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Effect of Different Cleaning Techniques on Shear Bond Strength of Contaminated Dental Ceramics

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Abstract

Saliva contamination has effect on bond strength between resin cement and ceramic restoration. This study aimed to evaluate the efficacy of cleaning methods of contaminated ceramics and their effect shear bond strength of zirconia and vita enamic to resin cement. In this study, two types of ceramics used; zirconia and vita enamic (n = 70). Each type of ceramic subjected to saliva contamination and divided according to methods of decontamination to 7 groups (n = 10); Group (1) control with no contamination. Group (2) contaminated with artificial saliva then cleaned using hydrofluoric acid for 30 seconds. Group (3) contaminated then cleaned using 37 % phosphoric acid for 30 seconds. Group (4) contaminated then cleaned using immersion in ultrasonic bath filled with distilled water for 5 minutes. Group (5) contaminated then cleaned using 70% alcohol, Group (6) contaminated then cleaned using vacuum drying for 30 seconds and Group (7) contaminated by immersion for 5 minutes in artificial saliva. Each group was subjected to shear bond strength test. Data were collected, tabulated, and statistically analyzed. Result of shear bond strength revealed that the highest value was in control group (10.4±0.53 Mpa) in zirconia and (13.35±0.19) in vita enamic followed by hydrofluoric acid (9.84±0.33) in zirconia and (12.84±0.37 Mpa) in vita enamic then phosphoric acid (9.67±0.49 MPa) in zirconia and (12.24±0.24 MPa) in vita enamic then ultrasonic path (9.4±0.48 MPa) in zirconia and (12.13±0.26 MPa) in vita enamic and alcohol (8.67±0.37 MPa) in zirconia and (11.67±0.24 MPa) in vita enamic then vacuum drying (7.72±0.38 MPa) in zirconia and (11.25±0.28 MPa) in vita enamic. Saliva contamination resulted in decrease bond strength between dental ceramics and resin cement. Clinical retreatment with hydrofluoric acid is preferred due to its ability to improve the cementation of dental ceramics. HF helps in achieving a mechanical union by exposing the crystals at the surface of the ceramic structure, creating areas of micro retention. Phosphoric acid is good method for decontamination from artificial saliva because it's successfully removed carbon-based contaminants on contaminated ceramic surfaces. Ultrasonic path and alcohol methods have little cleaning effect on saliva contaminated dental ceramics.

Keywords: Saliva**,** hydrofluoric acid, shear bond strength, zirconia, vita enamic

Full-length article **Corresponding Author*, e-mail:

1. Introduction

With the growing demand for esthetic treatments, there has been development and improvement in the composition of ceramics and cementing systems to improve the mechanical strength of the ceramic material, enhance bonding capacity to both the tooth and the restoration, and enhance esthetics [1]. The long-term success of ceramic restorations is directly dependent on a suitable cementation technique capable of establishing durable bond strength between restoration and substrate. Total ceramic restorations depend on adhesive cementation with resin cements to achieve a good clinical prognosis. [2]. Isolation of tooth surface from water or patient saliva and avoid contamination from operator hands during cementation is necessary to improve the bonding of ceramics restoration. The restorations must be cleaned appropriately before cementing in place, but in a manner that itself does not reduce bond strength by chemical modification of the surface. Thus, the cleaning technique must be compatible to the chemistry of the crown material and the bonding agent. [3]. The bonding protocol for silica-based glass ceramics and non-silica-based ceramics is different due to the materials composition. Silica-based glass ceramics use hydrofluoric acid to etch the bonding surface due to its effect on the silica particles. Non-silica-based ceramics, lacking these particles, will not etch if acid is applied. Therefore, a different bonding approach like air abrasion must be utilized.

Ceramic restorations are tried in and fitted to abutment teeth before cementation. During this process, the restoration surface is contaminated with saliva, blood, and commonly used rubber gloves, which most likely impairs the required bonding performance. [3]. Due to this problem, several cleaning methods have been tested in previous studies in order to remove the contamination and provide strong resin bond.

Methods of rinsing the contaminated surfaces with water, cleaning it ultrasonically in isopropanol or a combination of both methods resulted in a minor or no cleaning effect and therefore, are not recommended [4]. Another method of covering the surface with phosphoric acid gel showed a good efficiency in removing saliva, however was not effective in the removal of silicone residuals [5]. The most effective ceramic surface treatment methods recommended in previous studies is using hydrofluoric acid (HF) etching or air-particle abrasion [2]. The objective of this study is to compare the shear bond strength between contaminated and non-contaminated two different types of ceramics; hybrid ceramics and high translucent Zirconia. The null hypothesis was that saliva contamination affects the bond strength between ceramic surface and rein cement.

2. Materials and Methods

2.1. Preparation of specimens

One hundred and forty (140) samples were fabricated from two types of CAD/CAM ceramic blocks were used; High translucent Zirconia: Ceramill zolid HT + Preshade B1 Zirconia (70 samples) and Hybrid ceramic: Vita Enamic, VITA Zahnfabrik (Shade 1m1) (70 samples) in the form of discs (10mm diameter x 3mm thickness) .First the zirconia and vita enamic discs were installed on the tray of the electric isoMet micro saw 4000 where it was cut into slices using a saw diamond disk of 0.6mm thickness (Buehler, USA) under copious amounts of water to produce discs with final thicknesses of 3.0 mm. The thickness of the samples was assessed using a digital micrometer with the accuracy of 1um on five different sites (center and each corner of each sample) of each sample after sintering. After finishing all the discs were subjected to ultrasonic cleaning in both sides in distilled water to remove any residues on the surface and air dried.

The discs of zirconia or vita enamic were divided into 7 groups (each group n = 10), according to methods of decontamination and surface treatment technique. Group (1) control with no contamination. Group (2) contaminated then cleaned using hydrofluoric acid for 30 seconds. Group (3) contaminated then cleaned using 37 %phosphoric acid for 30 seconds. Group (4) contaminated then cleaned using immersion in ultrasonic bath filled with distilled water for 5 minutes. Group (5) contaminated then cleaned using 70% alcohol. Group (6) contaminated then cleaned using vacuum drying for 30 seconds. Group (7) contaminated by immersion for 5 minutes in artificial saliva.

2.2. Fixation of the ceramic specimen in an acrylic mold

Each one of the ceramic discs was embedded and fixed in a mold (12 mm in diameter and 3 cm in height) filled with acrylic resin to be used as mold or mounter during fabrication and testing.

2.3. Saliva contamination

Each sample, except for the control group (Group 1) was subjected for saliva contamination using artificial saliva solution before the bonding procedure. The artificial saliva was placed on the surface of zirconia and vita enamic samples using micro brush at 37°C for 60 second. Then the samples were rinsed with distilled water spray for 15 seconds and dried with oil free air dryer spray for 30 seconds. [26] **Group 7**: After saliva contamination, the surfaces of the samples were rinsed with distilled water spray and air dried for 10 seconds followed by application of G-ænial universal bond agent.

2.4. Cleaning methods

Group 2: After saliva contamination, the surfaces of ceramics slices or discs were etched with 9.5% HF acid for 30 seconds with the Inspiral™ brush tip of the syringe. Then washed with air/water spray for 30 seconds and dried for 10 seconds followed by application of the bonding agent.

Group 3: After saliva contamination, the surfaces of discs of zirconia or vita enamic were etched by 37% phosphoric for 30 seconds, rinsed with distilled water spray, and air dried for 10sec followed by application of G-ænial universal bond.

Group 4: After saliva contamination, the surfaces of discs of zirconia or vita enamic were cleaned using immersion in ultrasonic bath filled with distilled water for 5 minutes then air dried for 15 sec followed by application of G-ænial universal bond.

Group (5): after contamination with artificial saliva, 70% alcohol used for 15 seconds and air dried for 10sec. **Group (6):** after contamination with saliva samples cleaned using distilled water for 10 sec followed by insertion in auto clave Woson class B vacuum drying program for 30 seconds.

2.5. Shear bond strength test

A split Teflon mold [6] with diameter 3 mm and thickness 5 mm was used to shape the BisCem resin cement and to hold it in place on the specimens' surface until it set. Figure (3). The resin cement was mixed and applied through the tubes to the ceramic bonding area as follows: The cap was removed and discarded from the dual–syringe, and a small amount of material was dispensed onto a mixing pad to eliminate any voids in each chamber of the dual-syringe. A mixing tip was attached to the dual –syringe, base and catalyst paste of resin cement were mixed in a 1:1 ratio according to the manufacturer's instructions , then it was light cured (20 sec./surface) using Light curing light emitting diode (LED) with intensity 1000 mW/cm2 (Bluephase G2 wireless curing lamp-Ivoclar Vivadent).

All samples were individually and horizontally mounted on a computer controlled testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a load cell of 5 kN and data were recorded using computer software (Blue hill Lite; Instron Instruments). Samples were secured to the lower fixed compartment of testing machine by tightening screws. Shearing test was done by compressive mode of load applied at ceramic (Zirconia or vita enamic) resin interface using a half circle notch ended metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5 mm/min as shown in figure (4). The load required to debonding was recorded in Newton [7].

Failure was manifested by an audible sound and evidence of debonding between the resin cement and surface of ceramic sample. Shear bond strength was calculated by dividing the load at failure to bonding area to express the bond strength in MPa (11) $\tau = P/ \pi r2$ where; τ =shear bond strength (MPa), P = max load at failure (N), π =3.14 and r =radius of resin disc (mm).

2.6. Statistical Analysis

The analysis of the data was carried out using the IBM SPSS version 25 statistical package software. Normality of the data was tested using the Shapiro Wilk test. Data were expressed as mean ± SD and minimum and maximum of range for parametric quantitative data. Analyses were done between all groups for parametric quantitative data using One Way ANOVA test followed by post hoc LSD analysis between each two groups. P-value less than 0.05 were considered statistically significant.

3. Results

Regarding shear bond strength between the seven groups in Zirconia, there was a significant difference between groups, the highest value was in control group (10.4±0.53 Mpa), followed by hydrofluoric acid (9.84±0.33) group then phosphoric acid group (9.67±0.49 Mpa) followed by ultrasonic bath distilled water group (9.4±0.48 Mpa), then 70% alcohol group (8.67±0.37 Mpa) then vacuum drying group (7.72±0.38 Mpa) and lastly contaminated with saliva group (7.33±0.27 Mpa).

There was a significant difference between each two group except for:

- Group II and III showed insignificant differences P = 0.530
- Group II and IV showed insignificant differences P = 0.105
- Group III and IV showed insignificant differences P = 0.309

Group VI and VII showed insignificant differences P = 0.150. Shear bond strength mean values between the seven groups in vita enamic material, there was a significant difference between groups, the highest value was recorded in control group (13.35±0.19 Mpa), followed by hydrofluoric acid group (12.84±0.37 Mpa) then phosphoric acid (12.24±0.24 Mpa) then ultrasonic bath distilled water group (12.13±0.26 Mpa), followed by 70% alcohol group group (11.67±0.24 Mpa) then vacuum drying group (11.25±0.28 Mpa) and the lowest value was contaminated with saliva group (10.66±0.26 Mpa).

There was significant differences between each two group except for Group III and IV showed insignificant differences P = 0.533

4. Discussion

The increased demand of the patients for esthetics has driven the development of all ceramic materials [8]. There is an increase benefits for the use of all ceramics because they have enhanced esthetic properties, diminished plaque accumulation and high biocompatibility [9, 10]. Both ceramic and luting agent that used in this study was selected according to available products in the market in order to have a beneficial practical application for the dental profession. In this study we used CAD-CAM ceramic blocks (high translucent zirconia and vita enamic hybrid ceramics) due to their standardized manufacturing process with more homogeneous structure, reliable quality and better mechanical and physical properties. [11, 12]

The cementation procedures, which are affected by the type of ceramic materials, surface conditioning techniques, and cementing agents play a great role in the success of all-ceramic restorations [3,13]. Different types of ceramics need different surface conditioning methods as they have different chemical compositions [14,15] In general, manufacturers recommend specific pre-treatments for the inner ceramic surface before bonding to achieve a sufficient micro-mechanical and chemical interlocking between ceramic restorations and adhesive luting systems. During the intraoral try-in procedure, restoration surfaces come in close contact with the gingiva, causing contamination with saliva, sulcus fluid, and blood. Different cleaning protocols for zirconia were used and their influence on bond strength had been recorded. [16, 17]. Saliva contamination resulted in decrease bond strength value due to saliva might have left a thin invisible film on the zirconia surface, which might inhibit stable micro-mechanical retention and stable chemical bonds. Prior bonding all compounds of phosphate, such as phospholipids from saliva are considered to be problematic, because this may impair bonding effectiveness [18] due to formation of zirconium phosphate. Therefore, immediate elimination of these precipitates is crucial for adhesion.

Teresa P et al (2021) [19] found that mechanical properties of dental ceramics values were significantly reduced after one month of immersion in saliva. Results of this study show that hydrofluoric acid group was the most effective method in cleaning techniques from saliva contaminated zirconia and vita enamic followed by phosphoric acid then ultra-sonic cleaning path with distilled water group followed by 70% alcohol cleaning techniques while the vacuum drying group was the least effective method as decontamination technique from artificial saliva. This result is in agreement with the Patel D et al (2015) study which was designed to test resin to zirconia bond strength and its durability related to different cleaning methods of contaminated zirconia bonding surfaces. It was found that saliva contamination led to the lowest bond strength values. [20].

Hydrofluoric acid (HF) helps in achieving a mechanical union by exposing the crystals at the surface of the ceramic structure, creating areas of micro retention. As reported by Bona et al (2002) [14] that HF produces the most prominent etching pattern on dental ceramics. Many previous studies supported that HF acid is the most effective methods in cleaning of saliva contaminated ceramics. K et al (2009) concluded that among the different cleaning methods, HF is the most effective in removing contamination with saliva or a silicone disclosing medium. [21]**.** Abdulkader et al. [22] (2020) reported that bond strength value is significantly influenced by the surface treatment method and the HF acid group recorded statistically significantly higher SBS mean value than the aluminum oxide sandblasting method.

Previous studies like Cekic-Nagas et al. (2016) [23] concluded that HF acid gel treatment did not affect bond strength values. Hajjaj M S and Alzahrani S J [24] found that cleaning the contaminated zirconia surface with hydrofluoric acid did not restore SBS to the uncontaminated state, but it was significantly higher than simply rinsing with water.

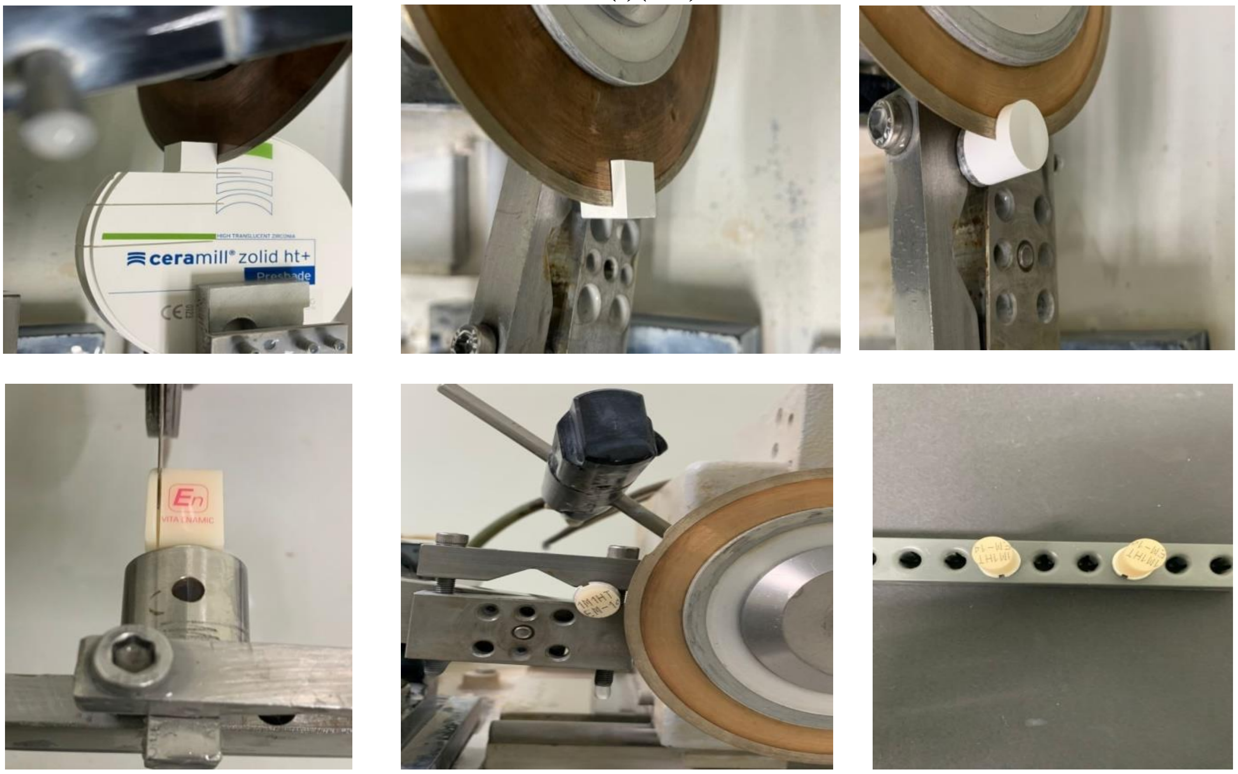

Figure 1. Construction of zirconia and vita eanmic samples

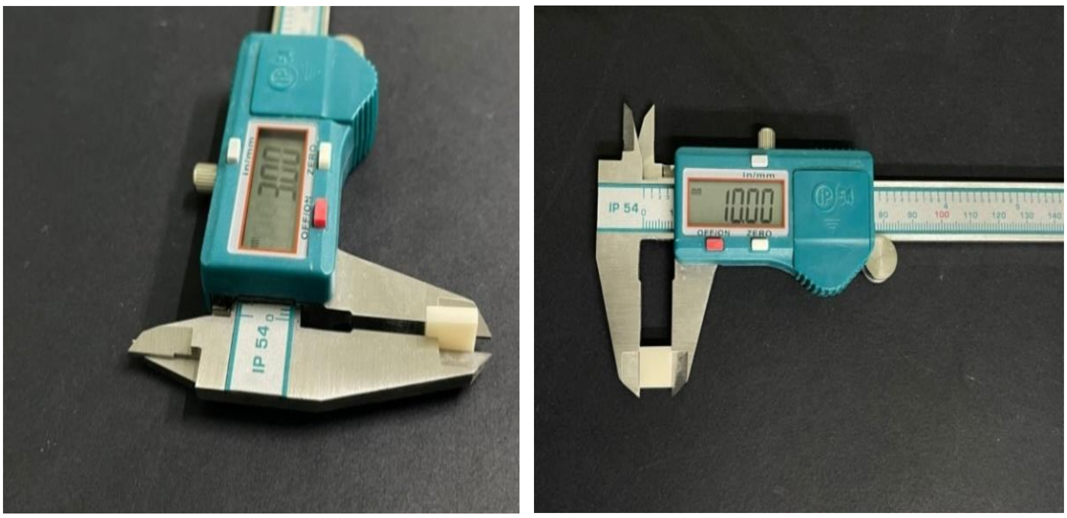

Figure 2. Calibration of zirconia discs by digital caliber after sintering

Figure 3. Application of BisCem resin cement

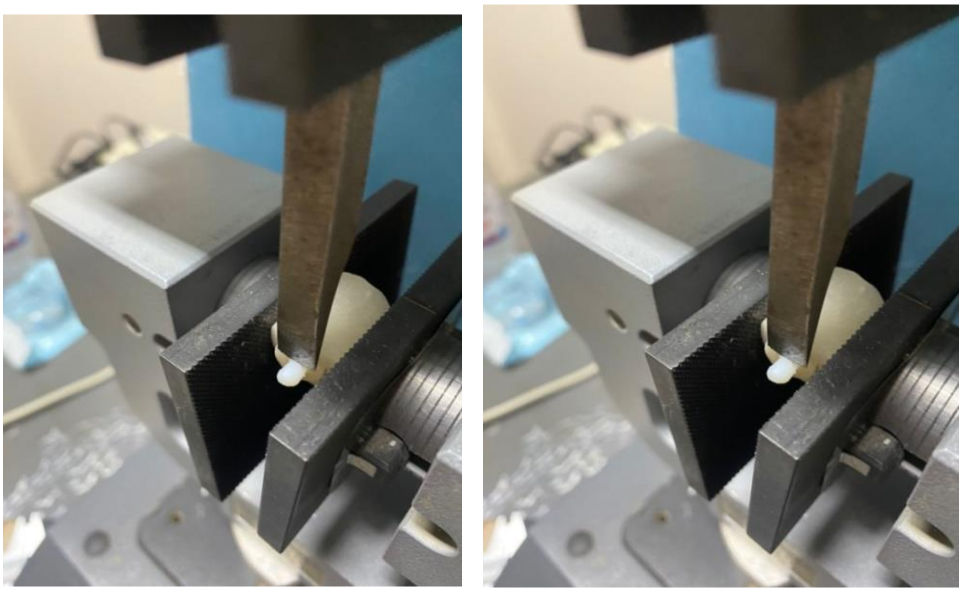

Figure 4. Sample placed on universal testing machine

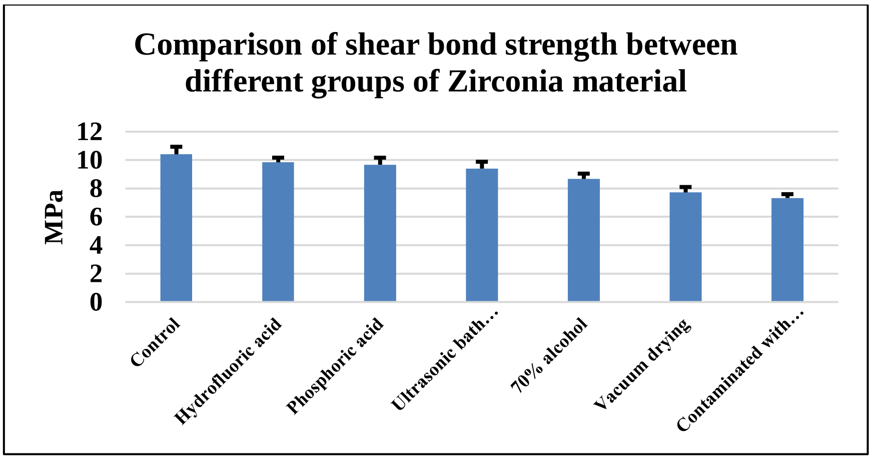

Figure 5. Histogram showing shear bond strength between different groups of Zirconia samples

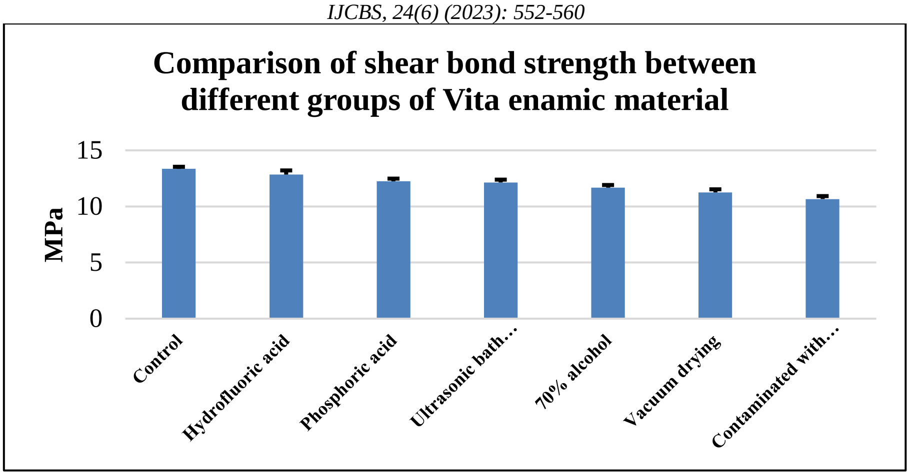

Figure 6. Histogram showing shear bond strength between different groups of vita enamic samples

Table 1. Comparison of shear bond strength between different groups of Zirconia samples

Zirconia	**Control (I)**	**Hydrofluoric acid (II)**	**Phosphoric acid (III)**	**Ultrasonic bath Distilled water (IV)**	**70% alcohol (V)**	**Vacuum drying (VI)**	**Contaminated with saliva (VII)**	**P value**
Shear bond strength	(9.86-11.04) 10.4±0.53	(9.32-10.21) 9.84±0.33	(8.97-10.33) 9.67±0.49	(8.95-10.13) 9.4±0.48	(8.27-9.16) 8.67±0.37	(7.15-8.13) 7.72±0.38	(7.04-7.63) 7.33±0.27	***<0.001****
P value between each two groups								
I		***0.042****	***0.010****	***0.001****	***<0.001****	***<0.001****	***<0.001****	
II			*0.530*	*0.105*	***<0.001****	***<0.001****	***<0.001****	
III				*0.309*	***0.001****	***<0.001****	***<0.001****	
IV					***0.010****	***<0.001****	***<0.001****	
V						***0.001****	***<0.001****	
VI							*0.150*	

One way ANOVA test for quantitative data between the seven groups followed by post hoc LSD analysis between each two groups.

****: Significant level at p value < 0.05***

Table 2. Comparison of shear bond strength between different groups of Vita enamic samples

Vita enamic	Control (I)	Hydrofluoric acid (II)	Phosphoric acid (III)	Ultrasonic bath Distilled water (IV)	70% alcohol (V)	Vacuum drying (VI)	Contaminated with saliva (VII)	P value
Shear bond strength	(13.13-13.63) 13.35±0.19	(12.36-13.35) 12.84±0.37	(11.97-12.53) 12.24±0.24	(11.86-12.46) 12.13±0.26	(11.35-11.95) 11.67±0.24	(10.98-11.63) 11.25±0.28	(10.33-10.95) 10.66±0.26	<0.001*
P value between each two groups								
I		0.005*	<0.001*	<0.001*	<0.001*	<0.001*	<0.001*	
II			0.001*	<0.001*	<0.001*	<0.001*	<0.001*	
III				0.533	0.002*	<0.001*	<0.001*	
IV					0.010*	<0.001*	<0.001*	
V						0.018*	<0.001*	
VI							0.002*	

One way ANOVA test for quantitative data between the seven groups followed by post hoc LSD analysis between each two groups.
**: Significant level at p value < 0.05*

Kweon and Hakansson (2006) [25] reported that the application of phosphoric acid successfully removed carbon-based contaminants on contaminated zirconia surfaces. However, this acid leaves an inorganic residue of the phosphate group on the surfaces, which negatively affects the chemical bond between MDP and zirconia and thus decreases the zirconia-resin bonding [26].

Yang B et al (2008) [4] recommended that phosphoric acid cleaning seemed to be an effective chemical cleaning method to remove salivary contamination. Quaas AC et al (2007) [6] and Elif Pak T et al (2016) [27] concluded that cleaning with phosphoric acid showed higher values than cleaning with alcohol or steam water. Sabrina F et al (2008) [18] reported that 37% phosphoric acid gel had high SBS values than 70% isopropanol and Nikpour L et al (2019) [28] study result showed that PA is better than ultra-sonic path cleaning or acetone. While Phark J H. et al. (2009) [5] recommended that combination of phosphoric acid with ultrasonic bath in ethanol is an efficient method for cleaning contaminated modified zirconia surfaces.

Using of ultrasonic cleaning as a surface cleaning method of zirconia ceramic is controversial. Some researchers found that it was more effective on polished zirconia surface. While, others reported that ultrasonic cleaning of dental restorations after sandblasting should be avoided because it decreased the adhesive strength of resin luting material. [29]. The results of the present study are coincident with those of Attia and Kern (2011) [30] and Lehmann F et al (2011) (29) who demonstrated that ultrasonic cleaning methods had little cleaning effect on bonding to contaminated zirconia.

Cleaning contaminated zirconia with water or alcohol was not an effective method for removing the protein contaminants in blood or saliva, as X-ray photoelectron spectroscopy (XPS) showed the presence of carbon, oxygen, and nitrogen on the surface after contamination and cleaning. (4) This residue would interfere with the bonding of zirconia to composite resin, resulting in a decrease in bond strength values.

Quaas et al (2007) [6] reported that cleaning with alcohol was not effective in strengthening the bond of resin cements to ceramic surfaces. Yang B et al (2008) [4] also showed that alcohol cleaning was not an effective method for removing organic contaminants. Similar to the results by Quaas et al. and Yang et al., it was found in this study that alcohol cleaning was not an effective surface cleaning method. Previous studies like Awad MM et al (2022) [31], Patel D (2015) [20], Philipp Güers et al (2019) (32) and Kim H-J et al (2022) [33] support results in this study and recommended that alcohol had little or no cleaning effect on contaminated zirconia.

Conclusions

1. Saliva contamination resulted in decrease bond strength between dental ceramics and resin cement.
2. Clinical retreatment with hydrofluoric acid is preferred due to its ability to improve the cementation of dental ceramics. HF helps in achieving a mechanical union by exposing the crystals at the surface of the ceramic structure, creating areas of micro retention.
3. Phosphoric acid is good method for decontamination from artificial saliva because it's successfully removed carbon-based contaminants on contaminated ceramic surfaces.

Recommendations

1. Proper isolation during cementation is preferred to avoid saliva contamination.

2. Cleaning methods like hydrofluoric acid and phosphoric acid is better than ultrasonic cleaning path or alcohol cleaning methods.

3. Further studies with application of primer and different contaminants in order to mimic wider variety of clinical situations are recommended.

Limitations of the study

1. Saliva was the only source of contamination in all groups. It is possible that the presence of blood, and/or silicone try-in pastes are also sources of surface contamination in clinical settings. Further studies with application of primer and different contaminants in order to mimic wider variety of clinical situations are recommended.

2. Recent different cleaning techniques as cleaning paste not used in this study.

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