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#### ORIGINAL ARTICLE

### Dentistry

# Shear Bond Strength of Two Self-Etching Adhesives to Air-Abraded Dentin: An in Vitro Study

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#### **Abstract**

**Background**: The aim of this research was to study the effect of air-abrasive treatment of dentin on the chemical composition of its surface and the adhesion strength of 2 self-etching adhesive systems (AS).

Methods and Results: Powders based on aluminum oxide (Al<sub>2</sub>O<sub>2</sub>) (27μm) (KaVo, Biberach, Germany), sodium bicarbonate (NaHCO3) (40µm) (AIR-FIOW Classic Comfort, EMS, Nyon, Switzerland), and erythritol (14µm) (AIR-FLOW Plus, EMS, Nyon, Switzerland) were used for the air-abrasive treatment of adhesive surfaces. Bonding steps were carried out with Single Bond Universal (SBU) (3M ESPE, USA) and Bond Force 2 (BF2) (Toquyama, Japan). The adhesion strength of composite to dentin was evaluated on 80 samples prepared in accordance with the Ultradent Shear Bond Strength test. All samples were divided into 4 groups depending on the method of dentin surface processing. In the samples of Group 1 (n=20), aluminum oxide was used for the air-abrasive treatment of dentin. In Group 2 (n=20) and Group 3 (n=20), samples were treated using powders based on sodium bicarbonate and erythritol, respectively. Group 4 (control, n=20) included tooth samples in which the dentin surface was not airabraded after preparation with carbide burs. Then, each group was divided into 2 subgroups (Sub-A and Sub-B) depending on the type of adhesive system used. Adhesive resin was applied and polymerized in accordance with the manufacturer's instructions. Single Bond Universal (SBU) was used for the samples of Sub-A, and Bond Force 2 (BF2) (Toquyama, Japan) was used for the samples of Sub-B. Scanning electron microscopy and determining the surface elemental composition of samples were carried out on an SEM-EVO MA 10 (Carl Zeiss) and energy dispersive X-ray spectrometer with EDS Aztec Energy Advanced X-Act (Oxford Instruments). It was concluded that air-abrasive treatment of the dentin surface does not enhance the adhesion strength of composite material when using self-etch AS. Also, it was noted that the pH level of self-etch AS is not a crucial feature in determining the strength of the filling-tooth interface. The resulting variations in the elemental composition of dentin surface after air-abrasion with various mixtures and their effect on the efficacy of the different AS require further in vitro studies.(International Journal of Biomedicine. 2022;12(4):591-595.).

Keywords: air-abrasion • adhesive systems • dentin surface

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#### **Abbreviations**

AA, air-abrasion; AS, adhesive systems; SBU, Single Bond Universal; BF2, Bond Force 2.

#### Introduction

Modern trends in minimally invasive cavity preparation, combined with the advent of self-etching adhesive systems (AS), have conceptually changed the philosophy of dental caries operative treatment.<sup>(1-5)</sup> Alternative methods of tooth

preparation allow performing dental care, considering the microscopic surface features of enamel and dentin. (6-11)

Removing biofilm and the smear layer without a clinically significant impact on hard tooth tissues became possible due to the introduction into the practice of low abrasive powders based on glycine, erythritol, trehalose, etc. (12-14) At the same

time, the variety of clinical situations and increasing complexity of comprehensive tooth care unfortunately do not allow the complete abandonment of coarse-grained air-abrasive mixtures based on aluminum oxide, sodium bicarbonate, calcium carbonate, and many other substances. (15-17) Recent attempts have also been made to combine the removal of stains and tissue debris of the smear layer with a guided biofilm therapy and the formation of a bioactive layer on the surface of the tooth to promote accelerated remineralization of dentin and enamel weakened by the carious pathology. (18,19)

Therefore, the current techniques of tooth surface preparation with consideration of the features of adhesive and restorative materials can contribute to reliable and long-term restorations.

The modern concept of simultaneous etching and hybridization of hard tooth tissues is another step toward minimally invasive dentistry, and it was embodied in AS of the sixth and seventh generations. (20,21) It is important to emphasize that one of the bad features of total-etch AS is considered to be incomplete infiltration by monomers of microgaps created after etching with phosphoric acid in the surface layers of enamel and dentin. (22,23) Moreover, moisture in the demineralized extracellular matrix of dentin causes gel polymerization of Etch&Rinse adhesive monomers resulting in the formation of hydrolytically unstable polymers in a hybrid layer. (24,25)

The above and many other shortcomings related to total-etch AS were considered when a new group of self-etching adhesives based on acidic monomers was introduced. The positive aspects of these adhesives are the absence of mandatory acid etching of dentin and the formation of a chemical bond between the hydroxyapatite of hard tooth tissues and adhesive resin monomers. (26,27) Also, the chemistry and mode of application of one-step bonding materials imply the incorporation of a smear layer into the structure of the adhesive layer. (28)

In this regard, it can be assumed that the composition and amount of the smear layer may affect the bond strength of the composite to tooth dentin when using self-etching adhesives.<sup>(29)</sup>

It is known that air-abrasion of prepared dentin can open a significant number of orifices of dentinal tubules and facilitate the formation of a thinner smear layer. (30) However, considering the experience of previous studies, this method may cause changes in the chemical composition of dentin surfaces and affect the strength of composite adhesion. (31,32)

The aim of this research was to study the effect of airabrasive treatment of dentin on the chemical composition of its surface and the adhesion strength of 2 self-etching adhesive systems (AS).

#### Materials and Methods

Powders based on aluminum oxide ( $Al_2O_3$ ) (27µm) (KaVo, Biberach, Germany), sodium bicarbonate (NaHCO3) (40µm) (AIR-FlOW Classic Comfort, EMS, Nyon, Switzerland), and erythritol (14µm) (AIR-FLOW Plus, EMS, Nyon, Switzerland) were used for the air-abrasive treatment of adhesive surfaces. Bonding steps were carried out with Single Bond Universal (SBU)

(3M ESPE, USA) and Bond Force 2 (BF2) (Toquyama, Japan). The adhesion strength of composite to dentin was evaluated on 80 samples prepared in accordance with the Ultradent Shear Bond Strength test.

To obtain a uniform smear layer, the exposed tooth surfaces on all samples were processed with carbide burs under constant water cooling and then washed with water spray for 30 seconds using an air-water syringe of a dental unit.

All samples were divided into 4 groups depending on the method of dentin surface processing. In the samples of Group 1 (n=20), aluminum oxide was used for the air-abrasive treatment of dentin. In Group 2 (n=20) and Group 3 (n=20), samples were treated using powders based on sodium bicarbonate and erythritol, respectively. Group 4 (control, n=20) included tooth samples in which the dentin surface was not air-abraded after preparation with carbide burs. In each case of air-abrasion, the nozzle of the handpiece was angulated at 45° to the dentin surface. The treatment was carried out with a constant flow of particles under 0.25MPa pressure for 30 seconds, slowly moving a nozzle with sweeping motions above the surface of tooth samples at a distance of 5mm, after which they were thoroughly washed with an air-water spray for 30 seconds.

Then, each group was divided into 2 subgroups (Sub-A and Sub-B) depending on the type of adhesive system used. Adhesive resin was applied and polymerized in accordance with the manufacturer's instructions. SBU was used for the samples of Sub-A, and BF2 (Toquyama, Japan) was used for the samples of Sub-B. The light-curing composite Herculite XRV (Kerr, Italy) served as a material of choice. The polymerization was carried out using a VALO lamp (Ultradent Products Inc., USA) in a standard mode. The one-day adhesive strength of bonded interfaces without aging simulation was evaluated on an UltraTester device (Ultradent Products Inc., USA). The speed of movement of the test clamp was set to 1mm/min. The top value of bonding failure was fixed in pounds.

Scanning electron microscopy and determining the surface elemental composition of samples were carried out on an SEM-EVO MA 10 (Carl Zeiss) and energy dispersive X-ray spectrometer with EDS Aztec Energy Advanced X-Act (Oxford Instruments). There were 12 additional tooth samples, which were divided into 3 groups. Each sample had 2 different areas on the dentin surface, which were subjected to air-abrasion and carbide bur (machined) processing. For air-abrasion, in the A/M group (n=4), B/M group (n=4), and E/M group (n=4), alumina particles, bicarbonate, and erythritol mixtures, respectively, were applied.

Statistical analysis was performed using StatSoft Statistica v6.0. For descriptive analysis, results are presented as mean $\pm$ standard deviation (SD). Multiple comparisons were made with one-way ANOVA and post-hoc Tukey HSD test. The Mann-Whitney U Test was used to compare the differences between the two independent groups. A probability value of P<0.05 was considered statistically significant.

#### **Results**

It was revealed that the adhesive strength of composite material to tooth dentin depends on the type of powder used for air-abrasion and the kind of adhesive system applied (Table 1). When compared with the control (Sub-4A), air-abrasion of the dentin surface with powders based on alumina (Sub-1A), sodium bicarbonate (Sub-2A), and erythritol (Sub-3A) did not adversely affect the strength of adhesion when SBU was used.

In the case of BF2, there was no statistical difference between the control (Sub-4B) and Sub-1B. However, we found a statistically significant decrease in the strength of adhesion in samples of Sub-2B by 1.2 times (P=0.012) and Sub-B3 by 1.7 times (P=0.000), compared to Sub-4B.

Changes in the elemental composition of dentin surfaces after air-abrasion with different mixtures were predictable to a certain extent (Table 2). Along with the minor variations in the level of basic elements of dentin, the appearance of aluminum and silicon ions was noted after exposure to powders based on aluminum oxide, sodium bicarbonate, and erythritol.

An increase in Al+ content by 8.3 times was found after air-abrasion of dentin surfaces in samples of the A/M group (P=0.000). Also, Na+ content grew by 1.28 times in the B/M group (P<0.05). The same tendency in Si+ content by 1.5 times was noted in the B/M and E/M samples; however, these changes were not statistically significant (P>0.05).

An unexpected 1.5-times elevation of Mg+ (P<0.05) was registered on dentin surfaces after air-abrasion with erythritol-based powder. However, the level of the element in the E/M group did not significantly differ from the value obtained after dentin treatment with Al<sub>2</sub>O<sub>3</sub> powder in A/M samples. The high content of C+ after dentin surface processing with a carbide bur in samples of the E/M group was assumed for the contamination of scanning areas.

#### **Discussion**

A huge amount of research has been devoted to the problem of integrating dental composite with dentin, which is directly related to a large number of factors that affect the quality of adhesion and hence the longevity of restorations. (33-36) Also, the complex ultrastructure of adhesive surfaces and technical problems associated with AS of fourth and fifth generations can negatively affect the quality of dental treatment when composite materials are applied. (37,38)

That is why the use of self-etching adhesive monomers on dentin appears to be the most promising. (39) However, considering the mode of interaction of these materials with hard tooth tissues, the method of its application, and the presence

Table 1.
Adhesion strength of composite to tooth dentin in groups (lb).

Group/ Subgroup	Group 1	Group 2	Group 3	Group 4	Statistics
Subgroup A	24.57±4.72	21.42±2.03	21.82±4.7	23.53±2.27	F=1.6220 P=0.2013
Subgroup B	15.56±1.72	13.57±2.3	9.9±2.96	16.47±2.35	$\begin{array}{c} F{=}15.0697\ P{=}0.000 \\ P_{1B{-}2B}{=}0.2567\ P_{1B{-}3B}{=}0.0000 \\ P_{1B{-}4B}{=}0.8265\ P_{2B{-}3B}{=}0.0074 \\ P_{2B{-}4B}{=}0.0457\ P_{3B{-}4B}{=}0.0000 \end{array}$
P-value	< 0.0001	< 0.0001	< 0.0001	< 0.0001	

Table 2.

Elemental composition of dentin surfaces after processing with different methods.

	Groups	C <sup>+</sup>	O-	Na <sup>+</sup>	$Mg^+$	Ca <sup>+</sup>	$P^+$	$Al^+$	Si <sup>+</sup>
	Al <sub>2</sub> O <sub>3</sub> abraded	16.02±0.56	41.23±1.45	0.55±0.1	0.75±0.05	28.1±0.67	13.62±0.3	0.25±0.05	0.07±0.05
A/M	P-value	>0.05	>0.05	>0.05	>0.05	< 0.05	< 0.05	0.000	>0.05
	Machined	15.53±0.8	39.45±0.39	0.57±0.052	0.67±0.05	29.4±0.47	14.23±0.21	0.03±0.05	0.05±0.05
B/M	Bicarbonate abraded	15.26±1.04	39.03±0.9	0.87±0.11	0.63±0.05	29.7±0.87	14.16±0.33	0.03±0.05	0.27±0.11
	P-value	>0.05	>0.05	< 0.05	>0.05	>0.05	>0.05	>0.05	>0.05
	Machined	16.25±1.15	38.3±0.66	$0.68\pm0.1$	0.57±0.12	29.87±1.22	14.12±0.57	$0.02\pm0.04$	0.18±0.1
Э/М	Erythritol abraded	16.82±0.65	40±0.49	0.62±0.07	0.82±0.04	27.78±0.31	14.08±0.71	0.02±0.04	0.18±0.07
	P-value	< 0.05	< 0.05	>0.05	< 0.05	< 0.05	< 0.05	>0.05	>0.05
	Machined	20.63±0.61	38.27±0.29	0.55±0.05	0.55±0.05	26.75±0.33	13.07±0.16	$0.07 \pm 0.05$	0.12±0.04

of a smear layer that is amorphous and weakly adhered to the underlying dentin may cause particular concern. (40

In the available literature, there is a sufficient amount of data indicating the effective use of air-abrasive methods concerning cleaning the tooth surface from dentin debris and enamel fragments that appear after traditional preparation with diamond and carbide burs. However, the results of studies that assume to improve the adhesion strength of a composite to abraded dentin may go either way. (18,41-43) In this regard, the main purpose of this study was to evaluate the effect of various air-abrasive mixtures on the adhesion strength of composite to dentin when self-etching AS are used. It is well known that adhesive materials of the sixth and seventh generations create mechanical and chemical bonds with dentin and enamel hydroxyapatite due to the unique properties of acidic monomers. (26,27) That is why, considering the appearance of possible changes on the surface of abraded dentin, a comparative analysis of its elemental composition was also carried out. It was previously noted that self-etching AS might have a different pH, and based on the pH, they are divided into very weak (ultra-mild, pH $\geq$ 2.5), weak (mild, pH $\approx$ 2), medium (intermediate, pH $\approx$ 1.5), and strong (strong, pH≤1). It has also been found that the depth of demineralization of hard tooth tissues directly relates to the pH level of self-etch AS.(44) Hence to reduce the level of subjectivity, we used SBU and BF2 adhesives in the study, which are slightly different from each other in terms of acidity and have pH of 2.7 and 2.8, respectively.

Results of the study demonstrated that in the case of SBU, an air-abrasion of the dentin surface with  ${\rm Al_2O_3}(27\mu m)$ , AIR-FLOW Classic, and AIR-FLOW Plus did not cause a clinically significant variation in values of adhesion strength of composite to dentin.

However, the same processing of the dentin surface led to a significant decrease in bond strength between resin and tooth when BF2 was applied, whereas the differences were significant after abrasion with sodium bicarbonate and erythritol-based powders.

Concerning changes in the elemental composition of the dentin surface after treatment with Al<sub>2</sub>O<sub>3</sub> powder (27µm), a significant increase in Al+ and a decrease in Ca+ and P+ were noted, presumably indicating the decrease in the number of hydroxyapatite crystals in the scanning sectors. However, the revealed changes were not a reason for significant variations in the bond strength values for the SBU and BF2. The accumulation of Na+ on the dentin surface after treatment with AIR-FLOW Classic powder could be one of the possible reasons for the deterioration of composite adhesion when BF2 was used. Also, the most dramatic reduction in composite bond strength with BF2 was observed after dentine treatment with AIR-FLOW Plus despite a relative increase in the surface level of Ca+ and P+. Therefore, it was concluded that air-abrasive treatment of the dentin surface does not enhance the adhesion strength of composite material when using self-etch AS. Also, it was noted that the pH level of self-etch AS is not a crucial feature in determining the strength of the filling-tooth interface. The resulting variations in the elemental composition of dentin surface after air-abrasion with various mixtures and their effect on the efficacy of the different AS require further in vitro studies.

#### **Competing Interests**

The authors declare that they have no competing interests.

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