Effect of Hydrofluoric Acid Etching Time on Shear Bond Strength of Resin Cement to Different CAD/CAM Ceramic Materials

Omar Mahmood Awad, Mohammed Rashid Hameed

Department of Restorative and Esthetic Dentistry, College of Dentistry, University of Baghdad.
Corresponding author: omarmhmudaiad@gmail.com

The clinical performance of ceramic restorations is directly dependent on the resin luting procedure, which necessitates using hydrofluoric (HF) acid etching and silanization of the bonding surface of ceramic restorations. This study aims to evaluate and compare the effects of various HF acid etching times and the type of ceramic materials regarding the resin cement's shear bond strength with ceramic. Ninety samples with dimensions of (14mm×12mm×3mm) were produced from three different ceramic materials and divided into three groups (n=30): feldspathic (CEREC blocs CPC; Sirona dental, Germany) (group FP), leucite reinforced (IPS Empress CAD; Ivoclar Vivadent, Shann, Liechtenstein) (group LR), and lithium disilicate (IPS e.max CAD; Ivoclar Vivadent, Shann, Liechtenstein) (group LD). The samples were embedded in self-polymerized acrylic blocks. After polishing and ultrasonic cleaning, the samples in each group were subdivided randomly into three subgroups (n=10): HF acid (4.5%) was used to etch Groups 1, 2, and 3 for 20, 60, and 90 seconds, respectively. The samples received silanization after ultrasonic cleaning, and a surface adhesive was applied. A luting resin cement cylinder was constructed using a silicone mold and tested for shear bond strength (SBS) after thermocycling. Data were analyzed using one-way ANOVA and Tukey's test (α=0.05). Significant effect for different etching times on shear bond strength (SBS) in each group ($p<0.05$). In the LD1 group, SBS was significantly lower than LD2 and LD3 ($p<0.05$). The LR2 showed higher SBS than LR3 ($p<0.05$). The FP3 showed significantly higher SBS than FP1 and FP2 ($p<0.05$). The mean SBS of the groups varied significantly ($p<0.05$) overall etching durations, with the LD1 and LR1 groups exhibiting significantly larger SBS than the FP1 group ($p<0.05$). SBS is significantly greater in the LD2 and LR2 groups than in the FP2 group ($p<0.05$). Moreover, SBS was significantly greater in LD2 than in LR2 ($p<0.05$). Comparing the LD3 to other materials, the SBS is noticeably greater ($p<0.05$). In conclusion, more than 60 seconds of etching can increase the shear bond strength for feldspathic and lithium disilicate CAD/CAM ceramics. However, etching greater than 60 seconds decreases the shear bond strength for leucite-reinforced ceramic.

Keywords: Hydrofluoric acid, CAD/CAM ceramics, shear bond strength, surface treatment,

resin cement.

1. Introduction

Glass-ceramic has been widely used in clinical situations due to its superior optical qualities, resilience to wear and fracture, good adhesion to the tooth surface, good marginal adaptation, good biological response, low thermal conductivity, and simplified manufacturing (Kang et al., 2013, Saadallah and Al-Azzawi 2017). Indirect restorations such as veneers, crowns, inlays, onlays, and fixed partial dentures have all been made using glass ceramic. There are numerous types of glass ceramics on the market, such as polymer-infiltrated ceramics, feldspathic, zirconia-reinforced lithium silicate, lithium disilicate, and leucite-reinforced ceramics (Wendler et al., 2017). Numerous investigations assessing the clinical durability of Lithium disilicate and feldspathic ceramics have discovered that monolithic crowns can have a lifespan rate of up to 100% in just two years. (Denissen et al., 2002) And above 94% in three years for partial crowns. Nevertheless, there have been reports of ceramic material failures in the literature, particularly about cavities at the adhesive interface and fractures, loss of retention, and detachment. (Al-Joboury et al., 2015, Abdulla, Majeed et al. 2020). Their preference is heavily reliant on the cementation procedure. Many restorations can be cemented with luting agents such as zinc phosphate, glass ionomer, or resin composite. Two separate interfaces must be considered when cementing a ceramic restoration to tooth structure (cement-enamel or dentine and cement-ceramic interfaces). If the adhesive seal failed at these interfaces, micro-leakage occurred, endangering the restorations' durability and clinical performance, resulting in recurrent cavities, pulpal response, postoperative sensitivity, discoloration, and finally, restorative de-bonding. Because most adhesive resin-based cement has adequate bonding qualities to enamel and dentine but poor bonding properties to unconditioned ceramic, most clinical failures are caused by the resin-cement ceramic interface. Surface activation through roughening and cleaning is necessary for chemical attachment and micromechanical bonding to the ceramic surface, which are the foundations of a stable resin bond (Blatz et al., 2003).

Since HF acid etching was first suggested as a ceramic surface pretreatment for resin bonding, surface treatment plays a vital role in achieving an optimal adhesion between resin composite and ceramic restoration (Kilinc et al., 2020), can have an impact on the material surfaces' microstructure as well. Surface treatments include mechanical methods for conditioning (sandblasting, laser irradiation, tribo-chemical, and pyro-chemical silica-coating) and chemical methods for conditioning (acid etching, silane application) (Chakmakchi 2017, Celik et al., 2018, Mansoureh et al., 2019, Sharad et al., 2024). By preferentially dissolving the glassy phase and exposing the crystalline phase, HF acid etching of the ceramic surface is useful in creating a retentive surface for micro-mechanical interlocking. By using a silane-coupling agent, etched surfaces will have higher surface energy and wettability and better covalent bond formation between the silica groups in ceramics and the methacrylate groups in resin. (Della Bona et al., 2004, Puppin-Rontani et al., 2017).

The concentration of acid, etching duration, temperature, and material composition all affect etching effectiveness (Tian et al., 2014). Investigations examining the effects of varying etching periods and acid concentrations on bond strength, the degree of roughness, and flexural strength (Amaral et al. 2011) . Different ceramics, however, could react differently to HF acid.

The ideal concentration and duration of HF acid etching for glass-ceramic restoration are poorly defined. Thus, it is essential to determine the proper HF etching duration to ensure a strong resin cement bond without compromising the strength of the ceramic. While HF strengthens the cement's bond with the ceramic, depending on the acid concentration and conditioning period, acid etching can also weaken the restoration's mechanical resistance. (de Menezes, Borges et al. 2009). There is disagreement in the literature regarding the impact of HF acid concentration and etching duration on the resin bond strength of ceramic materials made by various methods. In addition, a lower concentration of HF acid should be used because of its hazardous effects, as HF acid toxicity is concentration-related (Özcan, Allahbeickaraghi et al. 2012). Hence, the present study aims to select a suitable etching time according to the material (Lithium-disilicate, feldspathic, and reinforced with leucite) without affecting the mechanical strength. This investigation tried to evaluate the impact of varying HF exposure durations and the kind of ceramic substance on the resin cement bond strength.

2. Materials and Methods

A total of ninety samples of three ceramic materials: feldspathic (CEREC blocs CPC; Sirona Dental Germany) (group FP), reinforced with leucite (IPS Empress CAD; Ivoclar Vivadent Schaan Liechtenstein) (group LR), and lithium disilicate (IPS e.max CAD; Ivoclar Vivadent Schaan Liechtenstein) (group LD) with dimensions of (10 mm×8 mm×2 mm) were prepared for shear bond strength testing. All the samples were measured with a digital caliper. The examples were made from ceramic blocks using a water-cooled, low-speed handpiece with a diamond disc bur (40 mm in diameter and 1 mm in thickness). in the sectioning machine (Venturini, Prochnow et al. 2015, Prochnow, Venturini et al. 2017). According to the manufacturer's instructions, IPS e.max samples were crystallized using a VITA VACUMAT 4000 M furnace (VITA Zahnfabrik Germany). The samples were embedded in self-polymerized acrylic resin (acryl Plus, SpofaDental, Czech Republic) using a silicone mold. After finishing acrylic polymerization, the surface of ceramic samples was wet polished with 400-,600-, and 1200-grit silicon carbide paper to remove the irregularity and then cleaned ultrasonically in a water bath for 10 min.

Depending on the ceramic surface treatment, the samples in every group were separated into three subgroups randomly (n=10): HF acid (4.5%) was used to etch groups 1, 2, and 3 for 20, 60, and 90 seconds, respectively An adhesive tape with a 3 mm perforation delimited the area to receive the acid. The HF acid was applied to the surface using a micro-brush (Dentsply, New York, USA) for 20, 60, and 90 seconds, dried with a stream of air for 60 seconds after being washed off from the surface using an air/water jet. (Cengiz-Yanardag et al., 2019). Etching was done in a laboratory cabinet with ventilation while wearing masks, coat covers, and acid-resistant gloves to avoid HF acid's potentially harmful effects. For five minutes, the etching gel was removed using the neutralizing powder (sodium and calcium carbonate). Following acid etching, the samples received a 10-minute ultrasonic cleaning in a water bath. Afterward, a thin layer of a saline agent, Monobond N (Ceramic N, Ivoclar Vivadent, Schaan/Liechtenstein), was applied using a micro brush to the uncovered surface of every sample permitted to react for 60 seconds and then dried using oil-free air in accordance with the manufacturer's recommendations. Then, a thin coat of helibond (Ivoclar Vivadent, Schaan/Liechtenstein) was applied using a micro-brush and dried with compressed air (Sinaga et al., 2021)

A cylinder of luting resin cement was applied on the exposed ceramic surface according to the manufacturer's instructions using a specially constructed Teflon mold with a central hole (2 mm in height and 3 mm in diameter). A Teflon mold was used to standardize the adhesive area. A celluloid strip was positioned beneath a glass slide and forced under a 200g load, light cured for 20 seconds (1,200 Mw/cm2), then left for a 5-minute chemical curing (Kilinc et al., 2020).

Before the shear bond test, each sample performed 10,000 sessions of thermocycling with remaining times of 30 seconds and transfer times of 5 seconds, at 5 °C and 55 °C. (Morresi et al., 2014, Shakir and Hameed 2022).

SBS testing: The SBS was measured using a computerized universal testing machine (Zwick Roell Germany). The shear force was exerted at a speed of 0.5 mm/min and load cell of 5 Kg until the sample fractured. The stainless-steel shearing blade had a knife-shaped edge and was positioned perpendicular to the bottom of the resin cylinder on the adhesive connection (Falah et al., 2020, Jassim and Majeed 2023).

The formula calculated the shear bond strength: SBS= Force (Newton)/surface area (mm2). The ceramic samples' adhesive area was expressed by the area of circle $A = \pi r2$, where $\pi = 3.14$ and r = the cylinder's radius.

3. Results

Effect of etching time

There was a significant difference in shear bond strength (SBS) between different etching times in each group of material ($p<0.05$) (Table 1). In the LD1 group, SBS was significantly lower than in LD2 and LD3. ($p<0.05$). The LR2 group showed significantly greater SBS than LR3 ($p<0.05$). The FP3 group showed significantly higher SBS than FP1 and FP2, respectively ($p<0.05$) (Figure 1)

Effect of type of ceramic

There was a significant variation in the average SBS among the various groups. at all etching times ($p<0.05$), as shown in Table 2. The LD1 and LR1 show considerably greater SBS than FP1 ($p<0.05$). Also, LD2 and LR2 show significantly higher SBS than FP2 ($p<0.05$), and LD2 shows significantly more SBS than LR2 ($p<0.05$). The LD3 shows significantly greater SBS than LR3 and FP3, respectively ($p<0.05$) (Figure 1)

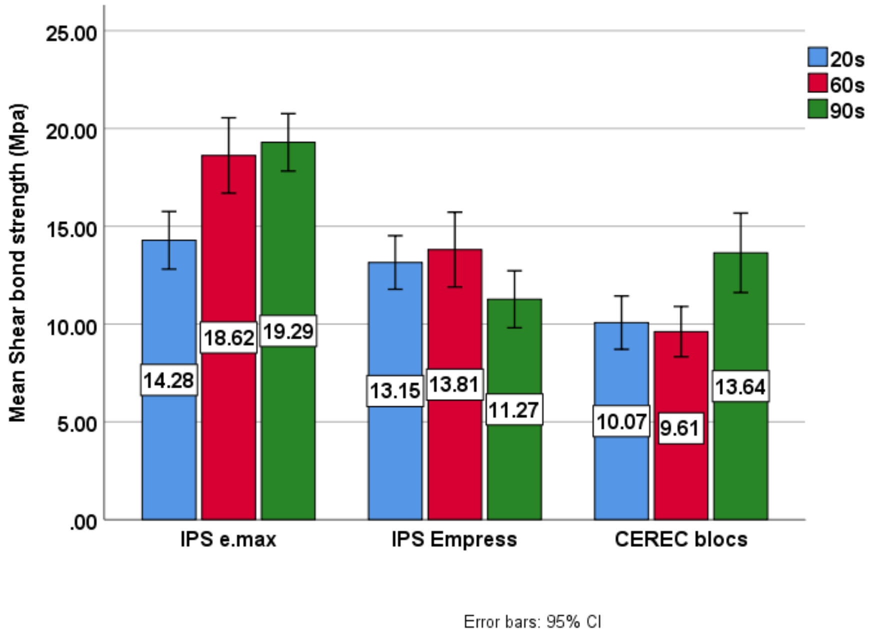

Figure 1: Bar chart of SBS in different groups and time

Table 1: Results of SBS after different etching times

group	Time	N	Mean	Std. Deviation	Minimum	Maximum	F	p-value*
IPS e.max	20 sec	10	14.28[a,b]	2.06	10.45	17.33	**14.151**	**0.000**
	60 sec	10	18.62[a]	2.69	15.14	23.15		
	90 sec	10	19.29[b]	2.05	16.25	22.13		
IPS Empress	20 sec	10	13.15	1.91	10.25	15.93	**3.491**	**0.045**
	60 sec	10	13.81[a]	2.67	9.87	17.43		
	90 sec	10	11.27[a]	2.03	8.56	15.09		
CEREC blocs	20 sec	10	10.07[a]	1.91	7.21	13.08	**9.783**	**0.001**
	60 sec	10	9.61[b]	1.80	7.16	12.06		
	90 sec	10	13.64[a,b]	2.84	9.28	17.24		

One-way ANOVA. Identical superscript small letters represent significant differences between relevant etching times in each group .

Table 1: Results of SBS of different ceramic groups.

Time	group	N	Mean	Std. Deviation	Minimum	Maximum	F	p-value*
shear 20	IPS e.max	10	14.28^{a}	2.06	10.45	17.33	12.342	**0.000**
	IPS Empress	10	13.15^{b}	1.91	10.25	15.93		
	CEREC blocs	10	$10.07^{a,b}$	1.91	7.21	13.08		
shear 60	IPS e.max	10	$18.62^{a,b}$	2.69	15.14	23.15	34.595	**0.000**
	IPS Empress	10	$13.81^{a,c}$	2.67	9.87	17.43		
	CEREC blocs	10	$9.61^{b,c}$	1.80	7.16	12.06		
shear 90	IPS e.max	10	$19.29^{a,b}$	2.05	16.25	22.13	31.083	**0.000**
	IPS Empress	10	11.27^{a}	2.03	8.56	15.09		
	CEREC blocs	10	13.64^{b}	2.84	9.28	17.24		

One-way ANOVA. Identical superscript small letters represent significant differences between relevant groups at each time.

4. Discussion

Appropriate adhesion between the ceramic and the tooth constituents is necessary for the clinical success of ceramic restorations over time (Blatz et al., 2003, Nagayassu et al., 2006). The processes of luting cement and its bonding resin adhering to the ceramic substrate, as well as to the enamel and dentin, are what allow a ceramic to be bonded to the tooth substance (Borges et al., 2007).

Surface activation through roughening and cleaning is necessary for micromechanical attachment and chemical linking to the ceramic surface, which are the foundations of a stable resin bond (Blatz et al.. 2003). Popular forms of treatment include grinding, acid etching, airborne particle abrasion with aluminum oxide, abrasion with diamond rotary instruments, and combinations of any of these techniques. A suitable surface roughness and texture can be achieved by acid etching with HF acid solutions (, Blatz et al., 2003, Nagayassu et al., 2006). Since the idea of etching porcelain surfaces was first proposed and the adhesive cementation of full-ceramic restorations was documented, several authors have shown that to maximize the bond strength, the concentrations and etching times need to be modified for every distinctive type of ceramic. (Özcan and Vallittu 2003).

The shear bond strength test was applied in this investigation. It is a commonly used, low-cost, and simple methodology for adhesion research (Sirisha et al., 2014). Alternative assays, including micro-tension and micro-shear, can also be employed. These assays feature a limited adhesive area, leading to few superficial failures and interior faults in the adhesive zone (Della Bona et al., 2004). Nevertheless, early interface failure in the micro-tension test can be caused by ceramic cutting operations, which lowers the assay's efficacy (Secilmis et al., 2016). Shear stress is the most common stress on the cement layer, according to some writers, which supports using this in vitro assay to assess this (Dal Piva et al., 2018).

Rougher feldspathic ceramic surfaces with more extended etching periods were identified by Zogheib and colleagues and others. (ranging from 20 to 180 seconds) (Zogheib et al., 2011). Thus, it is possible to achieve better bond strengths and safer patterns while dealing with HF if a longer etching period is used using HF acid. But if the etching time had been a bit longer, the ceramic degradation that followed might have been more severe and profound, weakening its structure (Zogheib et al., 2011). Consequently, given that this approach would not always produce a better-etched surface and bond strength, dentists should proceed with extreme caution when using an extended etching period with HF. Research indicates that the success of silane-only bonding relies on the bonding agent's capacity to fill empty spaces and create a tight link between the resin cement and ceramic material (Naves et al., 2010). On the other hand, applying an unfilled resin enhanced the binding strength and adaptability of the ceramic/resin cement interface by penetrating the etched surface flaws. The mean values of SBS were significantly larger in samples that received an unfilled resin with a silane coupling agent than in samples that received silane alone. This happened because the unfilled resin's viscosity was lower than the resin cement's (Naves et al., 2010, Sundfeld Neto et al., 2015).

This investigation partially accepted the first hypothesis, which suggested that the SBS between ceramic and resin cement would change depending on the time that HF etching was applied to ceramic surfaces. When compared to the values associated with more extended etching periods that were in line with the findings of previous investigations, the mean results demonstrated that lower values for SBS were obtained for smaller etching times for the IPS e.max and CEREC blocs groups, with a statistically significant difference. (Straface et al., 2019). In contrast to more extended etching periods, lower HF acid etching periods showed minor vitreous phase dissolution and were insufficient to dissolve the vitreous phase properly. As the roughness of the ceramic surface directly impacts the shear bond strength, this is probably explained by the existence of diminished microporosities, which promote the lower level of contact between the resin cement and ceramic surface, leading to less mechanical interlocking and smaller bond strengths (Kukiattrakoon and Thammasitboon 2007). However, the IPS Empress group demonstrated that etching times greater than sixty seconds resulted in a significant reduction in SBS in line with the findings of Other researchers (Barghi et al., 2006, Naves et al., 2010), Barghi and colleagues (2006) found that samples etched with HF gel for 90–180 seconds as opposed to 60 seconds resulted in lower bond strengths for a leucite-reinforced ceramic. Longer etching durations may result in stronger bonds, but only to a certain extent; beyond that, longer etching times may weaken bonds. This could be explained by crystalline residue accumulating on the ceramic surfaces (Magne and Cascione 2006) and the deeper dissolving depth brought about by longer etching times linked to inadequate resin cement penetration because of its high viscosity, resulting in an uneven cement-ceramic interface. Due to their fragile nature, ceramics may lose some of their mechanical strength if certain regions are left unfilled (Mecholsky Jr 1995) using main approaches: Two potential areas of stress concentration are 1) the pronounced geometry of the empty channels and 2) the delicate void area beneath the cement-ceramic entanglement, which could concentrate stress under mechanical loading. (Naves, Soares et al. 2010). Other studies showed contrasting results, (Sundfeld Neto et al., 2015) His research revealed that increased HF concentrations strengthened the link between resin cement and disilicate ceramic, but no significantly differentiable results were obtained for leucite-reinforced ceramics.

The second hypothesis, which states that differing ceramic materials significantly affect bond strength with similar HF etching periods, was accepted in light of our findings. They were using the same etching time. IPS e.max had the highest bond strength values than other materials. The IPS Empress had higher SBS than CEREC blocs ceramic at 20 and 60 conditioning times. Similar results were found by Kansu and others (Kansu and Gökdeniz 2011) and Verissimo and others (Veríssimo et al., 2019), who found that SEM scans revealed distinct ceramic surface topographies, with the treated groups having much more irregularities than the group that was not treated. Images from the SEM showed that the pressed disilicate subjected to 10% HF for 60 seconds had a rougher and more porous surface than it did after 20 seconds and greater adhesion values. Ramakrishnaiah et al. (2016) examined how various HF conditioning durations affected the porosity and roughness pattern of glass-ceramic surfaces, suggesting that longer exposures produced more profound, more irregular grooves, enhancing surface roughness. (Della Bona et al., 2004).

Regardless of the surface conditioning period, the IPS e.max ceramic exhibits significantly greater bond strengths and superior mechanical qualities due to its increased crystalline phase (70% ± 5%) compared to the IPS Empress and CEREC blocs. This finding appears to support the theory that the ceramic microstructure has an essential influence on the resin-ceramic adhesion zone's fracture resistance. (Kansu and Gökdeniz 2011).

However, it should be mentioned that this study has certain shortcomings. Firstly, only one HF concentration was applied. More SBS researches are required to assess the resin-ceramic interface following the application of various HF acid concentrations.

5. Conclusion

The following conclusion can be drawn within the limits of this investigation.

1. The different HF etching times influenced the bond strength of three ceramic materials to the resin cement

2. There was a statistical difference in SBS among the three ceramic materials. The IPS e.max showed the highest values of bond strength

3. The IPS Empress showed a decrease in bond strength when etched for more than 60 seconds

3. More than 60 seconds of etching can increase the shear bond strength for CEREC blocs and IPS e.max ceramics

By centering on the remarkable SBS results of e.max and their reply to erosion, practitioners gain a greater understanding of selecting the most suitable protocol for ceramic surface treatment. So, 60 second etching of the e.max can increase the SBS of resin cement as compared with 20 second. This is beneficial when there is needing to overcome the decrease in the retention of the restoration.

Acknowledgments

This research received support from the University of Baghdad, the University of Technology, and the University of Mosul, which provided valuable insight and expertise that significantly contributed to the research.

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