

## Effect Of Adhesive Application Protocols On Bond Strength Of Ceramic Veneer

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### **Abstract:**

**Aim:** The aim of this study is to evaluate Shear Bond Strength of lithium disilicate ceramic veneers using three adhesive application protocols (pre-curing, co-curing and no adhesive application) using two resin cements (light cure total etch and dual cure self-etch).

**Material and Methods:** By using CAD/CAM milling machine, 42 ceramic disc samples of dimensions 4 X 4 mm in diameter and 0.5mm in thickness from Lithium disilicate ceramic material (Up-cad blocks). Buccal surface of 42 premolar teeth were flattened and etched then divided into 3 groups first group (P) received bonding agent then curing, second group (C) received bonding agent with no curing, third group (N) did not receive any bonding agent. Then each group is further subdivided into two subgroups according type of cement: light-cured luting cement total etch and a dual-cured self-adhesive resin cement. All specimen underwent thermocycling aging and then shear bond strength test was carried out. A stereo microscope was used to measure mode of failure of each specimen.

**Results:** Regardless of type of cement co-curing application protocol had highest bond strength, followed by No adhesive application protocol, while pre-curing application protocol had the lowest bond strength. Regardless of adhesive application protocol Self-etch dual-cured cement had a higher bond strength than the total-etch light-cured cement. While for mode of failure statistical analysis, there was no significant difference in adhesive application protocol and cement types.

**Conclusions:** To Increase adhesive protocol the Co-curing is better than other methods. With regards to Cement types, self-etch dual cure cement gave a more reliable bond than the total etch light cure cement.

**Keywords:** Bond strength, Ceramic veneer, Adhesive protocol.

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### I. Introduction

Dental ceramics are materials used to producing dental prostheses to restore missing or damage dental structure, ceramics have Lots of properties as esthetic, precision, strength and biocompatible to tooth and gum. This material resembles the appearance, textures and shades of the natural tooth.<sup>(1)</sup> Recently, ceramic materials and CAD/CAM technology are advanced in order to allow the accomplishment of high aesthetic demands and to reduce the shortcoming of conventional materials and methods; i.e., low tensile strength, high hardness so make wear to antagonist, extreme brittleness, sintering shrinkage crack propagation and marginal gaps.<sup>(2)</sup>

The laminate veneer <sup>(3)</sup> is a thin layer of tooth colored like material can be made from porcelain, composite resin or plastic material used to restore defects (localized or generalized) as discoloration cannot treated by bleaching, enamel defect or cracks, close the diastema or multiple spacing, rebuilding chipped or fractured teeth and mal-positioned teeth by applied veneer to tooth surface this technique named laminating. Should be aware cannot be used with parafunctional habits (bruxism or clenching), occlusal problem, Insufficient tooth structure to bonding, severe crowding, bad oral hygiene, high caries index and endo-treated teeth.<sup>(4)</sup>

Can be classified according to application (direct and indirect) veneer: Direct laminate veneer (free hand process): applied directly on prepared tooth surface by dentist no need lab in this technique, more economic, not need more preparation but have some abuse as easily wear, discoloration and fracture due to lower mechanical properties. Indirect laminate veneer depends on dentist and lab so need more visit and cost this type have good resistant to wear, discoloration over time and tendency to fracture.<sup>(5)</sup> After uses the computer and developed dental material has lead up to create (CAD/CAM) technology.

This technology permits scanning, design and milling. CAD/CAM have 2 techniques the first named rapid prototyping is additive method (3D printing) can obtain 3D model by deposited layer by layer and second technique is subtractive method by milling of solid block or blank ceramic.<sup>(6)</sup>

Can be classified according to material used: Dental Veneers divided in three families: dental resin composite, dental ceramic and hybrids as (polymer infiltrated ceramic network, resin nanoceramics, flexible

nanoceramics). Dental ceramic else divided into three subcategories: Feldspathic porcelain & Synthetic glass matrix as (Leucite reinforced glass ceramics, lithium disilicate, zirconia reinforced glass ceramics, fluoroapatite glass ceramics), Glass infiltrated ceramics and polycrystalline ceramics as (Ultra translucent zirconia).<sup>(5)</sup>

The ceramic material divided into two groups: first group silica based (feldspathic porcelains, leucite-reinforced ceramics, lithium disilicate Ceramics) and second group (alumina, zirconia) so, this groups various in strength and protocol of cementation.<sup>(7)</sup> Lithium disilicate material (glass-ceramic) Characterized by that it has good adhesive properties and preservation of tooth structure Because of those characteristics have broadly marketed. Lithium disilicate ceramic is formed by crystalline phase embedded in the glassy matrix. the  $\text{Li}_2\text{Si}_2\text{O}_5$  have high mechanical properties and high fracture toughness. Lithium disilicate appropriate for the production of monolithic restorations or veneered restorations in the anterior and posterior region. The final strength in  $\text{Li}_2\text{Si}_2\text{O}_5$  depend on bonding protocol to tooth surface.<sup>(8)(9)</sup>

The prepared tooth etched by phosphoric acid gel 37% about 15 seconds then washed and air dried with oil-free air water syringe second step apply bonding agent to prepared surface spread by bonding brush ,While the lab should be etched lithium disilicate ceramic veneer by hydrofluoric acid before send to dentist clinic if the lab does not make this step the dentist must have etched intaglio surface of veneer by HF acid 5% for 20 second then washed and air dried with oil-free air water syringe then apply silane coupling agent for 60 seconds then drying then apply of resin cement.<sup>(10)</sup>

The curing of the cement below restoration done by light cure device through tack and wave technique ,after apply cement and seated of restoration on prepared tooth use small diameter light guide then cured for 1 second on the center after that use large diameter light guide in waved motion around 1 inch from the restoration for additional 3 second to allow the cement convert to semi gel phase (initial seating ) in this step can be remove of excess cement easily after that curing for 20 second for final setting.<sup>(11)(12)</sup>

It is still challenging to attach inorganic restoration to organic teeth tissues, this is why adhesive bonding is necessary for ceramic restorations and can enhance their long term effectiveness in the clinical. Additionally, it can reduce micro leaks, ensure marginal stability, and increase fracture strength of the tooth's and indirect restoration. Resin cements have confirmed to be effective over time and have become popular because of their capacity to attach to both dental hard tissues and indirect restorations<sup>(13)</sup>. Additionally, adhesive cements have a mechanical retention and aesthetics that are superior. Today, resin luting agents can be categorized by how their adhesive systems operate, into total-etch, self-etch, and self-adhesive agents. or according to polymerization reactions into light-curing, chemical-curing, and dual-curing<sup>(14)</sup> Light-curing resin cements need a special light beam to initiate their chemical-curing reactions. Light-curing resin cements have a special light source that must be activated in order to initiate the chemical reaction that results in the curing of the material. These cements are mainly used for restorations in the esthetic region. polymerization reaction affected by ceramic thickness, opacity of the restoration and proper use for light cure.<sup>(15)</sup> The Dual-curing systems, have chemical initiators so can be start polymerization with or without the light source, However, can be attained a stronger bond with additional light curing, which leads to a better adaptation to the marginal area. <sup>(16)</sup>The null hypothesis Neither adhesive application protocol nor type of resin cement has an effect on bond strength of laminate veneers.

## **II. Materials And Methods**

**Study design** The study was carried out as an in vitro study.

Exemption from the ethical committee Ain Shams University with the following number: Fpr21-( 150) – M

### **Power analysis**

A total number of 42 individuals will be randomly assigned 6 subgroups (7 cases per group). The effect size (0.6) was calculated according to Zhu et al. (2022) using sheer bond strength as a key parameter. The sample size was measured using  $\alpha=0.05$  with an actual power of 81%.

### **Materials:**

- Lithium silicate ceramic (Upcera UP-CAD).
- Self-Adhesive dual cured resin cement (Bisco –BisCem).
- Light cure veneer material resin cement (Choice <sup>TM2</sup> Veneer Cement).
- 9.5% buffered hydrofluoric acid gel. (Bisco Porcelain Primer).
- Pre Hydrolyzed porcelain silane (Bisco Silane primer).
- Phosphoric acid etching gel (Chem teeth etchant).
- Light cured dental adhesive (Bisco All Bond Universal).

### **Specimens' fabrication:**

Teeth specimens 42 premolars will be selected and examined for absence of cracks and defects. Buccal surface of each specimen will be flattened by round end taper stone to provide enamel area of 4mm x 4mm.Blocks

of the CAD CAM glass ceramic materials (Up-cad block) were cut into 42 ceramic slices measuring 4 x 4 mm and 0.5 mm thick using (Isomet 4000, Buehler, USA) that was lubricated with water and used a diamond disk with a thickness of 0.6 mm at a cutting speed of 2500 rpm. Following that, sintering firing was carried out in a furnace (Programat EP3010 furnace) in accordance with the manufacturer's instructions. specimens were cleaned using ultrasonic cleaner with Isopropanol 99% for the max CAD then specimens were ready for the bonding process.

**Sample grouping:**

Samples will be randomly divided into three groups according to the light activation of adhesive into:

Group (P): pre-curing application of adhesive N = 14

Group (C): co-curing application of adhesive N = 14

Group (N): no adhesive application N = 14

Then each group will be subdivided into two subgroups according to types of cements:

(L) light cure total etch resin cement N = 7

(D) dual cure self-etch resin cement N = 7

**Surface treatments:**

Before applying any surface treatments, each of the 42 slices had its surface thoroughly cleaned of any dirt and dried with 70% ethyl alcohol. For better standardization, after 20 seconds of etching with 9.5% hydrofluoric acid (porcelain etchant gel), the Up-Cad slices were cleaned and allowed to air dry using an oil-free air/water syringe. For sixty seconds, a silane coupling agent was administered. The specimens were then air dried using an oil-free airway syringe.

Surface treatment of teeth: To ease the handling and fixation during the shear test, all the specimen of premolars was embedded in an acrylic block, before any surface treatments done, 70% ethyl alcohol was used on each of 42 slices for cleaning the surface from any debris, and drying these surfaces very well.

Surface treatment of teeth 42 specimen will have divided into 3 groups:

According to adhesive application protocol (N=14):

Group (P): etching of tooth by phosphoric acid for 15 second, afterwards washed and air-dried with oil-free air/water syringe and application of bonding agent and curing for 20 seconds.

Group (C): etching of tooth by phosphoric acid for 15 second, afterwards washed and air-dried with oil-free air/water syringe and application of bonding agent and no curing.

Group (N): etching of tooth by phosphoric acid, afterwards washed and air-dried with oil-free air/water syringe and no application of bonding.

**Cementation procedures:**

Premolars teeth were etched for 15 seconds with 37% phosphoric acid (chem) and divided into six groups according to the adhesive protocol:

Pre-curing: application of Bisco All Bond Universal by bond brush for 20 seconds then air dried by gentle oil free air for 5 seconds then curing, after curing divided 14 samples into two group first group apply the light cure cement (Bisco CHOICE<sup>TM2</sup>) and second group apply the self-etch dual cure adhesive resin cement (BisCem adhesive resin cement) and ceramic veneer of each tooth then curing. (curing in two step).

Co-curing: application of Bisco All Bond by bond brush for the 20 second then air dried by gentle oil free for 5 second then divided 14 samples into two group first group apply the light cure cement (Bisco CHOICE<sup>TM2</sup>) and second group apply the self-etch dual cure adhesive resin cement (BisCem adhesive resin cement) and ceramic veneer of each tooth then curing together. (curing in one step).

No curing: no application of Bisco All Bond only in this step divided 14 samples into two group first group apply the light cure cement (Bisco CHOICE<sup>TM2</sup>) and second group apply the self-etch dual cure adhesive resin cement (BisCem adhesive resin cement) and ceramic veneer of each tooth then curing.

To ensure uniform load application, a specially designed cementation device was machined from stainless steel. It consists of: a) two horizontal metal plates rectangular in shape (upper and lower); b) two supporting vertical metal arms attached to the upper and lower horizontal metal plates; c) a T-shaped metallic rod attached to the upper metal plates that can move freely vertically; it also carries a disc-shaped plate at its upper end over which the required load will be placed; at its lower end, a Teflon rod with a 10 mm diameter tip was attached; and d) a 1 kg load was placed on the disc-shaped plate.

A 1 Kg constant load was applied on the disk-shaped plate at the upper end of the T-shaped metallic part of the cementation device, and was left for 3 minutes.

The excess cement was then removed with a sharp lancet, the resin material was polymerized from three directions for by a LED light curing unit with a mean light intensity of 2300 mW/cm<sup>2</sup> and <sup>1</sup> optical wavelength of 385-515 nm, for 40 seconds.

**Aging of the samples:**

Before measuring shear bond strength, all of the specimens were subjected to aging factor through a custom-made thermocycling machine where the thermocycling unit contains a hot water path (55°C±1) and a cold water path (5°C±1), the samples where immersed in each path for 30 seconds and dwell time 10 second. 5000 cycle where adjusted using the control board of the machine which is equivalent to 6 months of restorations serving in oral cavity.

**Shear bond strength test:**

Each block with its own bonded specimen was secured horizontally with tightening screws to the lower fixed compartment of a universal testing machine (Model 3345;) Instron Industrial Products, (Instron® Bluehill universal instron England) with a load cell of 500 kN and data were recorded using computer software. As hearing load with compressing mode of force was applied via materials testing machine at a crosshead speed of 1 mm/min. The load required to debonding was recorded in Newton. The load at failure was divided by bonding area to express the bond strength in MPa:  $\tau = P / \pi r^2$ . Where;  $\tau$ = shear bond strength (in MPa), **P**=load at failure (in N),  $\pi$ =3.14 and **r** = radius of micro-cylinder (in mm)

The deboned specimens' failure mode was examined using the Nikon SMZ745T Stereo microscope at 30X magnifications to determine the deboned region and confirm if the mechanism of failure is cohesive, adhesive, or mixed.

**Statistical analysis:**

Categorical data were presented as frequency and percentage values and were analyzed using the chi-square test. Numerical data are presented as mean and standard deviation values. They were checked for normality by viewing the data distribution and using Shapiro-Wilk's test. They were found to be normally distributed and were analyzed using two-way ANOVA. Simple effects were compared using the pooled error term from the main ANOVA model with p-values adjustment using the False Discovery Rate (DFR) method. The significance level was set at p<0.05 within all tests. Statistical analysis was performed with R statistical analysis software version 4.4.1 for windows.

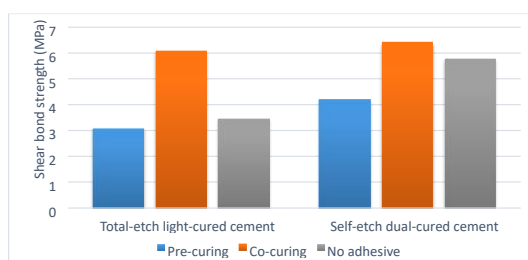
**III. Results**

**Regarding shear bond strength:** There was a significant interaction between both tested variables (p=0.049) in the shear bond strength between different adhesive protocol and cement types.

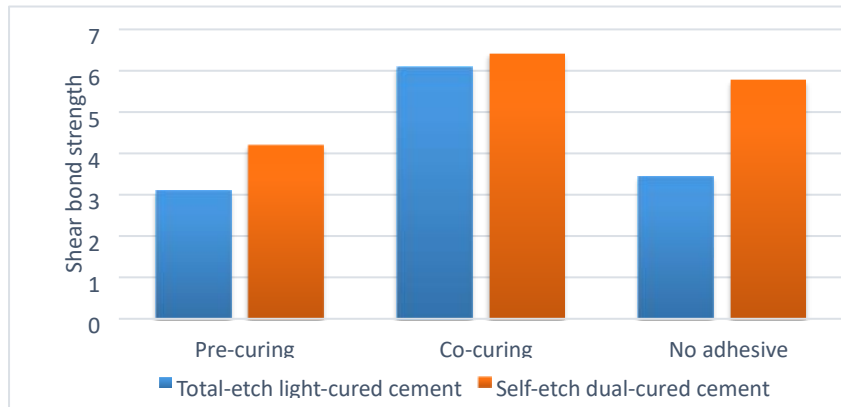
**Table (1):**

Application protocol Cement type	Shear bond strength (MPa)(Mean±SD)			p-value
	Pre-curing	Co-curing	No adhesive	
Total-etch light cured	3.08±0.79 <sup>B</sup>	6.08±1.24 <sup>A</sup>	3.43±0.42 <sup>B</sup>	<0.001*
Self-etch dual cured	4.19±0.68 <sup>B</sup>	6.41±1.43 <sup>A</sup>	5.76±1.30 <sup>A</sup>	0.001*
<b>p-value</b>	0.055ns	0.559ns	0.001*<	

Values with different superscripts within the same horizontal row are significantly different, \* significant (p<0.05), ns not significant.



**Figure (1):** Bar chart showing average shear bond strength (MPa) for different adhesive application protocols and cement types (A).

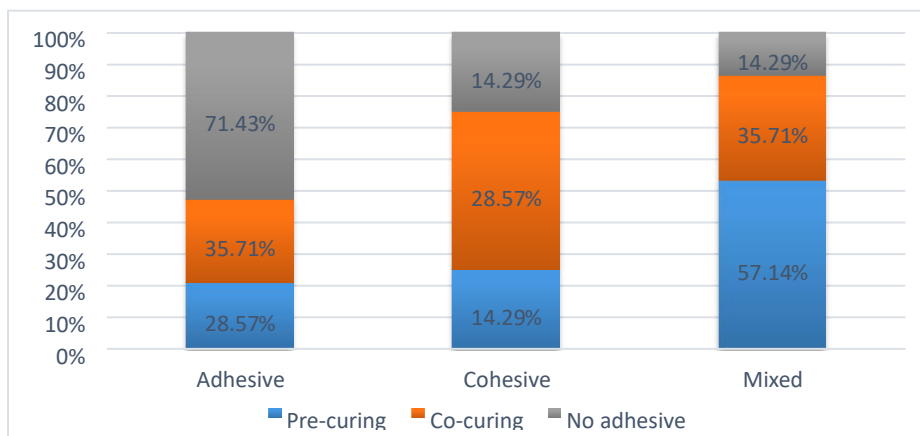


**Figure (2):** Bar chart showing average shear bond strength (MPa) for different adhesive application protocols and cement types (B)

**Regarding mode of failure:** According to effect of adhesive application protocol the majority of samples subjected to pre-curing had mixed failures. Most of the co-cured samples either had adhesive or mixed failures. In contrast, most of the samples with no adhesive had adhesive failures. However, the difference was not statistically significant ( $p=0.097$ ). Comparisons and summary statistics of mode of failure for different adhesive application protocols are presented in **table (2)**:

Failure mode	n (%)			p-value
	Pre-curing	Co-curing	No adhesive	
Adhesive	4 (28.57%)	5 (35.71%)	10 (71.43%)	0.097ns
Cohesive	2 (14.29%)	4 (28.57%)	2 (14.29%)	
Mixed	8 (57.14%)	5 (35.71%)	2 (14.29%)	

ns not significant.



**Figure (3):** Stacked bar chart showing the mode of failure distribution in different adhesive application protocols.

According to effect cement type the most of the samples cemented with the total-etch light-cured cement had adhesive failures, while most self-etch dual-cured samples had mixed failures. However, the difference was not statistically significant ( $p=0.054$ ). Comparisons and summary statistics of mode of failure for samples and cement types are presented in **table (3)**:

Failure mode	n (%)		p-value
	Total-etch light-cured	Self-etch dual-cured	
Adhesive	13 (61.90%)	6 (28.57%)	0.054ns
Cohesive	4 (19.05%)	4 (19.05%)	
Mixed	4 (19.05%)	11 (52.38%)	

ns not significant.

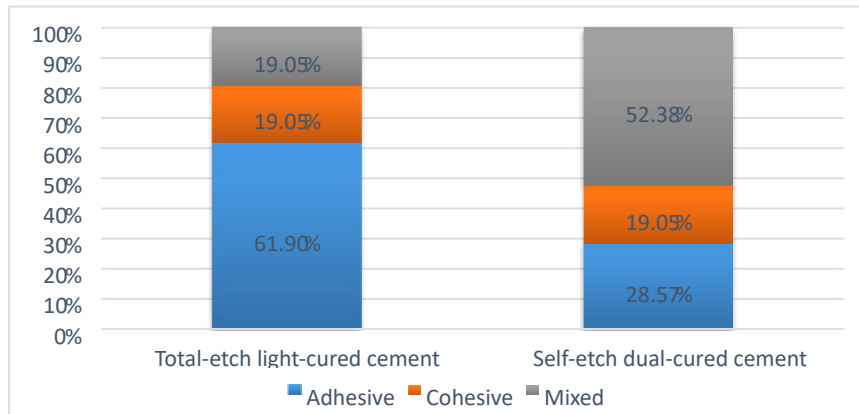


Figure (4): Stacked bar chart showing the mode of failure distribution in samples and cement types.

Comparisons and summary statistics of the mode of failure for different adhesive application protocols and cement types are presented in table (4):

Cement type	Failure mode	n (%)			p-value
		Pre-curing	Co-curing	No adhesive	
Total-etch light-cured	Adhesive	3 (42.86%)	4 (57.14%)	6 (85.71%)	0.279ns
	Cohesive	1 (14.29%)	2 (28.57%)	1 (14.29%)	
	Mixed	3 (42.86%)	1 (14.29%)	0 (0.00%)	
Self-etch dualcured	Adhesive	1 (14.29%)	1 (14.29%)	4 (57.14%)	0.311ns
	Cohesive	1 (14.29%)	2 (28.57%)	1 (14.29%)	
	Mixed	5 (71.43%)	4 (57.14%)	2 (28.57%)	
<b>p-value</b>		0.472ns	0.165ns	0.301ns	

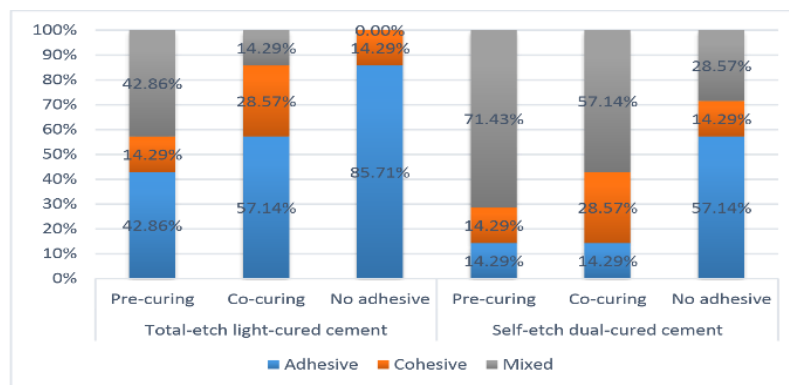


Figure (5): Stacked bar chart showing the mode of failure distribution in different adhesive application protocols and cement types (A).

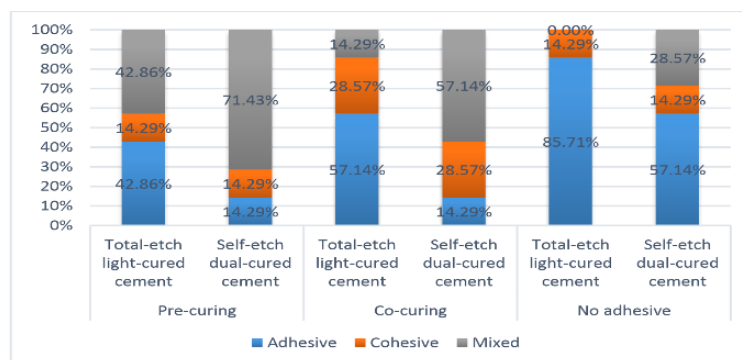


Figure (6): Stacked bar chart showing the mode of failure distribution in different adhesive application protocols and cement types (B).

#### IV. Discussion

Recently, the utilization of CAD/CAM technology in the recent dental practice, mainly in regards to fixed prosthesis, has led to a significant increase in the development of dental ceramic technology regarding the optical properties, microstructure and wide range of indications that led to the creation of numerous new products in a short period of time.<sup>(17)</sup>

The material employed in this research; UPCERA (Up-Cad) is a Lithium silicate ceramic that was recently introduced into the CAD/CAM materials.<sup>(18)</sup>

The performance of a ceramic restoration depends greatly on the quality and strength of the bond between the resin cement and the restoration. To enhance bonding of resin cements to ceramics, different types of surface treatments that can promote both chemical adhesion and micromechanical retention have been suggested.<sup>(19)</sup>

According to Kim et al. The outcome of the treatment surface mainly depends on the chemical composition of the ceramic. Hydrofluoric acid etching is the most effective procedure for treating a lithium-disilicate ceramic. The exposure of crystals is noted later the etching of the lithium-based ceramic. It may be observed that all glassy ceramic materials have a same composition of SiO<sub>2</sub>-60 %. However, the specific internal composition and the presence of other chemicals influence the effects of HF acid etching.<sup>(20)</sup>

According to Bajraktarova-Valjakova et al. several Upon examining the etching surface of IPS e-max CAD, one can observe the presence of mini-pores and channels of various sizes, accompanied by irregular ceramic particles. It is worth mentioning that the dissolving of the silica-matrix has led to the extrusion of elongated or bean-like crystals, as evidenced by previous studies, there are several approaches that can be employed to the treatment of ceramic surfaces that are bonded by adhesive. Without a doubt, the most effective method of treating silica-based ceramic is acid etching.<sup>(20)</sup>

According to Juliana Fraga Soares Bombonatti in 2018<sup>(21)</sup> demonstrates that the shear bond strength and the resin cement was not directly relation (i.e., light-curing or dual-curing). The lack of difference between cements could be demonstrate by the actuality that the light activation was directly applied over the cement. As a result, it is expected that the majority of the reaction take place at the expense of the light activation for both cements; all of the sampling were light activated immediately rather than waiting for chemical polymerization. It's notable that delaying the light activation could have an effect on the properties of dual-cured resin cements. Additionally, in a clinical situation, the thickness of the ceramic may have a major role on the light activation. Depending on the type, thickness, and color of the ceramic, the amount of radiant exposure may be insufficient due to the attenuation, this will lead to a lack of polymerization and compromise the process of polymerization.

Regarding the bonding' agents, the outcome was consistent with other research. The current investigation documented a higher average shear bond strength for the universal adhesive. This may be attributed to the concept that universal adhesive systems do not share the particularities of other simplified etch-and-rinse systems. In the case of simple self-etching adhesives, because the poor polymerized oxygen inhibited layer the residual monomers is react with tertiary amines named the reaction bimolecular redox lead to compromises the chemical reaction.<sup>(21)</sup>

According to J Luke Chapman Pre-curing of self-etching adhesives prior to the resin's cure increases the bond to dentin. The method of curing has no significant role in the strength of the enamel bond in self-etching adhesives.<sup>(22)</sup>

In the research, uses the SBS (shear bond strength) test to examine the bond strength of the CAD/CAM blocks. This test is advised a relatively simple test that allows for efficient detection of protocols for adhesive testing, as well as the profiling of different substrates in terms of depth and region.<sup>(23)</sup>

The failure mode analysis of the present experiment revealed that the majority of them were adhesive, which is indicative of the shear test's effectiveness in the evaluation of the bond strength between CAD/CAM materials and cement.

Limitations of this research was excluding other factors like a cyclic loading, multiple firing, different acidities in intraoral environment and another element that could influence the final outcome, this is a vitro study that concerns the environmental factors in the intraoral space as a whole, which can occur all at once.

#### V. Conclusions

Within the limitation of the current studies:

- To Increase adhesive protocol, the Co-curing is better than other tested methods.
- With regards to Cement types, self-etch dual cure cement gave a more reliable bond than the total etch light cure cement.

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