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Original article

Assessing the bond strength of short fiber composites to dentin using various air abrasion particles

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Abstract

Purpose: This study investigated the bond strength between short fiber-reinforced resin composite (SFC) and dentin following air abrasion with various types of abrasive particles.

Methods: A total of 120 human molars were prepared for a shear bond strength (SBS) test of the resin composite. The teeth were divided into 12 groups (n = 10/group) based on the air abrasion particle used. Half of the groups underwent phosphoric acid etching (10 s) prior to air abrasion, while the other half received no pretreatment. Air abrasion was performed using five types of particles, after which a two-part adhesive (G2 Bond) and SFC were applied on the treated surfaces. The SBS test was performed using a universal testing machine at a crosshead speed of 1.0 mm/min until failure. Two additional specimens from each group were prepared to evaluate the air abraded surface using scanning electron microscopy after being stored in simulated body fluid (SBF) for two weeks. Statistical analysis was performed using two-way analysis of variance and the Tukey test ($\alpha = 0.05$).

Results: The highest SBS values were observed in the control group (18.9 MPa), which did not undergo air abrasion. The use of different air abrasion particles affected the SBS of SFC to dentin ($P < 0.05$), as air abrasion with jet sand in the absence of pre-etching differed significantly from the other Cojet group ($P < 0.05$).

Conclusions: Air abrasion with various particles did not improve the bond strength between the SFC and dentin.

Keywords: Air abrasion, Etching, Shear bond strength

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1. Introduction

Dental caries, one of the most common, preventable, and chronic diseases, is still common in society despite widespread fluoride use, the availability of preventive health services, and improvements in oral hygiene practices[1,2]. Numerous options are available for the treatment of teeth with severe coronal tissue loss, including the following: amalgam, which is no longer used for environmental reasons; full crowns, which cause unnecessary material loss; and resin materials, which are often doubted owing to their low mechanical strength[3,4]. The most common reasons for failure in resin restorations, which are frequently used in dentistry because of their conservative cavity design and superior aesthetic properties, are secondary caries and restoration fractures, both of which are primarily due to polymerization shrinkage, which can lead to interfacial debonding, postoperative sensitivity, and recurrent caries[5]. It is necessary, therefore, to add minerals, such as calcium phosphate (CaP), to the demineralized dentin to promote remineralization[6].

Studies have continuously attempted to improve the mechanical properties of resin composites, which have recently gained popularity[7,8]. Previous studies have evaluated the addition of different particles to resin composites by changing the particle size and percentage[9,10]. Interest has also increased in fiber-reinforced composite (FRC) resins, which were designed in an effort to improve the mechanical properties of FRC without sacrificing the aesthetic properties[11]. Short fiber-reinforced composites (SFCs) are also used to strengthen both the remaining tooth structure and large composite restorations when high-stress restorations such as large mesio-occlusal-distal (MOD) restorations are needed. If a crack develops in the restoration, the material reinforced with short glass fibers resists further cracking[4,12]. Manufacturers specifically recommend the use of flowable SFCs to replace lost dentin, and recent studies have

WHAT IS ALREADY KNOWN ABOUT THE TOPIC?

» *Studies are continuously trying to improve the mechanical properties of resin composites. Additionally, there is limited research available on the effect of different surface treatments.*

WHAT THIS STUDY ADDS?

» *Air abrasion with various particles does not improve the bond strength between short fiber-reinforced resin composite and dentin.*

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shown that flowable SFCs exhibit the highest load-carrying capacity[12,13].

Effective bonding between the restoration and prepared tooth surface is crucial in adhesive dentistry. Although this bond takes the form of micromechanical bonding on the enamel surface, it is more complicated on the dentin surface[11,14]. The etch and rinse and self-etch systems differ based on the removal or modification of the smear layer[15]. Self-adhesive cement systems work by modifying the smear layer of the dentin. During the etching of dentin with acid, the smear layer is removed and the substrate is demineralized by creating a strong micromechanical bond[16,17]. Despite advancements in adhesive systems, the interface between the tooth tissue and restoration remains the weakest point of the restoration[15,18]. In addition to the development of adhesive systems, procedures such as selective dentin etching, air abrasion, the use of matrix metalloproteinase inhibitors, dentin pretreatment with laser, multiple-layer application, and selective laser etching have been utilized in an attempt to improve dentin bonding[19].

Air abrasion, which was developed in the 1940s, did not attract sufficient attention when it was introduced to the market, largely because the restorative materials used at that time required sharp angles, as adhesive dentistry had not yet been developed. Additionally, there were no high power suction tools at that time, making it difficult to evacuate the abrasive powder[14,20]. In air abrasion, abrasive particles hit the tooth at high speed and abrade the tooth structure; however, the most important variable in this system is the abrasive powder used[20,21]. Although alumina particles are the most commonly used abrasive particles, various other particles such as calcium carbonate (CC), glycine, and sodium bicarbonate have also been used for air abrasion[21,22]. Although alumina particles are generally preferred for cutting the tooth surface, other abrasive particles are often used to clean the tooth surface prior to bonding.

Alumina particles are irregularly shaped and approximately 30–90 µm in diameter. Particles larger than 90 µm are more abrasive and are used for cavity preparation, cutting dental tissue, and roughening various dental materials[23]. Additionally, alumina, which is used to remove the smear layer and roughen the enamel and dentin, can strengthen the bond by ensuring better infiltration of the adhesive resin into the dentin[24–26]. Glasses with densities lower than that of aluminum oxide have also been used for air abrasion. The environmental conditions of bioactive glass have been investigated due to its antibacterial properties and ability to promote dentin remineralization[23,27]. Surface modification by coating with bioactive glass particles promotes *in vitro* biomineralization. CC and CaP, which are biocompatible, absorbable, and osteoconductive materials successfully used in the roughening of the titanium implant surface, are also considered good alternatives for the tribochemical coating of the dentin surface[28–30]. These bioactive particles/materials may leach ions to tooth structure and aid in the remineralization of demineralized dentin[31]. However, the question arises: do these particles affect the dentin bond strength?

This study, therefore, aimed to examine the bond strength of flowable SFCs to dentin after air abrasion using different abrasive particles. The null hypotheses were as follows: (1) different abrasive particles do not affect the bond strength of SFC to dentin and (2) the application of acid etching to the dentin surface has no impact on bond strength.

Table 1. Groups tested in the present study

Abrasive particle type (n = 10)	Etching No/Yes	Group abbreviation
Control with no air abrasion	No	C0
	Yes	C1
Cojet sand	No	Co0
	Yes	Co1
Bioactive glass	No	BAG0
	Yes	BAG1
Aluminum oxide	No	AO0
	Yes	AO1
Calcium carbonate	No	CC0
	Yes	CC1
Calcium phosphate	No	CP0
	Yes	CP1

2. Materials and Methods

2.1. Preparation of dentin specimens and experimental design

In this study, 130 human teeth extracted for various periodontal or orthodontic reasons were used to measure bond strength, and all necessary approvals were obtained from the ethics committee (Approval number: 5/17 22.02.2017). The extracted teeth were stored in chloramine solution at 5 °C and randomly divided into 12 groups of 10 teeth. All teeth were ground flat (Federation of European Producers of Abrasives [FEPA] grit 180/320) using a grinding machine (Struers RotoPol 11; StruersA/S, Rodovre, Denmark) under water cooling just below the enamel-dentin junction. The teeth were then embedded with their occlusal surfaces facing outward in acrylic resin cylinders 4 mm in diameter and height. Finally, silicon carbide (SiC) abrasive paper (FEPA grit 600) was used to obtain a standard smear layer. Any teeth exhibiting visible pulp exposure or cracks were excluded and replaced, resulting in 130 specimens available for testing. The groups and their abbreviations are listed in **Table 1**, while the materials used in this study are listed in **Table 2**.

We etched half of the teeth for 10 s using phosphoric acid to remove the smear layer prior to air abrasion. The dentin surface was washed for 20 s and gently dried, and air abrasion was performed using a Cojet system (3M Espe, St Paul, MN, USA) at a pressure of 400 kPa. After each air abrasion test, the specimens were rinsed with deionized water.

A total of 20 intact human molars (n = 2/group) were selected for evaluation. Their roots and occlusal enamel were removed under water cooling, after which 3 mm thick dentin discs were obtained by grinding the dentin surface with 600 grit SiC sandpaper. Surface treatments were applied to half of the dentin discs (**Table 1**), while the other half was covered with paper (no air abrasion) to serve as a control. After air abrasion, the samples were rinsed with running water and gently air-dried before immersion in simulated body fluid (SBF). The prepared dentin discs were stored in an incubator at 37 °C for 2 weeks in SBF and then prepared as described by Kokubo and Takadama[32] for scanning electron microscopy (SEM) analysis. When removed from the SBF, the samples were rinsed under running water and dried in an incubator for 2 days.

Table 2. Materials used in this study (manufacturer or published data)

Materials	Manufacturer	Composition (wt%)
G2-Bond Universal (pH = 1.5)	GC, Tokyo, Japan	Primer: 4-MET, MDP, MDTP, dimethacrylate monomer, acetone, water, photoinitiator, filler Adhesive: dimethacrylate monomer, bis-GMA, filler, photoinitiator
everX Flow (dentin shade)	GC, Tokyo, Japan	resin (Bis-MEPP, TEGDMA, UDMA) Glass fibers (25), Barium glass (42–52), Silicon dioxide (Trace)
Phosphoric acid etching gel	3M ESPE, MN, USA	Silica thickened 35% phosphoric acid gel (pH = 0.5)
Aluminum oxide	BEGO, Bremen, Germany	99.6% aluminum oxide particles Particle size: 30–90 µm
Cojet sand particles	3M ESPE, MN, USA	95% silica-modified Al_2O_3 Particle size: 30 µm
Bioactive glass	Schott, Mainz, Germany	45.0% SiO_2, 24.5% CaO, 24.5% Na_2O, 6.0% P_2O_5 Particle size: 4 µm
Calcium phosphate ($Ca_3(PO4)_2$)	Chempur, Piekary Śląskie, Poland	≥ 90.0% ($Ca_3(PO_4)_2$) Particle size: < 50 µm
Calcium carbonate ($CaCO_3$)	Sigma Aldrich Darmstadt, Germany	≥ 99.0% $CaCO_3$ Particle size: < 50 µm

2.2. Bonding procedures and resin composite build-up

A commercially available adhesive system (G2-Bond Universal; GC, Tokyo, Japan) was used for all specimens according to the manufacturer's instructions. SFC (everX Flow Dentin; GC, Tokyo, Japan) was then applied to the dentin surface using plastic tubes with a diameter of 3.6 mm and a height of 4 mm. The SFC was then light-cured through the tube (mold) from the top and sides for 60 s (Elipar TM S10; 3M ESPE, St. Paul, MN) with an irradiance of 1200 mW/cm^2. The plastic mold was carefully removed and specimens were stored for 48 h in 37 °C water before testing.

2.3. Bond strength test and failure modes

To maintain stability during testing, the specimens were placed in an apparatus (Bencor Multi-TShear Assembly; Danville Engineering Inc., San Ramon, CA, USA) and loaded into a universal testing machine (Lloyd Instruments Ltd., Fareham, Hants, UK) with a circular cutting tip. The bond strength was determined in shear mode at a crosshead speed of 1.0 mm/min until fracture occurred. Shear bond strength (SBS) was calculated using a computer program (Nexygen 4.0; Lloyd Instruments Ltd., Fareham, Hants UK) which divided the maximum load at failure (N) by the bond area (mm^2), with the result recorded in megapascals (MPa). Adhesive and cohesive failure modes were determined using a light microscope (Stereomicroscope; Wild M3B, Heerbrugg, Switzerland).

2.4. SEM analysis

SEM (LEO, Oberkochen, Germany) was also utilized in this study. The specimens prepared for SEM analysis were dried in an incubator and coated with gold using a sputter coater (BAL-TEC SCD 050 Sputter Coater; Balzers, Liechtenstein). The SEM images were obtained at 20 kV acceleration voltage using 12 mm working distance in vacuum.

2.5. Statistical analysis

The distribution of the SBS values (MPa) was analyzed and found to be homogeneous. Therefore, a two-way analysis of variance (ANOVA) and Tukey's test were used to analyze the data. Statistical significance was determined using Tukey's post-hoc test. A P-value < 0.05 was considered to be statistically significant (SPSS version 23; IBM Corp., Chicago, IL, USA).

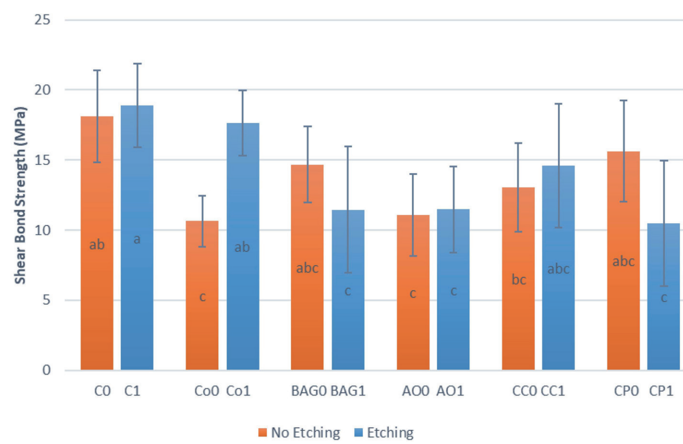

Fig. 1. Mean shear bond strength values to dentin and standard deviations. Groups with different letters have statistically significant differences among each other (significance level of $P < 0.05$).

3. Results

The mean SBS values for dentin and the standard deviations for all tested groups (MPa) are shown in **Figure 1**. There was no statistically significant difference between the etched and non-etched groups ($P = 0.745$); however, there were statistically significant differences among the groups where different air abrasion particles were utilized ($P < 0.05$). ANOVA revealed a statistically significant interaction between etching and air abrasion ($P < 0.05$) (**Fig. 1**).

The highest bond strength values among were observed in the C1 group (18.9 MPa), which did not undergo air abrasion, while the lowest values were observed in the CP1 group (10.49 MPa). The bond strengths of the BAG0 and CP0 groups were lower than those of the BAG1 and CP1 groups. Among all other groups, the bond strength

Table 3. Results of failure types

	Cohesive (dentin)	Adhesive
No treatment	10	0
No treatment with etching	8	2
Cojet	4	6
Cojet with etching	4	6
BAG	9	1
BAG with etching	5	5
Aluminum oxide	5	5
Aluminum oxide with etching	6	4
Calcium carbonate	4	6
Calcium carbonate with etching	9	1
Calcium phosphate	10	0
Calcium phosphate with etching	2	8

values were higher in the acid-treated groups.

A statistically significant ($P < 0.05$) difference was observed between the group that had no surface treatment (control), including etching, and those that had only air abrasion with the CoJet system using aluminum oxide. Furthermore, a statistically significant difference was observed between the group that had no surface treatment and those that received air abrasion with BAG1, AO1, or CP1 ($P < 0.05$). Additionally, the SBS values were ranked as follows: no air abrasion treatment (18.48 MPa) > Cojet (14.35 MPa) ≥ calcium carbonate (CC) (13.82 MPa) ≥ calcium phosphate (CaP) (13.07 MPa) ≥ bioactive glass (BAG) (13.06 MPa) ≥ aluminum oxide (AO) (11.29 MPa).

The results of the failure analyses are presented in **Table 3**. All specimens in the untreated and CaP-treated groups showed cohesive fractures (dentin). Among the groups that underwent air abrasion with CC, the pre-etching group showed more cohesive fractures, while the non-etching group showed more adhesive fractures.

The SEM results are shown in **Figure 2**. Dentinal tubules were observed in the etched specimens while the smear layer was observed in the unetched specimens. When air abrasion was performed after etching, the opened dentinal tubules were clogged with particles. The amount and shape of irregular mineral accumulation varied depending on the structure of the applied particles. Besides, more mineral deposition was observed in the groups subjected to air abrasion with AO than in the other groups. A calcified layer was visible in the groups where heavy abrasion was performed with BAG; however, it is difficult to call this layer an organized mineral layer. Non-homogeneous mineralized groups were also observed in the CP1 group, while in the CC1 group mineralized foci that persisted inside the tubules were observed.

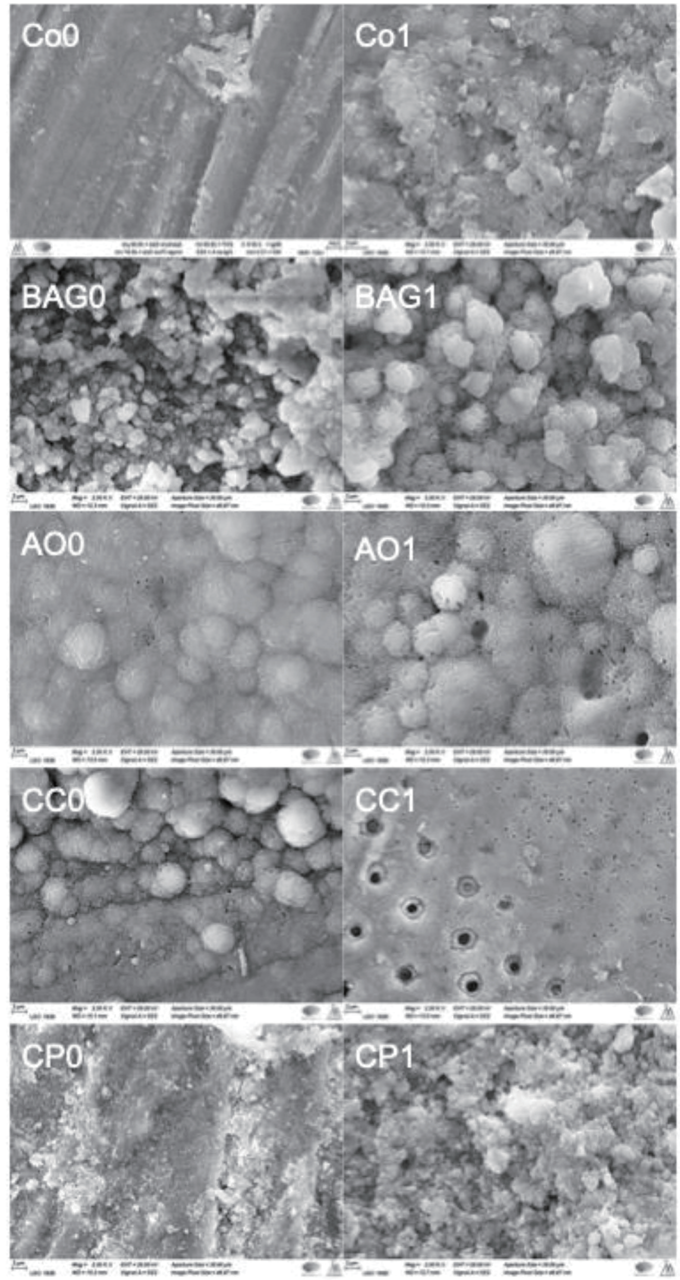

Fig. 2. Scanning electron microscopy images of samples kept in simulated body fluid for 2 weeks after air abrasion (2.50 K magnification)

4. Discussion

Despite recent advancements in adhesive systems, studies continue to evaluate the bond between the resin and dentin interface[19]. In this study, two different adhesive protocols and five different abrasive particles were used to condition dentin surfaces, after which the bond strength between SFC resin composites and the dentin were examined.

Adhesive systems are an important factor in the success of restorations, and self-etching adhesive systems that do not require phosphoric acid etching are widely used at this time[33]. The manufacturer of one of the newest adhesives, G2-Bond Universal (G2-B; GC, Tokyo, Japan), has stated that the Dual H-Technology reduces the risk of degradation and provides superior durability[34]. Additionally, this technology facilitates the transition from a hydrophilic to hydrophobic state and enhances the bond between the dental tissue and resin composite[33]. 2-hydroxyethyl methacrylate (HEMA), which is used in most adhesives to increase adhesion to dentin, is not used in G2-Bond Universal, which instead uses 4-methacryloxyethyl trimellitic acid (4-MET) 10-methacryloxydecyl dihydrogen phosphat (10-MDP), and methacryloyloxydecyl dihydrogen thiophosphate (MTDP) as functional monomers[35]. Owing to the HEMA-free composition, the bonding layer was hydrophobic, which reduced the possibility of water absorption, and subsequently, the risk of adhesive deterioration. Studies have shown that G2-Bond Universal is the most effective universal adhesive for clinical applications, especially when applied in self-etch mode[33].

Previous studies have reported that the surface preparation can directly affect the bond strength between the resin composite and dentin[36,37]. In accordance with previous studies, different abrasive particles were found to reduce the bond strength to dentin in the present study; therefore, the first null hypothesis, that different abrasive particles do not affect the bond strength of SFC to dentin, was rejected. Additionally, the protocols used in this study were insufficient to provide the desired dentin surface preparation, which should be considered in clinical practice.

The reason why the bond strength between the resin composite and dentin was negatively affected was because abrasive particles may create undesirable topographies on the dentin surface. For example, microscopic cracks can be created by abrasive particles on dentin, and dentin minerals may be lost[38]. These effects render the dentin surface unsuitable for adhesive bonding, resulting in a decreased bond strength.

The factors that affect the amount of dental tissue loss using the air abrasion method are the particle type, air pressure, and speed of the abrasive particles, which are determined by the distance between the tip of the Cojet and the tooth surface[21,39]. Cojet sand particles, one of the air abrasion particles used in this study, are 30µm spherical silica-coated aluminum oxide particles. In a previous study, however, 50 µm Al_2O_3 particles with a sharp-edged surface were also tested, and it was observed that increasing the particle size and treatment period might result in the degradation of restorative materials[40]. While the BAG particles used in the present study were 4 µm in size, the size of the CC and CaP particles was < 50 µm in size.

In this study, a statistically significant difference was observed between the group without air abrasion and the group subjected to air abrasion with Al_2O_3, which may be explained by the fact that aggressively abrasive Al_2O_3 particles may cause significant mineral loss from the dentin surfaces. In a previous study, Rinaudo *et al.*[41] reported that Al_2O_3 particles accumulated on the dentin surface as a result of air abrasion, negatively affecting the bond strength In another recent study using Cojet sand particles, conditioning the dentin surface in this manner did not have a significant effect on bond strength[42]. The results of the present study, therefore, are partially consistent with those of previous studies. No significant differences were observed between the etching groups after the air abrasion process and the control group; however, the bond strength of the unetched group was lower than that of the control group. This can be explained by the fact that the Cojet particles were not homogeneously distributed in the dentin during air abrasion. In a meta-analysis, Lima *et al.*[43] reported that air abrasion should be utilized only when the particle size is larger than 30 µm and the pressure is > 5 bar.

Because of their antibacterial properties and remineralization potential, bioactive glasses are good alternatives for air abrasion[44]. They have also been reported to be less corrosive than Al_2O_3 and may help prevent sensitivity by blocking dentinal tubules[27,45]. Sauro *et al.*[46] reported that air abrasion with bioactive glass does not negatively affect the bond strength. Similar to the results of the aforementioned study, no statistically significant difference was observed between the air abrasion with bioactive glass without acid etching group and the control group in the present study.

CC, which a laboratory study indicated has the potential to erode the enamel surface, was reported to be a less abrasive particle than alumina despite its abrasive potential[47,48]. Additionally, CaP-based materials have the potential to stimulate dentin remineralization. Bioactive glasses are biomaterials that contain CaP in their composition[6]. Because sodium bicarbonate, which was the first particle marketed for air abrasion, is highly corrosive and destructive, CC was one of the products introduced to replace it[49].

In a previous study, Frankenberger *et al.*[50] used sodium bicarbonate particles for air abrasion and reported that this application weakened the bond strength. Researchers have attributed this to the fact that sodium bicarbonate particles may cause excessive damage to dentin tissue. Similar to the results of our study, Tamura *et al.*[51] stated that the bond strength of the air abrasion groups to dentin was significantly lower than that of the control group. This difference may be attributed to the sodium bicarbonate powder remaining on the dentin surface, which may cause mechanical and chemical changes in the collagen fibrils, preventing adhesive resins from penetrating the dentin. The application of acid etching on dentin surfaces yielded different outcomes based on the adhesive particles, thus rejecting the second hypothesis that the application of acid etching to the dentin surface has no impact on bond strength. Although the application of etching enhanced the bond strength in the control group and the group in which Cojet particles, AO, and CC were utilized, this effect was not statistically significant, except in the group using Cojet particles. Conversely, etching adversely affected the bond strength in the groups that underwent air abrasion with BAG and CaP.

A previous study by Sellan *et al.*[52] demonstrated that a CaP-based desensitizer was effective in reducing dentin permeability and increasing the adhesive-dentin bond strength when combined with laser treatment. Similarly, another study demonstrated that a 1-min pretreatment with metastable CaP on etched dentin collagen fibrils can achieve biomimetic remineralization and increase microtensile bond strength[53]. To the best of the authors' knowledge, there is limited research available on the effects of different surface treatments, such as a combination of acid etching followed by air abrasion with different particles, on dentin bond strength. However, the results of the present study may be explained by the fact that the BAG and CaP particles are softer than the jet particles; therefore, the use of these particles may reduce the impact of acid etching. Additionally, the accumulation of these materials on the surface may block the dentinal tubules, which may negatively affect the bond strength between the resin and dentin.

The smear layer exposed during tooth preparation is a complex structure composed of collagen fibrils, organic residues, and mineral particles. Sauro *et al.*[54] measured dentin permeability after air abrasion and detected the presence of a compact multilayer smear layer on dentin surfaces. Additionally, a previous study showed that the smear layer was compacted after air abrasion with alumina particles on the dentin surface. This layer may weaken the bond strength by preventing the penetration of functional adhesive monomers[55]. Therefore, in half of the groups prepared in our study, the smear layer was removed by acid etching prior to air abrasion in an attempt to avoid the negative effects of air abrasion on the smear layer and observe the effects of the particles used on dentin composition and bond strength. Phosphoric acid application aims to remove preparation residues lodged within the dentinal tubules[56], but also removes the smear layer and demineralizes superficial hydroxyapatite crystals[57]. In the SEM images obtained in this study, dentinal tubules exposed only in the group subjected to etching were observed in a manner consistent with the existing literature. Following pre-etching, more particles accumulated in the group treated with the Cojet through air abrasion than in the group without etching. This difference may be attributed to the etching process, which rendered the exposed dentinal tubules more susceptible to particle accumulation. In the groups treated with BAG and CaP, the increased accumulation of irregularly mineralized tissue was observed on the dentin surface than in the other groups. In a study in which air abrasion was performed with sodium bicarbonate and glycine, it was reported that the remaining sodium bicarbonate powder on the dentin surface could affect the chemical and mechanical structures of the collagen fibrils, potentially impeding the penetration of the adhesive into the fibrils[51].

Materials used in the oral cavity are exposed to the dynamic conditions of the mouth. A significant limitation of our study was that the dynamic and thermal conditions within the oral cavity were not reflected in the experimental conditions. Another limitation was the lack of consideration of long-term usage conditions. Future studies evaluating dentin conditioning using the air-abrasion method should consider the dynamic and thermal conditions of the oral cavity; however, the cytotoxicity of these particles require further evaluation.

5. Conclusions

Air abrasion with various particles did not improve the bond strength between the SFC and dentin. Additionally, removing the smear layer prior to air abrasion did not affect the bond strength.

Acknowledgments

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Conflict of interest

There are no conflicts of interest to declare regarding this study.

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