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Influence of Different Surface Treatments on Bond Strength of a Self-Adhesive Resin Cement to CAD-CAM Materials

Influencia de los diferentes tratamientos superficiales en la unión del cemento resinoso autoadhesivo a los materiales CAD-CAM

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ABSTRACT: To evaluate the shear bond strength (SBS) of self-adhesive resin cement when used with two different computer-aided design (CAD)-computer-aided manufacturing (CAM) materials after various surface treatments. Nanoceramic resin Lava Ultimate (LU) and feldspathic ceramic Vita Mark II (VM) CAD-CAM block samples were prepared with 1.5-mm thickness, and a total of 90 samples were obtained (N=90), with five samples of each block. The samples were divided into the following five groups according to the surface treatments (n=9): group 1, untreated (control); group 2,5% hydrofluoric acid etching; group 3, Er: YAG laser irradiation; group 4, tribochemical silica coating (Cojet); and group 5, air-abrasion with Al_2O_3. After silane application, resin cement was applied on a transparent matrix (diameter, 3mm; height, 2mm) on the blocks. SBS was determined using a universal testing device at a crosshead speed of 1mm/min. Two-way analysis of variance (ANOVA) and Tukey's post hoc tests were used to analyze the SBS values. LU showed the highest SBS value in group 4. The average SBS values in groups 3 and were found to be lower than that in the control group ($p<0.05$). When VM was examined, while all surface treatments increased the SBS values significantly, the highest SBS value was observed in group 4 ($p<0.05$). This study revealed that all

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surface treatments used negatively affected the bond strength values of self-adhesive resin cement to LU, except for Cojet application. The SBS values of resin cement with VM increased in all surface treatment application groups.

KEYWORDS: CAD/CAM materials; Resin cement; Shear bond strength; Er: YAG laser.

RESUMEN: Evaluar la resistencia de unión al corte (SBS) del cemento de resina autoadhesivo cuando se utiliza con dos materiales diferentes de diseño asistido por computadora (CAD) y fabricación asistida por computadora (CAM) después de varios tratamientos superficiales. Se prepararon muestras de bloques CAD-CAM de resina Lava Ultimate (LU) y cerámica feldespática Vita Mark II (VM) con un espesor de 1,5mm, y se obtuvieron un total de 90 muestras (N=90), con cinco muestras de cada bloque. Las muestras se dividieron en los siguientes cinco grupos según los tratamientos superficiales (n=9): grupo 1, sin tratar (control); grupo 2, grabado con ácido fluorhídrico al 5%; grupo 3, irradiación con láser Er: YAG; grupo 4, recubrimiento triboquímico de sílice (Cojet); y grupo 5, aire-abrasión con Al_2O_3. Después de la aplicación de silano, se aplicó cemento de resina sobre una matriz transparente (diámetro, 3mm; altura, 2mm) sobre los bloques. La SBS se determinó usando un dispositivo de prueba universal a una velocidad de cruceta de 1mm/min. Se utilizaron análisis de varianza bidireccional (ANOVA) y pruebas post hoc de Tukey para analizar los valores de SBS. LU mostró el valor más alto de SBS en el grupo 4. Los valores promedio de SBS en los grupos 3 y fueron más bajos que en el grupo de control (p<0,05). Cuando se examinó VM, mientras que todos los tratamientos superficiales aumentaron significativamente los valores de SBS, el valor más alto de SBS se observó en el grupo 4 (p<0,05). Este estudio reveló que todos los tratamientos de superficie utilizados afectaron negativamente los valores de resistencia de la unión del cemento de resina autoadhesivo a LU, a excepción de la aplicación Cojet. Los valores de SBS del cemento de resina con VM aumentaron en todos los grupos de aplicación de tratamiento de superficie.

PALABRAS CLAVE: Materiales CAD/CAM; Cemento de resina; Resistencia al cizallamiento; Láser Er: YAG.

INTRODUCTION

In recent years, the development of digital systems has allowed three-dimensional modeling of restorations. Computer-aided design (CAD) and computer-aided manufacturing (CAM) have become popular in dentistry. This technology can be applied to inlays, onlays, veneers, and crowns because of its high esthetic capabilities, less technique-sensitive need, and requirement of minimal procedural steps compared to conventional restorations (1,2). With the use of indirect restorations, complications such as microleakage, secondary caries, postoperative sensitivity, and discoloration caused by polymerization shrinkage in conventional composite restorations can be overcome.

Indirect esthetic materials can be classified into two groups: ceramics (crystal or glass ceramics (feldspathic porcelain and glass ceramics) and composites. Glass ceramics are available in the form of powder, ingots, or CAD/CAM blocks. Indirect composite restorations are produced chemically, heat-cured, light-cured, or from CAD/CAM blocks (3-5). Glass-matrix ceramics and resin composites are frequently used materials for CAD/CAM restorations with improved physical and mechanical properties and wear resistance (6,7).

The manufacturers claim that with the production of resin nano-ceramic and dual-network ceramic restorations, the advantages of ceramic and composite restorations can be combined in the same material. Resin-modified blocks have undeniable advantages over feldspathic ceramics, such as increased fracture resistance, fast milling, and milling tolerance (8). These restorations are also easy to polish, can be finished in a single appointment session, and can be more easily repaired than CAD/CAM ceramic restorations (9,10). Despite these advantages, the weakest feature of ceramic and indirect composite restorations is the bond strength between the restoration and resin cement (6,11). In parallel, a recent study reported 10% debonding for full-coverage crowns with lava ultimate, which is a nano-ceramic resin composite (12).

CAD/CAM composite resins have a limited number of carbon-carbon double bonds on their surfaces because of their high conversion degree values. Therefore, surface treatment is required to ensure a reliable bond (13). Various studies have been conducted to investigate different surface treatments to increase the bond strength (6). The surface conditioning processes recommended in the literature to achieve better bonding results include sandblasting (aluminium oxide [Al_2O_3]), hydrofluoric acid etching, laser application, and tribochemical silica coating (14).

Sandblasting is a surface conditioning process that aims to increase mechanical retention by creating a rough surface with the use of Al_2O_3 particles. It also aims to increase mechanical retention by etching the restoration surface. Hydrofluoric acid etching does not result in satisfactory bonding to zirconia owing to its high crystal content and glassy phase. The tribochemical silica coating process not only roughens the ceramic surfaces, but also chemically activates them. As a result of blasting, the embedded silica and alumina particles chemically react with silane coupling agents (1,14).

Lasers are another method used to modify the surface conditions of restorative materials. With lasers, the surface treatment of restorative materials can be performed easily and safely. The Er: YAG laser is one of the most frequently used lasers in surface conditioning with a wavelength of 2940nm, and the use of accurate parameters can create a suitable surface to increase the bond strength (15).

The successful cementing of indirect restorations is an important factor in clinical success. The use of adhesive luting agents instead of conventional cements increases the marginal adaptation and fracture resistance of indirect restorations (1). Self-adhesive resin cements containing self-adhesive monomers are useful bonding agents in reducing the time required for surface treatment. They exhibit strong adhesion to dental materials, resin composites, or ceramics (16).

This study aimed to evaluate the effects of surface treatments on the shear bond strength (SBS) of two CAD/CAM materials with a self-adhesive resin cement. The null hypothesis of this study was that surface conditioning methods do not affect adhesion when compared with no surface treatment.

MATERIALS AND METHODS

Statistical analysis was performed to decide the number of samples in the groups. Considering α=0.05, ß=0.10, and 1-ß=0.90, a total of 90 samples (N=90) were included in the study, with nine composite samples in each group (n=9). The power of the test was P=0.9342.

This study tested the SBS of two different CAD/CAM restorative materials with a self-adhesive resin cement. The composition and manufacturers of the materials used in this study are listed in Table 1.

Resin-based nano ceramics (Lava Ultimate, 3M ESPE, St Paul, MN, USA; LU) and feldspathic glass-matrix ceramics (Vita Mark II, VITA Zahnfabrik Sackingen/Germany; VM) were used. The CAD-CAM blocks were sectioned using a water-cooled low-speed diamond saw (Isomet 1000, Buehler, Germany) with a thickness of 1.5mm. A total of 100 samples (N1=100) were obtained, 50 for each block. (N2=50) Then, they were randomly divided into five groups according to the surface treatment applied (n=10).

Group 1. No surface treatments were performed (control group). Silane (Monobond-S, Ivoclar Vivadent) was applied for 60s followed by air-drying for 20s.

Group 2. 5% Hydrofluoric acid (IPS Ceramic Etching Agent; HF) was applied on the surfaces for 15s then rinsed with water for 15s and air-dried. After HF etching, silane was applied for 60s and air-dried for 20s.

Group 3. Er:YAG laser (Smart 2940D Plus, Deka Laser; Florence, Italy) treated at a wavelength of 2940nm, 3 W power, 150mJ energy level, 20Hz frequency, and 700ms long pulse to scan the entire specimen surface from a distance of 10mm. After Er: YAG laser pretreatment, silane was applied for 60s and air-dried for 20s.

Group 4. Tribochemical silica coating (30-μm silica coated Al_2O_3) (CoJet Sand, 3M ESPE, Seefeld, Germany) was applied for 15s at 2.5 bar air pressure and a distance of 5mm from the surface of the samples. After Cojet application, silane was applied for 60s and air-dried for 20s.

Group 5. The specimens were sandblasted with 40-μm Al_2O_3 particles (Korox, Bego) under a pressure of 2.5 bar at a distance of 5mm for 15s. The specimens were then rinsed under running water for 30s, air-dried, and treated with silane for 60s and air-dried for 20s (Figure 1).

Table 1. Materials and their compositions used in this study.

Materials tested	Type	Chemical content	Manufacturer
Lava Ultimate CAD/ CAM Restorative	Resin-Based Nano Ceramic	80% inorganic (69% SiO_2, 31% ZrO_2) 20% organic	3M ESPE, St Paul, MN, USA
Vita MarkII	Feldspathic glass-matrix ceramic	56-64% SiO_2, 20-23% Al_2O_3, 6-9% Na_2O, 6-8% K_2O	VITA Zahnfabrik Sackingen/ Germany
RelyX Unicem	Self Adhesive Resin Cement	Methacrylated phosphoric acid esters, triethylene glycol dimethacrylate, silanized glass powder, silane treated silica, sodium persulfate, substituted pyrimidine, calcium hydroxide (filler=72 wt%; avg. <9.5μm)	3M ESPE, St Paul, MN, USA
Monobond-S	Silane coupling agent	Ethanol, 3-trimetho-xysilsylpropylmethacrylaat, methacrylated phosphoric acid ester	Ivoclar Vivadent

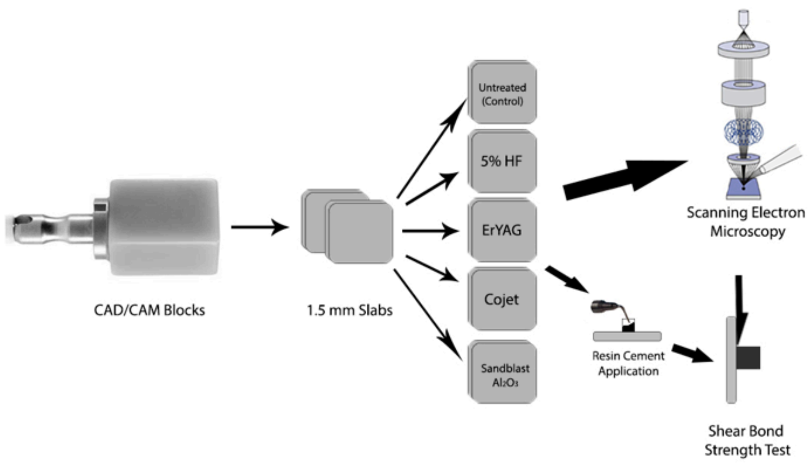

Figure 1. Study group design.

SEM ANALYSIS

An extra total of 10 samples (one sample for each subgroup) were prepared to represent each subgroup for scanning electron microscope (SEM) examination. Surface treatments were applied as described for the subgroups. The samples were sputter-coated with a gold layer (Polaron Range SC 7620; Quorum Technology, Newhaven, UK). Images were obtained using an SEM device (Jeol Ltd., JSM-5600, Tokyo, Japan) at 5000× magnification to examine the surface texture.

RESIN CEMENT BONDING AND SHEAR BOND STRENGTH TEST

A cylindrical transparent matrix (diameter, 3mm; thickness, 2mm) was placed in the middle of the specimens, and self-adhesive resin cement (RelyX Unicem) was applied to the matrix on the CAD/CAM slices. The excess resin cement was then removed and light polymerized for 40s from two lateral directions according to the manufacturer's instructions. Following polymerization of the resin cement, the transparent matrix was carefully cut away with a lancet. Before the SBS test, all the specimens were stored in distilled water (37°C for 24h). The specimens were then attached to a universal testing device (LF Plus, LLOYD Instruments, Ametek Inc., England) and subjected to shear force at a crosshead speed of 1mm/min until failure occurred. The SBS failure values were calculated in megapascals (MPa) by dividing the failure load in newtons (N) by the bonding area (mm²).

STATISTICAL ANALYSIS

The SBS test data were examined using a two-way ANOVA with post-hoc Bonferroni correction and Tukey tests were used when the parametric test assumptions were fulfilled according to the Kolmogorov-Simirnov test. The results are presented as means and standard deviations. The significance level was set to $p<0.05$ for all tests. All statistical analyses were performed and using IBM SPSS Statistics (version 25.0; IBM Corporation). Statistical significance was set at $p<0.05$.

RESULTS

The mean SBS (MPa) values for each group are listed in Table 2. The highest SBS value was observed in group 4 for both the CAD/CAM materials, LU and VM. For LU group 4 (Cojet), the mean SBS values were significantly higher than those of group 1 (control), group 2 (HF), group 3 (Er: YAG laser), and group 5 (Al_2O_3 sandblasting) ($p=0.029$). Groups 5 and 3 had significantly lower SBS values than group 1.

When VM mean values were examined, group 4 (Cojet) showed significantly higher SBS values than group 1 (control), group 2 (HF), group 3 (Er: YAG), and group 5 (Al_2O_3 sandblasting) ($p<0.05$). Group 2 (5% HF) and group 3 (Er: YAG laser) showed significantly higher SBS values than group 1 (control) ($p=0.012$). No difference was observed between group 5 (Al_2O_3 sandblasting) and group 1(control) ($p>0.05$).

SEM images are shown in Figure 2.

Table 2. The Mean and SD values of SBS test of surface treatment groups for two CAD/CAM materials.

	N	Lava Ultimate Mean±SD	Vita Mark2 Mean±SD	P Values
Goup1 (Control)	9	$14.55±4,09^{aA}$	$4.58±1,87^{aB}$	p=0,001*
Goup2 (Acid)	9	$13,21±2,87^{aA}$	$8.06±1,58^{bB}$	p=0,001*
Goup3 (Er:YAG)	9	$11.19±2,02^{bA}$	$7.50±2,33^{bB}$	p=0,001*
Goup4 (Cojet)	9	$15.72±2,70^{cA}$	$10.04±1,66^{cB}$	p=0,001*
Goup5 (Al2O3)	9	$10.63±2,31^{bA}$	$6.46±2,34^{aB}$	p=0,001*
		F=1,38 p=0,029*	F=7,04 p=0,012*	

*p<0,05

*In each row, groups with the different capital superscripts are significantly different and in each column, groups with the different lower case superscripts are significantly different (p<0.05).

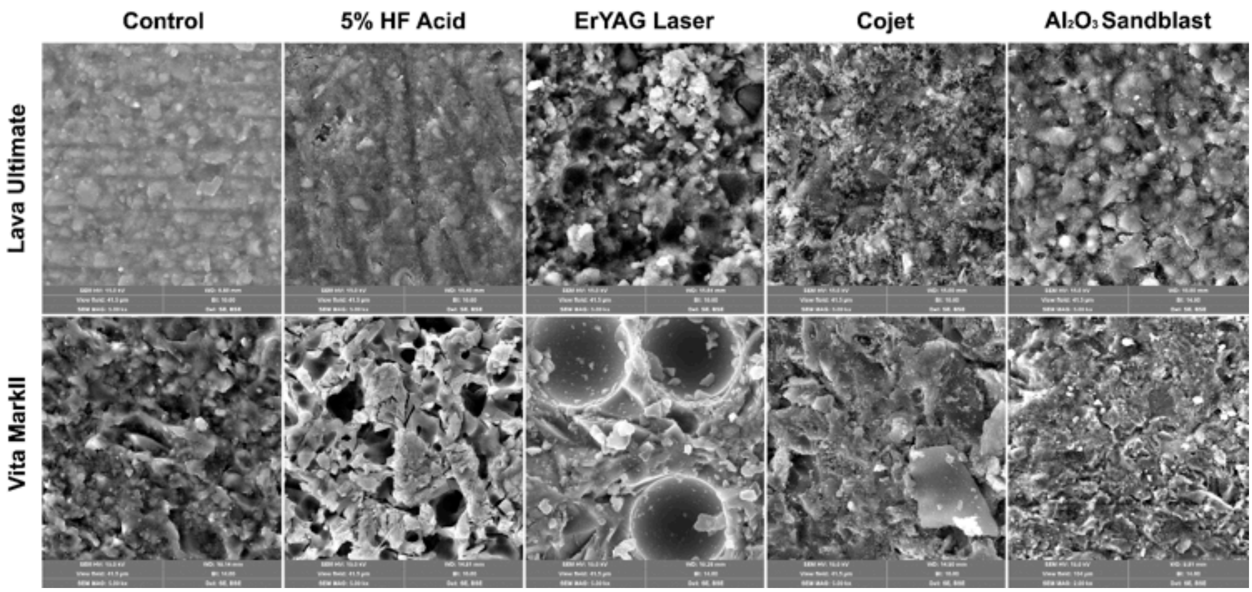

Figure 2. SEM images showing selected samples of surface treatment groups of CAD/CAM restorative materials.

DISCUSSION

Enhancing the bond strength between CAD/CAM restorations and resin cements is important for increasing fracture resistance and preserving the marginal integrity of the restorations (17,18). Mechanical or chemical pretreatments are required for adhesion surfaces (17).

A wide variety of surface conditioning methods have been suggested to increase the bond strength of restorations (19). Roughening with burs, HF or phosphoric acid etching, aluminum oxide abrasion (with or without silane coupling agents), tribochemical pretreatment with silica-coated alumina particles, and different laser applications are the most common surface conditioning methods in the literature (1,6,14,19,20). However, there is no consensus on the best surface conditioning method that provides optimal bond strength to indirect restorations (14).

This study aimed to evaluate the SBS of a self-adhesive resin cement with two different CAD-CAM materials after various surface treatments. The null hypothesis of the present study was rejected because the mean SBS values indicated that different surface treatment application methods affected the SBS of self-adhesive resin cement with two indirect restoration materials.

Chemical surface treatments, such as HF application, contribute to optimal bonding by increasing the surface roughness and surface energy in most acid-sensitive glass-containing materials such as ceramics and polymers (leucite-based ceramics and silica-based hybrid CAD/CAM materials) required for a strong micromechanical bond and wettability (17). In the literature, using different concentrations of HF is generally recommended for this purpose (21-23). Excessive acid etching of lithium disilicate ceramics with HF may weaken the bond strength; therefore, the etching duration must be balanced to prevent surface damage (24). In this study, 5% HF, which has been shown to create a rough surface on the most acid-sensitive indirect restorative materials, was used for 15s.

In the present study, HF did not affect the SBS of resin cement to Lava Ultimate ($p>0.05$) and increased the SBS values for VM, which is a glassy matrix feldspathic ceramic ($p<0.05$). HF created strong homogeneous patterns in the ceramic matrix of VM. This porous surface with microcavities in the glass matrix may enhance the bond strength of resin cement (22). The resin-based indirect composite used in this study had 20% organic content, which may have resulted in its low sensitivity to HF acid. Nagasawa *et al*. reported that the polymer network in the composition of polymer-infiltrated glass ceramics is not sensitive to the HF function and is not significantly affected by HF in terms of etching depth.(16) In addition, Elsaka *et al*. reported that etching resin-based composites for 90s increased their bond strength. (25) In this study, a short acid application duration (15s) and low concentration (5%) of etchant may have caused no change in SBS. In several studies, 9% HF was used at 20-120s (23,24,26).

Lasers are another method used for surface conditioning to increase the bond strength. Various studies have evaluated the effects of different lasers and laser intensities on the bond strength of indirect restorations and resin cements, and conflicting results have been obtained (27,28). The most frequently used lasers in the literature for surface pretreatment are Er: YAG, Er, Cs: YSGG, and Nd: YAG lasers (11, 28, 29). In this study, the Er: YAG laser was pretreated at a wavelength of 2940nm, 3 W power, 150mJ energy level, 20Hz frequency, and 700-ms long pulse to scan the entire specimen surface from a distance of 10mm. The SBS of resin cement to LU was negatively affected by Er: YAG laser pretreatment ($p<0.05$). Although laser treatment created a rougher surface, it did not improve the SBS, which is in accordance with the findings of Turker *et al*. (26). In their study, the mean SBS of RelyX U200 resin cement to LU in the Er: YAG laser group was (9.1± 5.4) not significantly different from that of the control group (9.4±2.9). The surface irregularities created by the Er: YAG laser may not have sufficient microdepth for the micromechanical retention of the resin cement (14). In addition, different intensities of Er: YAG laser may enhance the bond strength of resin-based indirect restorative materials.

Contrary to the results in LU, Er:YAG laser treatment SBS values were significantly higher than those of the control group for VM in this study ($p<0.05$). Several studies have reported that Er: YAG laser pre-treatment enhances the resin cement bond strength of CAD/CAM materials (1, 27,29,30).

Another method for conditioning surfaces is tribochemical silica airborne-particle abrasion, which is conducted by sandblasting with silica-coated particles instead of pure Al_2O_3 (17). In the present study, 30-μm silica coated Al_2O_3 (Cojet) was applied for 15s at 2.5 bar air pressure and 5 mm from the surface of the samples. Both indirect restoration CAD/CAM blocks (LU and VM) showed higher mean SBS values after Cojet pretreatment than the other groups ($p<0.05$). Tribochemical silica airborne-particles not only roughen the surface, they also support chemical retention by bonding silane and silica-coated restorative materials. The silica coating of restorative materials gives them the capacity to be reactive to silane. This may be the reason for the enhanced bond strength. Similar to the findings of the present study, Altan *et al*. (1) reported the highest SBS values in the Cojet groups of three CAD/CAM materials. In contrast, Papadopoulos *et al*. (17) found similar results between Al_2O_3 and Cojet for the bond strength of LU to resin cement.

Al_2O_3 abrasion is a widely used method for increasing mechanical retention by creating a rough surface using Al_2O_3 particles, and particles of various sizes have been used in several studies (15,17,20,29). In the present study, 40-μm Al_2O_3 particles under a pressure of 2.5 bar at a distance of 5mm for 15s were applied for sandblasting. There was no difference between the VM sandblasting and control groups according to the test results ($p>0.05$). In the LU groups, the sandblasting with Al_2O_3 group showed lower SBS values than the control group ($p<0.05$).

The lower mean SBS values of the indirect resin composite material is similar to the values reported by Strasser *et al*. (22) and Yoshihara *et al*. (31). The use of 40-μm Al_2O_3 at 2.5 bar pressure may damage the surface of the composite resin block. Strasser *et al*. (22) suggested that 50-μm Al_2O_3 at 1 bar is sufficient, which is lower than the pressure used in the present study. Similarly, Yoshihara *et al*. (31) reported that sandblasting caused cracks, 1-10μm in length, on the surface of a resin-based composite material (Shofu Block HC). These subsurface cracks are mostly seen inside the resin matrix and at the interface between the filler particles and resin matrix. Sandblasting also caused remarkable debonding of filler particles; therefore, the silane coupling agent could not compensate for this surface damage prior to bonding.

When the SEM images were examined, it was observed that HF did not cause enough change on the surface of the resin-based nano-ceramic (LU) for microretention. However, pit forms created by the dissolution of silica were observed on the surface of feldspathic glass-matrix ceramics (VM). The Er: YAG laser created larger deformations on the surface of VM than on LU. Cojet created irregular surfaces and Al_2O_3 created slight irregularities on the bonding surfaces in both groups.

The present study evaluated the effect of different surface treatments to SBS of self-adhesive resin cement of indirect resin restorations immediately after 24h; further research should be conducted on the adhesion of aged restorative materials. Other *in vitro* studies with longer aging intervals should be conducted in order to verify these surface conditioning strategies.

CONCLUSIONS

While Cojet application increased the SBS of the LU restorative material, other application methods did not differ from the control group. For VM, Al_2O_3 abrasion did not increase the SBS. However, other conditioning methods increased the bonding of resin cement to the CAD/CAM material. The application of Cojet yielded the highest SBS values. The use of appropriate surface pretreatment methods can enhance the bonding capacity of resin cements.

In this study, tribochemical pretreatment with silica-coated alumina particles (Cojet) appeared to be the most suitable surface treatment for indirect restoration materials and self-adhesive resin cement.

DISCLOSURE STATEMENT

The authors of this article certify they have no proprietary, financial, or other personal interest of any product, service, and/or company in this article.

AUTHOR CONTRIBUTION STATEMENT

Conceptualization and design: Ö.Ö.B and D.E.
Literature review: D.Ö.D. and A.K.
Methodology and validation: A.K. and Ö.Ö.B.
Formal analysis: Ö.Ö.B.
Investigation and data collection, Ş.M.
Resources: D.Ö.D.
Data analysis and interpretation: D.E.
Writing-original draft preparation: A.K.
Writing-review & editing: A.K.
Supervision: A.K.
Project administration and funding acquisition: Ö.Ö.B.

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