

Comparison of the effects of different surface treatment methods on the bonding strength of CAD/CAM blocks to repair composites

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ABSTRACT

Aims: The aim of our study is to evaluate the effects of surface treatments performed prior to composite resin repair on the shear bond strength of one ceramic CAD/CAM material (IPS e.max) and two different CAD/CAM materials with resin additions (Lava Ultimate and Vita Enamic) and to investigate the contribution of milling roughening to other surface treatments.

Methods: In our study, a total of 189 samples with a thickness of 2 mm were prepared, with 63 samples from each of the IPS e.max, Lava Ultimate, and Vita Enamic blocks. The test materials were divided into nine subgroups based on surface treatment techniques (phosphoric acid, HF acid, sandblasting, Cojet, milling + phosphoric acid, milling + HF acid control, milling + sandblasting, milling + Cojet, and control). After the thermal cycling process was applied, silane + bond + composite resin was applied to all sample surfaces. Subsequently, the samples were subjected to a shear bond strength test. Fracture types were evaluated using a stereomicroscope and SEM. Data were analyzed using the Mann-Whitney U, Kruskal-Wallis H, and post-hoc tests.

Results: It was found that the differences between all materials in terms of bonding strength were statistically significant (Vita Enamic > Lava Ultimate > IPS e.max) ($p < 0.05$). The bond strength of the control group was found to be statistically significantly lower than that of the HF acid surface treatment (HF acid > Cojet > Sandblasting > Phosphoric acid > Control) ($p < 0.05$). The highest number of adhesive fractures was observed in the IPS e.max material after all surface treatments, while the highest number of mixed fractures was detected in the Lava Ultimate and Vita Enamic materials. Although milling increased bond strength, it was not statistically significant ($p > 0.05$).

Conclusion: Surface treatments applied prior to the repair of ceramic materials with composite resin have increased the bonding strength.

Keywords: CAD/CAM ceramics, composite resin, surface roughening, bonding strength, repair

INTRODUCTION

One of the most preferred materials in indirect restoration in dentistry is dental ceramics. These materials, which can mimic the translucency and color depth of natural teeth, are highly resistant to wear and biocompatible.^{1,2} Compared to other materials, they cause less plaque accumulation. Their thermal expansion coefficients are similar to those of dental tissues, and they are corrosion-resistant. Due to the strong intermolecular bonds in their structure, they are resistant to chemical substances. Dental ceramics are primarily composed of feldspar, kaolin, and quartz.³ Feldspar (70-80%) provides transparency to the material, while kaolin (3-5%) provides opacity. Quartz (10-30%) increases dimensional stability and durability.⁴

Dental ceramics are classified according to their general structure as glass-matrix ceramics, polycrystalline ceramics, and resin-matrix ceramics.^{5,6} Based on their crystal structures,

they are categorized as aluminum oxide, magnesium aluminum oxide, zirconium oxide, leucite, lithium disilicate, lithium phosphate mica, and fluorapatite. Based on their manufacturing techniques, they are grouped into systems that are shaped by heat and pressure, castable, slip-cast, or prepared using CAD/CAM technology.⁴

In CAD/CAM systems, the three-dimensional design of the restoration is created in a computer environment after taking optical measurements with an intraoral scanner.⁷ Thus, there is no need for traditional measurement procedures and temporary crown treatment, and the risk of cross-infection is eliminated.⁸ Thanks to this system, which uses special blocks made from different materials, restoration production is completed in a single session without the need for laboratory procedures.⁹ Commonly preferred materials include feldspathic ceramics, glass ceramics reinforced with



lithium disilicate or leucite, oxide ceramics, glass ceramics reinforced with lithium silicate and zirconia particles, resin matrix ceramics, composites, polymers, and certain metals.¹⁰

The main structure of feldspathic ceramic blocks, which can mimic the natural tooth color due to their high glass content, is composed of silica and feldspar particles (30%).^{11,12} Prior to cementation, these materials undergo a roughening process using hydrofluoric (HF) acid.¹³ Glass ceramics reinforced with lithium disilicate (70%)¹⁴ are used only as substructure materials because they strengthen the substructure of ceramic restorations and reduce light transmission. Ceramics reinforced with leucite (17-25%) undergo greater shrinkage compared to feldspathic ceramics. Since they exhibit aesthetics and wear similar to natural teeth, they are preferred in metal-free inlay/onlay and crown restorations.¹⁵

In glass-infiltrated oxide ceramics, which have superior physical and mechanical properties, glass is infiltrated into the spaces between alumina or zirconia crystals in an attempt to eliminate the porous structure. The absence of glass in the composition of polycrystalline ceramics has increased their fracture resistance.¹⁶ Both glass-infiltrated and polycrystalline ceramics undergo a silica coating (tribochemical blasting) process on their inner surfaces using a Cojet device.¹⁷ Resin matrix ceramics, on the other hand, are produced to combine the positive properties of ceramics (75%) and composite resins. The voids between the sintered matrix structures of these materials are filled with polymer. This procedure reduces the risk of cracking commonly encountered in ceramic materials.¹⁸ Since resin matrix ceramics are polymerized at high temperatures during production, they do not require any firing or glazing after the carving process. They are commonly preferred for inlay/onlay, full crown, and laminate veneer restorations in patients with bruxism.¹⁹

For clinical applications, it is generally recommended to use CAD/CAM blocks (IPS e.max, Lava Ultimate, Vita Enamic, Cerec Blocks, VitaMark II, etc.) that offer the advantages of both ceramic and composite resin.^{20,21} IPS e.max blocks, reinforced with lithium disilicate crystals (40%), have a semi-crystalline glass-ceramic structure.^{22,23} Lava Ultimate blocks, composed of a resin and nano-ceramic mixture, contain nano-ceramic particles (80%) embedded in the resin matrix.²⁴ Vita Enamic hybrid ceramic blocks contain feldspar (86%) and polymer (14%).²³

The primary causes of failure in CAD/CAM-produced posterior restorations include improper tooth treatment, parafunctional habits, trauma, loss of adhesive bonding, internal stresses generated during production, and localized fractures caused by porosity within the material.^{25,26} Such situations require the restoration to be completely replaced or repaired. Replacing the restoration causes trauma to the tooth surface, time loss, and additional costs. Repairing the restoration allows it to regain function and aesthetics in a short time and at low cost.^{25,27}

Removing the restoration for repair outside the mouth increases the risk of fracture in the tooth.²⁷ This is why intraoral repair procedures have gained prominence. In intraoral repair of restorations, the use of composite resins capable of bonding to ceramic materials or the bonding of broken ceramic pieces using resin cement is considered.²⁸ The type, composition, and particle size of the composite resin

to be used are the most important factors affecting bonding strength and the success of the repair process.^{29,30}

To increase the bonding strength of the restoration materials to be used, it is recommended that the exposed fracture surfaces be subjected to certain mechanical or chemical processes (milling, acid or laser etching, sandblasting with aluminum oxide/silica-modified aluminum oxide particles).³¹⁻³³ Milling roughening removes the unsupported ceramic structure and smear layer, creating an area conducive to bonding. However, the use of a burr can cause cracks to form throughout the ceramic.³⁴ Acid etching (HF acid, phosphoric acid, or APF gel) is preferred, especially on glass ceramic surfaces.^{35,36} HF acid is recommended for surface treatment of ceramics containing more than 15% silica oxide.^{37,38}

On oxide ceramic or metal surfaces, the air abrasion/blasting method (spraying Al₂O₃ particles at a pressure of 2-2.5 bar, from a distance of 10 mm, for 10-15 seconds at a right angle to the ceramic surface) is used to ensure micromechanical retention.³⁹ The most significant disadvantage of this application is that it may cause surface damage during restoration and predispose to fractures in the long term.⁴⁰ In acid-resistant ceramics with high crystal content (aluminum or zirconium), the ceramic surface is more often roughened with silica-modified aluminum oxide particles.⁴¹ This method increases the silica content of the ceramic, enabling it to react better with the bonding silane agent.⁴²

The aim of our study is to determine the effect of different surface treatments on the bond strength of composite resin in fractures that may occur in two different CAD/CAM materials with resin addition (Lava Ultimate, Vita Enamic) and one ceramic CAD/CAM material (IPS e.max).

METHODS

This study did not require ethical approval as it did not involve any human subjects or animal experiments. All procedures were carried out in accordance with the ethical rules and the principles.

The materials used in the study are shown in **Table 1**.

The IPS e.max lithium disilicate glass ceramic test specimens used in the study were produced from CAD LT A2 C14-coded CAD/CAM blocks (12x14x18 mm). Lava Ultimate resin hybrid nano-ceramic test specimens were produced from A2 LT 14L-coded CAD/CAM blocks (12x14x18 mm). The Vita Enamic hybrid ceramic test samples were produced from CAD/CAM blocks (12x14x18 mm) coded 2M2-T EM-14. Using a minitom precision cutting device (at a speed of 200 rpm), 63 samples measuring 12x14 mm and 2 mm thick were prepared from the surface of all blocks. After the burrs on the edges of the samples were smoothed with a ceramic finishing bur, they were cleaned with distilled water. IPS e.max samples were crystallized in a porcelain furnace according to the manufacturer's recommendations.

All material samples were embedded in 3x3x2 cm autopolymerizing acrylic resin molds with the surfaces to be treated exposed. Prior to surface treatment, the samples were leveled with 600, 800, and 1200 grit silicon carbide abrasives. The samples were soaked in distilled water for 10 minutes and dried with an air spray. A total of 63 acrylic blocks per material were divided into nine subgroups of seven each for different surface treatments (**Figure 1**).



Table 1. Materials used in the study

Materials	Content of the materials	Lot number	Manufacturer
IPS e.max	Lithium disilicate glass ceramic (SiO ₂ 57-80%, Li ₂ O 11-19%, K ₂ O 0-13%, P ₂ O ₅ 0-11%, ZrO ₂ 0-8%, ZnO 0-8%, others and coloring oxides 0-12%)	V33396	Ivoclar Vivadent, Schann, Liechtenstein
Lava Ultimate	Resin nano-ceramic (BisGMA/TEGDMA, By weight 80% nano-ceramic)	N652686	3M, St. Paul, USA VITA, Zahnfabrik, Bad Sackingen, Germany
Vita Enamic	Hybrid Ceramics (UDMA, By weight 86% feldspar ceramics, 14% polymer, 58-63% SiO ₂ , 20-23% Al ₂ O ₃ , 9-11% Na ₂ O, 4-6% K ₂ O, 0.5-2% B ₂ O ₃ , <1% Zr ₂ O and CaO)	54210	VITA, Zahnfabrik, Bad Sackingen, Germany
Filtek Z250	BIS-GMA, UDMA, BIS- EMA, zirconia/silica fillers	N879677	3M ESPE, Seefeld, Germany
Ultradent Porcelain Etch	9% hydrofluoric acid	BDMZW	Ultradent Products Inc, USA
Panora 200 Etch	37% phosphoric acid	18025	Imicryl
Cojet	30 µm Al ₂ O ₃	644521	3M ESPE
Clearfil Ceramic Primer Plus	3-Methacryloxypropyl-trimethoxysilane 10-MDP, Ethanol	6K0027	Kuraray Noritake Dental Inc, Sakazu, Kurashiki, Okayama, Japan
Clearfil S ³ Bond	10-Methacryloyloxydodecyl dihydrogen phosphate (MDP) Bisphenol A diglycidyl methacrylate (Bis-GMA) 2-hydroxyethyl methacrylate (HEMA) Hydrophobic dimethacrylate dl-camphorquinone Ethyl alcohol, Water, Silanized colloidal silica	230015	Kuraray Noritake Dental Inc, Sakazu, Kurashiki, Okayama, Japan
Korox	50 µm	14171620912	Bego, Bremen, Germany

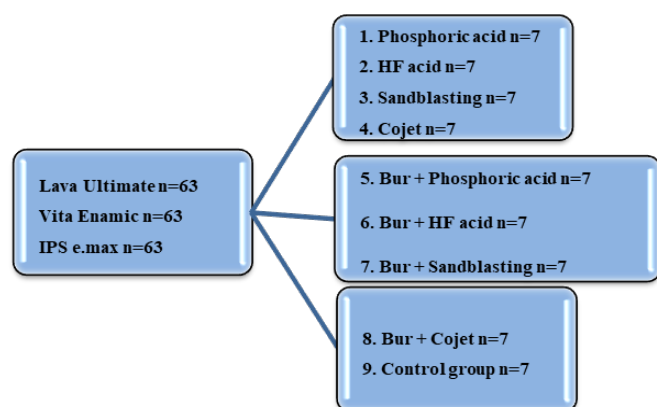


Figure 1. Distribution of test models according to groups

For each material, a 37% phosphoric acid gel was applied to the surface of the samples in the first group for 20 seconds, then washed with an air-water spray for 10 seconds and dried. For the second group of samples, a 9% HF acid gel was applied to the surface for 90 seconds, then washed with an air-water spray for 20 seconds and dried. The surface of the samples in the third group was treated with 50 µm Al₂O₃ sand at a pressure of 2.8 bar, from a distance of 10 mm, at an angle of 90°, for 10 seconds, using a sandblasting device. The sandblasted samples were cleaned in distilled water for 5 minutes. The surfaces of the samples in the fourth group were coated with silica using a Cojet system (at a pressure of 2.3 bar, from a distance of 10 mm, at an angle of 90°, for 15 seconds, with Si₂O sand particles of 30 µm in size) and cleaned with distilled water.

The surfaces of the samples in the fifth group were roughened using a green belt flame shape bur (150 µm) and 37% phosphoric acid gel. The surfaces of the samples in the sixth group were treated with green belt flame shape bur + 9% HF acid. The surfaces of the samples in the seventh group were roughened using a green belt flame shape bur + Al₂O₃ sandblasting process. The surfaces of the samples in the eighth group were treated with a green belt flame shape bur + Cojet process. The samples in the ninth group, which were not subjected to surface treatment, were separated as a control group and cleaned with distilled water.

Clearfil Ceramic Primer Plus was applied to the surface of all prepared samples and allowed to evaporate for 60 seconds. Clearfil S³ bond was applied using an application brush for 20 seconds and polymerized for 10 seconds using an LED light device with a light intensity of 800-1000 mW/cm², in accordance with the manufacturer's recommendations. After completing the surface treatments, composite resin material (Filtek Z250) was applied to the surface of all samples using plastic tubes with a diameter and height of 5 mm, using a layering technique, and polymerized with an LED light device. After removing the plastic tubes, light was applied for 60 seconds from four different angles. After the samples were kept in an oven for 24 hours (in distilled water at 37°C), they were subjected to 1000 thermal cycles at 5-55°C (with a soak time of 40 seconds and a transfer time of 6 seconds). They were then kept in the oven again for 24 hours.

The shear bond strength measurements of the samples were performed at the Research Laboratory of the Faculty of Dentistry, Erciyes University. During the measurement, the operator placed the metal tip of the universal testing machine (Instron), which ended in a knife edge, parallel to the composite-ceramic interface (Figure 2). Force was applied at a rate of 0.5 mm per minute. After obtaining the force value at the moment the composite sample separated from the ceramic surface, the shear bond strength was calculated using the following formula (in MPa) by dividing the force value by the surface area of the fracture zone (19.625 mm²).

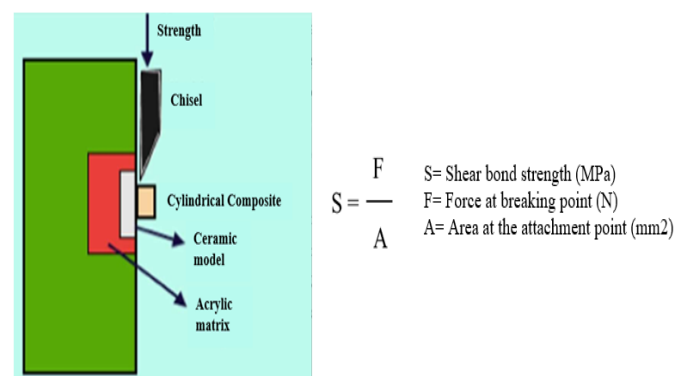


Figure 2. Shear test procedure



All samples were examined under a stereomicroscope (at 10x magnification) to determine the type of fracture. Fractures occurring at the composite/ceramic interface were classified as adhesive fractures, those occurring within the composite layer or ceramic were classified as cohesive fractures, and those involving both adhesive and cohesive fractures were classified as mixed fractures. Samples that fractured without being subjected to testing were classified as premature fractures. All samples were coated with approximately 45 Angstroms of Au/Pd (80% Au, 20% Pd) for 15 seconds and imaged and photographed under SEM at magnifications of x200, x500, x1000, and x2000. The obtained data were analyzed using IBM SPSS Statistics Version 22 software and statistically evaluated using the Shapiro-Wilk test. The Mann-Whitney U and Kruskal-Wallis H tests were used to examine the differences between the groups. If the differences were found to be significant, a post-hoc multiple comparison test was used.

RESULTS

In groups where milling was not applied, In IPS e.max material, the bond strength of the sandblasting surface treatment was found to be statistically significantly lower than that of the HF acid and phosphoric acid groups, and the bond strength of the Cojet and control groups was also found to be statistically significantly lower than that of the phosphoric acid group ($p < 0.05$). In Lava Ultimate material, the bond strength of the control group was found to be statistically significantly lower than that of the Cojet and HF acid surface treatments ($p < 0.05$). In Vita Enamic material, the bond strength of the phosphoric acid group was found to be statistically significantly lower than that of the HF acid and sandblasting surface treatments ($p < 0.05$) (Table 2).

In the Cojet, HF acid, and sandblasting surface treatment groups, the bonding strength of IPS e.max material was found

to be statistically significantly lower than that of Vita Enamic material ($p < 0.05$). In the phosphoric acid treatment group, no statistically significant differences were observed between the bond strengths of the materials ($p > 0.05$). In the control surface treatment group, the bond strengths of IPS e.max and Lava Ultimate materials were found to be statistically significantly lower than that of Vita Enamic material ($p < 0.05$) (Table 3).

In the groups treated with milling, in IPS e.max material, the bonding strength of Cojet and sandblasting surface treatments was found to be statistically significantly lower than that of HF acid surface treatments ($p < 0.05$). In Lava Ultimate material, the bond strength of the phosphoric acid group was found to be statistically significantly lower than that of Cojet and HF acid surface treatments ($p < 0.05$). In Vita Enamic material, the bond strength of the HF acid group was found to be statistically significantly lower than that of the Cojet surface treatment ($p < 0.05$) (Table 4).

In the Cojet and Sandblasting surface treatment groups, the bonding strength of IPS e.max material was found to be statistically significantly lower than that of Lava Ultimate and Vita Enamic materials ($p < 0.05$). In the HF acid surface treatment group, the bond strength of IPS e.max material was found to be statistically significantly lower than that of Lava Ultimate material ($p < 0.05$). In the phosphoric acid surface treatment group, the bond strengths of IPS e.max and Lava Ultimate materials were found to be statistically significantly lower than that of Vita Enamic material ($p < 0.05$) (Table 5).

The effect of milling on bonding strength for surface treatments was not found to be statistically significant ($p > 0.05$) (Table 6).

It was found that the differences between all materials in terms of bonding strength were statistically significant (Vita Enamic > Lava Ultimate > IPS e.max) ($p < 0.05$) (Figure 3).

Table 2. Bond strength of materials in the non-bur group according to different surface treatments

		Bond strength (Mpa)						Kruskal-Wallis H Test	
The group without bur application		n	Mean	Median	Min	Max	SD	Rank Avg.	p
IPS e.max	Cojet	7	5.39	5.22	3.81	7.78	1.37	13.71	26.033 0.001
	HF acid	7	9.21	8.89	7.63	12.73	1.71	25	
	Phosphoric acid	7	12.85	13.78	9.42	14.64	2	31.43	
	Sandblasting	7	3.44	2.49	1.36	6.75	2.1	7.43	
	Control	7	5.1	4.91	2.47	8.46	1.9	12.43	
	Total	35	7.2	6.23	1.36	14.64	3.85	4-2 4-3 5-3 1-3	
Lava Ultimate	Cojet	7	10.21	11.55	3.56	17.82	5.79	22	14.915 0.005
	HF acid	6*	12.87	12.71	9.68	17.09	2.38	24.83	
	Phosphoric acid	7	7.08	3.93	2.2	13.65	4.94	14	
	Sandblasting	7	8.75	6.01	4.09	15.96	5.06	21.14	
	Control	7	3.41	3.5	2.54	3.87	0.44	6.57	
	Total	34	8.33	5.51	2.2	17.82	5.11	5-1 5-2	
Vita Enamic	Cojet	7	12.94	12.87	9.36	18.27	3.01	11.57	17.012 0.002
	HF acid	7	16.47	15.72	13.01	18.84	2.28	24.71	
	Phosphoric acid	7	11.99	11.63	10.39	15.05	1.86	8	
	Sandblasting	7	17	17.31	13.29	19.95	2.11	26.14	
	Control	7	15.41	15.1	14.57	16.83	0.96	19.57	
	Total	35	14.76	15.02	9.36	19.95	2.84	3-2 3-4	

*Groups with premature fractures, Min: Minimum, Max: Maximum, SD: Standard deviation



Table 3. Bond strength of different surface treatments according to materials in the non-bur group

The group without bur application		Bond strength (Mpa)						Kruskal-Wallis H test	
		n	Mean	Median	Min	Max	SD	Rank Avg.	p
Cojet	IPS e.max	7	5.39	5.22	3.81	7.78	1.37	6.14	7.688 0.021
	Lava Ultimate	7	10.21	11.55	3.56	17.82	5.79	11.57	
	Vita Enamic	7	12.94	12.87	9.36	18.27	3.01	15.29	
	Total	21	9.51	9.36	3.56	18.27	4.85		
HF acid	IPS e.max	7	9.21	8.89	7.63	12.73	1.71	4.57	14.059 0.001
	Lava Ultimate	6*	12.87	12.71	9.68	17.09	2.38	10.5	
	Vita Enamic	7	16.47	15.72	13.01	18.84	2.28	16.43	
	Total	20	12.85	12.72	7.63	18.84	3.71		
Phosphoric acid	IPS e.max	7	12.85	13.78	9.42	14.64	2	14.43	5.351 0.069
	Lava Ultimate	7	7.08	3.93	2.2	13.65	4.94	6.86	
	Vita Enamic	7	11.99	11.63	10.39	15.05	1.86	11.71	
	Total	21	10.64	11.63	2.2	15.05	4.04		
Sandblasting	IPS e.max	7	3.44	2.49	1.36	6.75	2.1	5.14	14.382 0.001
	Lava Ultimate	7	8.75	6.01	4.09	15.96	5.06	10.21	
	Vita Enamic	7	17	17.31	13.29	19.95	2.11	17.64	
	Total	21	9.73	6.75	1.36	19.95	6.56		
Control	IPS e.max	7	5.1	4.91	2.47	8.46	1.9	10	15.636 0.001
	Lava Ultimate	7	3.41	3.5	2.54	3.87	0.44	5	
	Vita Enamic	7	15.41	15.1	14.57	16.83	0.96	18	
	Total	21	7.98	4.91	2.47	16.83	5.56		

*Groups with premature fractures, Min: Minimum, Max: Maximum, SD: Standard deviation

Table 4. Bond strength of materials in the bur applied group according to different surface treatments

Bur applied group		Bond strength (Mpa)						Kruskal-Wallis H test	
		n	Mean	Median	Min	Max	SD	Rank Avg.	p
IPS e.max	Cojet	7	2.42	1.94	1.09	3.96	1.2	8.43	16.797 0.001
	HF acid	7	10.95	10.54	8.4	16.25	2.6	23	
	Phosphoric acid	6*	3.77	3.28	3.11	5.18	0.87	13.67	
	Sandblasting	6*	2.44	1.81	1.08	5.16	1.56	8.17	
	Total	26	5.03	3.38	1.08	16.25	4.04		
Lava Ultimate	Cojet	7	13.89	14.67	9.41	16.37	2.4	17.29	14.968 0.002
	HF acid	7	15.52	15.93	10.39	21.99	4.25	19.29	
	Phosphoric acid	7	5.74	5.75	4.41	7.54	1.19	4.29	
	Sandblasting	6*	12.99	12.18	6.44	20.53	4.61	15.33	
	Total	27	12	11.89	4.41	21.99	5		
Vita Enamic	Cojet	6*	18.26	18.1	14.48	21.27	2.26	22.17	10.076 0.018
	HF acid	7	13.87	13.91	12.17	15.91	1.44	8.29	
	Phosphoric acid	7	15	15.61	11.84	16.95	1.86	13.29	
	Sandblasting	7	15.27	14.62	13.83	18.15	1.75	13.43	
	Total	27	15.5	14.74	11.84	21.27	2.35		

*Groups with premature fractures, Min: Minimum, Max: Maximum, SD: Standard deviation

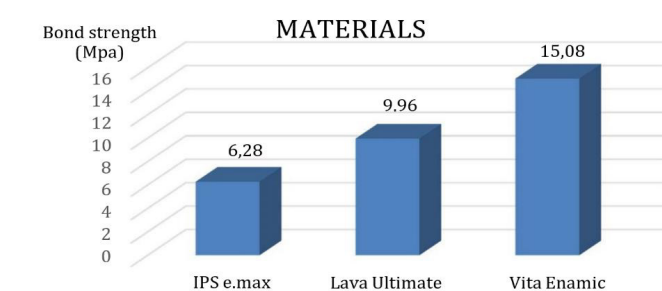


Figure 3. Differences between materials in terms of bond strength

The bond strength of the control group was found to be statistically significantly lower than that of the HF acid surface treatment (HF acid > Cojet > Sandblasting > Phosphoric acid > Control) ($p < 0.05$) (Figure 4).

The types of fractures identified at 10x magnification under a stereomicroscope at the ceramic-composite interface are shown in Table 7. After all surface treatments, the highest number of adhesive fractures was observed in IPS e.max material, while the highest number of mixed fractures was



Table 5. Bonding strength of different surface treatments according to materials in the bur applied group

Bur applied group		Bond strength (Mpa)						Kruskal-Wallis H test		
		n	Mean	Median	Min	Max	SD	Rank Avg.	H	p
Cojet	IPS e.max	7	2.42	1.94	1.09	3.96	1.22	4	15,287	0.001
	Lava Ultimate	7	13.89	14.67	9.41	16.37	2.4	11.71		
	Vita Enamic	6*	18.26	18.1	14.48	21.27	2.26	16.67		
	Total	20	11.18	14.58	1.09	21.27	7.1			
HF acid	IPS e.max	7	10.95	10.54	8.4	16.25	2.6	6.29	6,286	0.043
	Lava Ultimate	7	15.52	15.93	10.39	21.99	4.25	14.14		
	Vita Enamic	7	13.87	13.91	12.17	15.91	1.44	12.57		
	Total	21	13.45	13.08	8.4	21.99	3.44			
Phosphoric acid	IPS e.max	6*	3.77	3.28	3.11	5.18	0.87	4.17	15,567	0.001
	Lava Ultimate	7	5.74	5.75	4.41	7.54	1.19	9.43		
	Vita Enamic	7	15	15.61	11.84	16.95	1.86	17		
	Total	20	8.39	5.75	3.11	16.95	5.21			
Sandblasting	IPS e.max	6*	2.44	1.81	1.08	5.16	1.56	3.5	12,492	0.002
	Lava Ultimate	6*	12.99	12.18	6.44	20.53	4.61	11.5		
	Vita Enamic	7	15.27	14.62	13.83	18.15	1.75	14.29		
	Total	19	10.5	12.59	1.08	20.53	6.34			

*Groups with premature fractures, Min: Minimum, Max: Maximum, SD: Standard deviation

Table 6. Effect of bur application on bond strength

	Bond strength (Mpa)						Kruskal-Wallis H test		
	n	Mean	Median	Min	Max	SD	Rank Avg.	z	p
No bur applied	104	10.12	10.72	1.36	19.95	5.22	89.25	-0.944	0.345
Bur applied	80	10.92	12.03	1.08	21.99	5.85	96.73		
Total	184	10.46	11.59	1.08	21.99	5.5			

Min: Minimum, Max: Maximum, SD: Standard deviation

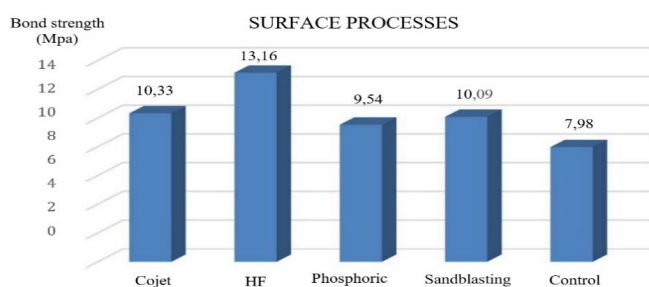


Figure 4. Differences between surface treatments in terms of bond strength

observed in Lava Ultimate material (except for milling + HF acid and milling + phosphoric acid). In the Vita Enamic material, mixed fractures were also the most common. In the control group, 100% adhesive fractures were observed in the IPS e.max and Lava Ultimate materials, while 71.4% cohesive fractures were observed in the Vita Enamic material (Figure 5-9).

SEM findings related to fracture types and materials observed at 2000x magnification using SEM are shown in Figure 6-9.

Table 7. Fracture types observed after shear bonding test

Surface procedure	Sandblasting	Bur+sandblasting	Cojet	Bur+Cojet	HF acid	Bur+HF acid	Phosphoric acid	Bur+phosphoric acid	Control
IPS e.max	85.7% Ad (6/7) 14.3% Kr (1/7)	100% Ad (6/6)*	85.7% Ad (6/7) 14.3% Kr (1/7)	85.7% Ad (6/7) 14.3% Kr (1/7)	71.4% Ad (5/7) 28.6% Kr (2/7)	85.7% Ad (6/7) 14.3% Kr (1/7)	57.1% Ad (4/7) 42.9% Kr (3/7)	100% Ad (6/6)*	100% Ad (7/7)
Vita Enamic	85.7% Kr (6/7) %14.3Koh (1/7)	57.1% Kr (4/7) 42.9% Koh (3/7)	85.7% Kr (6/7) 14.3%Koh (1/7)	50% Kr (3/6)* 50% Koh (3/6)*	100% Kr (7/7)	57.1% Kr (4/7) 42.9% Ad (3/7)	100% Kr (7/7) 28.6% Koh (2/7)	71.4% Kr (5/7) 28.6% Koh (2/7)	71.4% Koh (5/7) 28.6% Kr (2/7)
L a v a Ultimate	85.7% Kr (6/7) 14.3% Ad (1/7)	83.8% Kr (5/6)* 16.2% Ad (1/6)*	57.1% Kr (4/7) 42.9% Ad (3/7)	100% Kr (7/7)	66.6% Kr (4/6)* 33.4% Ad (2/6)*	100% Koh (7/7)	71.4% Kr (5/7) 28.6% Ad (2/7)	57.1% Ad (4/7) 42.9% Kr (3/7)	100% Ad (7/7)

*Premature fracture seen during thermal cycling, Ad: Adhesive fracture, Kr: Mixed type fracture, Koh: Cohesive fracture

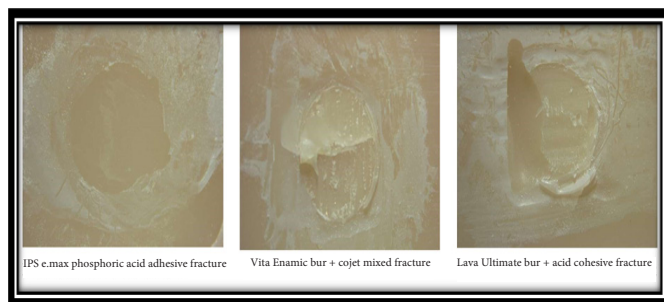


Figure 5. Fracture types

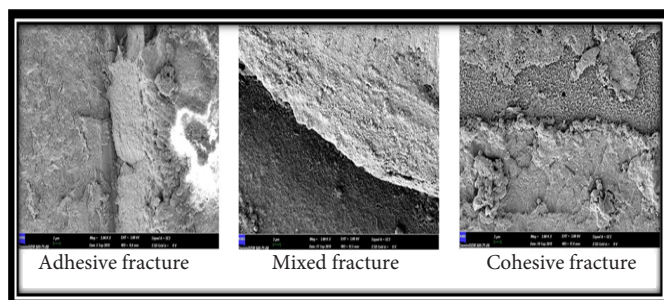


Figure 6. SEM (x2000) images of fracture types

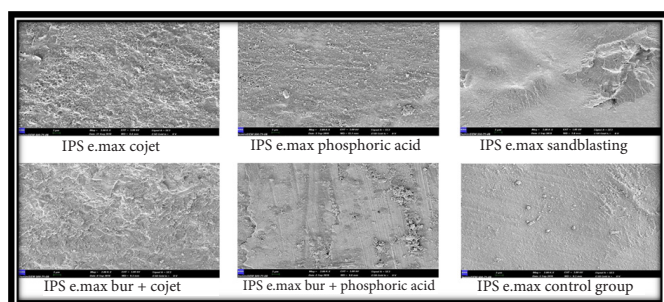


Figure 7. SEM (x2000) images of IPS e.max materials

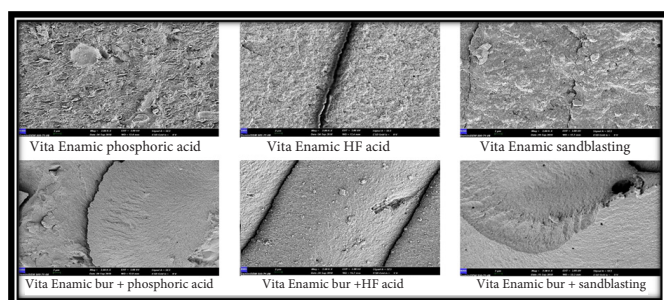


Figure 8. SEM (x2000) images of Vita Enamic materials

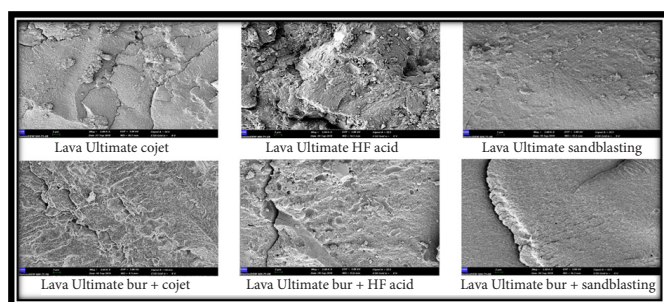


Figure 9. SEM (x2000) images of Lava Ultimate materials

DISCUSSION

The clinical lifespan of indirect restorations produced with CAD/CAM systems varies between 5 and 10 years on average.⁴³ The most important reason for their short lifespan

is their low fracture resistance.⁴⁴ Since it is not possible to add intraoral porcelain to ceramic restorations, repairing localized fractures with composite resins has become a viable option.^{24,45} The success of the composite resin repair procedure depends on the type of repair material and the bond strength between the ceramic and resin materials.⁴⁶ Hybrid composite resins exhibit higher bond strength than microfilled composites,^{34,39} so microhybrid-based composite resin was used in our study.

Different test methods are used to calculate bond strength. Although these tests may not fully reflect clinical performance, they are useful in deciding which adhesive system to use in practice. Laboratory tests enable the mechanical testing of bonding surfaces and the determination of variables.³⁹ In the in vitro evaluation of bond strength, different test methods such as tensile, microtensile, shear, and microshear are utilized. The most significant disadvantage of the tensile test method is the formation of heterogeneous stresses that can cause cohesive fractures at the bonding interface.⁴⁷

In the shear test method, a force is applied parallel to the bonding surface at a speed of 0.75 ± 0.3 mm/min until the bond between the two materials bonded with the adhesive agent breaks. A loading speed higher than 1 mm/min may cause incorrect results.⁴⁸ The shear bond strength is calculated by dividing the maximum force applied by the surface area of the bonded surface.⁴⁹ Kalra et al.⁵⁰ reported that the bond strength must be at least 8-9 MPa for successful intraoral material repair. In our study using the shear bond strength test, the force was applied at a loading rate of 0.5 mm/min. To ensure that the obtained data could be compared accurately, the sample surfaces were standardized using 600, 800, and 1200 grit sandpaper, respectively.

For the bonding success of composite resin, mechanical methods such as roughening the fracture surfaces with a diamond bur or acid, sandblasting, or silica coating are recommended.²⁷ Wiegand et al.⁵¹ reported that the highest bonding strength in terms of mechanical retention can be achieved by roughening with a burr (25-125-micron green band diamond burr). Jain et al.,³⁰ on the other hand, claimed that roughening with a burr causes microcracks on the ceramic surface.

HF acid, which is used to roughen silicate-containing ceramic surfaces, creates numerous pores in the matrix and crystal structure of the ceramic.³⁴ It has been stated that the concentration of HF acid and the roughening time should vary depending on the type of ceramic.⁵² Vidotti et al.⁵³ emphasized that 10% HF acid can dissolve the glass matrix of IPS e.max material. In this case, the crystal structure is exposed, and the increased surface area also increases the wettability of the ceramic.⁵⁴ Although extending the application time increases the bond strength,^{55,56} when HF acid is applied for a long time (>160 s), the glass phase dissolves faster than the crystalline phase, weakening the ceramic structure and reducing the bond strength.^{57,58} Mokhtarpour et al.⁵⁹ reported that increasing the HF acid concentration and extending the etching time leads to an increase in mixed and cohesive fractures.

In a study, it was stated that HF acid does not increase the bonding strength of Vita Enamic hybrid ceramic material, and this fact is attributed to the material's high leucite and zirconia content.⁶⁰ It has been reported that phosphoric acid, used for roughening ceramic surfaces, only enables the



cleaning of contaminated surfaces but does not cause changes at the microstructural level.⁶¹

Given that acid applications can have toxic effects on living tissue, it has been recommended that ceramic materials be roughened using a sandblasting method. The size of Al_2O_3 particles affects the adhesion of the repair material. Smaller particles are preferred in glass, hybrid, or nano-ceramics, while larger particles are used in rigid materials such as zirconium. It has been claimed that sandblasting results in a more irregular surface morphology compared to roughening with HF acid.⁶² Studies examining the ceramic surface of IPS e.max material after application of HF acid and Al_2O_3 using SEM reported that a more regular surface change was obtained after HF acid treatment.^{63,64} Elsaka²⁴ found the surface roughness of Vita Enamic and Lava Ultimate material samples treated with sandblasting and HF acid to be similar. Colares et al.⁶⁵ stated that the application of HF acid + silane is sufficient for the repair of IPS e.max lithium disilicate ceramic materials with composite resin and that sandblasting should be avoided.

In a study where sample surfaces were roughened using diamond milling and sandblasting methods, it was found that after milling, unidirectional indentations and protrusions formed, while after sandblasting, the indentations and protrusions were found to be in different directions and orientations.⁶⁶ Güngör et al.⁶⁷ reported that the highest surface roughness values were observed in the groups treated with milling, followed by sandblasting and acid application methods. In our study, we observed that the irregular areas detected on the surface of the samples after sandblasting prevented the ceramic surface from being completely wetted by the bonding agents and caused voids at the interface. This suggests that further roughening would not lead to higher bonding strength.

Chemical surface treatments used to increase bonding strength (silane coating agent and bond application) are at least as effective as mechanical methods.^{24,68} Due to their structure (being dual-functional), silanes can bond with both organic and inorganic materials. This property helps to form a strong bond between silica-containing ceramics and composite resin.⁶⁹ The same bond cannot be achieved with materials that do not contain silica.¹¹ Elsaka,¹² who applied a silica coating to Vita Enamic material using a Cojet device, noted that the application of silane to the surface resulted in the formation of covalent bonds between alumina, silica particles, and adhesive cement. Nagai et al.⁷⁰ also stated that silane application increased bonding strength.

The silica layer formed after applying the Cojet process to the ceramic surface provides the necessary environment for the silane to become active. Thanks to the chemical bond formed by the silane between the ceramic surface and the resin,²⁷ the ceramic-resin bond remains stable even after prolonged immersion in water and thermal cycling.³⁹ The ceramic-composite resin bond is exposed to chewing stresses in the mouth and is affected by thermal and chemical factors. Temperature changes in the mouth create stress between materials with different thermal expansion coefficients.²⁷ To simulate such effects in a laboratory setting, it is recommended to soak the samples in artificial saliva at different temperatures and for different durations,⁴¹ use devices such as chewing simulators, or perform thermal cycling.³⁹ In our study, the

samples were subjected to thermal cycling. In the samples subjected to milling, low bond strength values (<8-9 MPa) were measured in the Cojet, phosphoric acid, and sandblasting groups of the IPS e.max material, and in the sandblasting group of the IPS e.max material in the samples not subjected to milling. This result suggests that sandblasting and Cojet treatment are not sufficiently effective in samples containing glass ceramics.

In recent years, bonding systems containing MDP (10-methacryloxydiethyl hydrogen phosphate monomer) have gained prominence in the intraoral repair of ceramic materials.³⁰ It is claimed that treating the ceramic surface with MDP primer increases its bonding ability.^{19,42} In light of this information, Clearfil Ceramic Primer Plus was used in our study due to its MDP content.

The effect of combining mechanical and chemical processes on bond strength is controversial.^{20,71,72} Elsaka¹² reported that applying HF acid or sandblasting to Vita Enamic material in combination with a silane pretreatment was more effective than mechanical surface treatment alone, but no such difference was observed in Lava Ultimate material. Cekic-Nagas et al.,¹⁹ in a study using Vita Enamic, Lava Ultimate, and GC Cerasmart materials, found that the Vita Enamic material surface was rougher and more irregular than the others and exhibited higher bond strength. In our study, no significant difference in shear bond strength was detected between the groups with and without milling. This phenomenon is thought to be due to the use of an MDP-containing silane agent (hydrophobic siloxane bond) in all groups. When compared to resin-based materials such as Vita Enamic, Lava Ultimate, and ceramic-based IPS e.max materials, resin-based materials exhibited higher bond strength. We believe this result is due to the material containing more silica and a higher filler content. The higher bond strength of resin-containing hybrid ceramics (Vita Enamic and Lava Ultimate) is attributed to the materials' ability to form both micromechanical and chemical bonds.

In order to examine changes occurring on the surface of the material at a small scale, stereomicroscopes and SEM devices are used. In our study, premature fractures⁶³ observed in some samples before undergoing fracture testing were found to be adhesive fractures upon stereomicroscopic examination. While adhesive fractures were generally observed in IPS e.max material samples, mixed cohesive and adhesive fractures were observed in Vita Enamic and Lava Ultimate blocks, respectively. Our results are consistent with the findings of Peumans et al.,⁷³ who observed adhesive failure in IPS e.max materials,⁷² and Hu and colleagues, who reported cohesive fractures in Vita Enamic material samples treated with HF acid + silane.

CONCLUSION

Roughening processes applied to ceramic surfaces increase bond strength. The effect of milling on bond strength was not found to be statistically significant ($p>0.05$). The lowest bond strength was observed in the control groups where no surface treatment was applied, while the highest bond values were measured in the samples treated with HF acid. In IPS e.max samples with a high ceramic content, phosphoric acid and milling + HF acid application increased bonding strength. In Lava Ultimate samples made of resin-reinforced materials,



Cojet and HF acid application increased bonding, while in Vita Enamic samples, sandblasting and HF acid application increased bonding. SEM examination of the surfaces of materials treated with sandblasting and Cojet revealed microporosity on the surface.

ETHICAL DECLARATIONS

Ethics Committee Approval

This study did not require ethical approval as it did not involve any human subjects or animal experiments.

Informed Consent

Because the study has no study with human and human participants, no written informed consent form was obtained.

Referee Evaluation Process

Externally peer-reviewed.

Conflict of Interest Statement

The authors have no conflicts of interest to declare.

Financial Disclosure

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Author Contributions

All of the authors declare that they have all participated in the design, execution, and analysis of the paper, and that they have approved the final version.

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