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Effect of Different Surface Treatment Protocols on MicroTensileBond Strength of CAD/CAM Composite Blocks to Dentin (In-Vitro Study)

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Abstract

Aim: The aim of this study was to evaluate the effect of different surface treatment protocols of CAD/CAM composite blocks on the micro tensile bond strength of CAD/CAM composite blocks to dentin. **Methods and Materials:** A total of forty-eight human molars were used in this study. They were divided into four main groups (12 each) according to the CAD/CAM blocks surface treatment protocol; either with laboratory sandblasting as a control group, chairside air abrasion, phosphoric acid etching and a combination of chairside air abrasion and phosphoric acid etching. Each group was divided into two subgroups (6 each) according to the storage time either 24 hours or 6 months. Teeth were mounted into acrylic resin blocks to facilitate its usage. A total of forty-eight blocks were obtained from the selected CAD/CAM composite blocks (Brilliant, Coltène Whaledent, Switzerland). The surface treatments were done according to the assigned groups. The dentin surface of all specimens was treated with the etch and rinse mode using universal adhesive (One coat 7, Coltène Whaledent, Switzerland). Dual cured self etching cement (Duocem, Coltène Whaledent, Switzerland) was used to cement the cut blocks to dentin specimens. Specimens were stored according to the storage time either for 24 hours or 6 months. After storage for the assigned time, each specimen was mounted on the cutting machine and sectioned into beams of $1mm^2$ cross-section area. The central beams from each specimen were selected and attached to the specially designed attachment jig, which was mounted on a universal testing machine. A tensile load was applied via testing machine at a crosshead speed of 0.5 mm/min. The load (Newton) required for de-bonding of each beam was divided by the area to express bond in MPa. **Results:** There was a significant difference between different groups ($p<0.001$). The highest value was found in surface treatment combination (28.94±2.59), followed by conventional surface treatment (27.48±3.37), then chairside air abrasion (25.24±3.82), while the lowest value was found in phosphoric acid etching (21.36±2.95). Moreover, value measured at 24 hours (28.75±3.07) was significantly higher than value measured after 6 months (23.09±3.26) ($p<0.001$). **Conclusions:** The bond strength of CAD/CAM Resin composite blocks to dentin is highly affected by the surface treatment protocol of the block.

Keywords: CAD/CAM Composite Blocks, Surface Treatment Protocols, indirect restoration; composite; inlays.

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Introduction

Nowadays, there is a global trend towards addressing dental caries with minimally invasive (MI) techniques. (1) Indirect restorations as biomimetic partial-coverage indirect restorations are performed annually throughout the world. (2)

In the latest decade chairside recent advancements in CAD/CAM technology have boosted the usability and cost-effectiveness of the restorations, which has led to an increase in the popularity of CAD/CAM procedures. (3) CAD/CAM composite systems have been reported to have superior marginal adaptation (4) , accepted edge stability and good aesthetic and colour stability as benefits. Also, they provide upgraded repair options, less wear tendencies and less cost effectiveness.

Some materials need additional processes after milling to achieve durable bonding between the restorative material, resin cement, and tooth structure. The bond strength to indirect restorations has reportedly been improved by surface preparation before cementation.

Typically, sandblasting is strongly recommended by manufacturers to obtain good bonding. (5) Sandblasting increases the surface roughness of the adherent area therefore creates mechanical retention and cleans bonding surfaces. (6) (7)

Sandblasting machines can be classified into laboratory sandblasting and chairside sandblasting. Laboratory sandblasters need space and special setup that can be difficult to achieve in a clinical setting. On the other hand, chair-side air abrasion devices are smaller in size, readily available in the clinical dental setting and easier to use by clinicians.

Therefore, the aim of this study was to evaluate the effect of different surface treatment protocols on the micro-tensile bond strength of CAD/CAM composite blocks to dentin. The null hypothesis of this investigation is that different surface treatment methods have no effect on micro-tensile bond strength of CAD/CAM composite blocks to dentin.

Materials and methods

The following materials were used in this study

I.1. Nano hybrid composite blocks (Brilliant) (Coltène Whaledent, Switzerland)

I.2. 37% Acid etching gel (Gel S, Coltène Whaledent, Switzerland).

I.3. Universal adhesive (One coat 7, Coltène Whaledent, Switzerland)

I.4. Dual-cure Cement (Duocem, Coltène Whaledent, Switzerland)) for blocks cementation

I.5. Abrasive powder Aluminium Oxide 53 µm powder (Velopex, UK

All materials' specifications, compositions and manufacturers were written in Table (1).

The following devices were used in this study

1- **Aqua Care twin air abrasion and polishing Unit** (Aquacare, Velopex, UK).
2- **EliparTM Deep Cure** LED curing light (3M ESPE, Germany).
3- **Basic Eco fine sandblasting unit** (Renfert,Germany)

Table (1): materials' specification, compositions and manufacturers

Material	Specifications	Composition	Manufacturer
Brilliant Crios	Resin Composite Block	Dental glass Bariumglass Size < 1.0 µm,Amorphous silica SiO_2 Size < 20 nm, Resin matrix Cross-linked methacrylates and Inorganic pigments such as ferrous oxide or titanium dioxide	Coltène Whaledent, Switzerland
Gel S	Acid etching gel	37% phosphoric acidgel	Coltène Whaledent, Switzerland
One coat 7 active	Universal adhesive	Urethane dimethacrylate 2-hydroxyethyl methacrylate Photoinitiators Ethanol, Ethyl alcohol Water	Coltène Whaledent, Switzerland
Duocem	Dual-cured adhesive resin cement	Bis-GMA, DMA, silica fillers, benzoyl peroxide, amines, pigments, additives	Coltène Whaledent, Switzerland
Aluminum Oxide	Abrasive powder	Aluminum Oxide 53µm particle size	Velopex international, UK

Samples size calculation

The calculation of the sample size was done using G*Power 3.1.9.4 Software. Based on an earlier study ***(Reymus et al, 2018)***, if the mean difference between 2 groups is 10 MPa, 6 specimens per group were needed to be able to reject the null hypothesis with a total of 48 specimens. The two-tailed significance level for the t-test was set at 5% with a power of 80%.

Teeth selection

Freshly extracted human molars were collected. The teeth were thoroughly cleaned and disinfected before being microscopically examined under x10 magnification to check for defects. Teeth with any problems were excluded. The teeth selected were kept in glass jars with distilled water at 40^{C} until used within three months.

Sample grouping:

A total of forty-eight human molars were used in this study. They were divided into four main groups (12 each) according to the CAD/CAM blocks surface treatment protocol; either with laboratory sandblasting as a control group, chairside air abrasion, phosphoric acid etching and combination of chairside air abrasion and phosphoric acid etching. Each group was divided into two subgroups (6 each) according to the storage time either 24 hours or 6 months.

Preparation of dentine specimens:

Using water cooled diamond impregnated disc at low-speed micro-slicing machine (Isomet 5000, Buehler, Buehler Ltd, Lake Bluff, Illinois, USA) the roots were cut 1mm beneath the cemento–enamel junction, the cuts were done perpendicular to the long axis of the tooth. Following that a parallel cut was done occlusally in a mesiodistal direction to remove the occlusal enamel and superficial dentin exposing the midcoronal dentine and obtaining a flat and uniform dentin surface.

Mounting of teeth:

The prepared molars were fixed with their occlusal surface facing upwards in chemically activated acrylic resin that in especially fabricated Teflon molds. All specimens were labelled according to their respective groups.

CAD-CAM blocks preparation:

A total of forty-eight specimens were obtained from the CAD/ CAM blocks to be used in the study. Using the same micro-slicing machine, we obtained the final specimens with dimensions of (4x4x4mm). The specimens' dimensions were measured with digital caliper to ensure their accuracy.

Dentin surface pretreatment and Adhesive application:

The universal adhesive was used with the etch and rinse mode following the manufacturer's recommendations. After etching, rinsing and drying, universal adhesive (One coat 7) was applied using micro-brush and rubbed for 20 seconds , air-thinned and cured for 20 seconds using LED light-curing unit.

Pretreatment of CAD/CAM Blocks:

The surfaces of the prepared CAD/ CAM composite blocks were treated according to their assigned groups.

Group 1: Sandblasting with laboratory sandblaster (control).

Basic eco fine 230 V sandblasting unit was used to deliver the aluminum oxide particles (25-70 μm), the diameter of the tip nozzle was 0.8 mm working at 1cm from the block surface for 10 seconds at an air pressure of 4 bar. To ensure standardized distance and angulation, a premeasured micro-brush was fixed to the nozzle tip by duct tape.

Group 2: Air abrasion with chairside air abrasion unit (AquaCare),

Aquacare twin air abrasion unit was used to deliver the aluminium oxide (53 μm). It is equipped with a standard tip fixed on a hand-piece nozzle with a diameter of 0.6 mm (Silver-coded) working at 1 cm distance from the restoration surface for 10 seconds under continuous water shroud and at an air pressure of 5 bar (500 MPa).

Group 3: Phosphoric acid etching

37% phosphoric acid gel was applied on the block surface for one minute then rinsed with air-water spray for 10 seconds followed by drying by gentle air for 5 seconds.

Group 4: Combination of chairside Air abrasion with AquaCare and phosphoric acid etching.

Cementation procedure:

After different surface treatments, a thin coat of the universal adhesive was actively applied on the block surface using micro-brush, gently air- thinned and light cured for 20 seconds. A thin layer of resin cement was applied to the dentin surface, A light finger pressure was used to seat the restoration. Excess cement was and the exposed margins were then sealed with glycerin gel as oxygen inhibitor and then light cured for 30 seconds from all aspects.

Storage of specimens:

For the short storage time group (24h), specimens were stored in distilled water at room temperature for only 24h. On the other hand, the 6 months specimens were stored in distilled water at room temperature for 6 months and the water was changed every 3 days.

Microtensile bond strength testing:

The specimens were sectioned along the buccolingual and mesiodistal planes using a diamond disc (MTI Corporation, Richmond, CA, USA) and a low-speed micro-slicing device (Isomet, Buehler, Lake Bluff, IL, USA) with water cooling. to create beam-shaped specimens (bonding areas around 1mm2). From each specimen, two or three beams were obtained. After cutting, a test for binding strength was conducted. The testing tailored micro-tensile jig was used to secure the beam specimens with cyanoacrylate gel (Zapit; Dental Ventures of America, Corona, CA, USA). This jig was made to accommodate the TBS Instron Universal testing machine and was made to transmit tensile forces to the specimen solely without the need of torque (Bisco Inc.Schaumburg, IL, USA). At a cross-head speed of 0.5/minute, the tensile force was applied up until failure of the specimen occurred. The failure load in Newton was then documented. The ratio between the failure load and the beam area, which was verified using a digital calliper before to testing, was used to determine the bond strength.

Statistical analysis:

Numerical data were presented as mean and standard deviation (SD) values. They were explored for normality by checking the data distribution, and using Shapiro-Wilk test. Data were normally distributed and were analyzed using two-way mixed model ANOVA followed by Tukey's post hoc test. Comparison of main and simple effects were done utilizing one- way ANOVA followed by Tukey's post hoc test for independent variables and paired t-test for repeated measurements. P-values were adjusted for multiple comparisons utilizing Bonferroni correction. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows

Results

Effect of different variables and their interaction:

There was a significant interaction between surface treatment and time (p=0.016).

Table (2): Effect of different variables and their interactions on micro-tensile bond strength(MPa)

Source	Sum of Squares	df	Mean Square	f-value	p-value
Surface treatment	713.58	1	713.58	318.95	**<0.001***
Time	668.44	3	222.81	99.59	**<0.001***
Surface treatment * Time	24.46	3	8.15	3.64	**0.016***

df =degree of freedom*; significant ($p \leq 0.05$) ns; non-significant (p>0.05)

Effect of surface treatment within each time:

- **24 hours:**

There was a significant difference between different groups (p<0.001). The highest value wasfound in surface treatment combination (30.88±1.39), followed by conventional surface treatment (30.56±1.55), then chairside air abrasion (28.80±1.52), while the lowest value was found in phosphoric acid etching

(23.98±1.33). Post hoc pairwise comparisons showed conventional surface treatment and surface treatment combination to have significantly highervalues than other groups ($p<0.001$). In addition, they showed chairside air abrasion to have significantly higher value than phosphoric acid etching ($p<0.001$).

- **6 months:**

There was a significant difference between different groups ($p<0.001$). The highest value wasfound in surface treatment combination (26.82±1.78), followed by conventional surface treatment (24.41±0.83), then chairside air abrasion (22.01±1.72), while the lowest value was found in phosphoric acid etching (18.99±1.66). All post hoc pairwise comparisons were statistically significant ($p<0.001$).

Table (3): Mean and standard deviation (SD) values of micro-tensile bond strength (MPa) for different surfacetreatment treatments within each time

Time	Micro-tensile bond strength (MPa) (mean±SD)				p-value
	Conventional surface treatment	Chairside air abrasion	Phosphoric acid etching	Combination	
24 hours	$30.56±1.55^{A}$	$28.80±1.52^{B}$	$23.98±1.33^{C}$	$30.88±1.39^{A}$	**<0.001***
6 months	$24.41±0.83^{B}$	$22.01±1.72^{C}$	$18.99±1.66^{D}$	$26.82±1.78^{A}$	**<0.001***

Different superscript letters indicate a statistically significant difference within the same horizontal row *;significant ($p \leq 0.05$) ns; non-significant ($p>0.05$)

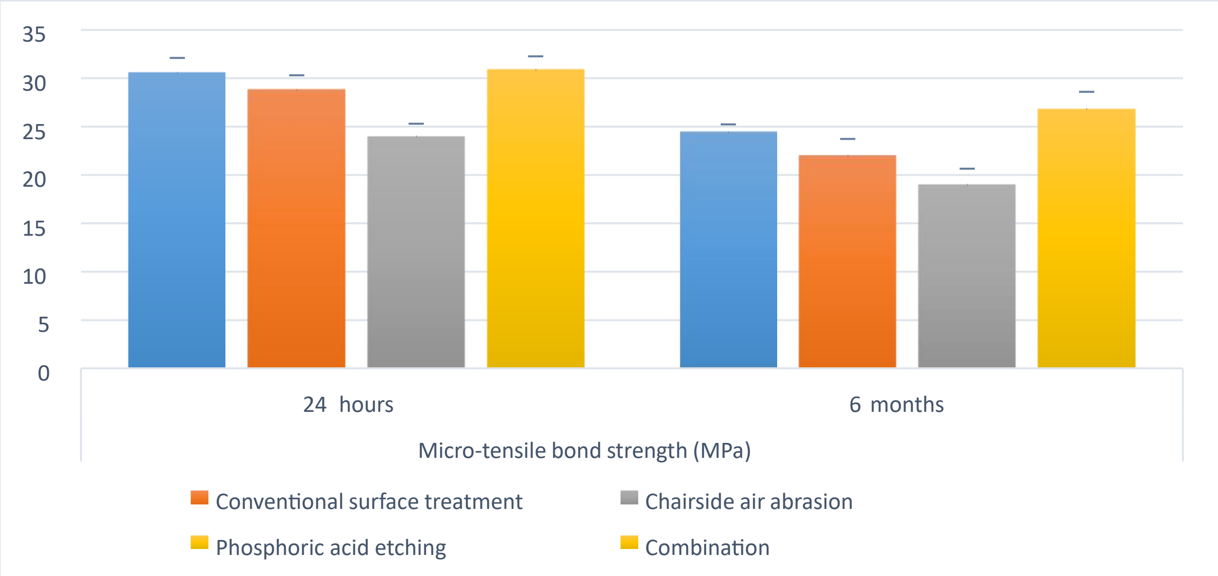

Figure (1): Bar chart showing mean and standard deviation (error bars) of micro-tensile bond strength (MPa)for different surface treatment treatments within each time

2- Effect of time within each treatment:

Conventional surface treatment:

The value measured at 24 hours (30.56±1.55) was significantly higher thanvalue measured after 6 months (24.41±0.83) ($p<0.001$).

Chairside air abrasion:

The value measured at 24 hours (28.80±1.52) was significantly higher thanvalue measured after 6 months (22.01±1.72) ($p<0.001$).

Phosphoric acid etching:

The value measured at 24 hours (23.98±1.33) was significantly higher thanvalue measured after 6 months (18.99±1.66) ($p<0.001$).

Combination:

The value measured at 24 hours (30.88±1.39) was significantly higher thanvalue measured after 6 months (26.82±1.78) ($p<0.001$).

Different superscript letters indicate a statistically significant difference within the same horizontal row *;

Table (4): Mean and standard deviation (SD) values of micro-tensile bond strength (MPa) fordifferent times within each treatment

Surface treatment	Micro-tensile bond strength(MPa) (mean±SD)		p-value
	24 hours	6 months	
Conventional surface treatment	30.56±1.55	24.41±0.83	**<0.001***
Chairside air abrasion	28.80±1.52	22.01±1.72	**<0.001***
Phosphoric acid etching	23.98±1.33	18.99±1.66	**<0.001***
Combination	30.88±1.39	26.82±1.78	**<0.001***

significant ($p \leq 0.05$) ns; non-significant ($p>0.05$)

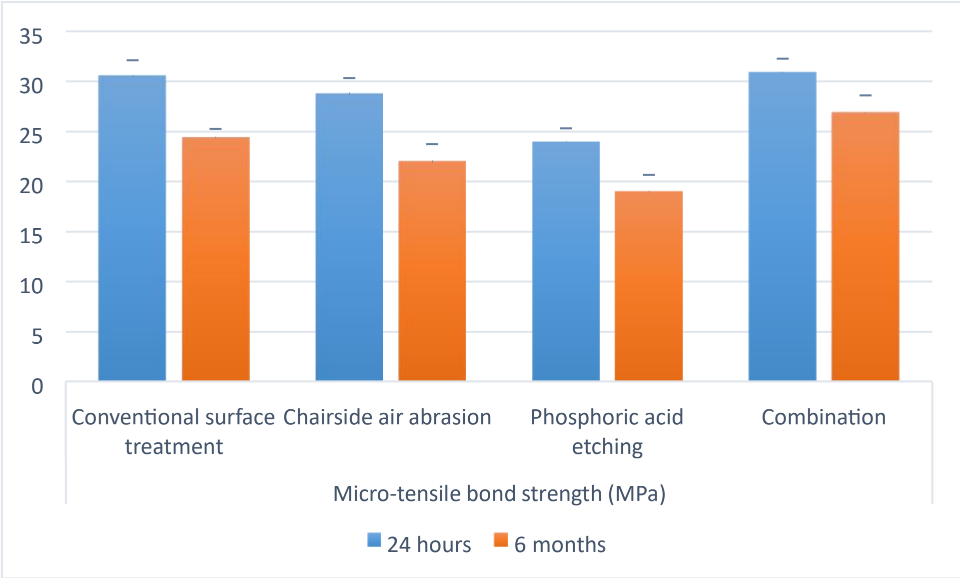

Figure (2): Bar chart showing mean and standard deviation (error bars) of micro-tensile bondstrength (MPa) for different times within each treatment

Discussion

The main goal of restorative dentistry is to return the teeth's biomechanical, functional, and aesthetic qualities. Indirect restorations can restore posterior teeth with significant tissue loss. Many factors, including the material, adhesive cementation to dentin/enamel, and the bonding technique, are important for applying indirect restorations (10). As a result of complex composition and intrinsic moisture of dentin, creating a strong bond between the resin adhesive and dentin is a difficult task (11). Thus, numerous attempts to enhance and maintain the resin dentin bonding have been attempted, and in this study, various surface treatment techniques of the CAD/CAM blocks are carefully examined.

Regarding the Effect of different surface treatment protocols,

there was a significant difference between different groups. The highest value was found in surface treatment combination (28.94±2.59), followed by conventional surface treatment (27.48±3.37), then chairside air abrasion (25.24±3.82), while the lowest value was found in phosphoric acid etching (21.36±2.95). This could be attributed to the combined effect of the mechanical preparation of the surface by air abrasion providing abundant micro- irregularities; then the chemical surface treatment with phosphoric acid that cleaned the smear layer formed by the mechanical preparation as well as increase the number of micro-irregularities by dissolving the resin matrix of the composite block obtaining clean and deep surface micropores for maximum adhesive infiltration. Another explanation is due to rising of the surface energy of the surface and wetting of the adhesive forming well and deeply infiltrated resin micro-tags that obtain maximum bond strength. While for the dry laboratory sandblasting, it offered only mechanical surface treatment with wide range of AlO_3 particles (25-70 μm) and high pressure (4 bar) and wide nozzle tip (0.8mm) obtained large and deep micro- irregularities resulting in deep micro-tags and high bond strength. On the other hand, chair side air abrasion with standard AlO_3 particles (53 μm) and narrow nozzle tip (0.6mm) in comparison to the laboratory sandblaster using coolant that might affect the number of particles delivered to the surface producing less or shallower micro- irregularities with subsequent lower bond strength. Lastly, the phosphoric acid produced the lowest bond strength. This might be due to the weak chemical effect of the acid on the composite block producing shallow micro-pores in comparison to the mechanical effect of sandblasting and air abrasion that adversely impacted the strength of the bond.

These results were in agreements with ***"Fathy H et al 2022"***(12) who found that the greatest bond strength outcomes were in the group of combination of both sand blasting using Al_2O_3 particles and acid etching and revealed that roughening the surfaces of the restorative material through mechanical conditioning (air abrasion or chemical etching) has been found to improve surface energy and wettability, enhancing the mechanical bonding between the resin cement and the CAD/CAM material . Additionally, "Reymus M et al 2019" (13) found that the extra air abrasion step increased TBS values and decreased rates of failure. Furthermore, special pretreatment techniques and air abrasion are necessary to ensure a strong bond between CAD/CAM resin composites and the luting composite and to obtain promising long-term results. Air abrasion and the removal of a smear layer appear to be the mechanical factors underlying these superior results. In addition(9)showed superior results in the comparison of different surface treatments in CAD CAM blocks in the groups of a combination of chemical and mechanical surface treatment using sandblasting and acid etching using phosphoric acid as they found that mechanical roughening is also necessary; in group 6 (H_3PO_4/Si), H_3PO_4 was not strong enough to induce visible surface roughening and the need for the mechanical roughening using Al_2O_3 sandblasting to get the highest values of bond strength.

On the other hand ***"Kubilay Barutcigil et al 2016"*** (14) disagreed with these results finding that using single bond universal without surface treatment showed superior bonds and this was due to the difference in the material tested and the CAD-CAM blocks used were mainly ceramic blocks, not composite blocks as in this study. Also, According to "Akah M et al 2022" (15), In the absence of an adhesive layer being added to the Brilliant Crios inlay restorations' fitting surface, the surface roughness created by CAD/CAM milling alone was able to create a stronger bond following artificial ageing than that established by air abrasion at high pressure. This is because the high pressure of sandblasting for 1 bar to 2 bar may have been the cause of a slight crack forming in the surface.

Concerning the effect of time, Value measured at 24 hours (28.75±3.07) was significantly higher than value measured after 6 months (23.09±3.26). There was general decrease in μTBS mean values of all groups in comparison to those after 24h. The main reason of this decrease is the impact of six months storage in distilled water on all bonded specimens. Bond degradation is a frequent consequence of extended aqueous storage. This could be as a result of the water absorption of the adhesive, particularly with the one step self-etch technique, which is characterized by its high hydrophilicity to enable its infiltration into the moist dentin. (16)

These findings were in line with those of "Lise DP et al 2017" (9) which discovered that high levels of bond strength were discovered after being stored for 3 weeks and that differences between the surface treatments didn't become, apparent until after more water storage time was done, which in turn altered the adhesive strength depending on bonds were achieved. ***"Pfeffer S et al 2020"*** (17) agreed in the microtensile bond strength of luting cements to a 3D printable composite finding that the bond strength of the self-adhesive resin cement was severely affected by long-term storage in water as well..

Concerning the effect of time on different groups, for the Conventional surface treatment, value measured at 24 hours (30.56±1.55) was significantly higher than value measured after 6 months (24.41±0.83). While for chairside air abrasion, Value measured at 24 hours (28.80±1.52) was significantly higher than value measured after 6 months (22.01±1.72). For Phosphoric acid etching, value measured at 24 hours (23.98±1.33) was significantly higher than value measured after 6 months (18.99±1.66). Finally, the combination group, value measured at 24 hours (30.88±1.39) was significantly higher than value measured after 6 months (26.82±1.78). The same attribution of bond deterioration due to hydrophilicity of the single step self-etch adhesive could be assigned. The good infiltration of the adhesive into the formed micro-irregularities resulted in well infiltrated resin micro-tags with the combination group showed more resistant tags to degradation even after six months storage (26.82±1.78).

Finally, it should be mentioned that many limitations were faced in this study as the used materials' cost and testing steps. Also, longer follow-up period is advised to evaluate the tested outcome. Therefore, the null hypothesis of this study was rejected. To summarize, the different surface treatment protocols of the CAD/CAM composite blocks have significant effect on the adhesive strength to dentin even after being stored for six months

Conclusions

Under the limitations of the current study the following conclusions were derived:

1. The bond strength of CAD/CAM Resin composite blocks to dentin is highly affected by the surface treatment protocol of the block.
2. Mechanical surface treatment of CAD/CAM Resin composite blocks either by conventional sandblasting or chair side air abrasion is considered the most efficient protocol to obtain good bonding to dentin.
3. Chemical surface treatment in combination with the mechanical protocol has a profound effect on the bond strength of CAD/CAM Resin composite to dentine.

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