

## Research Article

# Effects of Two Different Acid Etching and Surface Washing Methods on Bond Strength on Different CAD-CAM Blocks under Aging Protocols

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Aim. The purpose of this study is to investigate the effects of hydrofluoric acid and one-component ceramic primer and silane (Monobond Etch and Prime (MEP)) applications on lithium disilicate glass ceramics and zirconium-infiltrated lithium silicate glass ceramics, as well as the effect of ultrasonic and phosphoric acid surface washing methods on bond strength. Materials and Method. A total of 240 ceramic samples were prepared using two different CAD-CAM material blocks with a thickness of 2 mm made of lithium disilicate glass-ceramic (IPS e.max CAD) and zirconium-infiltrated lithium silicate glass ceramic blocks (Celtra Duo). The samples were cemented to the composite discs (Tetric N-Ceram) after two different acid treatments, and surface washing processes were applied to them. As such, 24 groups were formed, each with two different acid applications, three different washing processes, two different CAD-CAM blocks, and two different aging procedures (n = 10). Following the application of the acid, different washing processes are used. These were HF acid and washing only (HF+W), HF acid and ultrasonic washing (HF+US), HF acid and phosphoric acid (HF+PA), MEP with washing only (MEP+W), MEP and ultrasonic washing (MEP+US), and MEP and phosphoric acid (MEP+PA). The composite discs were cemented with dual cure adhesive cement (Multilink Automix) after the determined surface treatments were applied to the blocks. After surface applications, SEM analysis was conducted. Following exposure to two different thermal procedures, long-term (TL) and shortterm (TS), bond strengths were measured using an Instron universal test device. SPSS version 23.0 software was used to perform the statistical analyses. Histogram graphs and the Kolmogorov-Smirnov/Shapiro-Wilk test were used to assess the variables' conformity to the normal distribution. Results. The bond strength values of TS and TL in the Celtra Duo block were significantly higher than those in the e.max CAD block (p < 0.05). The TS-TL bonding strength value difference in the e.max CAD block was significantly higher than the surface measurements in the Celtra Duo block. While the highest bond strength value HF + US for TS in e.max CAD was 20.07 ± .31, the values of HF + US in Celtra Duo were significantly higher in terms of TL values when compared to other groups. Conclusion. Celtra Duo material demonstrated higher bond strength values after a short and long thermal cycle than e.max CAD material. In general, groups bonded with HF were less affected by the thermal cycle than groups treated with MEP.

#### 1. Introduction

Due to their featured properties such as aesthetic success, surface smoothness, durability, and biocompatibility, dental ceramics are widely used to replace missing teeth or teeth with substance loss. Advanced ceramic systems with improved mechanical properties have begun to be produced in response to the growing demand for more successful aesthetic restorations [1]. The advancement of CAD-CAM (computer-aided design/computer-aided manufacturing) technologies benefits the treatment process by standardizing more compatible restorations [2].

Because of their biocompatibility, aesthetic appearance, and mechanical properties, lithium disilicate glass ceramics are among the most preferred restorative materials for indirect restorations in both aesthetic and functional treatments. They have strong mechanical properties and meet aesthetic requirements for posterior and anterior tooth restoration [3].

Recently, a new CAD-CAM ceramic material that combines the aesthetic properties of lithium disilicate with the mechanical properties of zirconium has been developed. In addition to lithium oxide and silicon dioxide, zirconium dioxide (ZrO2) is present in the glass phase of zirconiainfiltrated lithium silicate glass ceramic (Celtra Duo), which prevents zirconium oxide crystallization. The zirconiainfiltrated lithium silicate glass ceramic contains 10% zirconia by weight, which is dissolved in the metasilicate glassy matrix. This content contributes to its high translucency. The homogeneous distribution of zirconia particles and high glass content increased the material's durability, polishing quality, and aesthetic properties [4].

The compatibility of tooth structure and restoration material is critical for the long-term clinical success of CAD-CAM indirect restorations. Hydrofluoric acid, followed by silane, is the most commonly used surface treatment for glass ceramic cementation. The mechanical resistance of ceramics has been reported to decrease depending on the concentration and duration of hydrofluoric acid (HF). Etching with HF also poses the risk of toxic accidents. Despite the fact that HF can sufficiently roughen ceramic surfaces, it is not recommended for oral use in most countries due to its negative side effects [5]. Consequently, onecomponent ceramic primer and silane (Monobond Etch and Prime) (MEP) have replaced HF as alternative methods. In a single step, this product combines etching and silane applications. It has been demonstrated that providing roughening and silane to glass-ceramic surfaces in a single step reduces clinical treatment time [6].

The formation of silica fluoride salts between the glass ceramic and the silica phase is another drawback of hydrofluoric acid. These salts may precipitate on the ceramic surface, reducing the strength of the ceramic-resin bond on the surface by obstructing resin infiltration [7]. Surface treatments such as etching with acidic fluoride-containing compounds are used in various studies to remove these formed salts [8]. There have been insufficient studies comparing the effects of ultrasonic washing and phosphoric acid applications on lithium disilicate and zirconium-infiltrated lithium silicate glass ceramics.

The purpose of this research is to compare the effects of hydrofluoric acid, Monobond Etch and Prime, ultrasonic washing, and phosphoric acid application on the bond strength of CAD-CAM blocks, which are highly preferred. The null hypothesis was that the bond strength of CAD-CAM blocks with composite is not affected by surface washing or aging protocols.

#### 2. Material and Method

The G<sup>\*</sup>Power 3.1.9.4 program was used to determine the sample size. When determining the sample size of the study, when the margin of error was 5% and the effect size of the evaluations was assumed to be d = 0.5, it was concluded that

at least 108 (at least 9 for each group) subjects should be included in the sampling with 95% power.

2.1. Preparation of the Ceramic Samples. Table 1 presents the materials used in the study. From two different CAD-CAM material blocks (n = 10 per CAD-CAM material), 240 ceramic samples that were 2 mm thick were prepared parallel to the long axis, using a water-cooled low-speed cutting device (Isomet 1000, Buehler Ltd., Lake Bluff, 1 L, USA) at 400 rpm. Semicrystalline lithium disilicate glass ceramics (IPS e.max CAD; Ivoclar Vivadent, Schaan, Liechstenstein) were fully crystallized in the Programat P300 (Ivoclar Vivadent, Schaan, Liechstenstein) furnace following the manufacturer's recommendations. To ensure standardization, all samples were smoothed with silicon carbide (SiC) abrasive papers (600, 900, and 1200) to obtain a smooth surface. After that, it was embedded in a self-curing acrylic (Meliodent; Bayer Dental, Newbury, UK). After embedding all of the samples in the acrylic block, they were placed in individually numbered boxes, and the surface treatment stage began. Both materials received six different surface treatments, including two acid treatments and three different washings (Table 2).

2.2. Preparation of the Composite Discs. Tetric N-Ceram (Ivoclar, Vivadent, Schaan, Liechtenstein) composite disc was prepared with a layer thickness of 3 mm and a diameter of 5 mm. The upper surface of the composite was polymerized for 40 seconds with an LED device (VALO, Ultradent Products Inc., South Jordan, UT) with a light intensity of 1200 mW/cm<sup>2</sup>.

2.3. Scanning Electron Microscopy (SEM) Evaluation. A scanning electron microscope (EVO LS-10, Zeiss, Cambridge, UK) at 20 kV was used to examine one sample from each group for morphology ( $\times$ 5000 and  $\times$ 1000 magnification). At a low scanning frequency, digital images of 20  $\mu$ m  $\times$  20  $\mu$ m were acquired for each sample surface (1 Hz).

2.4. Surface Treatments and Cementation. All ceramic samples were cleaned inside an ultrasonic cleaner for 180 seconds with 96% percent isopropanol and air-dried for standardization. The ceramic surfaces were treated with a 5% concentration of HF (IPS Ceramic etching gel, Ivoclar Vivadent, Schaan, Liechtenstein). As per the manufacturer's recommendations, the samples were roughened with HF for 20 seconds to IPS e.max CAD and HF to Celtra Duo for 30 seconds before being rinsed with air-water spray for 30 seconds. The silane coupling agent (Monobond S, Ivoclar, Vivadent, Schaan, Liechtenstein) was applied and dried for 60 seconds.

In the MEP groups, the surface of the samples was first brushed for 20 seconds and then applied for a total of 60 seconds, waiting for 40 seconds to react on the surface. It was then washed for 10 seconds.

In order to remove the residual infiltration agent remaining on the surface in the HF+PA and MEP+PA groups, 37% phosphoric acid (N-etch etching gel, Ivoclar Vivadent, Schaan, Liechtenstein) was applied to the surface

Material	Chemical composition	Manufacturer	
IPS e.max CAD	57-80% SiO2, 11-19% Li2O, K2O, MgO, Al2O3, P2O5, and other oxides	Ivoclar Vivadent, Schaan, Liechtenstein	
Celtra Duo	SiO2, Li2O, ZrO2 P2O5, Al2O3, K2O CeO2	Celtra Duo Dentsply, Konstanz, Germany	
Monobond Etch and Prime (MEP)	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylate phosphoric acid ester, bis (triethoxysilyl) ethane, silane methacrylate, colorant, ethanol, water	Ivoclar Vivadent, Schaan, Liechtenstein	
Monobond S (MP)	Ethanol, water, silane methacrylate	Ivoclar Vivadent, Schaan, Liechtenstein	
IPS ceramic etching gel	5% hydrofluoric acid	Ivoclar Vivadent, Schaan, Liechtenstein	
Multilink Automix	Dimethacrylates, HEMA (2-hydroxyethyl methacrylate), barium glass filler, Ba-Al-fluorosilicate glass, ytterbium trifluoride, silica, catalysts, stabilizer, pigments	Ivoclar Vivadent, Schaan, Liechtenstein	
N-Etch etching gel	37% phosphoric acid	Ivoclar Vivadent, Schaan, Liechtenstein	
Tetric N-Ceram	Fillers: barium glass, ytterbium trifluoride, barium alumino-fluorosilicate glass, silica Resins: BIS-GMA, UDMA, and TEGDMA	Ivoclar Vivadent, Schaan, Liechtenstein	

TABLE 1: Materials used in the study.

TABLE 2: Groups in the study.

	Groups
HF + W	Hydrofluoric acid and washing
HF + US	Hydrofluoric acid and ultrasonic washing
HF + PA	Hydrofluoric acid and phosphoric acid
MEP + W	Monobond Etch and Prime and washing
MEP + US	Monobond Etch and Prime and ultrasonic washing
MEP + PA	Monobond Etch and Prime and phosphoric acid

for 20 seconds and washed with plenty of water for 10 seconds and then dried.

Before cementation, an ultrasonic hand piece (NSK, Nakanishi Inc., Japan) was used to remove the residual infiltration agent that remained on the surface in the MEP + US and HF + US groups, following the manufacturer's instructions and operating in anhydrous mode. To avoid overheating the ultrasonic hand piece tip, the device was operated for short periods (< 5 seconds) for 1 minute.

The composite discs were placed on the surface using finger pressure after the determined surface treatments were applied to the blocks and then cemented with Multilink Automix (Ivoclar Vivadent, Schaan, Liechtenstein), a dual cure adhesive cement. Cotton pellets were used to clean the overflowing resin cement. Polymerization was accomplished by holding a light for 40 seconds with an LED device that emits visible light at 380-515 nm with an intensity of 1200 mW/cm<sup>2</sup>.

2.5. Thermal Aging and Testing Procedures. The samples were subjected to 2 different aging methods (TS and TL), and their shear bond strength values were investigated. For short-term (TS) aging (5000 thermal cycles), after being kept in distilled water for 24 hours, they were placed in a thermal cycle (Thermocycler, SD Mechatronik Thermocycler THE-1100, Feldkirchen-Westerham, Germany). For long-term (TL) aging (20000 thermal cycles), the samples were sub-

jected to a thermocycling, after being kept in distilled water for 24 hours, in both 2 water baths at 5°C and 55°C with a 20-second waiting time and a transfer time of 10 seconds.

Shear bond tests were performed on the samples to determine their bond strength using an Instron universal testing machine (Shimadzu universal machine). A separation force of 90° was applied to the bonding interface at a loading speed of 0.5 mm/min. When the composite sample was separated from the ceramic surface, the force value was recorded in "N," and shear bond values in MPa were calculated by dividing the surface area. The SBS values were calculated using the following formula:

$$SBS(MPa) = \frac{Shear load(N)}{Surface area (mm^2)}$$
(1)

2.6. Statistical Analysis. SPSS version 23.0 was used to perform the statistical analyses (IBM SPSS Statistics for Windows, v23.0; IBM Corp). Histogram graphs and the Kolmogorov-Smirnov/Shapiro-Wilk test were used to assess the variables' conformity to the normal distribution. Mean and standard deviation (SD) values were used to present the descriptive analyses. To compare the blocks, an independent two sample *t*-test was used. To compare surface treatments, the one-way ANOVA test was used. To identify groups with significant differences, the Tukey HSD multiple comparison tests were used. Cases with a p value less than 0.05 were considered statistically significant.

#### 3. Results

The bond strength values of TS and TL in the Celtra Duo block were significantly higher than those in the e.max CAD block (p < 0.05) (Table 3).

In the e.max CAD block, all surface treatments differed in terms of mean bond strength values at TS values. The highest bond strength value HF+US in e.max CAD was  $20.07 \pm .31$ , while the lowest measurement MEP+W was  $16.08 \pm .20$ . In terms of TL values in e.max CAD, there

		Blo	ck		
Surface treatments	e.ma	e.max CAD		Celtra Duo	
	TS	TL	TS	TL	
	Mean $\pm$ SD	Mean ± SD	Mean ± SD	Mean ± SD	
HF+W	17.71±.41 <sup>Aa</sup>	$9.48 \pm .25^{Aa}$	$19.02 \pm .32^{Ba}$	$11.76 \pm .28^{Ba}$	
HF + PA	$18.96 \pm .27^{Ab}$	$11.84 \pm .35^{Ab}$	$20.11 \pm .32^{Bb}$	$13.32 \pm .18^{Bb}$	
HF + US	$20.07 \pm .31^{Ac}$	13.72±.19 <sup>Ac</sup>	$22.29 \pm .19^{Bb}$	$16.33 \pm .18^{Bc}$	
MEP + W	$16.08 \pm .20^{Ad}$	7.63±.27 <sup>Ad</sup>	17.23±.13 <sup>Bc</sup>	$9.47 \pm .22^{\mathrm{Bd}}$	
MEP + PA	$13.09 \pm .30^{Ae}$	$5.88 \pm .38^{Ae}$	$14.75 \pm .25^{Ba}$	$7.81{\pm}.39^{Ba}$	
MEP + US	$18.22 \pm .10^{Af}$	$11.55 \pm .18^{Ab}$	$20.31 \pm .20^{Bb}$	$14.11 \pm .25^{Be}$	

TABLE 3: Comparison of bond strength values according to surface treatments and blocks.

Mean values (MPa) with the same letter do not differ significantly from each other. Capital letters indicate significant differences between TSs and between TLs (p < 0.05); lower case letters indicate differences between surface treatments in each column (p < 0.05).

was no significant difference between HF + PA and MEP + US values, but other groups were statistically different from each other. Bond strength values of MEP + W were lower than other groups, while HF + US was higher.

The HF + US measurement in the Celtra Duo block was found to be significantly higher than the HF + W, MEP + W, and MEP + PA measurements. MEP + W was discovered to be below all other groups. The measurements for HF + PA, HF + US, and MEP + US are all similar. In terms of TL values, HF + US was found to have significantly higher values than the other groups. MEP + W levels were found to be significantly lower than those of the other groups. The difference between HF + W and MEP + PA was not statistically significant. Furthermore, changes in TS-TL values were found to be statistically significant in two separate blocks and across all surface treatments.

In terms of block and surface treatments, the difference between TS and TL values was compared. In the e.max CAD block, the HF+W and MEP+W difference values were significantly higher than the other group measurements. While the HF+US and MEP+US difference values were similar, they were significantly lower than the other groups. While the HF+US difference values in the Celtra Duo block were similar to the MEP + US difference values, they were significantly lower than the other groups. Only the values of MEP + PA were found to be similar when the TL-TS difference of bond strengths between blocks was compared. The difference in TS-TL bonding values in the e.max CAD block was significantly higher than the difference in bonding strength values in the Celtra Duo block (Table 4). When analyzing Celtra Duo and e.max CAD in fracture types, it was observed that Celtra Duo resulted in less adhesive failure (Figures 1 and 2). SEM analysis confirmed that the results and morphological surface changes were seen on the surface of specimens (Figures 3 and 4).

#### 4. Discussion

This study investigated the effects of hydrofluoric acid, Monobond Etch and Prime, ultrasonic washing, and phosphoric acid application on the bond strength of CAD-CAM blocks. Based on the results of the study, the null hypothesis was that surface washing or aging protocols TABLE 4: Mean and multiple comparison of TS and TL difference values in terms of block and surface treatment.

	IPS e.max CAD	Celtra Duo
	Mean ± SD	Mean ± SD
HF+W	8.23±.42 <sup>A,a</sup>	7.26±.41 <sup>B,a</sup>
HF + PA	7.12±.25 <sup>A,b</sup>	$6.79 \pm .33^{B,a}$
HF + US	6.35±.33 <sup>A,c</sup>	$5.96 \pm .25^{B,c}$
MEP + W	8.45±.17 <sup>A,a</sup>	$7.76 \pm .28^{B,a}$
MEP + PA	7.21±.53 <sup>A,b</sup>	$6.94 \pm .58^{A,a}$
MEP + US	$6.67 \pm .21^{A,b,c}$	$6.20 \pm .34^{B,c}$

While lower case letters indicate the differences within the surface treatments, upper case letters indicate the comparison of each surface treatment (of the same type) in terms of blocks (p < 0.05).



FIGURE 1: Fracture types on e.max CAD.

would not affect the bond strength of CAD-CAM blocks with composite that was rejected.

Surface preparation and adhesive cement preference are just as important as the clinician's choice of restoration material for long-term success. Long-term complications such as decementation, discoloration, and microleakage can be avoided with proper surface preparation. The null hypothesis was that the bond strength of CAD-CAM blocks with composite is not affected by surface washing or aging protocols.



FIGURE 2: Fracture types on Celtra Duo.

A stronger bonding is achieved by replacing ceramic interior surfaces with various surface treatments [9]. By dissolving the glass matrix and crystals in the ceramic with acid, an irregular surface topography is created. The application of hydrofluoric acid is based on the fact that the fluoride in the acid structure has a higher affinity for silicon than it does for oxygen, causing the glass matrix structure of the ceramic to dissolve and microprotrusions to form in the insoluble areas [9, 10]. The application time for hydrofluoric acid is 60 seconds at a concentration of 5-10%. However, this time and rate vary depending on the glass content of the ceramic material. The etching time of glass ceramic material reinforced with lithium disilicate, which has a higher glass matrix content, is specified by the manufacturer as 20 seconds, whereas for feldspathic and leucite reinforced ceramics, this time is recommended as 40-60 seconds [10]. In this study, HF was applied for 30 seconds on Celtra Duo and 20 seconds on e.max CAD as per the manufacturer's instructions.

After mechanical roughening of the ceramic inner surface, the application of silane increases bond strength [10]. Other acidic adhesives hydrolyze the silane monomer when it is added to a nonaqueous solution [11]. Because silane monomers are hydrolyzed and dehydrated when added to an aqueous medium, they have a short shelf life [12]. Some manufacturers, however, have recently developed a primer that contains silane monomer dissolved in ethanol and anhydrous acid [12, 13]. Self-etch primers have been proposed to reduce the toxicity of HF acid while also shortening the cementation process. Previous research [14] compared a self-acid primer (Monobond Etch & Prime, Ivoclar Vivadent, Schaan, Liechtenstein) to another primer from the same company (Monobond Plus, Ivoclar Vivadent). In comparison to the acidic primer application, more surface roughness and shear value were obtained. However, there has not been enough research yet on the surface treatments of 10% zirconium-infiltrated lithium silicate ceramic, the changes in its microstructure as a result of HF and MEP applications, and the differences in bond strength compared to lithium disilicate. The reaction kinetics of ceramics and various acids are influenced not only by time and acid concentration on the ceramic surface but also by the physical structure of the ceramic substrate [15]. As a result, surface preparation application and adhesion should be investigated in ceramic materials with varying chemical or physical structures. The most appropriate roughening agent, concentration, and application time for the relevant ceramic material can thus be determined [15]. As a result, the effects of HF and MEP on the bond strengths of Celtra Duo and e.max CAD, as well as various surface washing methods, were compared.

Thermal cycling is a recognized method for simulating the clinical performance and long-term durability of a tooth/restoration complex. Thermal cycling is one of the most common procedures used in clinical practice to simulate the physiological aging of biomaterials; as a result, it is frequently used in experimental studies to evaluate material performance [16]. The thermal cycling process was set at 5°C to 55°C in this study, with a dwell time of 20 seconds per bath solution and a transfer time of 10 seconds. According to the literature, the short-term thermal cycle may result in insufficient results when measuring the quality of the materials used [17]. In our study, we used a short-term thermal cycle on half of our samples and a long-term thermal cycle on the other half. We defined short-term thermal aging in the mouth (TS) as 5,000 thermal cycles representing 6 months and long-term thermal aging (TL) as a total of 20,000 thermal cycles representing 24 months in the mouth [16].

When silica-based ceramics are etched with hydrofluoric acid, insoluble silica fluoride salts form as a by-product on the surface. These by-products can interfere with the bonding strength if these salts are not removed. Then, the use of various cleaning methods to remove these by-products increases the bonding [18]. Phosphoric acid, which is used to abrade the tooth surface, is also commonly used on the inner surfaces of restorations to clean the surface and strengthen the bonding. It has been claimed that ultrasonic cleaning of the acid etched ceramic surface improves bonding strength by removing loose crystals from the surface. The primary goal of this cleaning procedure is to ensure that the ceramic surface is clean, ready, and has high surface energy before applying silane [15].

In the present study, we compared the bond strength of ultrasonic washing (US), phosphoric acid (PA), and washing only (W) processes after acidification of ceramic surfaces, and we discovered that ultrasonic washing had the highest bond strength values. The MEP + PA group had the lowest bond strength value. In the similar study of Lyann et al., it was reported that phosphoric acid could not sufficiently affect the surface of the ceramic [19]. However, in another research, 40% phosphoric acid was applied for 5 seconds and 60 seconds, and no significant morphological changes on the ceramic surface were observed [20]. Phosphoric acid's effect on ceramics may have been weak in the present study, and the reason for the MEP + PA group having the lowest bonding value may be that phosphoric acid also reduced the effect of MEP. In addition, Dos Sandos et al. evaluated the effect of different protocols used to remove the remaining hydrofluoric acid on the shear bond strength (SBS) between lithium disilicate and resin cement. They found that



FIGURE 3: SEM images after surface treatments on e.max CAD material: (a)  $HF + W \times 5000$ , (b)  $HF + PA \times 5000$ , (c)  $HF + US \times 5000$ , (d)  $MEP + W \times 5000$ , (e)  $MEP + PA \times 5000$ , (f)  $MEP + US \times 5000$ , (g)  $HF + W \times 1000$ , (h)  $HF + PA \times 1000$ , (i)  $HF + US \times 1000$ , (j)  $MEP + W \times 1000$ , (k)  $MEP + PA \times 1000$ , and (l)  $MEP + US \times 1000$ .



FIGURE 4: SEM images after surface treatments on Celtra Duo material: (a) HF + W  $\times$ 5000, (b) HF + PA  $\times$ 5000, (c) HF + US  $\times$ 5000, (d) MEP + W  $\times$ 5000, (e) MEP + PA  $\times$ 5000, (f) MEP + US  $\times$ 5000, (g) HF + W  $\times$ 1000, (h) HF + PA  $\times$ 1000, (i) HF + US  $\times$ 1000, (j) MEP + W  $\times$ 1000, (k) MEP + PA  $\times$ 1000, and (l) MEP + US  $\times$ 1000.

the ultrasound cleaning after hydrofluoric acid application resulted in a surface without fluorosilicate precipitates and a higher SBS value which is consistent with this study. Also, it was concluded that the use of phosphoric acid did not completely remove the precipitates of fluorine deposited on the specimens' surface, which was verified by the SEM images [21].

Prado et al. discovered that the strength of the samples to which they applied HF was higher than the strength of the samples to which they applied MEP in their study evaluating the bond strength after MEP and HF applications. Hydrofluoric acid reacts with the glassy matrix of erosive ceramics in its mechanism of action. Because the matrix in glass ceramics is mostly silica, and the reaction produces hexafluorosilicate, the glassy matrix is selectively removed, exposing the crystalline structure. Hydrofluoric acid, when compared to other abrasives, produces rougher ceramic surfaces [22]. The bond strength values of the samples to which we applied HF were higher than those of the samples to which we applied MEP in our study. Prado et al. discovered that HF caused more topographical surface changes in SEM images [23]. Surfaces roughened with HF were rougher, and the surface changes were more visible in our study's SEM images than those roughened with MEP (Figures 3 and 4).

When we compared Celtra Duo and e.max CAD in fracture types, we discovered that Celtra Duo resulted in less adhesive failure (Figures 1 and 2). Furthermore, in our study, the bond strength values of Celtra Duo were found to be higher than the values of e.max CAD. Celtra Duo's improved bonding may be due to its unique fine, homogeneous crystal structure, which is more regular and has fewer microvoids [24]. Lithium disilicate ceramics were characterized by interlocking needle-shaped crystals embedded in a glassy matrix in a microstructural comparison, whereas zirconium-infiltrated lithium silicate ceramics exhibited a homogeneous fine crystal structure with round and rodlike crystals [24, 25].

In our study, the bond strength values of our samples decreased significantly when compared to 5,000 in groups that received 20,000 thermal cycles (Table 3). As a result, our hypothesis stated that long-term thermal aging has no effect on the bond strength of surface-treated CAD-CAM blocks that was rejected. In a study that investigated the effect of 5,000 to 10,000 thermal cycles on bond strength after using MEP and HF in lithium disilicate ceramics, Lyann et al. discovered that 10,000 thermal cycles had significantly lower bond strength than 5,000 [19]. In this regard, additional clinical studies can also be conducted to support this. This could be considered as a limitation.

#### 5. Conclusion

Considering the limits of this investigation, the following conclusions were drawn. Although MEP, which is produced as an alternative to the harmful effects of HF acid, gives clinically acceptable results in terms of bond strength, it has been concluded that it is not superior to HF. In cases where high bond strength is the first priority, zirconia-reinforced glass ceramics should be preferred over lithium disilicate glass ceramics. In addition, it is possible to say that ultrasonic washing gives better results than washing with phosphoric acid in clinical routine.

#### Data Availability

The statistical data used to support the findings of this study are available from the corresponding author upon request.

#### **Conflicts of Interest**

The authors declare that they have no conflicts of interest.

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