANOVA was used to compare the obtained results ( $p < 0.05$ ).

**Results:** Specimens treated with 45S5 bioglass showed statistically significant reductions in ( $\Delta z$ ) values when compared to the other three groups ( $p < 0.05$ ). Treatment of demineralized enamel by fluoride prior to bonding significantly decreased shear bond strength values, however 45S5 bioglass treatment did not affect the shear bond strength of adhesive system to enamel ( $p < 0.05$ ). The acid-resistance of the interface between resin-enamel was significantly improved when fluoride or bioglass were applied onto the enamel surface prior to demineralization.

**Conclusions:** 45S5 bioglass paste has high potential to be used as a remineralizing temporary filling material.

## 1. Introduction

The worldwide acceptance in the Operative dental field for the minimal intervention concept [1,2] is responsible for the advancement of various dental techniques and material technologies nowadays. This concept mainly relies on preserving as much as possible from tooth structure to ensure that the teeth will be functioning for life [1,2]. The introduction of the functional monomer 10 MDP (10-Methacryloyloxydecyl dihydrogen phosphate) in dental adhesive was one of the great achievements that improved the bonding of composites to enamel and dentin [3–5]. One of the advantages of utilizing the MDP containing adhesives was preserving the enamel /resin interface from secondary acidic attacks through the formation of an acid-base resistant zone [3,6,7]. However, most of the experiments employed to test the enamel/resin interface utilized sound enamel as a substrate.

Previous studies showed that bonding to hypomineralized enamel

may result in significant decrease in bond strength when compared to bonding to sound enamel [8] which may be attributed to increased inter crystalline porosity of the hypomineralized enamel, moisture retention within the inter-rods spaces [9] and the higher protein contents [10]. It was suggested that applying 5% NaOCl to etched enamel followed by applying a resin infiltration system would improve bond strength values [8]. However, other study suggested that increased Tetraethyleneglycol Dimethacrylate content of resin infiltration systems may jeopardize the bonding interface durability due to its increased hydrophilicity that will cause its degradation by time [11]. Moreover, the lack of efficient remineralizing agents hindered the introduction of techniques that adopt remineralization of enamel prior to bonding.

Bioactive glasses were introduced in the late 60s as an aid in treatment of comminuted fractures [12] and as a coating material for orthopedic implants [12]. Recently, a potent remineralizing agent mainly composed of a bioactive glass was introduced [13–16] which

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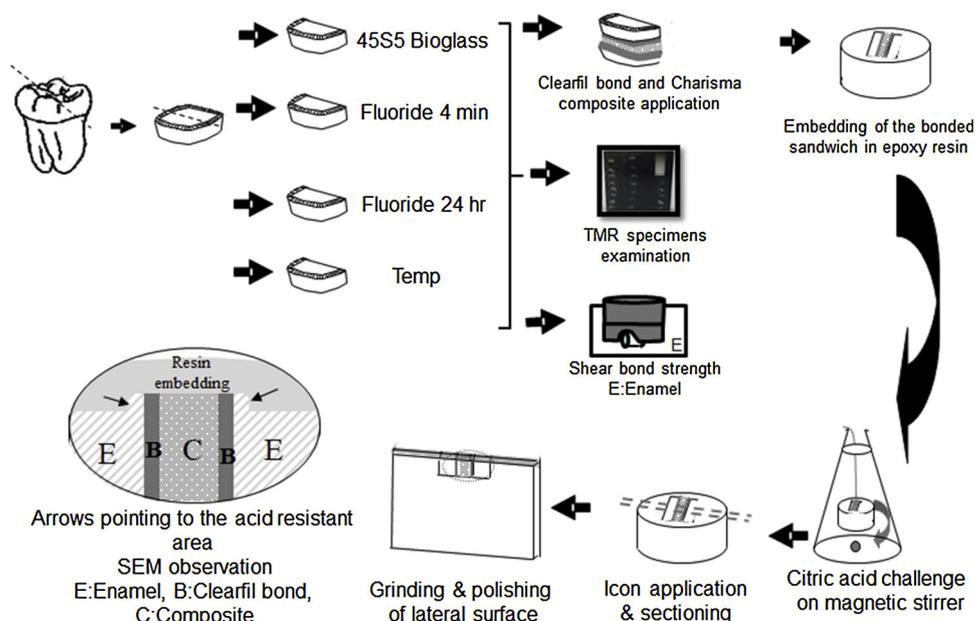


Fig. 1. Summary of the experimental procedures.

was capable of improving the mechanical properties of the demineralized enamel within a short duration [17]; however, no studies were conducted to assess the influence of applying this agent on demineralized enamel prior to bonding to dental adhesive systems

The null hypotheses adopted in this study were that the 45S5 bioglass paste and a topical fluoride agent, will remineralize the enamel lesion, and will improve the acid resistance of the enamel resin interface, and will not affect the shear bond strength of a bonding system to demineralized enamel.

2. Materials and methods

2.1. Specimens' preparation

Summary for the experimental procedures and materials tested are summarized in (Fig. 1), and (Table 1). Based on 80% power of test, enamel discs were obtained from the buccal and lingual surfaces of 66 freshly extracted human non-carious premolars. The buccal and lingual surfaces of the premolars were sectioned and embedded in acrylic blocks. The enamel surfaces of the samples were slightly ground with water-cooled silicon carbide discs to obtain flat enamel surfaces. All specimens were challenged with buffered demineralization solution (pH 4.5) [18]. The demineralized specimens were assigned into four groups (n = 33). The groups were prepared as follows: (Temp) group had a temporary filling material (Cavition; GC, Tokyo, Japan) applied onto its enamel surfaces for 24 h, (Fluoride 4 min) group had a fluoride gel (1.23% acidulated-phosphate-fluoride, Gelato Gel, NJ, USA) applied for 4 min and then gently washed by deionized water according to the manufacturer instructions followed by placing a temporary filling

material (Cavition; GC, Tokyo, Japan) for 24 h ; (fluoride 24 h) group had a fluoride gel (1.23% acidulated-phosphate-fluoride, Gelato Gel, NJ, USA) applied on the enamel surface followed by placing the temporary filling material for 24 h without washing the fluoride gel; and (Bioglass) group specimens had bioglass applied on its surface. After 24 h of storage in distilled water all remnants of temporary filling material and bioglass were removed and washed by deionized water.

2.2. 45S5 bioglass application

45S5 bioglass powder (5 μm average particle, composed of 24.5 wt % Na<sub>2</sub>O, 24.4 wt% CaO, 6 wt% P<sub>2</sub>O<sub>5</sub>, and 45 wt% SiO<sub>2</sub>) was mixed on a glass slab as was previously described [13–17] with diluted phosphoric acid to form a paste having a pH 2 (Table 1). The bioglass paste was applied on the tested specimens in the (bioglass) group by microbrush (Microbrush International, Grafton, WI, USA). A layer of a bonding agent (Clearfil Universal Bond Quick, Kuraray Medical; Tokyo, Japan) was applied over the 45S5 bioglass paste (that was applied on the enamel surface) and then light-cured Table 1. After storage in de-ionized water for 24 h, the thin layer of the bonding agent was gently removed by means of an excavator, and then rinsed with water spray for 30 s followed by cleaning the specimens' enamel surfaces by brush installed on a low speed handpiece with water irrigation to get rid of any remnant of the 45S5 bioglass paste.

2.3. Transversal microradiograph (TMR) examination

10 specimens from each group were embedded in resin material to maintain the stability of the enamel sample, that were cross sectioned

Table 1  
Materials used in this study.

Materials	Composition	Procedures
45S5 Bioglass 0.2 ml of phosphoric acid	Weight Percent 45%SiO <sub>2</sub> , 24.5%Na <sub>2</sub> O, 24.4%CaO, 6%P <sub>2</sub> O <sub>5</sub>	Mi x 0.1 gram of 45S5 Bioglass 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 self-etching adhesive system light cure (10 s)

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

to obtain enamel-resin slabs that are approximately 800 μm in thickness using a low-speed diamond saw (Isomet 5000; Buehler, Lake Bluff, Illinois, USA). The enamel-resin slabs were then manually thinned to obtain enamel slabs having a maximum thickness of 100 μm. Images were taken from the central enamel lesion slice using 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 TMR images, together with 15 aluminum step-wedges were digitized using a digital camera attached to a microscope (ML 8500, Meiji, Techno, Japan). The mineral loss ( $\Delta Z$ , vol % μm) and the lesion depths values were calculated as was previously reported [2,18–21]. The definitions of lesion depth and the mineral loss ( $\Delta Z$ , vol % μm) followed the previously published data [2,19–20]. One way ANOVA was used to compare the obtained results ( $p < 0.05$ ).

2.4. Acid resistant experiment

8 samples from each group were used in this experiment. A light curing bonding system (Clearfil Universal Bond Quick, Kuraray Medical; Tokyo, Japan) was applied onto the treated enamel 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 (Charisma submicro-hybrid resin composite; HeraeusKulzer, Hanau, Germany) was applied on the bonded enamel surfaces that was further embedded in a self-curing epoxy resin (Epoxy cure resin, Buehler; Lake Bluff, IL, USA). The resin/enamel interface was ground with abrasive papers to ensure having flat surfaces. The specimens were protected by nail varnish leaving a treatment window composed of the enamel resin interface. The specimens were exposed to an erosive challenge from a buffered demineralizing solution of 1% Citric acid for 18 min [21] 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 [4]. The specimens were then cross-cut, finished, polished, dried and gold coated to be ready for the SEM observation table [4].

2.5. Shear bond strength

15 specimens from each group were etched by 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 (Charisma submicro-hybrid resin composite; Heraeus Kulzer, Hanau, Germany) was carried out onto the enamel 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 at a crosshead speed of 0.5 mm/minute. All results were analyzed using One way ANOVA  $p < 0.05$ .

3. Results

3.1. Transversal microradiography (TMR) analyzation

Representative TMR images for all groups are shown in (Fig. 2) Means and standard deviations for the  $\Delta Z$ , mineral regain, and Lesion Depths for all groups are shown in (Tables 2 and 3). (Bioglass) group showed the most significant decrease in  $\Delta Z$  value when compared to all other groups ( $p = 0.001$ ), while there was no statistical significance in  $\Delta Z$  values among the remaining three groups ( $p = 0.05$ ).

Lesion depth statistical analysis showed that there were no

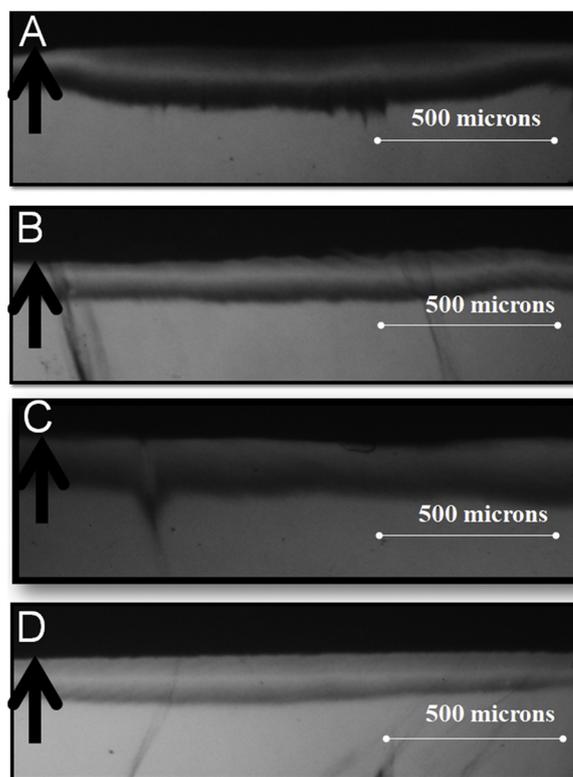


Fig. 2. Representative Transversal Microradiograph images for; (A) (Temp) group; Temporary filling material was applied onto the enamel surfaces after the demineralization challenge, (B) (Fluoride 4 min) group; Fluoride gel was applied for 4 min according to manufacturer instructions after the demineralization challenge, (C) (Fluoride 24 h) group; Fluoride gel applied for 24 h after the demineralization challenge, and (D) (Bioglass) group; Bioglass paste applied followed by temporary coverage of a bonding agent for 24 h after the demineralization challenge. Black arrows in all images are pointing to areas protected by nail-varnish, n = 10.

Table 2 Results of TMR mineral loss ( $\Delta Z$ , vol. %) μm).

	Temp	Fluoride 4 min	Fluoride 24 H	Bioglass
Mineral loss ( $\Delta Z$ , vol. % μm)	Mean 6480	5900	5390	2350
	SD 1400	294.67	479.57	155.13
remineralization percentage	0%	8.90%	16.80%	63.70%

n = 10.

Table 3 Results of lesion depth in microns.

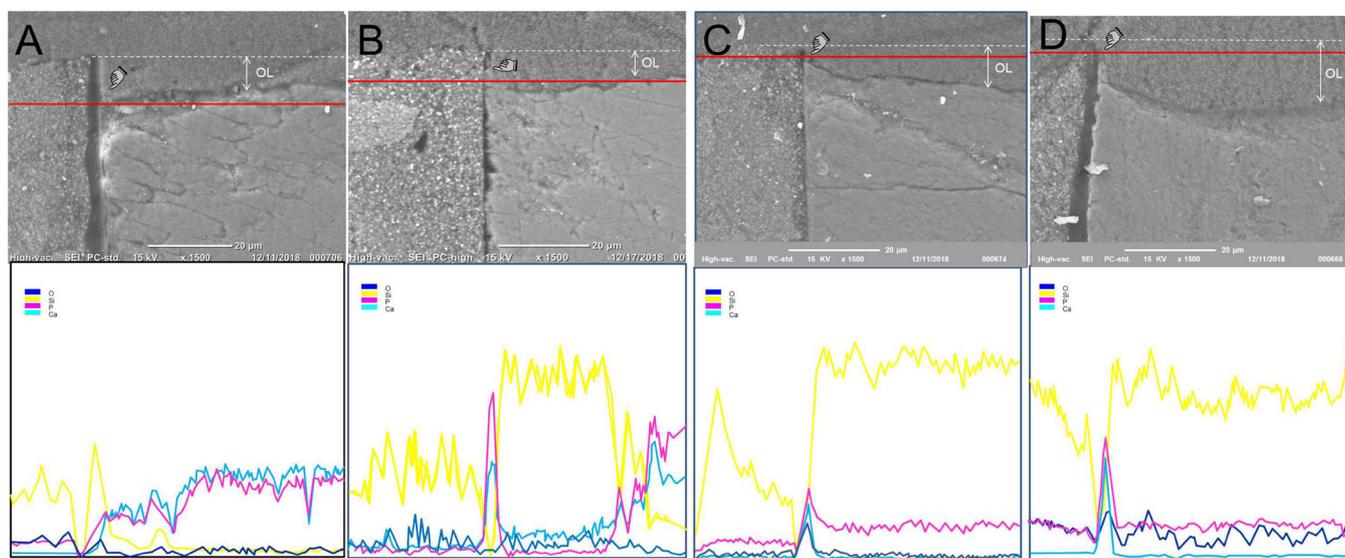
	Temp	Fluoride 4 min	Fluoride 24 H	Bioglass
Lesion depth	Mean 233 <sup>a</sup>	253 <sup>a</sup>	240 <sup>a</sup>	203 <sup>a</sup>
	SD 41	11	42	43
Percentage of decrease in lesion depth	0%	4%	0%	10%

Values with different superscripts have significant differences  $p = .0225$ , n = 10.

significant differences among the tested 4 groups ( $p < 0.05$ ).

3.2. Acid resistant experiment

The SEM-EDS morphological examination for the citric acid challenged specimens is summarized in (Fig. 3). An outer lesion (OL) was



**Fig. 3.** SEM-EDS morphological examination for the citric acid challenged specimens. (A) (Temp) group; No acid resistant area (ARA) observed (B) (Fluoride 4 M in. group. Finger pointer pointing to the formed (ARA) at the resin enamel interface, (C) (Fluoride 24 h) group; Finger pointer pointing to the formed (ARA) at the resin enamel interface (D) (Bioglass) group Finger pointer pointing to the formed (ARA) at the resin enamel interface. An outer lesion (OL) was formed in all groups,  $n = 8$ .

formed in all groups, and an acid resistant area (ARA) was observed in all groups except in the (Temp) group which showed complete demineralization of the enamel-bond interface at the cavo-surface margin. The average thickness of the (ARA) for the (Fluoride 4 min) and (Fluoride 24 hours) samples were  $1.53\ \mu\text{m}$  and for (Bioglass) samples was  $(2.8\ \mu\text{m})$ .

### 3.3. Shear bond strength

Shear bond strength values are summarized in (Table 4). Shear bond strength of specimens treated with fluoride for 4 min and 24 h showed significant decrease in the bond strength when compared to the shear bond strength values of enamel specimens treated by temporary filling material or bioglass  $p < 0.05$ . Failure modes for the specimens after shear bond strength are shown in (Fig. 4).

## 4. Discussion

The null hypotheses were partially accepted. The 45S5 bioglass paste and the fluoride gel improved the acid resistance of the resin/enamel interface to erosive attack. 45S5 bioglass successfully remineralized the enamel subsurface, and it did not decrease the shear bond strength to the tested bonding system, however, fluoride gel decreased shear bond strength and did not significantly remineralize the enamel sub-surface.

For assessing the enamel remineralization capacity of the agents tested in the current experiment the Transversal microradiography was utilized which is the golden standard method employed in demineralization/remineralization experiments [2,18,19]. Previous research [22] showed that sub-surface lesions with an average depth of  $75\ \mu\text{m}$  needs daily remineralization by saliva for 30 days [22] to significantly decrease its lesion depth. This might suggest the insignificant changes observed in the lesion depth for the bioglass group that was applied

**Table 4**  
Mean and standard deviation of Shear bond strength in mega pascal.

	Temp	Fluoride 4 min	Fluoride 24 H	Bioglass
Shear Bond strength	52.24 <sup>a</sup>	29.59 <sup>b</sup>	28.16 <sup>b</sup>	49.78 <sup>a</sup>
SD	3.9	8.73	9.89	8

Values with different superscripts have significant differences  $p < 0.05$ .

only for 24 h on a deep subsurface lesion with an approximate depth of  $200\ \mu\text{m}$ , however the mineral content of the artificial demineralized enamel lesion showed significant improvement upon the application of the 45S5 bioglass which may explain the significant mechanical improvement observed previously for the demineralized enamel that was remineralized by a similar paste [17].

Fluoride agent utilized in the current experiment did not efficiently remineralize the sub-surface demineralized enamel lesions and showed significant decrease in the bond strength values. This agrees with a previous report [23] which suggested that fluoride reacts with enamel calcium and precipitates calcium fluoride ( $\text{CaF}_2$ ) globules on the outer enamel surface. Upon decreasing the pH of the  $\text{CaF}_2$  by means of phosphoric acid etching, the  $\text{CaF}_2$  salts penetrate the demineralized enamel and deposit within the inter-prismatic spaces. The aforementioned enamel remineralizing mechanism of fluoride decreases the permeability of the enamel and limits the penetration of adhesive resin within the enamel structure which may explain the decreased bond strength values for the fluoride groups tested in the current experiment. The slow and weak remineralization of demineralized enamel observed in the current experiment concur with previous research which demonstrated that daily application of fluoride for a minimum of five weeks is necessary to improve the mineral density and lesion depth of active sub-surface lesions having a depth of  $90\ \mu\text{m}$  [23]. Also, a previous research showed that fluoride has weak remineralization capacity when used to treat arrested enamel subsurface lesions in oral cavities due to the blocking of the lesion porosities by organic materials [24].

On the other hand, 45S5 Bioglass significantly remineralized the examined enamel subsurfaces and did not significantly affect the shear bond strength values between demineralized enamel and the tested bonding system which may be attributed to the deep remineralizing effect exerted by the bioglass paste. 45S5 bioglass is a bioactive glass that needs at least 2 h to complete its bioactive cycle [12], and thus it was previously suggested that there will be a continuous release of calcium and phosphate ions from the bioglass network into the body of the demineralized enamel lesion [17]. A previous X-ray diffraction study [25] showed that the calcium and phosphate compounds released from the bioglass paste will preserve its amorphous state with no crystallization for 24 h [25]. Thus it is speculated that the aforementioned calcium and phosphate ions will have enough chance to penetrate into the enamel demineralized sub-surface for 24 h rather than only precipitating onto the enamel surface and hinder the resin

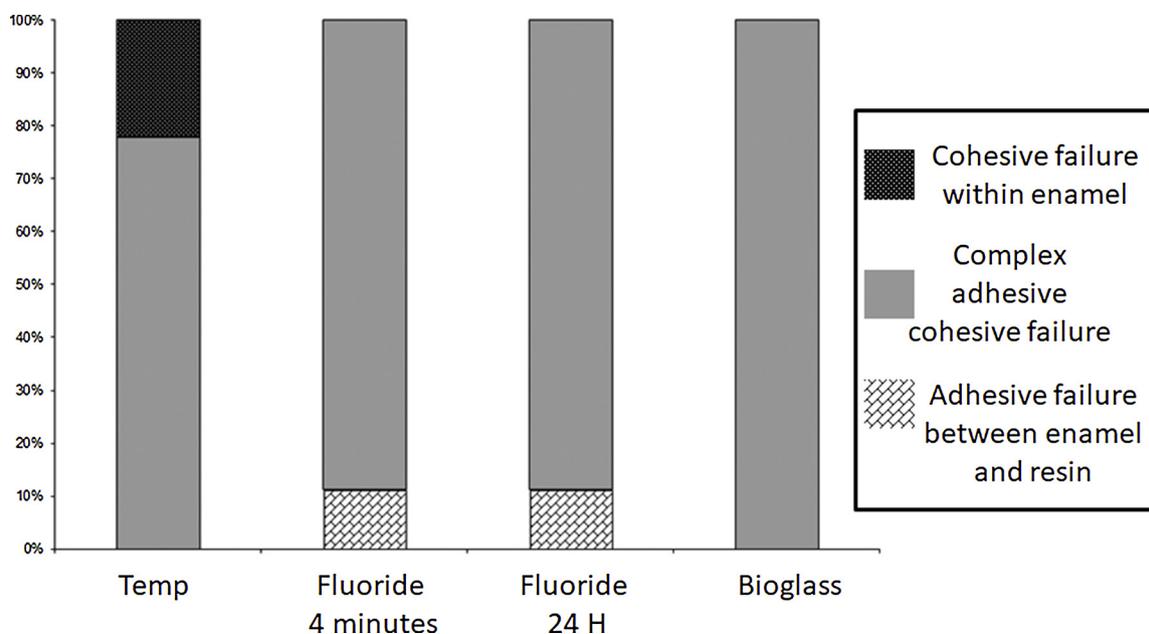


Fig. 4. Failure modes for the specimens after shear bond strength.

penetration. In the current study thorough mechanical cleaning of the tested enamel surfaces was done to avoid the presence of any fluoride or bioglass remnants that may interfere with the adhesive bonding procedures.

The high bond strength obtained between the demineralized enamel and the tested bonding system utilized in the current experiment may be attributed to the formation of deep resin tags in demineralized enamel porous surface [26] which increased the surface area exposed to bonding [26]. Previous studies recommended that one bottle self-etching adhesive containing 10MDP resin should be applied on sound enamel after phosphoric acid etching to protect the bonded enamel cavo-surface margin by forming an enamel acid-base resistant zone [27]. However in the current study it was clear that bonding of the MDP containing bonding system to demineralized enamel may jeopardize the longevity of the bonded restorations because the enamel/bond interface was completely demineralized. It is speculated that the decrease of the hydroxyapatite crystals of the demineralized enamel surface lead to decreased formation of the acid resistant calcium salts formed between the hydroxyapatite crystals and the MDP [27]. Moreover, it may be suggested that 45S5 Bioglass paste and fluoride applications rendered the demineralized enamel margins more resistant to secondary erosive attack due to the availability of enough inorganic enamel minerals capable of reacting with the MDP to form sufficient insoluble salts.

One of the widely used temporary filling materials nowadays that was tested in the current study showed no significant remineralization for the tested enamel specimens. Previous research showed that the temporary filling material examined in the current experiment may exert negative effect on pulpal cell counts if placed directly in contact with these cells [16]. However the suggested 45S5 bioglass paste has a potential to serve as a temporary filling material capable of remineralizing the demineralized enamel margins prior to the restorative phase. Previously, it was [28] shown that fluoride and calcium phosphate releasing restorative materials have limited remineralization capacity to demineralized enamel margin due to the limited amount of minerals released [28] from the aforementioned materials.

Within the limitations of this study it may be concluded that 45S5 bioglass paste utilized in the current experiment may serve as a remineralizing temporary material capable of remineralizing the demineralized enamel without affecting the bond strength of resin restoration to enamel. Also it improved the acid resistance of the enamel margins to any further acidic attacks at the enamel resin interface.

#### Conflict of interest

The authors wish to declare that there is no conflict of interest.

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