# Effect of vacuum plasma treatment on the shear bond strength of 3D-printed resin and self-adhesive resin cement

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This study investigated the effect of vacuum plasma treatment on the shear bond strength (SBS) of three-dimensional (3D)-printed resin and resin cement. Specimens were categorized based on airborne-particle abrasion (APA), plasma, and bonding agent treatments. Long-term adhesive stability was examined by comparing their SBSs before and after thermocycling. The group with the bonding agent applied after plasma treatment and before thermocycling exhibited the highest SBS of 23.9±2.2 MPa. No significant SBS difference was observed among the remaining groups, except for that subjected only to plasma treatment after thermocycling. The wettability of distilled water improved in the groups treated with plasma, whereas that of the bonding agent decreased in the group treated only with APA. Surface analysis revealed a roughened surface on the plasma-treated 3D-printed resin. Therefore, vacuum-plasma treatment before thermocycling can enhance the SBS of 3D-printed resin without compromising its properties.

Keywords: 3D printing resin, Vacuum plasma, Surface treatment, Shear bond strength, Thermocycling

# INTRODUCTION

With the advancement of computer-aided design/computer-aided manufacturing (CAD/CAM) technology, various dental prostheses can now be produced using three-dimensional (3D) printing methods<sup>1)</sup>. In addition, 3D printing resins are widely utilized for inlays, crowns, and dentures. In recent years, 3D printing resins with improved properties have been developed for constructing definitive prostheses. which offer several advantages over those fabricated using milling techniques. For example, the absence of cutting tools in 3D printing allows for unrestricted movement, resulting in superior marginal and internal fit<sup>2,3)</sup>. In addition, 3D-printed resins cause less wear on opposing teeth<sup>4)</sup>.

For dental restorations to function properly and be maintained in the oral cavity for long periods, they must achieve adequate shear bond strength (SBS) with dental resin cement. Restorations composed of 3D-printed resins demonstrate higher polymerization rates than those fabricated using traditional methods and lower amounts of residual monomers than those prepared by milling<sup>5)</sup>. Additionally, after polymerization, the amount of monomer released increases, whereas that of residual monomer decreases<sup>6)</sup>. The reduced number of functional groups available for binding to dental cement makes it challenging to achieve adequate SBS<sup>7)</sup>. Various surface treatments have been studied to address this challenge and enhance SBS.

One common mechanical surface treatment method for enhancing the adhesive strength between polymer materials and cement is airborne-particle abrasion (APA). It involves spraying alumina particles onto the material surface to remove the matrix and filler of the polymer and roughen the surface to increase the surface area, thereby increasing mechanical retention. This treatment also removes the smear layer or organic material present on the surface<sup>8,9</sup>. APA treatment of CAD/CAM provisional restorative resin has been shown to enhance the bond strength compared with traditional provisional restorative resin<sup>10</sup>. Additionally, tensile bond strength tests of CAD/CAM resin crowns with teeth have demonstrated that surface treatment with APA results in higher bond strength than no treatment<sup>7</sup>. However, APA treatment of CAD/CAM resins introduces localized compressive stresses and microcracks on the surface and subsurface, making it challenging to obtain a uniform surface. Therefore, caution is required with APA treatment because the degree of damage varies depending on the type of resin and APA conditions<sup>11</sup>).

Recently, plasma-based surface treatments, which rely on ionized plasma, have been proposed to modify the polymer surface while mitigating the limitations of APA. Notably, they can increase the surface energy and wettability without altering the material properties<sup>12)</sup>, and have been widely applied as an alternative to traditional mechanical surface treatments to improve the bonding strength of materials<sup>13)</sup>. Plasma treatments can be categorized as atmospheric-pressure or vacuum treatments, depending on the processing conditions. Most studies on dental materials have used non-thermal atmospheric plasma (NTAP), and the corresponding treatments have been conducted in the presence of a specific gas, typically O<sub>2</sub> or Ar<sup>12,14-18)</sup>. These gases are used to generate plasma, which forms free radicals on a polymer surface, creating various functional groups<sup>19,20)</sup>.

Alternatively, plasma can be discharged under vacuum by processing it without using additional gas. These methods produce high-energy radicals that chemically modify the material surface through

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incomplete ionization<sup>21,22)</sup>. Vacuum plasma is characterized by higher electron energy than NTAP and enhanced convenience because the need for additional gas injection is eliminated. Specifically, vacuum plasma can alter the material surface to enhance adhesion owing to its high electron energy and can effectively remove surface impurities through vigorous interactions with them. Consequently, it is more effective for surface cleaning<sup>23)</sup> than NTAP and can more notably improve the bonding process.

However, studies on the identification of appropriate surface treatments and bonding conditions for 3D printing resins used in definitive prosthetics are scarce. Therefore, this study evaluated the SBS between a 3D printing resin for definitive prosthetics and resin cement after vacuum-plasma treatment. Specifically, the SBSs of the materials before and after thermocycling were compared to assess their adhesive stability. The null hypothesis of this study was that there is no difference in the SBS of 3D printing resin after plasma treatment.

# MATERIALS AND METHODS

# Specimen preparation

The compositions of the materials used in this study are shown in Table 1. Cylindrical 3D-printed resin specimens (Tera Harz TC-80DP, Graphy, Seoul, Korea) with a diameter of 7.0 mm and height of 3.5 mm, were designed using a CAD software (MeshMixer, Autodesk, San Rafael, CA, USA). Supports were designed using a 3D printing software (UNIZ Maker 1.5.4.35, UniZ Technology, San Diego, CA, USA) and positioned on the opposite side of

the test area, and each specimen was laminated parallel to its long axis. G\*Power software (version 3.1.9.4) was employed to determine the sample size required for this study. Twelve samples per group, totaling 120 specimens, were sufficient to achieve 95% confidence ( $\alpha$ =0.05) and an effect size of 0.365. These specimens were printed using a 3D printer (SprintRay Pro 95, SprintRay, Los Angeles, CA, USA) with the digital light processing method, according to the manufacturer's recommendations. The printed specimens were ultrasonically cleaned with 90% isopropyl alcohol and then completely dried and cured using a post-curing machine (Cure M 102H, Graphy). The surface designated for resin cement application was polished with 1000-grit silicon carbon abrasive papers under irrigation to ensure uniformity. The prepared resins were divided into five groups according to the surface treatment, as listed in Table 2.

For the APA surface treatment, a sandblaster was used to vertically spray 50- $\mu$ m alumina oxide (Al<sub>2</sub>O<sub>3</sub>) particles onto the specimen surface at a pressure of 0.2 MPa for 10 s from a distance of 10 mm. Following treatment, the specimens were ultrasonically cleaned to remove residual alumina particles.

A commercially available plasma device (ACTILINK, Plasmapp, Daejeon, Korea) was used for the vacuum-plasma treatment. Each resin specimen was placed on the electrode within the dedicated container in the device and subjected to surface treatment for 120 s (Fig. 1).

For the groups receiving the adhesive (Single Bond Universal (SBU), 3M ESPE, St. Paul, MN, USA), a thin layer was applied to the specimen surface according to the manufacturer's instructions. This was followed by

Table 1 Compositions of materials used in this study

Product name	Manufacturer	Composition			
Tera Harz TC-80DP (A2)	Graphy, Seoul, Korea	Urethane dimethacrylate-based dental resin, phosphine oxides, pigment			
Single Bond Universal	3M ESPE, St. Paul, MN, USA	MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane			
RelyX U200	3M ESPE	Base: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst: methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives			

Table 2 Study groups and surface treatment methods

	Group name	Procedure		
	С	Control; no surface treatment		
C	A	Airborne particle abrasion		
Group	P	Plasma		
	AP	Plasma after airborne particle abrasion		
	Group name-B	Group with SBU added		

Control: no surface treatment; SBU: single bond universal

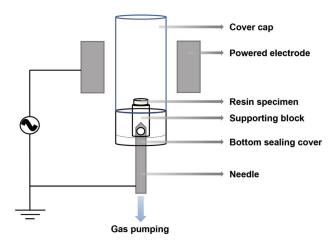


Fig. 1 Configuration of the plasma source.

air-drying for 5 s at a distance of 15 mm from the surface under a pressure of 0.25 MPa and light-curing for 10 s.

For the SBS test, a mold with a diameter of 25 mm and height of 15 mm was fabricated using a model resin (S-Plastic, S100M, Graphy) designed for the specimen to be attached to its top surface. The printed resin specimens were embedded in the mold with their treated surfaces exposed for cement bonding. Self-adhesive resin cement (RelyX U200, 3M ESPE) was applied to the resin within a bonding area of 4.45 mm² using a plastic jig (Ultradent) and light-cured for 20 s according to the manufacturer's instructions.

The specimens from each group were divided into two sets: before and after thermocycling. Thermocycling was performed using a thermal cyclic tester (R&B, Daejeon, Korea) with 10,000 cycles per group. Each cycle consisted of 30 s immersion in a 5°C water bath, a 5 s rest period, and 30 s immersion in a 55°C water bath. After treatment, all specimens were stored in distilled water at 37°C for 24 h.

#### SBS

The SBS between the 3D-printed resin and resin cement was determined in accordance with ISO 29022-2013<sup>24)</sup> using a universal testing machine (Bisco, Schaumburg, IL, USA). First, the crosshead notch was aligned with the resin cement and positioned flush against the resin specimen surface. The crosshead was then advanced until it contacted the resin cement. Subsequently, the specimen was loaded at a crosshead speed of 1.0±0.1 mm/min until failure, and the load was measured using a force gauge. For each specimen, the maximum recorded load was divided by the surface area (4.45 mm²) to obtain the SBS in MPa.

The failure mode was categorized as adhesive failure between the 3D printing resin and resin cement, cohesive failure within the 3D-printed resin or resin cement, or mixed failure, *i.e.*, both adhesive and cohesive failure. A frequency analysis was conducted for each failure mode.

# Contact angle

A contact-angle analyzer (SmartDrop, Femtofab, Seongnam, Korea) was used to measure the contact angles of distilled water and SBU adhesive to evaluate wettability following various surface treatments. Contact angles were measured across four groups: C, A, P, and AP, with three specimens analyzed per group $^{25}$ . For each specimen, 1  $\mu L$  of liquid was dispensed onto the center of the flat specimen surface, and measurements were obtained in a sessile-drop state.

# Surface roughness

A 3D optical surface profiler (Contour GT-X3 BASE, Bruker, Bremen, Germany) was used to compare and analyze the changes in the surface roughness ( $R_{\rm a}$ ) of the 3D-printed resin surfaces subjected to different surface treatments. One representative specimen from each group was analyzed. The surface roughness was measured at nine points, and the average value was used<sup>26-28)</sup>.

# Scanning electron microscopy (SEM)

An SEM instrument (S-3000N, Hitachi, Tokyo, Japan) was used to identify surface differences on the 3D-printed resin following surface treatment. All specimens were coated with platinum to a thickness of 100 nm using an ion-sputter coater (E-1010, Hitachi). SEM analysis was performed on the surface of one representative specimen from each group at a magnification of 2,000×.

# Statistical analysis

All data were statistically analyzed using SPSS 26.0 software (SPSS, Chicago, IL, USA). First, the Kolmogorov–Smirnov and Shapiro–Wilk tests were conducted to confirm the normal distribution of the data and Levene's test was conducted to confirm the homogeneity of variances. All SBS values were normally distributed and met the assumption of equal variances (Levene's test: F=1.616, p=0.119). To assess the main effects of each surface treatment and thermocycling, and their interaction effect on SBS, a two-way analysis of variance (ANOVA) with Bonferroni's post-hoc test was conducted at a 95% confidence level ( $\alpha$ =0.05).

# **RESULTS**

#### SRS

The two-way ANOVA results are in Table 3. The SBS exhibited significant differences depending on the surface treatment and thermocycling and the interaction between the two also significantly affected SBS.

The SBS results by group are presented in Table 4. Before thermocycling, Group P-B exhibited the highest SBS; however, the difference between Groups P-B and AP-B was not statistically significant (p=0.231). The SBS of Group P-B was significantly higher than that of all other groups except AP-B (p<0.001 for all groups). Even after thermocycling, the SBS of Group P-B remained significantly higher than that of Group P (p<0.001). Additionally, the SBU-treated groups

Table 3 Results of two-way ANOVA

	Sum of squares	Df	Mean squares	F	<i>p</i> -value
Surface treatment methods	2,373.190	6	395.532	29.856	0.000
Thermocycling	188.595	1	188.595	14.236	0.000
Surface treatment methods× Thermocycling	177.105	6	29.517	2.228	0.043
Error	2,040.188	154	13.248	_	_

Table 4 Mean and standard deviation of shear bond strength values of experimental groups (MPa) (n=12)

	C	C-B	A	A-B	P	P-B	AP-B
Before thermocycling	$13.0 \pm 3.5$ A,c	$16.7{\pm}3.9^{\mathrm{A,bc}}$	$14.9\pm4.8^{\mathrm{A,c}}$	$16.2{\pm}4.9^{\mathrm{A,bc}}$	$13.4 \pm 3.6$ A,c	$23.9\pm2.2^{\mathrm{A,a}}$	20.0±3.8 <sup>A,ab</sup>
After thermocycling	$10.0{\pm}1.2^{\rm{\;B,b}}$	$16.9{\pm}4.1^{\rm{\;A,a}}$	$11.7 \pm 4.3$ B,b	$16.6{\pm}4.4^{\rm{\;A,a}}$	$8.8{\pm}2.1^{\mathrm{~B,b}}$	$19.3 {\pm} 2.6^{\mathrm{B,a}}$	$19.9 \pm 3.4^{\mathrm{A,a}}$

SBU: single bond universal; C: control (no surface treatment); C-B: control with SBU; A: airborne particle abrasion (APA); A-B: APA with SBU; P: plasma; P-B: plasma with SBU; AP-B: plasma after APA with SBU. Means with different lowercase letters in each row present statistically significant differences (p<0.05). Means with different uppercase letters in each column present statistically significant differences (p<0.05).

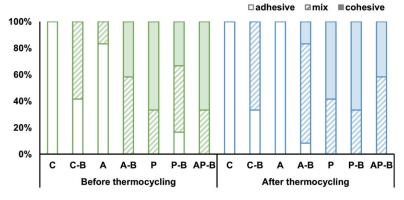


Fig. 2 Failure mode between the 3D printing resin and the resin cement in all groups after SBS test.
SBU: single bond universal; C: control (no surface treatment); C-B: control with SBU; A: airborne particle abrasion (APA); A-B: APA with SBU; P: plasma; P-B: plasma with SBU; AP-B: plasma after APA with SBU.

exhibited significantly higher SBS than the untreated groups. However, no statistically significant difference in SBS was found among the SBU-treated groups. Thermocycling significantly reduced the SBSs of Groups C, A, P, and P-B (p<0.05), whereas it did not significantly affect those of Groups C-B, A-B, and AP-B.

The frequencies and representative images of the adhesion-failure patterns are shown in Fig. 2. Evidently, adhesive failure was most prevalent in Group P, whereas in all other groups, cohesive failure and mixed failure were more common.

#### Contact angle

Contact-angle measurements with distilled water revealed that Groups P and AP exhibited increased

surface spreading, indicating that the 3D-printed resins exhibited enhanced wettability after plasma treatment (Fig. 3, Table 5). By contrast, the contact angle of Group A increased, suggesting reduced wettability for SBU. However, Groups P and AP showed wettability similar to that of Group C (Fig. 4, Table 5).

#### Surface roughness

Table 6 and Fig. 5 present the average  $R_{\rm a}$  values of the 3D-printed resin specimens subjected to different surface treatments. The surface roughness of all surface-treated groups was higher than that of Group C.

#### SEM

To confirm the influence of the surface treatment, the

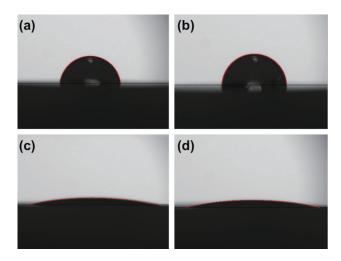


Fig. 3 Contact angle images of distilled water droplets on 3D-printed resins after different surface treatments.
(A) C: control (no surface treatment); (B) A: airborne particle abrasion (APA); (C) P: plasma;

(D) AP: plasma after APA.

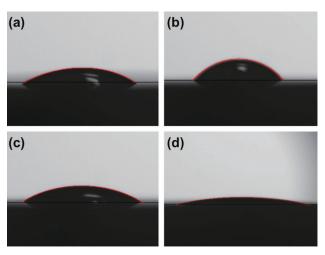


Fig. 4 Contact angle images and values of bonding agent (Single Bond Universal, 3M ESPE) droplets on 3D-printed resins after different surface treatments.

(A) C: control (no surface treatment); (B) A: airborne particle abrasion (APA); (C) P: plasma; (D) AP: plasma after APA.

Table 5 Contact angles of distilled water and bonding agent droplets on 3D-printed resin after different surface treatments (n=3)

		C	A	P	AP
B	Minimum	80.3	78.9	11.5	13.7
Distilled	Maximum	87.2	87.4	16.2	17.7
water	$Mean\pm SD$	83.5±3.48	$84.0 \pm 4.50$	13.6±2.39	$16.0\pm2.07$
SBU	Minimum	30.40	48.02	33.05	29.84
	Maximum	39.61	52.93	36.55	38.30
	Mean±SD	$38.2 \pm 3.79$	$52.3\pm2.09$	$38.6 \pm 1.48$	36.4±3.63

SBU: single bond universal; C: control (no surface treatment); A: airborne particle abrasion (APA); P: plasma; AP: plasma after APA

Table 6 Mean and standard deviation of surface roughness ( $R_a$ ,  $\mu$ m) of 3D-printed resin after different surface treatments (n=1, nine points of each specimen)

	C	A	Р	AP
Surface roughness (µm)	$0.39\pm0.03$	$0.78 \pm 0.09$	$0.72 \pm 0.14$	$0.71 \pm 0.05$

C: control (no surface treatment); A: airborne particle abrasion (APA); P: plasma; AP: plasma after APA

surface of one representative 3D-printed resin specimen per group was examined at 2,000× magnification *via* SEM (Fig. 6). The Group C specimen exhibited a smooth surface with regular micro-polished grooves induced by the SiC polishing paper. The Group A specimen exhibited a rough surface with irregular and deep grooves, whereas the Group P specimen exhibited a deeper micro-groovelike structure, forming an overall rough surface. The Group AP specimen had deep and wide grooves similar to the Group A specimen, with slightly finer and smaller grooves similar to those in the Group P specimen.

# DISCUSSION

This study assessed the influence of vacuum-plasma treatment on the SBS of 3D-printed resins for definitive prostheses with resin cement, both before and after thermocycling. The two-way ANOVA revealed that Group P-B exhibited the highest SBS before thermocycling, although no statistically significant difference was observed compared with Group AP-B. After thermocycling, Group AP-B exhibited the highest SBS; however, no statistically significant differences

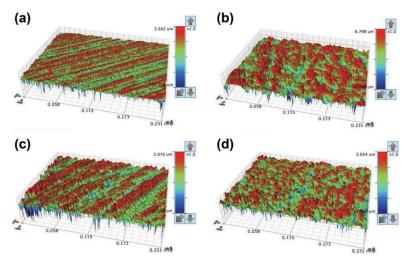


Fig. 5 Surface roughness (Ra, μm) of 3D-printed resins subjected to different surface treatments.
(A) C: control (no surface treatment); (B) A: airborne particle abrasion (APA); (C) P: plasma; (D) AP: plasma after APA.

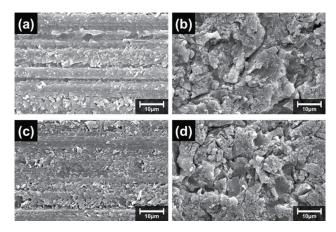


Fig. 6 SEM images of 3D-printed resins after different surface treatments (×2,000 magnification).
(A) C: control (no surface treatment); (B) A: airborne particle abrasion (APA); (C) P: plasma; (D) AP: plasma after APA.

were observed among Groups AP-B, C-B, A-B, and P-B. The adhesive-failure rate was higher in Group P, which had the lowest SBS values, and adhesive failures were usually observed at lower SBS. Therefore, the null hypothesis of no difference in SBS after plasma treatment was rejected.

APA treatment is generally recommended to enhance the adhesion of CAD/CAM resin materials  $^{29}$ . It cleans and roughens the resin surface, thereby improving its micromechanical retention  $^{30}$ . The recommended pressure for APA processing of CAD/CAM resins, typically 0.1–0.2 MPa, is lower than that required for ceramic or metallic materials owing to the lower Vickers hardness of CAD/CAM resins  $^{31}$ . A previous study confirmed that spraying 50- $\mu$ m  $Al_2O_3$  at 0.2 MPa during APA treatment increases

the SBS between a 3D-printed resin and resin cement<sup>32)</sup>. However, another study conducted under the same conditions found that APA treatment, while roughening the surface, also causes 1–10-µm-long cracks and filler exposure in various CAD/CAM resins<sup>11)</sup>. This highlights the potential for APA treatment to damage the resin surface and create stress zones, possibly leading to degradation of physical properties. Furthermore, APA treatment is influenced by various factors, including particle size, spray distance, duration, and operator skill<sup>33</sup>. One study reported that APA treatment did not significantly increase SBS when repairing 3D-printed resin with conventional temporary resin<sup>34)</sup>. Therefore, caution is advised when using APA on 3D-printed resins, and alternative surface treatments should be explored to improve their adhesive strength without compromising their properties.

Plasma treatment chemically and morphologically modifies surfaces through the action of high-energy electrons, ions, and free radicals 12,14). In this study, vacuum-plasma treatment decreased the contact angle and increased hydrophilicity of 3D-printed resins compared with those in Groups C and A. Additionally, the average  $R_a$  value of the 3D-printed resin surfaces increased. Notably, plasma treatment alone produced surface roughness comparable to that achieved with APA. SEM analysis confirmed that the surfaces of the 3D-printed resins subjected to vacuum-plasma treatment were rougher than those in Group C, with finer pores and a fine uneven structure. Thus, plasma surface treatment has two key effects: First, it cleans the surface by removing impurities. The vacuum plasma used in this study featured high electron energy and can efficiently react with the polymer surface to remove impurities such as carbon elements<sup>23)</sup>. Lee et al.<sup>35)</sup> demonstrated that vacuum-plasma treatment of implants can improve osseointegration by efficiently removing hydrocarbons without altering surface properties. Second, it increases the number of hydrophilic groups, such as hydroxyl (-OH) and carboxyl (C=O)<sup>36)</sup>. When these hydrophilic groups bind to the polymer surface, the adhesive strength of the resin is improved owing to the increased surface energy and reduced contact angle<sup>37)</sup>.

Several researchers have investigated the effects of NTAP on various dental materials. Liebermann et al.16 reported that argon plasma treatment of polymethyl methacrylate increases the surface energy without affecting  $R_{\rm a}$  but decreases the tensile bond strength with self-adhesive resin cement. According to Schwitalla et al. 17), argon/oxygen plasma treatment of polyetheretherketone (PEEK) increases hydrophilicity without affecting  $R_a$ . Dede et al.<sup>38)</sup> employed SEM analysis and observed no surface changes in polyetherketoneketone subjected to NTAP treatment and no impact on SBS to veneering resin. However, these studies reported that surface treatment with sandblasting and plasma could increase SBS to veneering resin. Other studies have shown that plasma treatment of zirconia increases surface hydrophilicity, and the application of a primer containing 10methacryloyloxydecyl dihydrogen phosphate (MDP) increases SBS to resin cement<sup>18,39)</sup>. Typically, NTAP aims to generate plasma at atmospheric pressure by introducing additional gases, such as oxygen and argon to create a specific plasma. Supplying these gases requires the installation of gas cylinders, piping systems, continuous gas replenishment, and equipment maintenance<sup>35)</sup>. The vacuum plasma used in this study avoids these requirements by not requiring specific gases, thus eliminating the maintenance requirements and facilitating more stable and convenient clinical application.

In this study, the bonding agent applied during the plasma treatment significantly affected the SBS of the 3D-printed resins. Group P (13.4±3.6 MPa), for which a binder was not used, exhibited a significantly lower SBS than Group P-B (23.9±2.2 MPa). The SBU used in this study contains 10-MDP, which improves the flowability of cement and can copolymerize with resin cement to form strong bonds. In addition, it can increase the SBS with resin cement by incorporating silane, which increases the surface energy by reducing the surface tension of the matrix<sup>40</sup>.

10-MDP is a phosphate ether monomer that enables micromechanical bonding through acid etching and features both hydrophilic and hydrophobic properties. In this study, the group treated with plasma and SBU containing 10-MDP exhibited significantly higher SBS before thermocycling. Additionally, after thermocycling, groups treated with SBU exhibited significantly higher SBS than the untreated ones, regardless of the surface treatment, demonstrating the importance of using SBU containing 10-MDP for bonding. After plasma treatment, mechanical bonding occurs on the surface of the 3D-printed specimen owing to plasma and corrosion effects, and a hydrophilic surface is formed on which

the hydrophilic mechanism of 10-MDP attaches and chemically bonds with the specimen. Subsequently, a polymerizable group and hydrophobic resin cement are combined on the upper part. The use of SBU after plasma treatment can be expected to induce higher chemical bonding when SBU is used because of the hydrophilic effect of the surface by plasma. In the case of Group A-B, the surface roughness increased, but the wettability for SBU was low, and the SBS results were similar to those of Group C-B. Based on these results, plasma treatment can be predicted to affect the improvement of SBS by increasing surface wettability more than increasing the surface area through mechanical surface treatment with APA.

Previous studies have shown that applying a pretreatment agent containing silane to the surface of CAD/CAM resin can improve chemical bonding and increase the bond strength<sup>41,42)</sup>. Akin *et al.* reported that the use of a dedicated bonding agent on light-cured dentures made of urethane dimethacrylate can increase the bond strength with the artificial tooth<sup>43)</sup>. However, other studies have reported that the use of a bonding agent does not affect the bond strength of CAD/CAM resin<sup>10)</sup>. Thus, further studies are required to evaluate the effects of different bonding agents on SBS during plasma treatment.

Thermocycling is a common method for artificial aging and is used to simulate the long-term use of dental restorative materials in the oral cavity<sup>44</sup>. A previous study reported that 10,000 thermocycling treatments approximate one year in the oral cavity<sup>45)</sup>. In this study, the 3D-printed resin and resin cement specimens underwent 10,000 thermocycles to simulate one year of clinical period to evaluate the effect of thermocycling on SBS. No significant differences were observed in the SBS values of Groups A-B and AP-B treated with APA before and after thermocycling. However, Groups P and P-B exhibited significant decreases in SBS after thermocycling. These results are consistent with those of other studies that reported reduced SBS after plasma treatment and thermocycling<sup>14,46,47)</sup>. These results suggest that the low resistance to hydrolysis of the hydrogen bonds after plasma application during thermocycling may have reduced the SBS<sup>14)</sup>. In this study, the SBS of Group P-B was similar to that of Group AP-B after thermocycling. However, Group P-B showed the highest proportion (66.7%) of cohesive failure, indicating a strong bond between the 3D-printed resin and the resin cement. These results demonstrate that treating 3D-printed resins with both plasma and the SBU adhesive without APA is a convenient surface treatment for clinical applications, particularly for the intaglio surface of crowns. However, because the SBS of Group P-B did not significantly differ from those of the other groups, except Groups C, A, and P, further investigations of the effects of long-term thermocycling are required for clinical application.

The results of this study indicate that the vacuumplasma-based surface treatment can effectively improve the adhesive strength of 3D-printed resins for definitive prostheses without the detrimental effects of APA treatment, such as the cracks and irregular surfaces, highlighting its potential for clinical applications. However, this study used limited materials and only one plasma process. Therefore, future studies should evaluate adhesive strength using various materials and different plasma-treatment conditions, such as varying treatment processes and durations. Additionally, as this study conducted only one year of thermocycling, SBS should be evaluated after more thermocycles to assess long-term stability.

# CONCLUSIONS

This study examined the SBS of 3D-printed resins and self-adhesive resin cement after application of different surface treatment methods, including vacuum-plasmabased processing. The following conclusions can be drawn from the results obtained in this study:

- Applying a bonding agent after vacuum-plasma treatment significantly increased the SBS between the 3D-printed resin and self-adhesive resin cement.
- Plasma treatment alone increased the surface roughness of the 3D-printed resin without surface damage in the form of cracks.

# **ACKNOWLEDGMENTS**

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# CONFLICT OF INTEREST

The authors declare no competing interests.

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