



Evaluation of Micro tensile Bond Strength of Lithium Silicate Ceramics Conditioned with Different Silane Primers

Mahmoud Soliman¹, Lamiaa Dawood², Walid El-zordk³

1: Clinical demonstrator of Fixed prosthodontics Dept. Faculty of Dentistry, Mansoura University, Egypt

2: Professor of Fixed Prosthodontics, Fixed Prosthodontics Dept. Faculty of Dentistry, Mansoura University, Egypt

3: Associate Professor of Fixed Prosthodontics, Fixed Prosthodontics Dept. Faculty of Dentistry, Mansoura University, Egypt

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ABSTRACT: The mechanical characteristics of glass ceramics may be affected by hydrofluoric acid (HF) etching. Furthermore, because of its toxicity and volatility, HF has been called into doubt regarding potential dangers to human health. MEP evaluated for achieving durable bond strength for different glass ceramic materials with less hazardous effects than HF. Also, universal primer evaluated for achieving durable bond strength for different glass ceramic materials without need for other primers for metallic or zirconia restorations. The objectives of this in-Vitro study are to evaluate the microtensile bond strength of different heat pressable glass ceramics (lithium disilicate / zirconia-reinforced lithium silicate / zirconia-reinforced lithium disilicate) to dentin using dual cure resin cement and different primers (conventional silane / self-etch silane / silane containing MDP). Sound natural molar teeth extracted due to periodontal causes were collected, disinfected, cleaned, and cut occlusally for exposure of coronal dentin using low speed diamond saw (Isomet). Wax cuboid rectangles were designed on software and milled using milling machine. These rectangles randomly divided into 3 groups for production of 3 pressable glass ceramics. Then, cementation of ceramic rectangles on dentin using different silane solutions and dual cure resin cement was done. After cementation, each specimen was cut into microbars (1mm²) using isomet. Evaluation of microbars using stereomicroscope to select intact microbars (20 microbars for each subgroup). Aging process by thermocycling for 5000 cycles (5-55⁰ C) was performed. Then, microtensile bond strength test was performed. Microtensile bond strength values for each group were collected, tabulated and statistically analyzed using one way and two way ANOVA tests. Also, Bonferroni's post hoc test was performed. Failure mode detection was performed using stereomicroscope (50 X) and scanning electron microscope (75 X, 250 X, and 500 X). Then, failure mode analysis underwent statistical

analysis. MEP provided statistically significant lower bond strength for all ceramic materials used. Vita Ambria provide statistically significant higher bond strength than celtra press. No significant difference in bond strength using Bis-silane and Monobond N. MEP resulted in the lowest bond strength for all glass ceramics used. Bond strength after HF acid etching was affected by the chemical composition of glass ceramic material.

KEYWORDS: HF, MEP, Monobond N, lithium disilicate glass ceramics, zirconia reinforced lithium silicate glass ceramics, zirconia reinforced lithium disilicate glass ceramics.

I. INTRODUCTION

Ceramics are widely used in dentistry due to their esthetic characteristics, good mechanical properties, biocompatibility, and favorable cumulative survival rates. Dental ceramics are generally classified into glass ceramics, oxide ceramics, and hybrid ceramics according to their composition. The compositions and processing techniques of glass ceramics have been developed over the years. Glass ceramics are now mostly composed of lithium disilicate-based pressable ingots or computer-aided design and computer-aided manufacturing blocks.¹

Lithium disilicate glass-ceramic has good biocompatibility because of reduced plaque retention and good adhesion and proliferation of human epithelial cells² and human gingival fibroblasts³, especially when its surface is smooth after polishing. Lithium silicate ceramics have greater translucency than typical zirconia ceramics.⁴

As a result, lithium silicate ceramics can be employed in the anterior region without the addition of a layer of veneering porcelain, reducing the danger of porcelain chipping.⁵ Minimally invasive tooth preparations can be established for glass ceramic restorations due to the high bond strength between the glass ceramic restoration and tooth structure.⁶ Several studies showed that lithium



disilicate glass ceramic restorations showed good survival and success rates.⁷⁻⁹

Because of its net form processing, decreased porosity, higher flexural strength, and superior marginal fit, the hot pressing technique based on the viscous flow of glass ceramics has found widespread application in dental restoration.¹⁰

Zirconia-reinforced lithium silicate glass-ceramics was presented in 2013 as a stronger substitute to the lithium disilicate ceramic material, Celtra (Dentsply Sirona) and Suprinity PC (Vita) are commercial lines of this material.¹¹ The main crystalline phase in these materials is lithium silicate with zirconium dioxide crystals (~10%). After the crystallization process of this material, the nucleated lithium silicate crystals have average dimension of (0.5 to 1 μm) that is up to 6 times less than that detected for lithium disilicate crystals present in lithium disilicate glass-ceramics. Zirconia particles hinder the crystal growth in the material and are responsible for fine crystallization of the material. Smaller crystals improve mechanical properties of the material. Additionally, compared to lithium disilicate glass-ceramics, these materials have good optical characteristics.¹²

Zirconia component could act as a crystal phase reinforcing the material and avoiding crack spreading.¹³ zirconia reinforced lithium silicate ceramics have higher fracture toughness and Vickers hardness than lithium disilicate glass ceramic.^{14, 15} Dentsply Sirona Company presented zirconia reinforced lithium silicate as Celtra Duo for CAD/CAM fabrication and Celtra Press for heat pressing. Celtra Press has several advantages; superior flexural strength of about 500 MPa (after power firing)¹⁶, and Outstanding flow properties during pressing. Crystals in the Celtra Press pellet are smaller in size leading to better compressibility and flowability (lower viscosity) during the pressing process than e.max press. Thin sections of restorations can be pressed with less number of sprues. In combination with the newly introduced investment, only a minimal reaction layer is formed. Celtra Press has a lower pressing temperature (50–60°C) than conventional lithium disilicate glass ceramics. The lower pressing temperature greatly reduces the hardness of the reaction layer, resulting in simplicity of its removal. Simpler and faster polishing is due to small crystal size.¹³

Vita Ambria is a recent zirconia-reinforced lithium disilicate glass ceramic. It has natural translucency, opalescence and fluorescence. Because the material and investment material are

ideally matched, it achieved efficient and highly accurate press results with a minimized reaction layer, and its flexural strength is more than 500 MPa.¹⁷

Adhesion has been well-documented to play a role in the strengthening of glass ceramics. Weak bonding between glass ceramics and resin cement could cause uneven stress distribution. As a result, cohesive failure of the resin cement may occur leading to weakening of the unsupported restoration under the functional load.¹⁸ Glass-ceramic restoration material is bonded to dental surfaces using an adhesive system. Micromechanical interlocking and chemical bonding are the main mechanisms involved in attaching dental glass ceramics.¹¹ Hydrofluoric acid was used to etch glass-rich ceramics, resulting in micromechanical interlocking.¹⁹ Additional chemical bonding is accomplished by silanization utilizing ceramic primers containing silane coupling agents, the most commonly utilized of which is methacryloxypropyl trimethoxysilane. When the alkoxy groups of the bifunctional silane molecule hydrolyze to silanols, monomer adsorption to the ceramic substrate happens via condensation. In addition, the silane coupling agent enhances the surface energy of the ceramic, which improves wettability.²⁰

Dentists liked hydrofluoric acid because of its low cost, effectiveness, and simplicity of usage. By interacting with silicon dioxide, hydrofluoric acid partly dissolves the glass matrix phase. As a result, hydrofluoric acid forms a network of micro-porosities, producing a micro-retentive pattern for resin cement interlocking, resulting in greater mean bond strengths than a non-etched glass ceramic surface.²¹

However, the mechanical characteristics of glass ceramics may be affected by HF acid etching.²² Furthermore, due to its toxicity and volatility, HF acid has been questioned for its dangerous effects on human health, particularly when it comes into touch with the eyes or skin or during intraoral restorative repair. HF acid is very corrosive and may be absorbed into the blood and bone through the skin; at higher concentrations, it can potentially cause cardiac arrest.²³

Recently, a less toxic self-etching primer was developed to lessen technique sensitivity and allow acid etching of glass-ceramic restorations. According to the manufacturer, this material allows for the superficial etching of ceramic restorations with ammonium polyfluoride and silanization with trimethoxypropyl methacrylate. Furthermore, the manufacturer states that the roughened surface is less noticeable than the surface generated during



HF acid treatment but still provides acceptable adhesion.²⁴

Dental primers have undergone alterations due to chemical developments and have made enormous advances over the last two decades. Universal primers (including 10-methacryloyloxydecyl dihydrogen phosphate and silane) are compounds that might be utilized to attach different restorations involving zirconia ceramics, glass ceramics, and metals. Without the need for a separate ceramic primer, in universal

primer, active and stable silane coupling agents may establish chemical bonds with glass-ceramic surfaces.²⁵

II. MATERIALS AND METHODS

Materials used in the present study were tabulated according to the product name, material type, chemical composition, lot number, and their manufacturer. (Table 1)

Table no 1: Materials used in this study

Product name	Material type	Chemical composition	Lot number	Manufacturer
IPS e. max press	Lithium disilicate glass ceramic	SiO ₂ : 57.0–80.0%, Li ₂ O: 11.0–19.0% K ₂ O: 0.0–13.0%, P ₂ O ₅ : 0.0–11.0% ZrO ₂ : 0.0–8.0%, ZnO: 0.0–8.0% Coloring oxides: 0.0–12.0%	Z02JF3	Ivoclar Vivadent AG, Schaan, Liechtenstein
Celtra Press	Zirconia reinforced lithium silicate glass ceramic		16004070	Dentsply-Sirona, Bensheim, Germany
Vita Ambria	Zirconia reinforced lithium disilicate glass ceramic	SiO ₂ : 58–66%, Li ₂ O: 12–16% Al ₂ O ₃ : 1–4%, K ₂ O: 1 + 4% P ₂ O ₅ : 2–6%, ZrO ₂ : 8–12% B ₂ O ₃ : 1–45%, CeO ₂ : 0–4% V ₂ O ₅ : <1%, Tb ₂ O ₃ : 1–4% Er ₂ O ₄ : <1%, Pr ₆ O ₁₁ : <1%	79162	Vita Zahnfabrik, Bad Sachingen, Germany
IPS Ceramic Etching Gel	Hydrofluoric acid	<5% Hydrofluoric acid	Z02FJY	Ivoclar Vivadent AG, Schaan, Liechtenstein
Porcelain primer	Prehydrolyzed silane primer	Ethanol and silane	210000163 8	Bisco Inc., Schaumburg, IL, USA
Monobond N	Universal primer	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate	Z02M0Y	Ivoclar Vivadent AG, Schaan, Liechtenstein
Monobond Etch & Prime	Self-etching glass ceramic primer	Tetrabutylammonium-dihydrogen trifluoride ≤10%, silane system (based on trimethoxypropyl methacrylate) 1–<2.5%, methacrylate phosphoric ester 3%–<10%, butanol 10%–<25%, water, and colorant	Z01butylV LY	Ivoclar Vivadent AG, Schaan, Liechtenstein
Tetric N-Bond Universal	Light curing dental adhesive	Methacrylate, ethanol, water, highly dispersed silicon dioxide, initiators, and stabilizers.	Z02LXJ	Ivoclar Vivadent AG, Schaan, Liechtenstein



Variolink Esthetic DC	Dual-curing resin-based dental luting material	Urethane dimethacrylate and further methacrylate monomers, 38% inorganic fillers including ytterbium trifluoride and spheroid mixed oxide Initiators, stabilizers and pigments	Z02FL9	Ivoclar Vivadent AG, Schaan, Liechtenstein
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Maxillary molars used in this study were collected from Oral and Maxillofacial Surgery Department, Faculty of Dentistry, Mansoura University. Teeth were extracted due to periodontal causes from patients with average age 45-60 years. Selected teeth were inspected using magnifying loupe 5X (UNIVET s.r.l. Via Giovanni Prati, Italy) to ensure the absence of any caries or fractures and also examined under visible light trans-illumination (Elipar, 3M ESPE Dental, St. Paul, MN, USA) to check presence of any cracks. Any tooth showed any defect was excluded. The Research Ethics Committee of Mansoura University's Faculty of Dentistry in Egypt gave permission to utilize human natural teeth (No. 19030821)

Teeth were disinfected for 7 days with 1:10 diluted 5.25% sodium hypochlorite household bleach (Clorox bleach, Clorox co., Cairo, Egypt) following CDC (Centers for Disease Control and Prevention, Atlanta, Georgia, USA) recommendations. Teeth were debrided and cleaned with an ultrasonic scaler (UDS-K, Guilin Woodpecker, China) at low power and under abundant water cooling to eliminate any calculus and soft tissue remains without the creation of micro-cracks. Then teeth were polished using a polishing paste (Zx-pro medium, Dan Dent Co, USA) and flat white bristle brushes (Barista proph brushes, AZDENT, China) mounted in low speed, latch-type contra-angle handpiece (BB-EC, NSK-Nakanishi International, Japan). To avoid dehydration, teeth were kept in distilled water at room temperature during the entire research time. To prevent bacterial growth, water was changed once a week.²⁶

To accurately centralize the teeth in the acrylic resin blocks, a specially-constructed centralizing metal device was employed.²⁷ The self-cured acrylic resin (acrostone cold cure, Egypt) was used. The central fossa of the occlusal surface of each tooth was fixed to the vertical metallic rod (upper moving arm) with sticky wax (Relief wax, ORTHO Organizers, Germany) so that the long axis of the tooth was parallel to the vertical rod. Sticky wax will fracture rather than deform if any movement of tooth occurs during fixation. One gram powder and 0.5 mL liquid were mixed until

reach the dough stage and then applied inside the splitted teflon ring which assembled in the copper ring. The vertical metallic rod was lowered with tooth fixed to it until the tooth was embedded in the self-cured acrylic resin, so that, 2mm distance between acrylic resin border and cemento-enamel junction was obtained.

Acrylic block -with the tooth fixed inside it- was fixed in the low speed diamond saw machine (Isomet 1000; Buehler, Lake Bluff, IL, USA), so that the disc was perpendicular to the axial surface of the tooth and the distance between the disc and cusp tip was 2.5 mm. Cutting was done under water cooling. After exposure of the dentin surface, the occlusal surfaces of all teeth were polished with 600 # grit silicon carbide paper (Egyptian Abrasives co., Cairo, Egypt), under water cooling for 30 s.²⁸

Design of cuboid rectangle with dimensions of 8 mm length × 8 mm width × 3 mm thickness was done on Exocad software (exocad GmbH, Align Technology, USA). Milling of wax blank to obtain wax cuboid rectangles of required dimensions was done. The weight of wax-up which was sufficient for one small ceramic ingot was 0.7 g or less according to manufacturer instructions. Thus, three wax rectangles were attached to ring base of 200 g investing ring using wax sprue formers. The difference between the weight of the empty and the loaded ring base was the definitive wax weight (0.67g) which was sufficient for one small ceramic ingot. Mixing of the investment material (Bellavest SH investment material) was done according to manufacturer instructions. After investing, the investing rings were randomly divided into three groups for three ceramic materials used.

Each ring was placed in the burn out furnace. After burn out of wax, pressing of ceramic material using the pressing furnace (Multimate cube press furnace, Dentsply Sirona, Germany) was performed according the manufacturer instructions for each material. After pressing, the investing ring was removed from the pressing furnace and allowed to bench cool. Rough divesting was performed with sandblasting device (Basic classic fine sandblasting unit, Remfert,



Germany) using 50 µm AL₂O₃ particles at pressure of 4 bar. Fine divesting and removal of reaction layer was done with sandblasting using 50 µm AL₂O₃ particles at pressure of 2 bar according to the manufacturer instructions. The sprue was cut using low speed thin diamond disk (#34789520, Visio.lign Toolkit, Bredent, Germany). Finishing and polishing of each surface of each block was done using special finishing and polishing tools

(DIAPRO HP- Set HP 360, EVE, GmbH, Germany), mounted on low-speed straight handpiece. The ceramic blocks were then ultrasonically cleaned in distilled water for 5 minutes in an ultrasonic cleaner (XH-E412, Baku3550, China). Each ceramic rectangle was randomly separated into three subgroups based on the surface treatment applied. **Figure (1)**

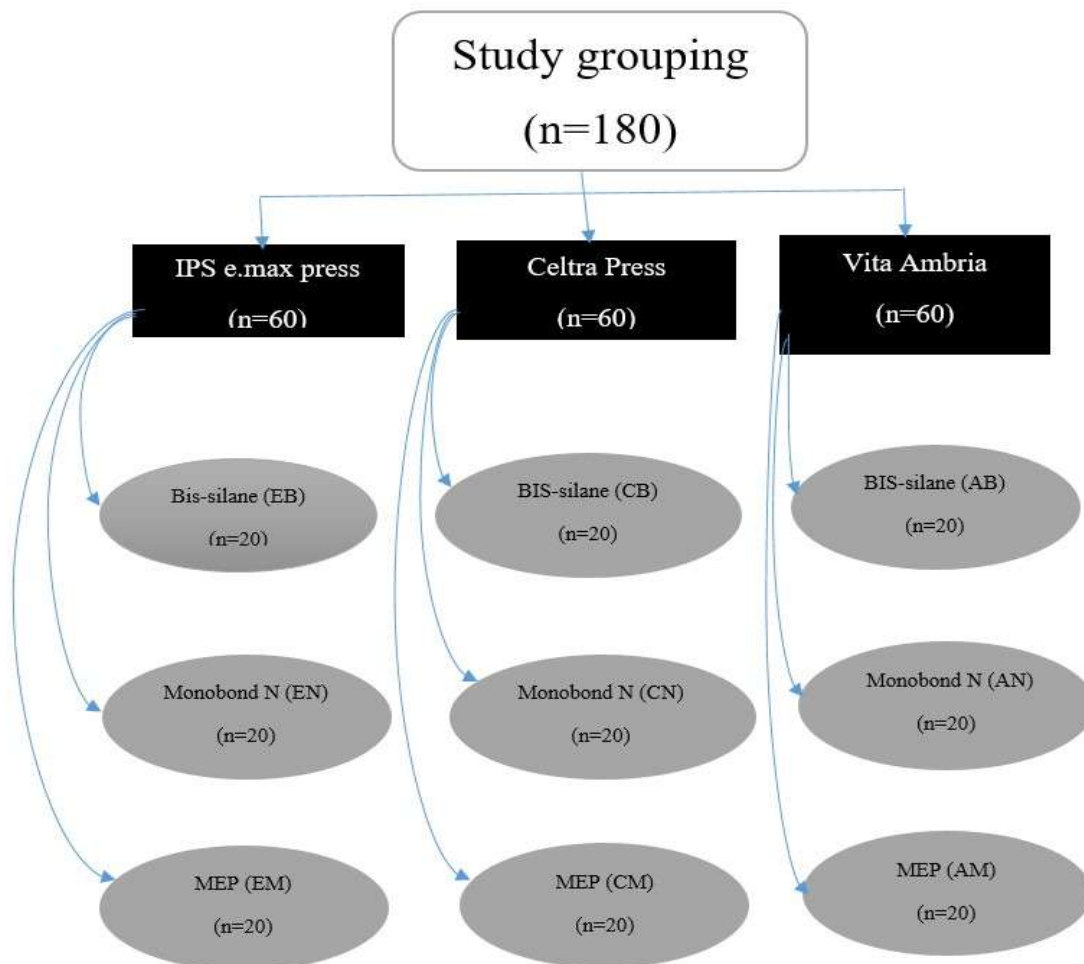


Figure no 1: study grouping

Dentine bonding agent (Tetric N-Bond Universal) was applied on dentine surface and agitated for 20 sec. The adhesive was dispersed with oil free compressed air until a glossy, firm layer of adhesive resulted. Then light curing for 10 seconds was done according to manufacturer instructions using a LED light curing unit (BlueLEX LD-106, Monitex, china) with light intensity: 1000 mW/cm².

Each ceramic block was fixed in putty consistency rubber base material (silaxil, Lascod, Italy), after mixing with its catalyst (Enersyl,

Lascod, Italy), to facilitate ceramic surface treatment. For Bis-silane group, HF was applied on the ceramic surface of each material using micro-brush and the surface was etched according to manufacturer instructions. For e.max press, the material was etched with 5% HF for 20 seconds. For Celtra Press, the material was etched with 5% HF for 30 seconds. For Vita Ambria, the material was etched with 5% HF for 20 seconds. Each ceramic rectangle was washed using air water spray for 30 seconds, then air dried. Silane coupling agent (Bis-silane) was applied for 30 seconds using



micro-brush then air dried according to manufacturer instructions. For Monobond N group, ceramic rectangles of each material were etched as in Bis-silane group, then, silane coupling agent (Monobond N) was applied for 60 seconds using micro-brush then air dried according to the manufacturer instructions. For MEP group, ceramic specimens were not etched using HF unlike the Bis-silane and Monobond N groups. Monobond Etch & Prime was applied on the ceramic surface and agitated for 20 seconds using micro-brush, left passive for 40 seconds then, water rinsed and finally air dried according to the manufacturer instructions.

The ceramic specimens were bonded to dentine surface using a specially-designed cementation device with static loading of 1 kg for 5 min to ensure formation of uniform cement layer thickness.²⁹ To avoid sliding of the ceramic rectangle on the dentine surface during load application, the button which applied the load on the ceramic rectangle had a cavity with the same dimensions of the ceramic specimen but less depth. The cement was initially light-cured from all surfaces for 3 sec at right angle using the LED light curing unit. The excess was gently removed by disposal micro-brush. The bonded specimen was light-cured from all directions for 10 seconds for each side according to the manufacturer's instructions.

Each specimen was fixed from its acrylic resin block in the metallic part of the cutting machine (Isomet 4000, shelter Ltd, Lake Bluff, IL, USA). The cutting process was done in two stages with 0.35 mm thickness diamond blade (20LC, 11e4225, wafering blade, Buheler, USA) with constant water cooling. After the cutting process, the external microbars of the tested specimens were demarcated to exclude them to avoid the possibility of microcracks or enamel bonding. The microbars

from the center of the specimen were used in the testing process. The dimension of each 1mm² microbar was checked by the digital caliper (ISZ-1108-300, Insize, Japan). The microbars were examined with stereomicroscope (MA 100 Nikon stereomicroscope Japan) at 50 X magnification to select 20 intact microbars without any microcracks for each subgroup. The selected microbars were stored in distilled water at 37 °C for 24 hours before thermocycling process.

The microbars were subjected to 5000 thermocycles in water baths (SD Mechatronics Thermocycler, Westerham, Germany) in temperature range from 5 °C and 55 °C with dwell time of 30 seconds in each water bath and transfer time of 5 seconds, simulating 6 months clinical physiological aging.²³⁻³⁰ Microtensile bond strength test was performed for each micro-bar. The stereomicroscope was used to examine the microbars at 50 X magnification after microtensile bond strength testing and failure to record the mode of failure which maybe adhesive, cohesive or mixed. Scanning Electron Microscope (Jeol, JSM-6510LV, Japan) was used to evaluate the three modes of failure after microtensile test at 75 X, 250 X, and 500 X magnifications.

All data were gathered, tabulated, and statistically analyzed with SPSS TM software (Version 23, IBM, USA). After confirming normality using the Shapiro-Wilk test, the descriptive statistics for quantitative data were computed in the form of mean standard deviation. The number and percentage were used to convey qualitative data. The significance of difference was determined using the one-way ANOVA test, two-way ANOVA test, and Bonferroni's post hoc test in the analytical statistics. The threshold of statistical significance for the obtained results was fixed at 0.05.

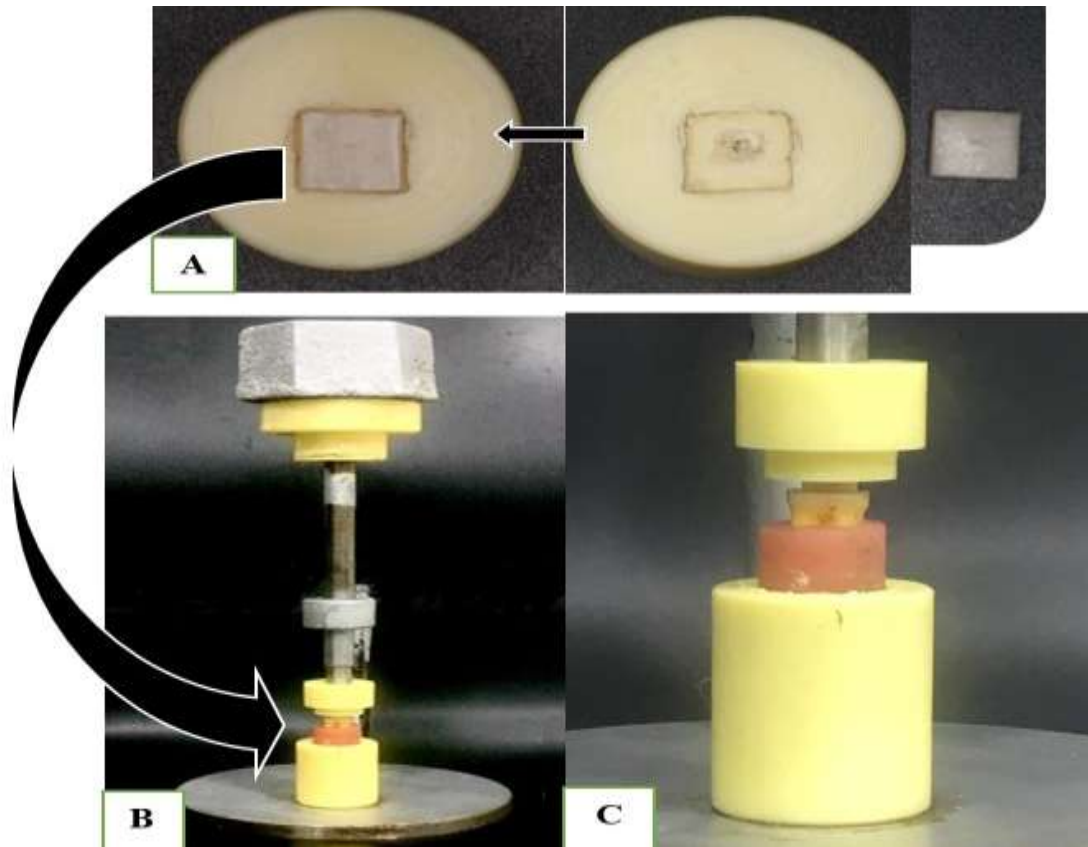


Figure no 2: A) Adaptation of the ceramic specimen on the button to avoid slippage during load application, B) The cementation device, C) Close up view for cementation with the ceramic rectangle cemented on dentine surface

III. RESULT

A. One-way ANOVA test

One-way ANOVA test (Table 2) was used to assess the effect of the type of ceramic material and the different surface treatment on μ TBS. There was significant difference in bond strength regarding Bis-silane (between CB and EB) ($P=0.01$). There was no significant difference in bond strength regarding Monobond N ($P=0.14$). There was no significant difference in bond strength regarding Monobond Etch &Prime ($P=0.38$). There was significant difference in bond

strength regarding e.max ($P< 0.001$). E1 had significantly higher bond strength than E3 ($P<0.001$). E2 had significantly higher bond strength than E3 ($P=0.03$). There was a significant difference in bond strength regarding Celtra ($P=0.002$). CN had significantly higher bond strength than CE ($P=0.002$). There was a significant difference in bond strength regarding Ambria ($P<0.001$). AB had significantly higher bond strength than AM ($P=0.01$). AB had significantly higher bond strength than AM ($P<0.001$).

Table no 2: One-way ANOVA test

		Materials			ANOVA
		IPS e.max Press	Celtra Press	Vita Ambria	P value
Surface treatments	Bis-silane	13.27±3.15 ^{ba}	10.41±3.00 ^{aAB}	12.39±2.84 ^{abA}	0.01*
	Monobond N	11.41±3.33 ^{aA}	12.07±2.91 ^{aA}	13.38±3.17 ^{aA}	0.14
	MEP	8.89±2.7	8.98±1.94	9.80±2.07	0.38



		7 ^{aB}	a ^B	a ^B	
ANOVA	P value	<0.001*	0.002*	<0.001*	

Data expressed as mean&SD
P: Probability *: significance ≤0.05
Different superscript lowercase letters show statistically significant differences between ceramic materials in the same line.
In the same line comparing different ceramic materials, similar superscript lowercase letters show no statistically significant difference.
Different superscript capital letters show statistically significant differences between surface treatments in the same column.
Similar superscript capital letters imply that there is no statistically significant difference between various surface treatments in the same column.

B. Two-way ANOVA test

Two-way ANOVA test (Table 3) was used to assess the effect of different surface treatments and glass ceramic materials on μTBS. It showed that surface treatment had a significant effect on μTBS to resin cement (P value <0.001),

also the type of ceramic material used has significant effect on μTBS (P value = 0.032). The interaction between the surface treatment and the type of ceramic material had significant effect on μTBS (P value = 0.046).

Table no 3: Two way ANOVA test

	Type III Sum of Squares	df	Mean Square	F	P value
Corrected Model	481.644 ^a	8	60.206	7.492	<0.001*
Intercept	22484.068	1	22484.068	2797.832	<0.001*
Ceramic materials	56.272	2	28.136	3.501	0.032*
Surface treatments	345.640	2	172.820	21.505	<0.001*
Ceramic materials * Surface treatments	79.733	4	19.933	2.480	0.046*
Error	1374.198	171	8.036		
Total	24339.911	180			
Corrected Total	1855.843	179			

P: Probability *: significance ≤0.05

C. Bonferroni's Post-hoc test

The Bonferroni's post-hoc test is a multiple comparisons stepwise process used to detect sample means that differ significantly from one another. When an analysis of variance (ANOVA) reveals a significant difference between three or more sample means, it is employed as a post hoc test. Bonferroni's Post-hoc for the effect

of glass ceramic materials on bond strength showed in (Table 4). It showed that Vita Ambria has a significantly higher bond strength than Celtra Press. Bonferroni's Post-hoc for the effect of surface treatments on bond strength showed in (Table 5). It showed that MEP has significantly lower bond strength than Bis silane and Monobond N.

Table no 4: Bonferroni's Post-hoc for the effect of glass ceramic materials on bond strength

	Total E max	Total Celtra	Total Ambria	P value
Mean±SD	11.19±3.54 ^{ab}	10.49±2.91 ^a	11.86±3.09 ^b	0.032*
Post-hoc	P1=0.027*	P2=0.6	P3=0.53	

Data expressed as mean &SD

P: Probability, *: significance ≤ 0.05

Different superscript lowercase letters indicate statistically significant difference in the same line comparing different ceramic materials.

Similar superscript lowercase letters indicate no statistically significant difference in the same line comparing different ceramic materials.

Table no 5: Bonferroni's Post-hoc for the effect of surface treatments on bond strength

	Total Bis-silane	Total Monobond N	Total MEP	P value
Mean \pm SD	12.02 \pm 3.19 ^a	12.28 \pm 3.20 ^a	9.22 \pm 2.29 ^b	<0.001*
Post-hoc	P1=<0.001*	P2=1.00	P3=<0.001*	

Data expressed as mean&SD,P: Probability, *: significance ≤ 0.05

Different superscript lowercase letters show statistically significant differences between surface treatments in the same line.

Similar superscript lowercase letters imply that there is no statistically significant change in the same line when different surface treatments are compared.

D. Failure analysis

1) Stereomicroscopic examination of modes of failure:

The stereomicroscope was used to examine the microbars after microtensile bond strength testing and fracture to record the mode of

failure which was adhesive failure between resin cement and glass ceramic or between resin cement and dentin, cohesive failure within resin cement or within glass ceramic or mixed failure which was combination between adhesive and cohesive failures(**Figure 4:A-E**).

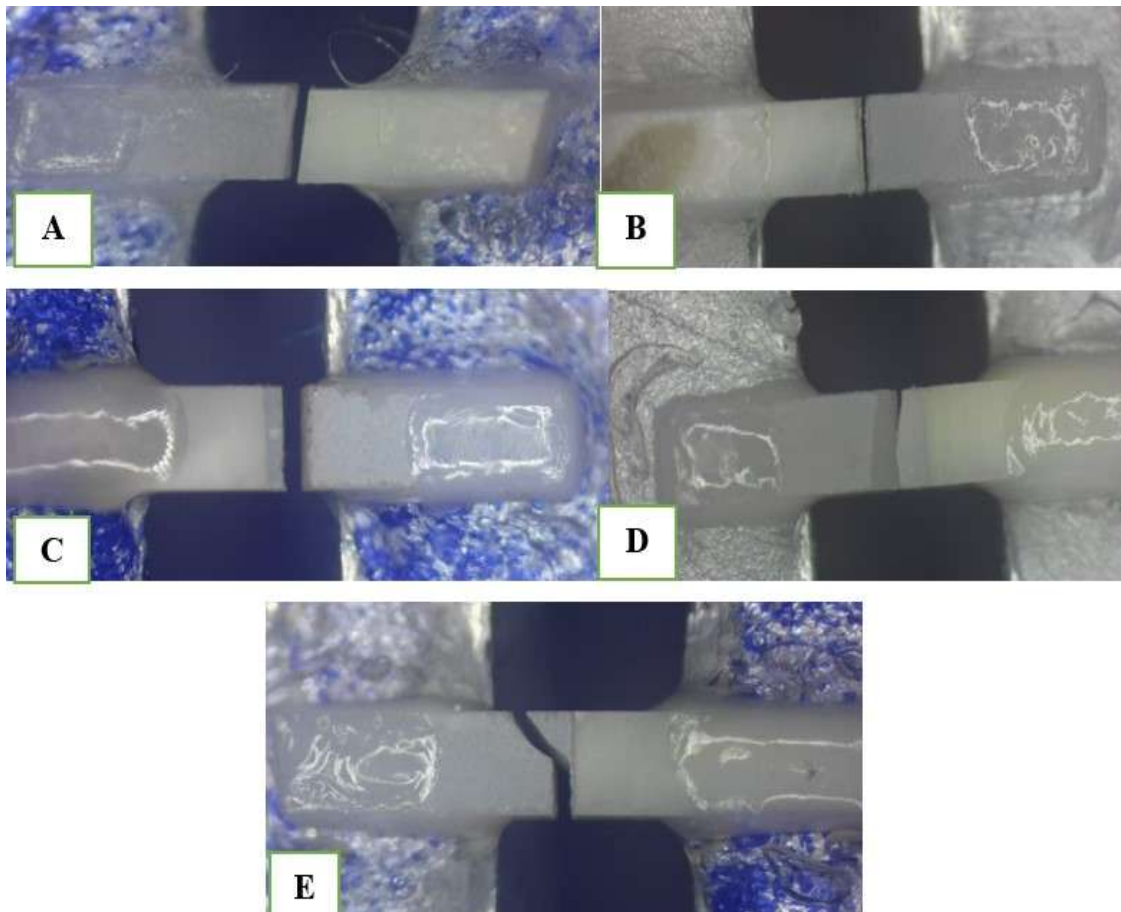


Figure 4: Stereomicroscopic examination of modes of failure

- A) Adhesive failure between resin cement and dentine
- B) Cohesive failure within resin cement
- C) Adhesive failure between resin cement and glass ceramic
- D) cohesive failure within glass ceramic
- E) mixed failure

2) Scanning electron microscopic examination of fractured surfaces

Threemodes of failures after μ TBS were shown under scanningelectron microscope at magnification x 500 (**Figures 5**).

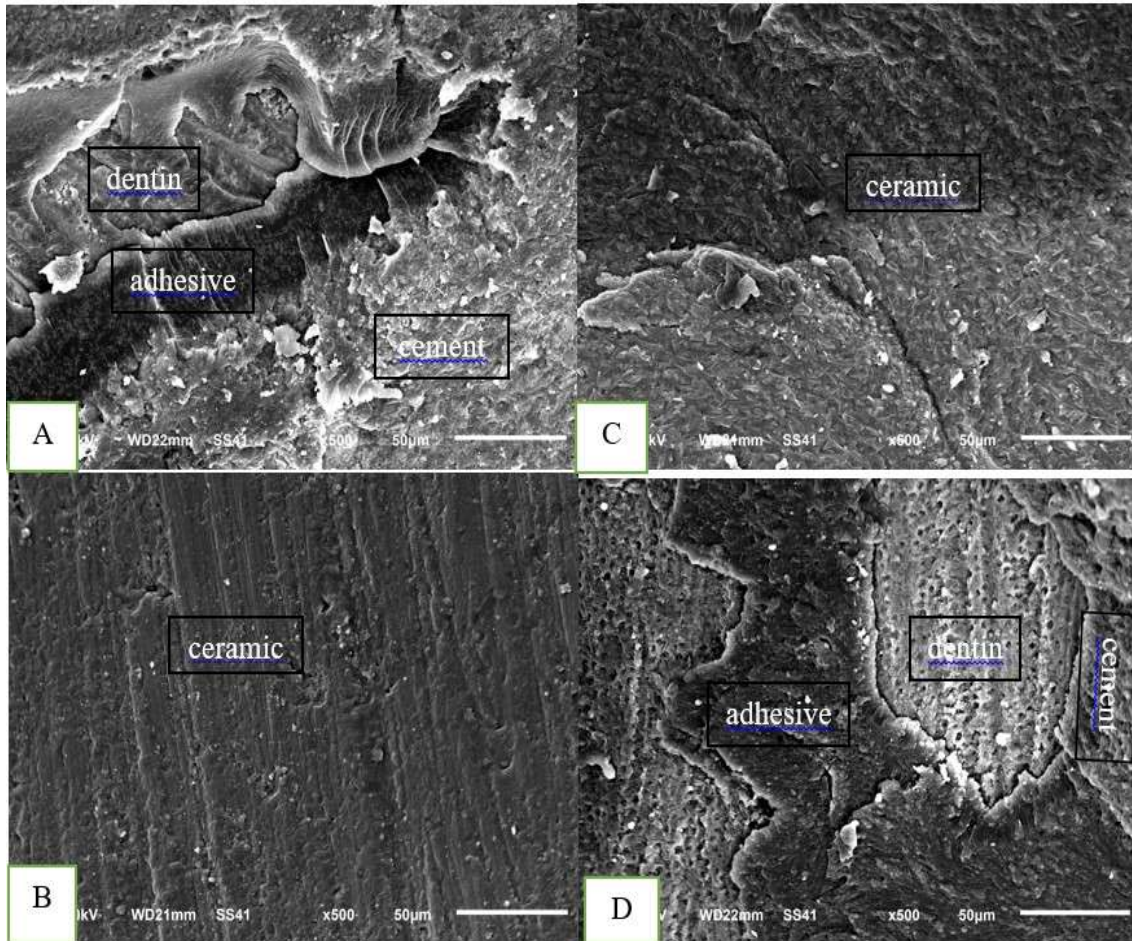


Figure 5: Scanning electron microscopic examination of fractured surfaces

- A) mixed failure
- B) Adhesive failure between resin cement and ceramic
- C) cohesive failure within ceramic material
- D) adhesive failure between dentine and ceramic material

3) Statistical analysis

Failure modes between studied groups were statistically analyzed showing that type of material had significant effect on mode of failure while type of surface treatment did not has significant effect on mode of failure (**Tables 7&8**).

Table no 7: comparison of mode of failure in different materials, data are expressed as number (%)

	IPS e.max press	Celtra Press	Vita Ambria	Chi square	Sig
Adhesive	30(50%)	18(30%)	19 (31.7%)	13.3	0.01*
cohesive	7(11.7%)	20(33.3%)	23(38.3%)		
Mixed	23(38.3%)	22(36.7%)	18(30%)		

Type of material has significant effect on mode of failure.

**Table no 8: comparison of mode of failure for different types of surface treatments, data are expressed as number (%)**

	Bis-silane	Monobond N	MEP	Chi square	Sig
Adhesive	22 (36.7%)	16(26.7%)	29(48.3%)	6.6	0.158
Cohesive	17(28.3%)	21(35%)	12(20%)		
Mixed	21(35%)	23(38.3%)	19(31.7%)		
Type of surface treatment does not has significant effect on mode of failure.					

IV. DISCUSSION

Retention of glass ceramic restoration is very important for long term success, so that bonding techniques must be improved. Also, durable and strong bonding improve marginal adaptation, prevent micro-leakage, and increase fracture resistance of tooth and restoration. Strong resin bonding relies on micromechanical interlocking using HF and chemical bonding to ceramic surface using silane coupling agent. But, HF is harmful and technique sensitive so self-etch silane was developed as it is assumed to be less dangerous and technique sensitive than HF.

In this in-Vitro study, human natural upper molars were selected, as they have large dimensions resulting in large dentin bonding surface. Natural teeth were used due to their bonding characteristics, modulus of elasticity, and strength, simulating the clinical conditions better than bovine or acrylic teeth.⁴⁸

For cutting of occlusal enamel in one plane, Isomet was used with the disc perpendicular to the axial surface of the tooth and the distance between the disc and cusp tip was 2.5 mm to ensure complete enamel cutting. Cutting was done under water cooling to avoid heat generation. After exposure of the dentin surface, occlusal surface of all samples were polished with 600 # grit silicon carbide paper, under water cooling for 30 s for production of flat dentin surface with standardized smear layer.

Wax patterns were designed and milled using CAD/CAM as it is easier, more accurate, less time consuming, more standardized than manual wax-up. Design with the dimensions of 8 mm length × 8 mm width × 3 mm thickness was done on software. These dimensions were selected as the average width and length of exposed dentine surfaces was 8 mm and 8 mm respectively. Also, this thickness was selected as it is appropriate for μ TBS, reduces stresses during cutting the specimen into microbars, and allows for light curing of dual curing cement material during cementation.

Etching of dentin using orthophosphoric acid was not recommended, as it is time consuming and leads to collapse of dentinal tubules and

collagen fibrils interfering with adhesive resin infiltration and hybrid layer formation. Dentin etching causes demineralization for several micrometers in depth, which is not entirely hybridized by the adhesive solution, leaving exposed collagen fibrils in the deepest places. Endogenous proteases, such as matrix metalloproteinase enzymes, can degrade these exposed collagen fibrils. Etching of dentin activate matrix metalloproteinase enzymes which cause degradation of unsupported collagen fibrils leading to adhesive failure over time.³¹ Active application of universal adhesive using a scrubbing technique accelerated evaporation of solvent and led to better penetration of monomers inside smear layer, thus improving bond strength of adhesive to dentin.

Variolink Esthetic DC resin cement was a dual cured resin cement which was used instead of light cured resin cement as the thickness of ceramic material was 3 mm through which light might not reach all parts of the cement. μ TBS test is one of the most common methods in testing adhesion between different materials. Microtensile bond strength test has several advantages over other bond strength tests. This test is economic as small number of teeth, small amount of ceramic and cementing materials can be used to obtain several micro-specimens. Better and uniform stress distribution at the real interface when compared to SBS. SBS has extremely non-uniform stress distribution concentrated in the substrate. Bond strengths are greater than those measured by traditional tensile and SBS tests because of a lower amount of imperfections in the substrate or at the bond interface. Because of the small diffusional distances, accelerated environmental aging can be obtained by thermal cycling or water storage. However, the microtensile test necessitates further sample processing following the bonding procedure, making the test more challenging and technology dependent.³²

The first null hypothesis in the present study was rejected as self-etch silane provided lower bond strength than that of conventional silane. The second null hypothesis was also



rejected since the bond strength of the various glass ceramics employed varied.

Regarding the ceramic material used, with the conventional silane, group EB showed the highest bond strength value (13.27 ± 3.15 MPa), while group CB showed the lowest one (10.41 ± 3.00 MPa). There was statistically significant difference between EB and CB groups and this may be due to the different composition between the two materials used as lithium disilicate glass ceramics may be better etched than zirconia reinforced lithium silicate glass ceramics. Also, this may be due to the difference in etching time between the two materials, as the increased etching time for celtra press (30 sec.) may lead to over etching of the ceramic material than the 20 seconds etching of IPS e.max press leading to decreased bond strength. Also, zirconia particles in Celtra are more acid resistant decreasing its etching ability than lithium disilicate glass ceramics. However, these results disagreed with **Al-Thagafi et al (2016)**⁴⁹ who found that zirconia reinforced lithium silicate ceramic (Vita suprinity) (27.1 ± 1.4 MPa) had significantly higher μ TBS than lithium disilicate ceramic (IPS e.max CAD) (22.4 ± 5.7 MPa) using conventional silane (silane without MDP). This may be due to this study used milled rather than pressed ceramics and prolonged HF exposure for 60 S.

There was no significant difference between CB group and AB group, with AB group slightly higher bond strength than CB. Although they have the same content of zirconia (about 10%), the composition was different. So, lithium disilicate may be better etched than lithium silicate. Also, the difference in etching time between two materials (20 sec. for Ambria versus 30 sec. for celtra) may affect the bond strength as previously mentioned.

There was no significant difference between EB group and AB group, with EB group providing slightly higher bond strength than AB group. This may be due to zirconia content of Ambria which is more resistant to etching.

Regarding the ceramic material used, with Monobond N, group AN showed the highest bond strength value (13.38 ± 3.17 MPa), while group EN showed the lowest one (11.41 ± 3.33 MPa). There was no significant difference between EN and CN groups, there was no significant difference between EN and AN groups, and there was no significant difference between CN and AN groups. While CN & AN groups have slightly higher bond strength than EN group. This might be attributed to the zirconia component in Ambria and Celtra (about 10%) which react with MDP in the universal

primer forming stable chemical bonds which might eliminate the negative effect of aging and improve the adhesive durability of resin cement to ceramic. Hydroxyl group of MDP monomer could form stable chemical bond with hydroxyl group of zirconia and resist hydrolysis degradation. Furthermore, decyl group in MDP prevented water penetration at the interface between dihydrogen phosphate and zirconia. These results of our study agreed with **Aboushelib and Sleem(2014)**⁵⁰ who found that microtensile bond strength of celtra ceramic was higher than IPS e.max ceramic using HF acid and Monobond Plus. Although, these results disagreed with **martins et al (2022)**⁵¹ who found that lithium disilicate glass ceramic provided better bond strength (18.66 ± 3.49 MPa) than zirconia reinforced lithium silicate glass ceramic (16.81 ± 2.62 MPa) using Monobond Plus. This may be due to use of milled ceramics rather than pressed ceramics.

Regarding the ceramic material used, with self-etch silane, AM group showed the highest bond strength value (9.80 ± 2.07 MPa), while EM group showed the lowest one (8.89 ± 2.77 MPa). There was no significant difference between EM and CM groups, there was no significant difference between EM and AM groups, and there was no significant difference between CM and AM groups. While CM & AM groups have slightly higher bond strength than EM group which may be due to the MDP component of MEP that react with zirconia in the zirconia-containing glass ceramics. These results agreed with **Donmez et al(2020)**⁵² who found that self-etch silane provided better bond strength for zirconia reinforced lithium silicate ceramics (Vita Suprinity) than for lithium disilicate glass ceramics (IPS e.max CAD), although ceramics used were milled not pressed ceramics.

Regarding the surface treatment used, with IPS e.max ceramic, EB group showed the highest bond strength value (13.27 ± 3.15 MPa), while EM group showed the lowest one (8.89 ± 2.77 MPa). There was statistically significant difference between EB and EM groups and there was statistically significant difference between EN and EM groups. This might be due to lower etching ability of tetrabutylammonium dihydrogen trifluoride component of MEP due to milder acidity compared to HF precluding a durable micromechanical bonding.³⁷ Reduced wettability of ceramic surface following surface treatment with self-etching silane suggests that the subsequent microstructural changes may have resulted in the creation of a layer comprising debris, making the surface less wettable. Furthermore, the presence of fluoride in self-etch silane material reduces the



substrate's wettability. Furthermore, the existence of F ions residue can be attributed to the material's interaction with the glassy component, which produces insoluble silica-fluoride salts that persist as residue or deposit on the surface.⁵³ Tetrabutyl ammonium dihydrogen trifluoride component in self-etch silane may reduce the efficacy of silane.³⁸ Chemical adhesion may be impaired while utilizing this silane since the technique for this new product requires silane water rinse after application. Also, acidic pH of MEP might result in activation and self-condensation of silane reducing its effect in chemical bonding with glass ceramic. These results agreed with **swank et al (2018)**³⁸ who found that bond strength using Bis-silane was significantly higher than that using Monobond Etch and Prime and bond strength using Monobond Plus was significantly higher than that using Monobond Etch and Prime. Other previous studies found that bond strength using Monobond Plus was significantly higher than that using Monobond Etch and Prime.^{24, 30, 40, 41}

These results disagreed with **Wille et al(2017)**⁴² who found that TBS to lithium disilicate glass ceramic (IPS e.max CAD) provided by self-etching silane (MEP) was comparable to conventional HF etching and silane priming (Monobond Plus). Also, these results disagreed with previous studies.^{5, 43-46} This was maybe however self-etching silane can't dissolve the glassy phase as profound as HF but silane will spread more because of active application. Use of HF resulted in significantly rougher surface. However, this morphological difference changed only the wettability and did not affect the bond strength. Acidic pH of methacrylate phosphate monomer was buffered using butanol and 1, 3 butanediol solvents in order to preserve silanol reactivity and decrease self-condensation during storage.³⁰ Also, incomplete removal of solvent and reaction by-products after application of conventional silane might affect bond strength with glass ceramic. The time recommended by the manufacturer for air blowing might be insufficient to eliminate these products from the silane layer.

Regarding the surface treatment used, with Celtra Press ceramic, CN group showed the highest bond strength value (12.07 ± 2.91 MPa), while CM group showed the lowest one (8.98 ± 1.94 MPa). There was statistically significant difference between CN and CM groups. This may be due to lower etching ability of tetrabutylammonium dihydrogen trifluoride component of MEP as previously mentioned. These results agreed with **Martins et al (2022)**⁵¹ who found that bond strength of celtra ceramic was significantly higher

using Monobond Plus (16.81 ± 2.62 MPa) than using self-etch silane (14.12 ± 3.51 MPa). There was no significant difference between CB and CM groups with CB group showing higher bond strength than CM group. There was no significant difference between CB and CN groups with CN group showing higher bond strength than CB group. This may be because zirconia component in Celtra (about 10%) react with MDP in universal primer as previously mentioned.

Regarding the surface treatment used, with Vita Ambria ceramic, AN group showed the highest bond strength value (13.38 ± 3.17 MPa), while AM group showed the lowest one (9.8 ± 2.07 MPa). There was statistically significant difference between AN and AM groups. This may be due to lower etching ability of tetrabutylammonium dihydrogen trifluoride component of MEP as previously mentioned. Also, zirconia component in Ambria (about 10%) react with MDP in universal primer as previously mentioned. There was statistically significant difference between AN and AM groups. There was statistically significant difference between AB and AM groups. This may be due to lower etching ability of tetrabutylammonium dihydrogen trifluoride component of MEP as previously mentioned. There was no significant difference between AB and AN groups with AN group showing higher bond strength than AB group. This may be due to zirconia component in Ambria (about 10%) reacting with MDP in universal primer as previously mentioned. There are no previous studies investigating the bond strength to Vita Ambria.

In our study, values of bond strength results were less than that of previous studies. This might be that previous studies performed bond strength test between composite or resin cement and ceramic material which was usually more than bond strength between dentin and ceramic. Achieving effective dentin bonding was much more challenging. This might be due to structure of dentine. Dentine had hybrid composition as it was composed of about 70% inorganic content and 30% organic content. Also, moisture of dentin compared to enamel made dentin bonding more difficult.³¹

Limitations of this study, Laboratory research design, like other in-vitro investigations, cannot perfectly replicate clinical situations. Lack of comparison with non-thermocycled group because the aim was to test aged interfaces only. The role of hydrolysis in different types of silane treatments using different ceramics was not investigated. The lithium disilicate samples utilized



were microbars rather than crowns. Bonding to dentin was variable due to different number and diameter of dentinal tubules. Aging was only 5000 thermocycling not more and there was no water storage.

V. CONCLUSION

Within the limitations of this in-vitro study, it was concluded that;

- 1) Bond strength after HF acid etching was affected by the chemical composition of glass ceramic material.
- 2) MDP component contained in silane solution increased the bond strength with glass ceramics containing zirconia
- 3) MEP resulted in the lowest bond strength for all glass ceramics used.

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