

# Effect of Dentin Surface Treatment Using a Non-Thermal Argon Plasma Brush on the Bond Strength of a Self-Adhesive Resin Composite

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## Abstract

**Background and Aim:** Improving the bond strength at the resin-dentin interface is an important challenge in adhesive dentistry. This study examined the effect of low-power, non-thermal atmospheric pressure plasma (NT-APP) treatments on the adhesion of a self-adhesive resin composite to dentin.

**Materials and Methods:** In this experimental in-vitro study, buccal enamel of extracted bovine incisors was removed using a high-speed diamond bur. The specimens were randomly divided into two groups according to the plasma treatment and thermocycling. The non-thermal atmospheric argon plasma brush was used in this study. One subgroup was subjected to the microshear bond strength ( $\mu$ SBS) test after 24 hours, whereas the other subgroup was subjected to artificial aging with thermocycling for 5000 cycles before being tested. Each specimen was attached to a testing jig and loaded at a crosshead speed of 1.0 mm/minute in a universal testing machine until failure occurred. Data were analyzed using two-factor repeated measures analysis of variance (ANOVA).

**Results:** The results of  $\mu$ SBS testing showed that with plasma treatment, the average  $\mu$ SBS increased to  $34.20 \pm 12.12$  MPa compared to  $19.47 \pm 7.4$  MPa in the controls ( $P=0.002$ ). After 5000 cycles of thermocycling, the adhesive-dentin bonding strengths of the plasma-treated specimens slightly decreased from  $34.20 \pm 12.12$  MPa to  $33.64 \pm 5.6$  MPa ( $P=0.886$ ), while the strengths of the untreated specimens reduced from  $19.47 \pm 7.4$  MPa to  $19.10 \pm 5.1$  MPa ( $P=0.461$ ). Plasma treatment improved the  $\mu$ SBS compared to the control group. After thermocycling, the  $\mu$ SBS did not decrease in the plasma and control (non-plasma) groups.

**Conclusion:** Plasma treatment using NT-APP improves the adhesion of self-adhesive flowable resin composites to dentin.

**Key Words:** Composite Resins, Non-Thermal Atmospheric Pressure Plasma, Dental Bonding

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## Introduction

Due to their aesthetic properties and noninvasive tooth preparations, the use of composite resin

restorations has significantly increased among dentists and patients [1]. The long-term success of a composite resin restoration depends on the

durability of the bond strength [2]. Most current dental adhesive systems show satisfactory immediate results in terms of the bond strength [3]. However, the adhesive-dentin interface is the weakest link within the adhesion complex of tooth-colored restorations, and the durability of aged resin-dentin adhesion is questionable. Previous long-term studies have shown that the bond strength of resin to dentin decreases over time [3,4]. A new strategy to produce a long-term durable dentin adhesion is needed. Recently, plasma has attracted attention in the biomedical field [5,6].

Plasma is the fourth state of matter and is the most copious state in the universe [7]; it exists in various forms, including fire, which is known for millions of years since the early Stone Age. Plasma is not a human invention and is present in nature as fire in the sun and stars, in the tails of comets, and as flashes of lightning [7]. In medicine and biology, plasma refers to the non-cellular fluid component of blood. The term was introduced into physics by Irving Langmuir in 1928 [7]. Non-thermal plasmas are partly-ionized gases containing exceptionally reactive particles, including electronically-excited atoms, molecules, and ionic and free radical species; the gas phase is stable at room temperature [8,9]. Depending on the plasma chemistry or the composition of the gas, these highly reactive plasma species react with, clean, and etch surfaces, bond with different substrates, or combine to create a thin layer of plasma that changes the surface properties [8,9]. Treatment with non-thermal plasma allows for the modification of dentinal surfaces to improve the interfacial bonding of dentin and composite restorations [10,11]. It has been stated that short-term treatment with plasma could change the chemical structure of exposed collagen fibrils and increase the hydrophilicity of the dentin surface, which allow better penetration of the adhesive into dental collagen fibrils and enhance the adhesive-dentin bond strength [10,11]. Treatment of dentin using a non-thermal plasma brush results in an increase of about 60% in the bonding at the adhesive-dentin interface compared to untreated controls [12,13].

A simplified adhesive protocol to reduce the duration of treatment is highly desirable in adhesive dentistry. A new category of composites

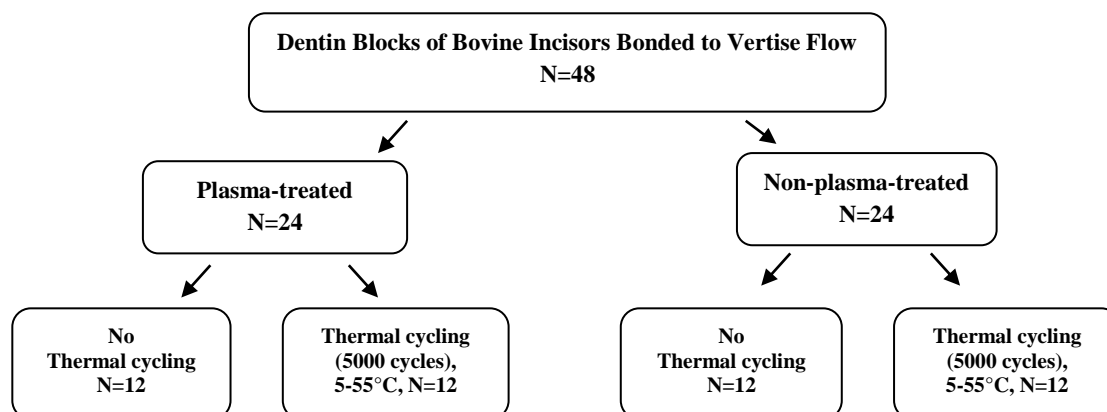
has been introduced; according to the manufacturers' claim, these self-adhesive (flowable) composites do not require any acid-etching or bonding protocol prior to the application [12,13]. Vertise™ Flow self-adhesive composite (Kerr, Orange, CA, USA) comprises phosphoric acid ester methacrylate and glycerol phosphate dimethacrylate (GPDM) as functional monomers [12,13]. The aim of this study was to evaluate and verify the effectiveness of plasma treatment of dentinal surfaces for improving the interfacial bonding of a self-adhesive composite resin and dentin by performing a microshear bond strength ( $\mu$ SBS) test.

### Materials and Methods

In this experimental study (ethical code: TUMS.DENTISTRY.REC.1397.017), 48 extracted bovine incisors were immersed in a solution of 0.5% Chloramine-T for one week after removing soft tissue debris and then stored in distilled water at 4°C until the experiment. The crowns of bovine incisors were separated from the roots and sectioned perpendicular to their long axis. The teeth were embedded in acrylic molds with a self-curing resin. Buccal enamel was removed using a high-speed diamond bur. The exposed dentin surface was polished with 600-grit silicon carbide abrasive papers under water cooling. The specimens were randomly divided into two groups according to the plasma treatment (with or without) and two subgroups according to thermocycling (n=12). The study design is shown in Diagram 1.

#### *The use of a non-thermal atmospheric pressure plasma (NT-APP) jet:*

The non-thermal atmospheric plasma brush employed in this study was designed by the Nik fanavar plasma, Parham Co., Tehran, Iran. The APP was used for 30 seconds with compressed argon gas (ultrahigh purity) as the plasma gas supply with argon gas flow rate of 3000 sccm. A glow discharge by the direct current power source was ignited between the two electrodes in a walled Teflon chamber. One of the electrodes was attached to a ballasted resistor which controlled the discharge current. The other electrode was grounded for electrical safety. The formed plasma discharge could be blown out of the chamber to



**Diagram 1.** Study design for different groups

create a brush-shaped non-thermal plasma jet, which was generated in about 2 seconds [14]. A self-adhesive composite (Vertise™ Flow; Kerr, Orange, CA, USA) was selected for this study. The composite was applied according to the manufacturer's instructions (Table 1). First, a 0.5-mm-thick layer was placed on the dentin surface and light-cured by a light-emitting diode (LED) curing unit (Bluephase® Style, Ivoclar Vivadent, Schaan, Liechtenstein) with the intensity of 1200 mW/cm<sup>2</sup> for 20 seconds. A cylindrical translucent mold (Saint Gobain Performance Plastics™ Tygon™ R-3603 Lab Tubing, Miami Lakes, FL, USA) with the diameter of 1 mm and the height of 3 mm was used. Nanohybrid Filtek™ Z250 XT resin composite (A2 Shade; 3M ESPE AG, Seefeld, Germany) was filled into the cylindrical translucent mold by a resin applicator, and packing was performed using a dycal applicator. After resin filling, the cylindrical translucent mold was light-cured by the LED curing light with the intensity of 1200 mW/cm<sup>2</sup> for 20 seconds.

#### *Microtensile bond strength (μSBS) testing:*

After storage in deionized water in an incubator at 37°C for 24 hours, each Tygon tubing was cut using a #12 blade. The specimens of each group were further divided into two subgroups according to the μSBS testing time: one subgroup was immediately submitted to the μSBS test, whereas the other subgroup was submitted to artificial aging with thermocycling for 5,000 cycles between 5°C and 55°C with a 20-second dwell time and a

10-second transferring time before being tested [14,15]. Each specimen was attached to a testing jig and loaded at a crosshead speed of 1.0 mm/minute in a universal testing machine (Zwick/Roell Z050, Ulm, Germany) until failure occurred.

#### *Statistical analysis:*

The μSBS data were analyzed using statistical software (SPSS 24.0; IBM Corp., Armonk, NY, USA). Two-way analysis of variance (ANOVA) was used to assess the effects of the plasma treatments and the measuring time (artificial aging). Because the interaction was not significant ( $P=0.33$ ), t-test was used for comparisons of the μSBS within the factors of treatment and measuring time. The analyses were performed at a significance level of  $\alpha=0.05$ .

## **Results**

Table 2 shows the μSBS values for all test groups. The two-factor repeated measures ANOVA revealed that the plasma treatment had a significant effect on the bond strength ( $P=0.002$ ), but the effect of artificial aging ( $P=0.397$ ) and the interaction effect ( $P=0.067$ ) were not significant. The plasma-treated groups showed a significantly higher bond strength than the control group, both at 24 hours and after thermocycling. However, there were no significant differences between the plasma-treated groups at either measurement times. After artificial aging, the mean bond strength of the control group and the conventional plasma

**Table 1.** Composition of the flowable composite and its application according to the manufacturer's instructions

| Material and Manufacturer  | Matrix components (monomers) | Filler type and diameter   | Filler load           | Application  |
|----------------------------|------------------------------|--|-----------------------|--|
| Vertise™ Flow, Kerr Dental | GPDM, HEMA, MeHQ             | prepolymerized particles, Ba-glass, SiO <sub>2</sub> , YbF <sub>3</sub> , ZnO (1-μm particle size) | average 70% by weight | Brush a thin layer (<0.5 mm) of Vertise™ Flow for 20 seconds and light cure for 20 seconds. Then, apply Vertise™ Flow with a Luer-Lock tip in layers not exceeding 2 mm. Light cure each layer for 20 seconds. |

GPDM=glycerol phosphate dimethacrylate, HEMA=hydroxyethyl methacrylate, MeHQ=hydroquinone monomethyl ether

**Table 2.** Microshear bond strength (μSBS; MPa) of the resin composite to the dentin surfaces

| Study groups                  | After 24 hours (n=12) | After thermocycling (5,000 cycles; n=12) |
|-------------------------------|-----------------------|--|
| Control (no plasma treatment) | 19.47±7.4             | 19.10±5.1                                |
| Argon plasma treatment        | 34.20±12.12           | 33.64±5.6                                |

group did not significantly decrease from the initial mean bond strength.

## Discussion

In restorative dentistry, high bond strength between the restoration and dentin has the potential to decrease secondary caries, tooth sensitivity, and marginal discoloration; dental caries is known to be a major epidemiological and economic burden in the developing and developed countries [16,17]. Plasma, the fourth state of matter, can quickly change surface energy without affecting the bulk properties of materials. Non-thermal plasma has been used recently to modify enamel [18] and dentin surfaces [19] and also to improve the interfacial bonding of dental composite restorations to dentin and enamel surfaces [20,21]. Moreover, a previous study has proven that non-thermal plasma treatment would improve the bond strength between dentin and self-etch adhesives [22]. The present study assessed whether NT-APP application by itself has a potential to increase the bond strength of self-adhesive composites without using adhesives or dentin

etching prior to the application of the composite resin.

While micro-mechanical interlocking is essential for attaining a good bonding in the clinical setting, the potential advantage of supplementary chemical interaction between functional monomers and tooth-substrate components has lately reclaimed attention [23]. Each dental adhesive comprises a specific functional monomer that determines its adhesive performance to dental tissue [24]. Vertise™ Flow contains GPDM monomers which are well-known from the OptiBond FL adhesive system [25]. GPDM features a rather short spacer chain and high hydrophilicity that result in a strong etching effect and a better dentin wettability but also a relatively low chemical bonding potential to hydroxyapatite compared to other self-adhesive monomers [26].

In the present study, Vertise™ Flow resulted in thin and sparse tags compared to a different study, where tags were partially branched [27]. Not only when measured immediately after application [28], but especially after aging [29,30], self-adhesive composites present significantly lower bond

strengths than conventional composites that were applied with a bonding system in the etch-and-rinse or self-etch technique. Chemical bonding of GPDM to the calcium present in hydroxyapatite has been positively demonstrated [31]. Unlike etch-and-rinse adhesives, self-adhesive composites are applied directly on the smear layer of dentin surface; etching and subsequent penetration of monomers into the demineralized dentin are carried out as a single step, which is achieved by including polymerizable acidic monomers into the formula [32]. These polymerizable acidic monomers can react with other resin monomers in the adhesive formula to produce an effective bonding. The acidic groups in these monomers can etch mineral debris, remove smear layers, and help resin monomers to penetrate into dentinal tubules [31,32].

Because of about 70% filler content in self-adhesive composites, they have lower wettability on dentin surface and higher viscosity that lead to less resin monomer penetration and decreased bond strength of self-adhesive composites [32].

Our results showed that the  $\mu$ SBS for the NT-APP group (34.20 MPa) is higher than that of the control group (19.47 MPa) after 24 hours. The same patterns have been seen after 5,000 cycles of aging, with the  $\mu$ SBS in the NT-APP group (33.64 MPa) and in the control group (19.10 MPa). There are no studies in the literature on the evaluation of  $\mu$ SBS values after NT-APP surface treatment or comparing  $\mu$ SBS between the control (no plasma treatment) and NT-APP-treated groups with the use of Vertise™ Flow self-adhesive composite. The higher bond strength in the plasma-treated group can be justified by the high Bis-GMA content in the hybrid layer. Bis-GMA has superior mechanical properties compared to monomethacrylates, such as HEMA (hydroxyethyl methacrylate), but because of its hydrophobicity and large molecular size, it does not sufficiently penetrate the wet dentinal surface [33]. The plasma-treated group would transfer excess water from the interfibrillar space without collapse of the collagen network, resulting in improved Bis-GMA penetration and bond strength [34]. Treatment of dentin surfaces with NT-APP is a promising approach to improve the penetration of adhesive and resin bond strength to dentin [34].

Optical emission spectroscopy (OES) from a study by Huang et al [34] indicated that the argon plasma brush induced the production of photo-emitting species from nitrogen, oxygen, and nitrous oxide as a result of the interactions between argon plasmas and ambient air. The surface interaction of the reactive oxygen species in the plasma could be incorporated into the type I collagen molecule as carbonyl groups containing oxygen. The addition of more carbonyl groups to the collagen fibrils has been hypothesized to increase the hydrogen-bonding interactions of the collagen fibrils with the adhesive that is subsequently applied to the dentin surfaces for test specimen preparation [35]. Another potential advantage of the increased number of carbonyl groups is the electrical repulsive forces that could cause the collagen fiber to be partly separated into smaller fibril collections or even single fibrils [36]; this would significantly improve the penetration of the adhesive into collagen fibrils and increase the interface area between the adhesive and collagen and consequently significantly enhance the interfacial bonding strength [37]. NT-APP after thermocycling could decrease the bond strength of the composite. Interestingly, bond strengths of the self-adhesive systems after thermal cycling were not different in the present study and in the study by Brueckner et al [20].

The effect of plasma treatment on dentin adhesion should be further investigated with different adhesive systems. In addition, future studies on plasma treatment need to evaluate the long-term durability of dentin adhesion and clinical performance.

## Conclusion

Plasma treatment using NT-APP improves the adhesion of self-adhesive flowable resin composites to dentin.

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