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## Effect of universal adhesives and surface treatments on shear bond strength to hybrid CAD/CAM Ceramics

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### Abstract

**Aim:** The purpose of this study is to investigate the effect of changes in hybrid ceramics to resin cement after the application of different surface treatments.

**Material and Method:** In our work, 330 pieces of 3 different hybrid CAD-CAM ceramics were used. The ceramics were divided into 5 subgroups and their surfaces were treated differently. These were set as bonding with hydrofluoric acid, bonding with CoJet, Silane application with hydrofluoric acid, only bonding application, and control group. The shear bond strength was measured with a universal tester, INSTRON. Values from the measurements were analyzed by one-way ANOVA and the Tukey HSD test was used to determine significant differences ( $p = 0,05$ ).

**Results:** Bonding values between CAD/CAM hybrid ceramic blocks subjected to the same surface treatment were statistically significant in all HF applied samples. ( $p < 0,05$ ) The highest bonding values were observed in HF + silane samples ( $52,24 \pm 7,4$  MPa) in the Vita Enamic group and the lowest bonding values were detected in HF + UQ samples in the Lava Ultimate group. ( $21, 26 \pm 4,4$ MPa)

**Conclusion:** Application of HF + Silane and HF + Adhesive can contribute to clinical success by increasing the resin cement joint strength of hybrid CAD/CAM ceramics.

**Keywords:** CAD/CAM, hybrid ceramics, surface treatment, universal bond, bond strength

### Introduction

In the case of restorations involving multiple teeth and tooth surfaces, extensive defects or cusp fractures, and in order to provide better mechanical properties to the restoration, indirect methods are generally preferred [1]. Ceramic and composite resin-based materials are used as an indirect restoration material. Hybrid ceramics; is a material designed to combine the good properties of ceramics, such as durability and color stability, and the good properties of composites such as flexural strength and low abrasive effect [2].

CAD/CAM is a computer-aided design and computer-aided production. CAD/CAM systems improved to provide long-lasting aesthetic indirect restorations [3]. New generation hybrid CAD/CAM blocks contain composite resin. These blocks have better mechanical properties compared to dental composites since they are polymerized at much higher temperatures and pressures [4].

Ceramic restorations must be exposed to some surface treatment prior to cementation. This stage is a very important step for the long life of restoration [5]. This treated surface, combined with silane, can demonstrate high bonding values. Indirect restorations have adhered to the tooth preparation with an adhesive cement. The cementation of glass ceramics was defined as hydrofluoric acid, silanization and resin cement, respectively. In indirect composites air-abrasion can be ensured by a good bonding with silanization [6, 7]. The micro-mechanical bonding between resin cement restoration affects the mechanical properties and clinical performance of restorations [5].

The newest product of adhesive systems is called "universal adhesive. This adhesive has considerably simplified and accelerated the adhesive protocol, and these systems actually represent a very important change in adhesive dentistry [8].

Universal adhesives should not be confused with the 7th generation all in one adhesive system. Universal adhesives have a wide range of indications suitable for use in a variety of procedures, such as etch&rinse, self-etch and selective-etch. In addition, direct and indirect restorations, as well as self-cure, light cure, and dual cure resin cements can also be used in combination.

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These adhesive systems are not only bonded to enamel and dentin but can also be bonded to restorative materials such as zirconium, metal, composite, silica-containing ceramic without the need for an extra agent such as silane [8].

All universal adhesives contain phosphate esters (MDP) as primary adhesive functional monomers. This versatile amphiphilic functional monomer provides a chemical bonding with the hydrophobic methacrylate group and methacrylate-based restoratives and cements, while hydrophilic phosphate group has the potential to chemically bond with dental tissues, metal, and zirconium [9-11].

In addition, some of the universal adhesive producers added silane to the adhesive formulation, suggesting that a single universal adhesive could be used instead of a separate silane application [8].

There is a study examining the bond strength of different surface applications of hybrid ceramics with resin cement.<sup>12</sup> However, no study has been found to investigate the bonding of universal adhesives to resin ceramics in hybrid ceramics.

The aim of our study is to investigate the effect of universal adhesives and different surface applications on the bond strength of resin cement to hybrid CAD/CAM ceramics with the shear bond strength test (SBS).

Our first hypothesis is that there is no difference between the hybrid CAD/CAM materials using similar surface treatments and the second hypothesis is that different surface treatments do not affect the performance of connecting the dental resin to the cement.

### Material and Method

This study was supported by Atatürk University Scientific Research Projects Coordinatorship (BAP)(THD-2018-6364) and approved by the Ethics Committee of Faculty of Dentistry (EKR-07/2017).

The applications of this study were performed at Atatürk University, Faculty of Dentistry, and Restorative Dentistry Laboratory. Three different hybrid CAD/CAM ceramics and 3 different adhesive systems were used in the study (Table. 1).

**Table 1:** Cad/Cam Materials and Universal Adhesives used in this study

CAD/CAM Hybrid Ceramic	Manufacturer	Composition	LOT No
<i>Vita Enamic (VE)</i>	Vita Zahnfabrik	Feldspar matrix and polymer material, UDMA, TEGDMA	59370
<i>Lava Ultimate (LU)</i>	3M ESPE	Bis-GMA, UDMA, Bis-EMA, TEGDMA, Silica Zirconia	N721602
<i>GC Cerasmart (GC)</i>	GC America	Silica, barium glass filler, Bis-MEPP, UDMA, DMA	1601061
Universal Adhesive			
<i>Scotchbond Universal Bond (SU)</i>	3M ESPE, St. Paul, ABD	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, fillers, ethanol, silan	658426
<i>G- Premio Bond (GP)</i>	GC Corporation, Tokyo, Japan	MDP, phosphoric acid ester monomer, dimethacrylate, 4-MET, MEPS, acetone, silicon dioxide	160413A
<i>Clearfil Universal Bond Quick (UQ)</i>	Kuraray, Medical, Sakazu, Okayama, Japan	MDP, amid monomer	AQ0052

### 1. Preparation of ceramic samples

In this study, 3 different hybrid Cad-Cam ceramics were used. These are Vita Enamic (Vita Zahnfabrik, Bad Sackingen, Germany), Lava Ultimate (3M ESPE, St. Paul, MN, USA) and GC Cerasmart (GC Corp., Tokyo, Japan)(Fig.1 and Table 1)

CAD/CAM hybrid ceramic blocks were cut and sliced to 12x14x1.5mm under water cooling with a diamond saw using the ISOMET device (ISOMET; Buehler, Lake Bluff, IL, USA). Then, these samples were divided into 4 equal parts with a yellow-belt flame-tipped diamond bur under water-cooling with an aerator (6x7x1.5mm). (Fig 2. and 3.)

A total of 330 ceramic samples were prepared with 110 samples in each ceramic group. Samples were embedded in a 14X25cm cylindrical acrylic block (Imicryl; Konya Turkey) and wet ground finished to #600, 800 and 1200 grid in sequence. The samples were ultrasonically cleaned (2 min in the distilled water, and air-dried for 1 min.).

### 2. Surface treatments of ceramic samples

Ceramic samples were randomly divided into 3 different universal adhesives: Scotchbond Universal(3M ESPE, St. Paul, USA), G-Premio Bond (GC Corporation, Tokyo, Japan), Clearfil Universal Bond Quick (Kuraray, Medical, Sakazu, Okayama, Japan). 4 different surface treatments were applied in each adhesive groups (Fig 4. and 5):

1. Adhesive; the samples were subjected to universal adhesive treatment in accordance with the manufacturer's instructions.
  - Scotchbond Universal (SU), apply and rub it for the 20s, 5s gently air dry, 10s light cure.
  - G-Premio Bond (GP), apply and leave undisturbed 10s

after application, dry thoroughly 5s under maximum air pressure, and 10s light cure.

- Clearfil Universal Bond Quick (UQ), apply adhesive then brush and rub it in for 10s, no waiting, dry by mild pressure more than 5s, and 10s light cure
2. CoJet and adhesive; 30 µm aluminum-trioxide particles coated with silica (Cojet sand, 3M ESPE, St.Paul, MN, USA), that was applied for 4 s at a pressure of 2.8 bar with a distance of 10 mm between the nozzle and the surface, the samples were ultrasonically cleaned and apply adhesive same as number 1.
  3. Hydrofluoric acid and adhesive; etched with 9% Hydrofluoric acid (Ultradent Porcelain Etch, Ultradent Dental Products, South Jordan, UT, USA) for 60s, then rinsed for the 60 s and air-dried, and apply adhesive same as number 1.
  4. Hydrofluoric acid and silane; hydrofluoric acid same as number 3., apply a puddle coat of Silane (Ultradent Dental Products, South Jordan, UT, USA) for 60 seconds, and dry.

All light-curing procedures were performed with LED curing light (Guilin Woodpecker, Medical Instrument, Guilin Guangxi, China- 800 - 1000 mW/cm<sup>2</sup>). 10 ceramic samples were used as a control group without surface treatment.

### 3. Resin cement application

Custom-made silicone disks (internal diameter, 2,5 mm; thickness, 2,5 mm) were fixed over the ceramic samples, and the cement Rely-X Ultimate (3M ESPE Dental Products, St. Paul, MN) was injected inside the silicon mold. All cements were light-cured for 20 s with an LED curing light

(Woodpecker, Medical Instrument, Guilin Guangxi, China), which was operated in standard mode. The light-curing unit emitted 800-1000 mW/cm<sup>2</sup> irradiance, as measured by the incorporated radiometer (Woodpecker LM-1 Light Meter). Once curing was completed on the silicone mold, the additional cure was performed each four sides of resin cement for the 20s. Then the molds were removed, and specimens were incubated in a dark/wet environment at 37 ± 1 °C for 24 h before testing.

**4. SBS testing**

Test specimens were placed inside the testing device, which was fixed in a universal testing machine (Instron 3350, Instron industrial products, Grove City, US). Shear loading was applied at the interface between the cement and ceramic surface at a cross-speed of 1 mm/min. The maximum debonding force (F, in N) for each specimen was recorded and used in calculating the SBS value (in MPa), according to the equation: SBS = F/A, where A is the cross-sectional area (in mm).

**5. Statistical analysis**

Statistical analysis was performed with SPSS 20.0 for

Windows (SPSS Inc., Chicago IL, USA). Descriptive statistics were generated. One-way ANOVA and Tukey HSD post hoc tests were used to examine differences among surface treatments (α = 0.05 significance level).

**Results**

The mean, standard deviation, and range of SBS values for the bonding of different surface treatments to different hybrid ceramics are summarized in Table 2. Statistically significant differences were found between the same surface treatment of dental ceramic samples and between different surface treatments of the same dental ceramics, as indicated by one-way ANOVA (Table 2.) (p<0,05).

In all CAD/CAM hybrid ceramic blocks, the lowest bond strength values were determined in the control groups where no surface treatment was applied (Vita Enamic: 10,11±2,5 MPa, Lava Ultimate: 13,6±5,6 MPa, GC Cerasmart: 8,03± 1,4 MPa)

In all CAD/CAM hybrid ceramic blocks, the highest bond strength values were determined in the samples we applied silane with hydrofluoric acid as surface treatment (p <0.05) (Vita Enamic: 52,24±7,4 MPa, Lava Ultimate: 39,65±9,3 MPa, GC Cerasmart: 37,85±8,4 MPa).

**Table 2:** Mean ± standard deviation values (MPa) of shear bond strength test data, and Tukey HSD multiple comparison test results

	VITA	LAVA	GC	p
Control	10,11±2,5 <sup>a,AB</sup>	13,6±5,6 <sup>ab, B</sup>	8,03±1,4 <sup>a, A</sup>	0,012*
GP	17,29±3,2 <sup>ab,B</sup>	12,44±2,6 <sup>a, A</sup>	19,59±5,5 <sup>bc, B</sup>	0,003*
UQ	18,44±5,1 <sup>ab,A</sup>	20,6±4,6 <sup>abcd,A</sup>	19,42±4,8 <sup>bc, A</sup>	0,631
SU	22,05±5,3 <sup>b,A</sup>	19,78±3,7 <sup>abc,A</sup>	18,22±6,2 <sup>b, A</sup>	0,309
CoJet +SU	23,17±5,4 <sup>bc A</sup>	25,92±6,3 <sup>cd, A</sup>	22,25±6,6 <sup>bcd,A</sup>	0,432
CoJet +GP	23,17±5,5 <sup>bc,A</sup>	23,89±6,6 <sup>cd, A</sup>	27,83±5,4 <sup>cd, A</sup>	0,213
CoJet +UQ	24,63±5,8 <sup>bcd,A</sup>	29,04±5,7 <sup>d, A</sup>	27,58±6 <sup>bcd, A</sup>	0,279
HF+UQ	34,29±11 <sup>cde,B</sup>	21,26±4,4 <sup>bcd,A</sup>	26,78±6,7 <sup>bcd,AB</sup>	0,006*
HF+GP	35,39±11 <sup>de,B</sup>	21,68±3,4 <sup>bcd,A</sup>	29,27±6,3 <sup>de,AB</sup>	0,003*
HF+SU	35,99±11,2 <sup>e,B</sup>	21,59±5,6 <sup>bcd,A</sup>	22,66±6,7 <sup>bcd, A</sup>	0,001*
HF+SILANE	52,24±7,4 <sup>f, B</sup>	39,65±9,3 <sup>e, A</sup>	37,85±8,4 <sup>e, A</sup>	0,002*
p	0,001*	0,001*	0,001*	

\*p<0,05 Different lowercase letters in the same column and different uppercase letters in the same row indicate statistical significance.

The bonding values between ceramic blocks with the same surface treatment were found to be statistically significant only in Lava Ultimate ceramics with GP universal adhesive and HF&adhesive treated samples (p <0.05). In the Vita

Enamic group, the HF&Silane samples with HF&adhesive showed the highest values significantly from other ceramics (P <0.05). However, Lava Ultimate and GC Cerasmart groups had similar bonding values (p>0,05).

**Table 3:** Mean (MPa), standard deviation (±) and Tukey HSD test results of ceramic samples applied with Scotchbond Universal Bond (SU)

	Control	SU	CoJet +SU	HF+SU	HF+Silane
VITA	10,11±2,5 <sup>a</sup>	22,05±5,3 <sup>b</sup>	23,17±5,4 <sup>bc</sup>	35,99±11,2 <sup>d</sup>	52,24±7,4 <sup>e</sup>
LAVA	13,6±5,6 <sup>a</sup>	19,78±3,7 <sup>ab</sup>	25,92±6,3 <sup>bc</sup>	21,59±5,6 <sup>abc</sup>	39,65±9,3 <sup>d</sup>
GC	8,03±1,4 <sup>a</sup>	18,22±6,2 <sup>b</sup>	22,25±6,6 <sup>bc</sup>	22,66±6,7 <sup>bc</sup>	37,85±8,4 <sup>d</sup>

\*p<0,05 Different lowercase letters in the same row indicate statistical significance.

In Table 3., it is seen that the Vita Enamic group has a much higher bonding value than other ceramic groups with

Scotchbond Universal(SU) Bond applied after the application of hydrofluoric acid (p<0,05).

**Table 4:** Mean(MPa), standard deviation (±) and Tukey HSD test results of ceramic samples applied with G-Premio Bond(GP)

	Control	GP	CoJet +GP	HF+GP	HF+Silane
VITA	10,11±2,5 <sup>a</sup>	17,29±3,2 <sup>ab</sup>	23,17±5,5 <sup>bc</sup>	35,39±11 <sup>de</sup>	52,24±7,4 <sup>f</sup>
LAVA	13,6±5,6 <sup>ab</sup>	12,44±2,6 <sup>a</sup>	23,89±6,6 <sup>cd</sup>	21,68±3,4 <sup>bcd</sup>	39,65±9,3 <sup>e</sup>
GC	8,03±1,4 <sup>a</sup>	19,59±5,5 <sup>bc</sup>	27,83±5,4 <sup>cd</sup>	29,27±6,3 <sup>de</sup>	37,85±8,4 <sup>e</sup>

\*p<0,05 Different lowercase letters in the same row indicate statistical significance.

In Table 4., in the Vita Enamic and GC Cerasmart groups, G-Premio bond application, following the application of

hydrofluoric acid, further increased the bonding compared to the Lava Ultimate group. (p<0,05)

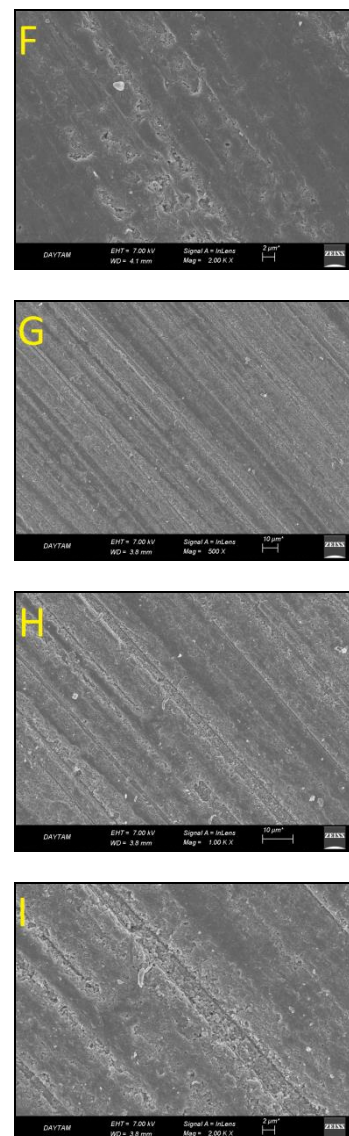
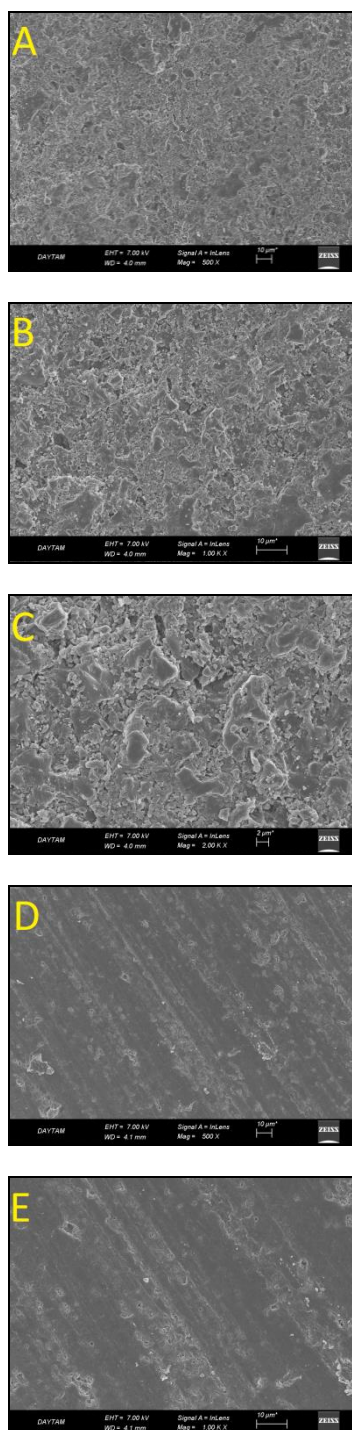
**Table 4:** Mean (MPa), standard deviation ( $\pm$ ) and Tukey HSD test results of ceramic samples applied with *Universal Bond Quick(UQ)*

	Control	UQ	CoJet +UQ	HF+UQ	HF+Silan
VITA	10,11 $\pm$ 2,5 <sup>a</sup>	18,44 $\pm$ 5,1 <sup>ab</sup>	24,63 $\pm$ 5,8 <sup>bcd</sup>	34,29 $\pm$ 11 <sup>cde</sup>	52,24 $\pm$ 7,4 <sup>f</sup>
LAVA	13,6 $\pm$ 5,6 <sup>a</sup>	20,6 $\pm$ 4,6 <sup>abc</sup>	29,04 $\pm$ 5,7 <sup>c</sup>	21,26 $\pm$ 4,4 <sup>abc</sup>	39,65 $\pm$ 9,3 <sup>d</sup>
GC	8,03 $\pm$ 1,4 <sup>a</sup>	19,42 $\pm$ 4,8 <sup>b</sup>	27,58 $\pm$ 6 <sup>bc</sup>	26,78 $\pm$ 6,7 <sup>bc</sup>	37,85 $\pm$ 8,4 <sup>d</sup>

\* $p < 0,05$  Different lowercase letters in the same row indicate statistical significance.

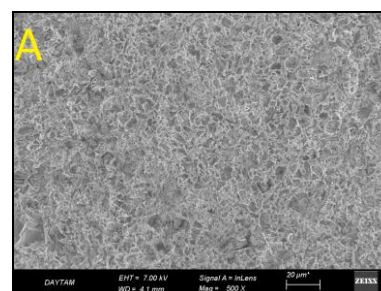
Table 5., shows that the Vita Enamic group has a much higher binding value than the other ceramic groups with Universal Bond Quick applied following the application of hydrofluoric acid. ( $p < 0,05$ )

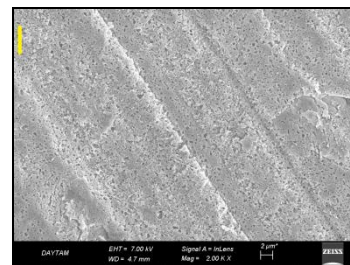
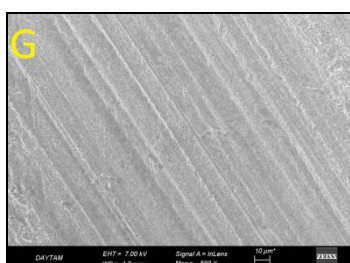
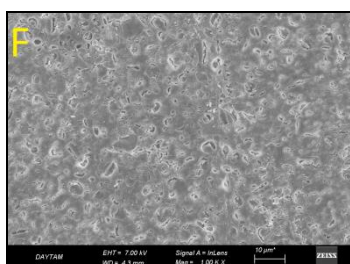
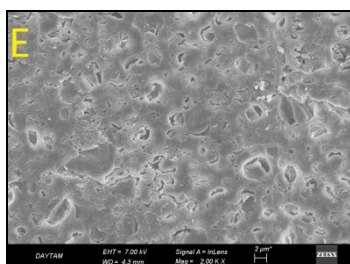
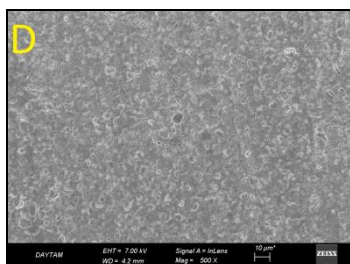
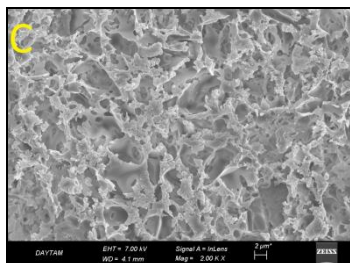
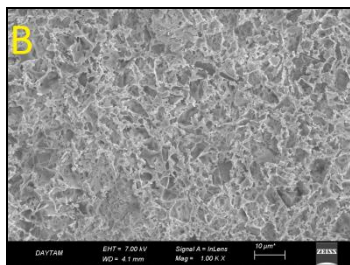
Representative SEM micrographs of 1200 grid sanding, HF and Cojet surfaces are presented in Figs. 1-3 respectively. Treatment with hydrofluoric acid and Cojet resulted in increased surface roughness with irregularities and pores on the treated surfaces of all materials tested.



**Fig 1:** SEM images obtained after sanding 1200 grids on ceramic surfaces. A: VITA 500x, B: VITA 1000x, C: VITA 2000x, D: LAVA 500x, E: LAVA 1000x, F: LAVA 2000x, G: GC 500x, H: GC 1000x, F: GC 2000x

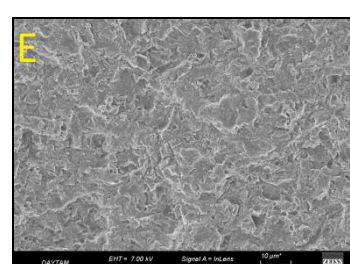
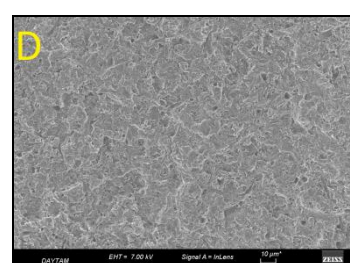
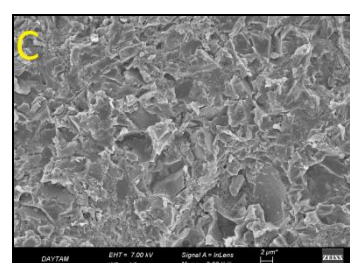
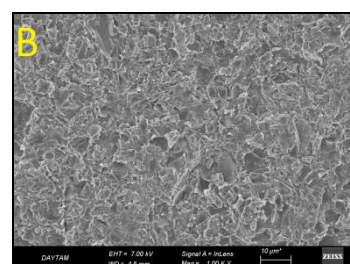
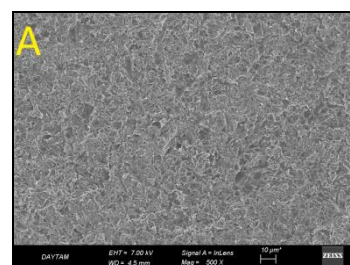
After 1200 grid sanding process; The Lava Ultimate and GC Cerasmart group show smoother surfaces than the Vita Enamic group. Vita Enamic has more porous structure was found. (Fig.1)

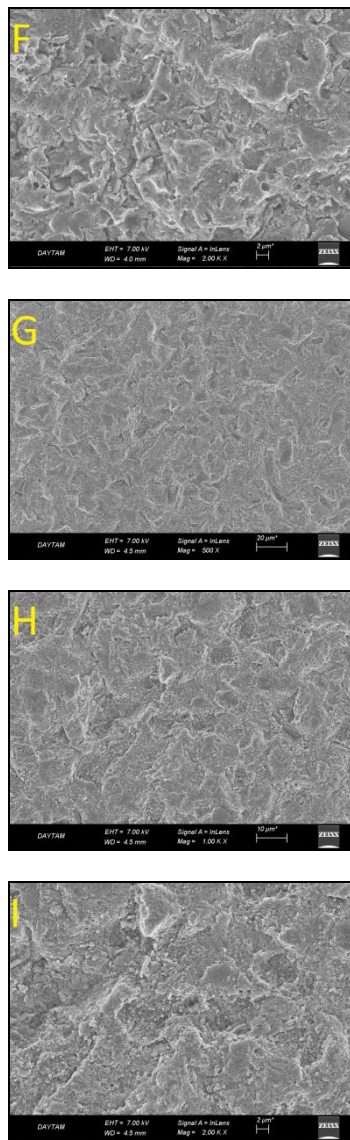




**Fig 2:** SEM images obtained after 9% HF acid application on ceramic surfaces. A: VITA 500x, B: VITA 1000x, C: VITA 2000x, D: LAVA 500x, E: LAVA 1000x, F: LAVA 2000x, G: GC 500x, H: GC 1000x, I: GC 2000x

After 9% HF acid application; Vita Enamic and Lava Ultimate ceramics seem to have more porosity than GC Cerasmart. (Fig. 2)





**Fig 3:** SEM images obtained from the CoJet application ready for ceramic surfaces. A: VITA 500x, B: VITA 1000x, C: VITA 2000x, D: LAVA 500x, E: LAVA 1000x, F: LAVA 2000x, G: GC 500x, H: GC 1000x, F: GC 2000x

After the CoJet application; it is observed that ceramic samples show surface properties close to each other. (Fig. 3)

### Discussion

Adhesive cementation processes are among the factors that demonstrate the clinical success of indirect restorations [13-15]. It has been reported that the use of resin cements increases the success in clinical and laboratory studies comparing traditional and resin cements [16-19].

Since a firm bond between the tooth and the restoration is critical to the longevity of the restoration, the optimal cementation strategy for new restorative materials should be sought.

There are two main methods for establishing the connection between ceramic and resin cement: micromechanical locking and chemical bonding. Micromechanical interlocking is achieved by acid application or air abrasion, while chemical bonding is achieved by silane binding agent.<sup>6</sup> Hydrofluoric acid roughening is the most recommended method in the literature for surface roughening of glass ceramics, which can be acidified [20-24].

Yen TW and *et al.* [25] reported that HF acid definitely improves the bonding in dental CAD/CAM hybrid blocks

with resin content by dissolving the silica-containing glassy matrix portion. In our study, it was found that the bonding was positively affected in all three ceramic groups containing HF acid.

The difference between the ceramic groups applied the same surface treatment in terms of bond strength to resin cement was found in HF + adhesive and GP treated samples. Among the samples treated with hydrofluoric acid, the highest binding yield value was observed in the Vita Enamic group. Thus, our first hypothesis was partially rejected.

Cekiç-Nagas I, and *et al.* [26] found that Vita Enamic specimens showed higher bonding strength to resin cement than Lava Ultimate and GC Cerasmart specimens. They attributed the high filler content (86% by mass) to reply to the high bonding ability to resin cement.

Campos F and *et al.* [12] reported that Vita Enamic with HF showed higher bond strength than feldspathic ceramic samples with HF. They have linked this to the fact that the use of silane and the composite content in the Vita Enamic reinforce the linkage between the resin cement monomers.

In our study, Vita Enamic group showed similar results with silane applications, which showed the highest value in all three adhesive applications among the universal adhesive applications following hydrofluoric acid.

In the Vita Enamic and Lava Ultimate groups (Vita + GP, Vita + UQ, Lava + GP, Lava + UQ, Lava + SU), where only universal adhesives were applied, low values were obtained similar to the control group. Furthermore, all other surface treatments positively affected the bonding strength to the resin cement. In this case, our second hypothesis was partially rejected.

Recommended surface applications for adhesive cementation are indicated as hydrofluoric acid [27, 28] for acidifying ceramic, or as air-abrasion for indirect composite restorations in which the acid may not be effective [29, 30]. And these surface treatments always improve the bonding positively.

Park JH and *et al.* [31] in their study with the hybrid ceramic Lava Ultimate, the highest bond strength to resin cement was found to be in samples using universal adhesive with air-abrasion. In addition, universal adhesive samples with hydrofluoric acid showed the lowest binding values.

In our study, the CoJet application in Lava Ultimate group showed higher bond strength than hydrofluoric acid application. The reason for this is that it contains zirconia and hydrofluoric acid is not very effective.

Blatz *et al.* [32] different adhesive resin cement 50-µm aluminum oxide powders with sandblasted and non-sandblasted ceramic samples compared the bonding strength of their work, they stated that air abrasion increases the bond strength. In our study, in all three dental Cad-Cam hybrid blocks, there was no statistically significant difference between the resin bond strengths of CoJet + Adhesive treated samples compared to the adhesive applied samples alone, but the increase was determined.

In our study, it was observed that universal adhesive application increased the bond strength with resin cement. In the Lava Ultimate ceramic group alone, the application of the G-Premio bond showed almost the same bond strength as the control group.

In the present study, SEM images of all surface treatments were obtained in Vita Enamic samples and rougher surfaces. Only in Cojet application, all ceramics showed similar surface porosity. There are studies showing that the effect of surface applications on the bonding is strictly dependent on the content of the material. However, it is a fact that surface

application processes certainly change the surface topography and contribute positively to the bonding<sup>[33]</sup>.

Our study suggests that the use of universal adhesive as a surface treatment for some CAD/CAM hybrid ceramics may effectively enhance the resin bond strength between resin cement and Cad/Cam ceramics. This may depend not only on the content of the CAD/CAM material but also on many factors such as production. It is important that the newly developed materials are compatible and facilitate clinical practice. Therefore, more in-vitro and in-vivo studies are needed to develop new adhesive systems, Cad/Cam materials, and resin cement bonding.

## Conclusions

Based on the results presented, the following conclusions can be made:

1. Among all CAD/CAM hybrid ceramic blocks, the highest bond strength values were determined in samples treated with HF + Silane (Vita Enamic: 52.24MPa, Lava Ultimate: 39.65MPa, GC Cerasmart: 37.85MPa).
2. In the Vita Enamic group; The application of HF + universal adhesive showed resistance to resin cement bonding similar to those applied to HF + Silane.
3. In the Lava Ultimate group; Although CoJet + Universal adhesive application showed higher resin bond strength than HF + Universal adhesive application, it was not statistically significant.
4. In the GC Cerasmart group; HF + GP samples showed similar resins cement bond strength as HF + Silane samples.
5. The 1200 grid sanding process and 9% HF application showed a more porous structure in the Vita Enamic group than in the other two ceramic groups.

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