**Original Article**

# Effect of Bioactive Glass air Abrasion on Shear Bond Strength of Two Adhesive Resins to Decalcified Enamel

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**Abstract**

**Objective:** Bioactive glass air abrasion is a conservative technique to remove initial decalcified tissue and caries. This study examined the shear bond strength of composite resin to sound and decalcified enamel air-abraded by bioactive glass (BAG) or alumina using etch-and-rinse and self-etch adhesives.

**Materials and Methods:** Forty-eight permanent molars were root-amputated and sectioned mesiodistally. The obtained 96 specimens were mounted in acrylic resin; the buccal and lingual surfaces remained exposed. A demineralizing solution was used to decalcify half the specimens. Both sound and decalcified specimens were divided into two groups of alumina and bioactive glass air abrasion. In each group, the specimens were subdivided into two subgroups of Clearfil SE Bond or OptiBond FL adhesives (n=12). Composite resin cylinders were bonded on enamel surfaces cured and underwent thermocycling. The specimens were tested for shear bond strength. Data were analyzed using SPSS 16.0 and three-way ANOVA ($\alpha$=0.05). Similar to the experimental groups, the enamel surface of one specimen underwent SEM evaluation.

**Results:** No significant differences were observed in composite resin bond strength subsequent to alumina or bioactive glass air abrasion preparation techniques (P=0.987). There were no statistically significant differences between the bond strength of etch-and-rinse and self-etch adhesive groups (P=1).

Also, decalcified or intact enamel groups had no significant difference (P=0.918). However, SEM analysis showed much less enamel irregularities with BAG air abrasion compared to alumina air abrasion.

**Conclusion:** Under the limitations of this study, preparation of both intact and decalcified enamel surfaces with bioactive glass air abrasion results in similar bond strength of composite resin in comparison with alumina air abrasion using etch-&-rinse or self-etch adhesives.

**Key words:** Air abrasion; bond strength; composite resin; enamel

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## INTRODUCTION

New techniques and materials have yielded new concepts for tooth surface preparation and caries removal in conservative adhesive dentistry. Air abrasion is a promising technique for ideal preparation of minimally invasive cavities [1]. In 1940, Robert Black introduced air abrasion as an alternative to manual and rotary preparation. Air abrasion usually targets the tooth structure with a high-speed stream of purified aluminum oxide particles using air pressure. This technique has some advantages, including no vibration, pressure or noise (associated with the use of rotary systems). In addition, there is less need for local anesthesia [1, 2]. Another advantage of this technique is formation of a rough enamel surface; which is more suitable for bonding procedures. Tooth surface preparation by air abrasion removes small amounts of tooth structure and produces irregular cavity contours, compatible with adhesive restorations.[3] Some studies have evaluated this technique as an alternative to the acid-etch technique [2, 3] In addition, air abrasion enhances the detection of occlusal surface carious lesions [3].

At present, air abrasion is mainly used to enhance the diagnosis of carious lesions and avoid the probing technique which may facilitate the progression of lesions [4]. However, selection of a powder for selective removal of carious lesions might influence the quality and longevity of the bond between the tooth structure and the adhesive/composite resin, brought about with the use of currently available adhesive systems [5-7]. However, air abrasion is more appropriate for small cavities due to subtle changes in temperature. In addition, air abrasion is leaves a smooth surface contrary to round burs [2]. Nonetheless, due to the absence of a tactile feedback, alumina air abrasion technique may lead to over-preparation of cavities because sound tooth structures may be removed as well [4]. Removal of enamel caries with air abrasion using sodium bicarbonate ($NaHCO_3$) results in less over-excavation but more under-excavation in comparison to the use of alumina powder [4].

Newly developed abrasive powders more selectively remove enamel and dentin caries, resulting in a more conservative treatment compared to the use of conventional alumina powder. Bioactive glass technique selectively cleanses enamel surfaces after orthodontic brackets are debonded, outperforming carbide burs [8]. In a study, bioactive glass was used to remove artificial carious lesions in enamel and the technique was shown to be selective compared to air abrasion with alumina [9].

Hench introduced bioactive glasses; which are silicate-based glasses and have calcium and phosphate in their chemical composition similar to hydroxyapatite in bone. Bioactive glass enhances cell-to-material adhesion in biological environments [10]. In abrasion with bioactive glass, particles interact with dentin, enhancing remineralization via the release of minerals. Efflandt et al. [10] showed the interaction of bioactive glasses with human dentin in vitro after being soaked in whole saliva; they also showed that bioactive glass 45S5 adhered to dentin under these conditions. Bioactive glass abrasive particles should contact dentin in order for the interactions to take place.

The bonding process to tooth structure further evolved after improving the technique, achieving adequate bonds in moist environments, and increasing resistance to demineralization [11[. Van Meerbeek, et al. classified adhesive systems based on their interaction with tooth structures and the number of steps involved as: etch-and-rinse (two- and three-step adhesives), self-etch (one- and two-step adhesives) and glass-ionomers. Etch-and-rinse adhesive systems consist of two or three steps depending on the separation of primer and the bonding agent or combining them in one bottle. The adhesion technique consists of at least two steps. In the conventional technique, there are three steps during which the conditioner or the acid etchant is applied, followed by the primer or the adhesion-promoting agent; finally, the bonding agent or the adhesive resin is applied. In the two-step technique, the priming and bonding steps are combined; however, there is still a separate etch and rinse step [11]. In the self-etch systems, acidic monomers are used to

demineralize and impregnate the tooth structure substrates simultaneously [12].
Application of self-etch systems has increased steadily because they save time and are easy to use. In high-risk caries patients preventive resin restorative procedures using early minimally invasive interventions might be effective; in such patients the peripheral sound enamel should be preserved [13, 14]. In such cases, occlusal lesions usually pose diagnostic problems and fluoride-rich acid-resistant enamel may be removed by burs or alumina air abrasion technique [4]. As a result, bioactive glass air abrasion technique should be used judiciously as a minimally invasive technique [15]. In numerous clinical situations, the clinician locates the decalcified enamel region, especially in the depth of occlusal fissures or in cervical areas adjacent to cervical caries; therefore, the aim of this in vitro study was to evaluate the shear bond strength of composite resin to sound and decalcified enamel abraded by alumina and bioactive glass air abrasion techniques by application of etch-and-rinse and self-etch bonding systems.

## MATERIALS AND METHODS

This study was approved by the Ethics Committee for Research of Isfahan University of Medical Sciences. Forty-eight extracted intact human third molars, free from cracks, erosion, hypoplastic enamel lesions and irregularities were collected and stored in 0.2% thymol solution at 4°C. Twenty-four hours before the experiment, the teeth were rinsed with normal saline solution.
Then, the crowns were separated from the roots, sectioned mesiodistally by diamond discs (D&Z, Diamante, Germany) using a trimming machine (Krupp Dental Dentarapid GmbH, Germany) and embedded in cylindrical acrylic resin molds (Acropars, Marlic Medical Co.), with the buccal and lingual surfaces exposed and placed horizontally. Then, the enamel surfaces (n=96) were abraded by 200, 400 and 600-grit wet silicon carbide papers, respectively to create smooth horizontal surfaces.
Half of the enamel surfaces (n=48) were covered with nail varnish, except for a window with a dimension of 6×6 $mm^2$ [16]. The specimens were immersed in a demineralizing solution (containing 2.2 mM of $CaCl_2$, 2.2 mM of $KH_2PO_4$, 0.05 M of acetic acid; pH=4.4) at 37°C for 96 hours [16].
Demineralized enamel surfaces underwent alumina and bioactive glass air abrasion using an air abrasion unit (DentoPrep, Ronvig Co, Denmark). In both groups, Abradent microabrasive device (60 psi, 0.6 mm of inner diameter, distance of 10 mm and 90° tipping angle) was used. Alumina particles measuring 50 µ in diameter (Heinrich, Germany) were used, with a powder flow rate of 3 g/min [15]. Bioactive glass group specimens were treated in the same manner except that the bioactive glass powder (Nova Bone Products, LLC Alachua, Florida, USA) was utilized. Prepared specimens in each group were divided into two subgroups for the bonding of composite resin with a self-etch adhesive system (Clearfil SE Bond, Kuraray Co, Osaka, Japan) and an etch-and-rinse adhesive system (OptiBond FL, Kerr Co., USA). After the application of each adhesive resin according to the manufacturers' recommendations (Table 1), cylindrical plastic molds with an internal diameter of 2 mm and a height of 1 mm (Orthorings, Ortho Organizers Inc, Carlsbad, CA, USA) were placed on prepared enamel surfaces at room temperature (22±1°C) and light cured composite resin (Clearfil AP-X, A3, Kuraray, Osaka, Japan) was placed in each mold. The composite resin samples in the molds were photo-polymerized using a light-curing unit (Coltolux 2.5, Coltene AG, Feldwiesenstrasse Altstätten/ Switzerland) using a light intensity of 480 $mW/cm^2$ for 40 seconds; the intensity of light was evaluated by an LED radiometer (LED Radiometer, Model 100, Kerr, USA).
The light-curing tip was placed 1 mm away from the composite resin surface.

The bonded specimens were preserved in a humid environment at 37°C for 24 hours, followed by 1000 rounds of thermocycling at 5°C/55°C with a dwell time of 30 seconds and a 10-second transfer time. The specimens finally underwent shear bond strength test in a universal testing machine (Walter & Bai, K21046, Lohningen, Switzerland). A knife-edge blade, 0.5 mm in terminal thickness, was placed in the machine, directing the shearing force perpendicular to the composite–enamel interface at a crosshead speed of 1 mm/min.

Data analysis was carried out using SPSS 16.0 and three-way ANOVA. Statistical significance was defined at $P< 0.05$.

The fracture modes of composite resin cylinders on enamel surfaces were evaluated under a light microscope at ×16 magnification and categorized as follows:

1. Cohesive fracture: fracture in the composite resin or tooth structure
2. Adhesive fracture: fracture in the adhesive interface
3. Mixed fracture: adhesive/cohesive fracture (Table 5).

One sound third molar was selected for SEM analysis. The tooth was sectioned mesiodistally into two halves and one half was demineralized as previously described. Subsequently, each specimen surface was divided into three zones: Zone A: abraded with alumina; Zone B: abraded with bioactive glass and Zone C: non-abraded surface.

After air abrasion, subsequent to a 10-minute ultrasonication procedure, the specimens were dehydrated for 24 hours and sputter-coated with platinum-gold to a thickness of 10 nm for SEM analysis.

**Table1.** Materials used in the study and the mode of their application according to manufacturers' instructions

| | | |
|---|---|---|
| OptiBond FL(Kerr Co, USA)<br>MMEP,<br>GMA, HEMA,<br>rium<br>factor A174 | 1. Etch with phosphoric acid (15 seconds<br>2. Rinse for (15 seconds) and dry (5 seconds),<br>3. Apply primer and rub for 15 seconds.<br>4.Dry for 5 seconds,<br>5. Apply adhesive in a uniform thin layer,<br>6. Light cure for 30 seconds. | Etchant: 37.5% H3PO4 FL Prime: HEMA, GPDM,<br>water, ethanol, CQ, BHT FL Adhesive: Bis-GDMA, CQ, ODMAB, filler (fumed SiO2, ba aluminoborosilicate, Na2SiF6), coupling (approximately 48wt% filled) |
| Clearfil SE Bond<br>late (Kuraray Co, Japan)<br>ethanol – P<br>drophilic | 1. Apply primer for 20 seconds.<br>2. Use of dry air<br>3. Apply adhesive<br>4. Light cure for 10 seconds. | Primer: 10- MDP, 2 HEMA Hydrophilic dimetha-cry<br>comphorquinone N,N-diethanol – P-toluidine<br>Water<br>Bond: 10- MDP,2 HEMA Bis – GMA N,N di toluidine Silanated colloidal silica (10)% Hy di-methacrylate<br>DL-Camphorquinone |
| Alumina Powder<br>(Heinrich, Germany) | Put into powder jar and press the operating button | $Al_2O_3$ with the size of 50µ |
| Bio-active Glass Powder<br>(Nova Bone, USA) | Grinded into particles less than 50µ<br>P2O5 then put into powder jar<br>and press the operating button | 45% SiO2, 24.5% Na2O, 24.5% CaO, 6% |
| Clearfil SE AP-X composite resin<br>(Clearfil AP-X,A3, Kuraray, Japan) | Put in a plastic mold and cure for 40 seconds | Bis GMA<br>TEG DMA<br>Silanated barium glass filler<br>Silanated silica filler<br>Silanated colloidal silica<br>DL-Camphorquinone<br>Catalysts<br>Accelerators<br>Pigments |

Different magnifications were used to prepare SEM images at a distance of 20 mm. An accelerating voltage of 15.0 kV was applied during the analysis.

## RESULTS

The shear bond strength (SBS) values in MPa (mean ± SD), minimum/maximum values and 95% confidence intervals (CI) for the groups are presented in Table 2. ANOVA did not show any significant differences between alumina and bioactive glass air abrasion techniques (P=0.660; Table 2).

Three-way ANOVA revealed that bond strength values were not influenced by the ‘‘air abrasive material’’ (f=2.443; P=0.121), ‘‘adhesive system’’ (f=0.337; P=0.563) or “enamel surface condition” (f=0.619; P=0.443).

Moreover, the interactions among the three variables were not significant either (f=0.223; P=0.638).

The fracture modes are presented in Table 3. According to the results, the frequency of cohesive failures was higher in enamel groups abraded with alumina.

**Table 2.** Bond Strength of the Specimens in different groups

| Groups | Group Definitions | Mean | CI 95% | | | | |
|---|---|---|---|---|---|---|---|
| | | | SD | LB | UB | Min | Max |
| **1** | Decalcified Enamel Alumina Abraded ,CSEB | 12.97 | 5.38 | 10.10 | 15.83 | 7.39 | 22.94 |
| **2** | Decalcified Enamel Bioactive Abraded, CSEB | 15.66 | 4.35 | 12.57 | 18.76 | 9.14 | 24.09 |
| **3** | Decalcified Enamel Alumina Abraded, OFL | 14.13 | 5.03 | 11.54 | 16.73 | 4.41 | 22.37 |
| **4** | Decalcified Enamel Bioactive Abraded, OFL | 17.88 | 5.92 | 14.79 | 20.97 | 8.6 | 26.48 |
| **5** | Sound Enamel Alumina Abraded, CSEB | 14.38 | 6.05 | 11.61 | 17.14 | 5.72 | 24.69 |
| **6** | Sound Enamel Bioactive Abraded, CSEB | 14.82 | 4.79 | 11.96 | 17.68 | 7.70 | 23.29 |
| **7** | Sound Enamel Alumina Abraded, OFL | 14.33 | 5.84 | 11.65 | 17.00 | 5.8 | 28.29 |
| **8** | Sound Enamel Bioactive abraded, OFL | 13.88 | 5.50 | 10.79 | 16.97 | 5.61 | 24.62 |

Abbreviation: CI, confidence interval; LB, Lower Bound; UB, upper bound; OFL, Optibond FL, CSEB, Clearfil SE Bond

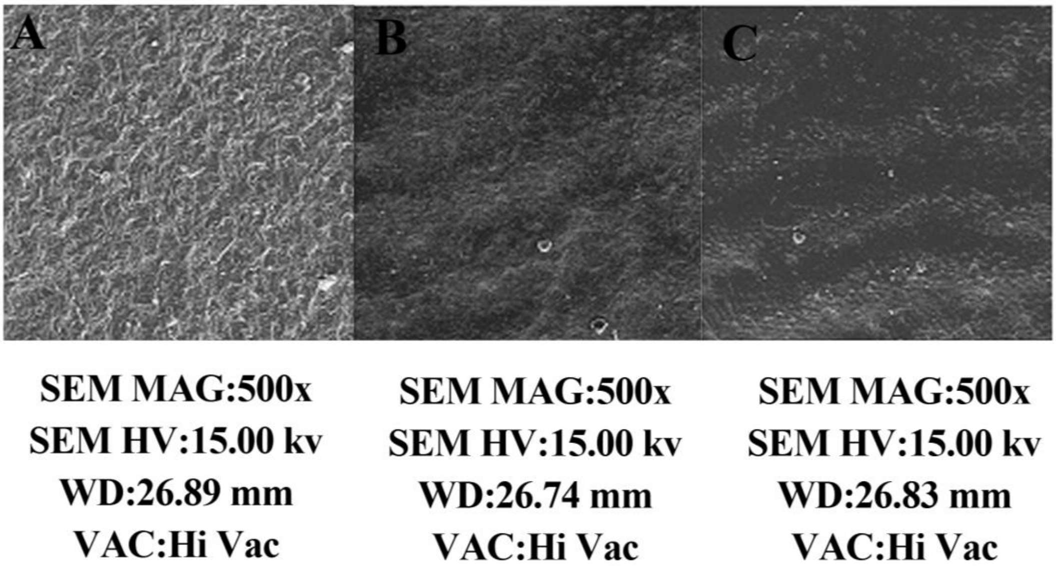


**Fig 1.** SEM micrographs of decalcified enamel: A) Alumina abraded B) Bioactive glass abraded C) No abrasion (Original magnifications: X500)

Principles of conservative dentistry insist that tooth structures should be preserved as much as possible.

Air abrasion technique is used to remove carious lesions and has some advantages including conservative removal of carious tooth structure and cavity preparation with no noise, vibration or pain, with rounded internal and cavo-surface angles [19].

Conventionally, alumina which is an abrasive material is used in the air abrasion technique [20, 21]. Some investigators have recently attempted to use bioactive glass powder as an abrasive material, reporting that bioactive glass air abrasion eliminates carious lesions more selectively compared to alumina air abrasion technique [15].

In addition, in high-risk patients with deep occlusal fissures, it is prudent to place a prophylactic resin restoration. In many clinical situations, decalcified enamel is found in deep fissures [6, 14]. Bioactive glass air abrasion does not remove superficial layers of enamel lesions that are rich in fluoride and more resistant to acid attacks [15].

In the present study, no significant differences were observed between alumina and bioactive glass air abrasion in shear bond strength of composite resin. However, group 4, in which bioactive glass air abrasion technique was used on decalcified enamel, exhibited the highest mean SBS among the study groups. It has been suggested that particles used in bioactive glass air abrasion technique can penetrate into the tooth structure and exchange ions with the tooth surfaces; this might be responsible for the higher bond strength [18]. Banerjee et al. [15] suggested that bioactive glass air abrasion technique has the capacity to selectively remove decalcified enamel, without removing the sound enamel. Demineralized enamel has greater porosity and is softer compared to sound enamel; therefore, its mechanical properties are compromised and there are ample reasons to be more selective during bioactive glass air abrasion.

Also, bioactive glass air abrasion completely removes the demineralized enamel from the lesions, without clinically significant over-preparation of sound tooth structure [15].

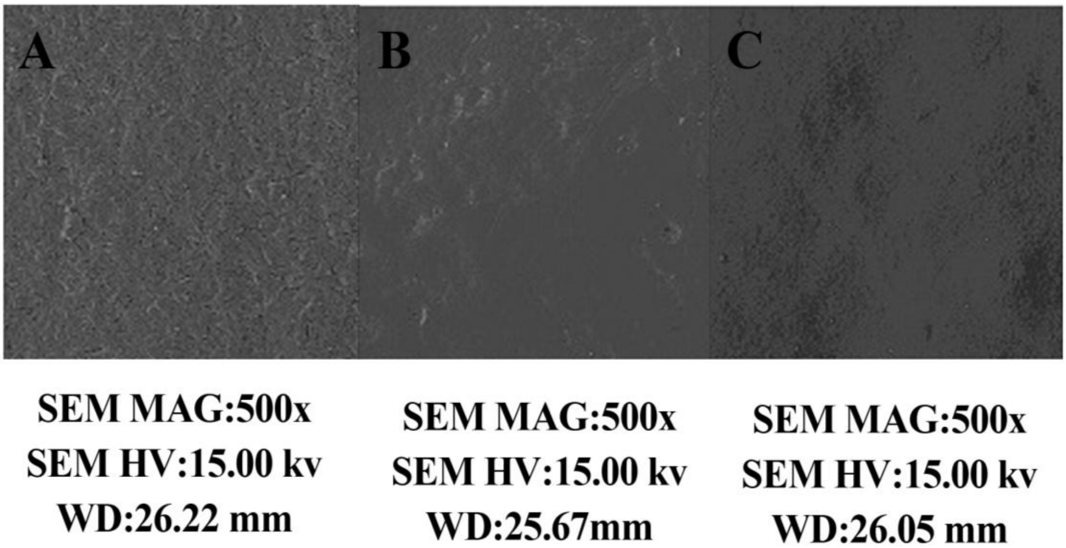


**Fig 2.** SEM micrographs of sound enamel: A) Alumina abraded B) Bioactive glass abraded C) No abrasion (Original magnifications: ×500)

SEM photomicrographs have revealed that the surface roughness produced by alumina technique is more specific compared to that produced by bioactive glass technique. However, according to the results of our study in relation to bond strength, application of bioactive glass as an abrasive material in air abrasion technique does not exert a deleterious effect on the bond strength of composite resin. Therefore, given its selective action in removing carious lesions [15, 21, 22], it might be considered superior to alumina. A study by Paolinelis et al. [18] showed that bioactive glass has the capacity to cut carious dentin more slowly than alumina powder. Saoro et al. reported that abrasion procedures with bioactive glass, along with polyacrylic acid, might improve the longevity of the bond and the healing potential of resin-modified glass-ionomer bonded to dentin [7]. Recent years have witnessed a growing interest in one-step adhesive systems as they do not have separate acid etch and bonding steps. These systems typically use methacrylated phosphoric acid esters; subsequent to application to enamel surface, the phosphate group dissolves and removes calcium ions from hydroxyapatite crystals and becomes incorporated in the network before polymerization of the primer, resulting in neutralization of the acid. In the present study, univariate ANOVA did not show any significant differences in the bond strength of composite resin after the application of three-step etch-and-rinse and two-step self-etch adhesive systems, consistent with the results of a study by Peutzfeldt, [23] who reported that self-etch adhesives might be suitable alternatives to the acid-etch technique in fissure sealant therapy.

On the other hand, a study by Sengun et al. [24] showed that three-step adhesive systems yield higher bond strength values compared to self-etch adhesive systems. Theoretically, an additional etching procedure can increase the bond strength of composite resin since more porosity is produced. On the other hand, it has been suggested that air abrasion increases the surface area available for adhesion, results in formation of more resin tags [25] and increases the bond strength values [25, 26]. In relation to dentin, a thinner smear layer is left after air abrasion [27] and while air abrasion does not negate the need for an etching step, the acid might easily penetrate into the resultant thinner smear layer [1, 28].

A decrease in the thickness of the smear layer might promote formation of the hybrid layer and resin tags with the use of self-etch adhesives, [29] which have been reported to give rise to a thinner hybrid layer [30]. In the present study, air abrasion with alumina or bioactive glass on sound or demineralized enamel did not result in significant differences in bond strength of composite resin. Baysal et al. [31] studied the effect of microabrasion and casein phosphopeptide-amorphous calcium phosphate on the shear bond strength of orthodontic brackets to demineralized enamel, reporting a higher bond strength in groups without demineralization compared to groups with demineralized enamel and without any pretreatment.

In addition, Attin et al. reported a lower bond strength of orthodontic brackets to demineralized enamel [32].

The differences between these two studies and the present study might be attributed to differences in enamel demineralization techniques and also different pretreatment protocols after demineralization. However, Zhang et al. [33] carried out an optical profilometric study of surface roughness alterations of enamel during in vitro demineralization, reporting that while the overall tooth surface characteristics do not change after demineralization, fine features of the surface become accentuated, demonstrating an increase in surface roughness [33].

In the present study, air abrasion was carried out continuously for 10 seconds on sound and decalcified enamel. Peruchi et al. [34] reported that use of the conventional tip in continuous mode for 10 seconds resulted in a slight abrasion on the enamel surface (average width and depth of 1116.90 μm and 54.64 μm, respectively).

**Table 3.** Different Fracture Modes in the Study Groups, N(%)

| **Modes of Fracture Total Groups** | **Adhesive** | **Cohesive** | **Mixed** |
|---|---|---|---|
| 1(Alumina Abraded ,SE 12(100%) Decalcified Enamel) | 6(50%) | 1(8.4%) | 5(41.6%) |
| 2(Bioactive Abraded, SE 12(100%) Decalcified Enamel) | 9(75%) | 1(8.4%) | 2(16.6%) |
| 3(Alumina Abraded, OFL 12(100%) Decalcified Enamel) | 6(50%) | 1(8.4%) | 5(41.6%) |
| 4(Decalcified Enamel 12(100%) Bioactive Abraded, OFL) | 8(66.6%) | 1(8.4%) | 3(25%) |
| 5(Alumina Abraded, SE 12(100%) Sound Enamel) | 6(50%) | 1(8.4%) | 5(41.6%) |
| 6(Bioactive Abraded, SE 12(100%) Sound Enamel) | 9(75%) | 0(0%) | 3(25%) |
| 7(Alumina Abraded, OFL 12(100%) Sound enamel) | 5(41.6%) | 1(8.4%) | 6(50%) |
| 8(Bioactive abraded, OFL 12(100%)Sound Enamel) | 11(91.6%) | 0(0%) | 1(8.4%) |

Such a condition is recommended for shallow cuts on tooth surfaces for the removal of resin or organic plugs in sulci and fissures.

On the other hand, Kumar [16] produced enamel lesions by placing the teeth in demineralizing solutions for 96 hours, which resulted in enamel lesions 120–200 µm in depth. As a result, it can be concluded that after air abrasion with alumina, the decalcified enamel was not thoroughly removed and bonding was carried out on decalcified enamel. Since it has been reported that air abrasion with bioactive glass is more conservative and selective compared to that with alumina, [8, 9] less decalcified enamel is removed after air abrasion with bioactive glass. Therefore, the authors of the present study believe that decalcified enamel remained after air abrasion with bioactive glass, as well. However, further studies are required to analyze enamel substrate.

In this study, air abrasion with bioactive glass as an abrasive on sound or demineralized enamel did not result in statistically significant differences in bond strength values of composite resin compared to alumina. In this study, bioactive glass particles (NOVA BONE) with a mean particle size of 50 µ were used after grinding to <50 µ. The pressure of the unit was adjusted at 70 psi and the duration of application was set at 10 seconds. It is recommended that in future studies bioactive glass powder be used with different particle sizes and the variable parameters of air abrasion device be changed. Air abrasion using alumina powder resulted in faster and easier bulk removal of both sound and demineralized enamel. Banerjee et al. reported that even at low air pressures, alumina air abrasion removed stains very effectively from both carious and sound enamel, which was evidenced by an increase in wear at the margins of abraded and non-abraded sound enamel surfaces [8, 15]. The apparent selectivity of bioactive glass might be explained by differences in the physical properties of carious and sound enamel. Demineralized enamel is more porous and softer compared to sound enamel, compromising its mechanical properties. It has been hypothesized that the selectivity of the bioactive glass powder might be explained by the close matching of the physical properties of the abrasive with those of the substrate [15]. According to SEM results, both bioactive glass and alumina air abrasion techniques cause microscopic changes in surface characteristics of the enamel surface in all samples, including the demineralized and sound enamel, consistent with the findings described previously [15, 33]. The results of SEM analyses of the tooth surface in both sound and decalcified enamel revealed more roughness in the samples abraded with alumina. Further investigations are required in relation to the mechanical and biochemical characteristics of enamel bioactive glass air abrasion.

## CONCLUSION

Under the limitations of this study, bioactive glass air abrasion technique resulted in immediate bond strength similar to the conventional method. This technique may be a suitable alternative for preparation of normal and/or decalcified enamel for resin bonding.

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