EGYPTIAN
DENTAL JOURNAL

Vol.67, 2711:2723, July, 2021

Print ISSN 0070-9484 • Online ISSN 2090-2360

Conservative Dentistry and Endodontics

www.eda-egypt.org • Codex : 64/21.07 • DOI : 10.21608/edj.2021.68272.1554

CAD/CAM SURFACE TREATMENT AND TYPE OF ADHESIVE AFFECT CAD/CAM COMPOSITE BONDING TO DENTIN

Lina S. Marmoush*, Aya.S. Samaha ** *and* Farid S. El-Askary ***

ABSTRACT

Objective: To evaluate the effect of CAD/CAM surface treatments and different adhesives on the shear bond strength (SBS) of a CAD/CAM composite to dentin.

Materials and Methods: In total, 120-flat dentin surfaces were prepared, and divided into 12 groups (n=10) according to: 1- CAD/CAM surface treatment, and 2: Type of adhesive. A total of 120 CAD/CAM composite rods (2mmx2mmx4mm) were prepared, and sandblasted. The rods were left without further treatment, ultrasonically cleaned in distilled water, or acid etched using 35% phosphoric acid. The dual- and self-cured adhesives were applied to dentin, in etch-and-rinse or self-etching modes and the rods were cemented using dual-cured resin cement. The SBS test was run at a crosshead speed of 0.5 mm/min until failure. Data were analyzed using Two-Way ANOVA/ Tukey test, $P=0.05$.

Results: Factors "surface treatment", "adhesive" and "surface treatment x adhesive" had significant effect on SBS ($P<0.05$). Sandblasting had the highest significant SBS among surface treatments ($P<0.05$). Self-cured adhesive showed higher SBS compared to dual-cured adhesive in etch-and-rinse mode ($P<0.05$). Mixed failure was the predominate failure (59.2%).

Conclusion: Sandblasting improved the SBS of a CAD/CAM composite to dentin. The use of dual-cured universal and self-cured adhesives in their simplified self-etching approach is preferrable.

KEYWORDS: CAD/CAM composite, Surface treatments, Universal adhesive, Self-cured adhesive, Shear bond strength.

INTRODUCTION

A reliable bond between indirect composites and adhesive resin cements has always been a challenge. The higher the degree of resin polymerization of the CAD/CAM composites, with almost no residual C=C for chemical bonding, was thought to be the reason[1,2]. Therefore, different surface treatments have been proposed to improve the

* Demonstrator, and Master's Degree Student, Operative Dentistry Department, Faculty of Dentistry, Ain Shams University.
** Lecturer, Operative Dentistry Department, Faculty of Dentistry, Ain Shams University.
*** Professor, Operative Dentistry Department, Faculty of Dentistry, Ain Shams University.

bond between CAD/CAM composites and resin cements. One of the most recommended surface treatments by manufacturers is sandblasting using 50µm Al_2O_3[1]. Unfortunately, the precise surface treatment is not always clearly provided by the manufacturers. Surface treatments can be varied from just sandblasting followed by air-drying, up to ultrasonication in distilled water, or steam cleaning for a non-advised time. Alcohol may be used as final cleaning, but it is an optional step. As most manufacturers do not recommend the use of acid etching after sandblasting, during the clinical try-in step, the fitting surfaces of the indirect restorations is at risk of saliva contamination. After the try in step, some authors recommended ultrasonication and/or phosphoric acid etching to clean the contaminated surfaces[2,3].

Previous studies showed disagreement in their results regarding the effect of the different surface treatments and surface cleaning methods on bonding of CAD/CAM composites. There was no significant difference in the bond strength of resin cement to a CAD/CAM composite using the different surface treatment methods[3,4]. On the contrary, it was shown that ultrasonic or acid etching treatments of the sandblasted CAD/CAM composite had a negative effect on the bond strength of resin cement[2].

Dentin adhesives were used with conventional resin cements to achieve a strong and durable bond between tooth/resin cements/indirect restoration. Universal adhesives were introduced essentially as self-etching adhesives, which by the operator's preference can be also used in the etch-and-rinse mode[5]. It was reported that light- and dual-cured universal adhesives showed similar bond strength when, they used in etch-and-rinse and self-etching modes[6,7]. However, the cumulative retention rate after 24-month of clinical evaluation showed that universal adhesives in the etch-and-rinse mode was significantly higher in their retention rate compared to the same adhesives when, used in the self-etching mode[8].

The incompatibility between simplified adhesives and dual- and self-cured resin composites was extensively discussed in the previous studies[9–12], which encouraged dental manufacturers to switch simplified light-cured adhesives to dual-cured ones. This is achieved by providing the so-called "self-cured activators" to be mixed with the light-cured adhesive[13–15]. The dual-cured adhesives polymerize basically by light activation, which in areas of difficult light access, their bonding effectiveness could be compromised[16]. For this reason, some manufacturers developed self-cured adhesives to be used in areas with deficient light activation. Self-cured adhesives depend on the chemical initiator/co-initiator system using the benzoyl peroxide as the initiator and tertiary amine as the co-initiator[17].

The aim of this study was to evaluate the effect of CAD/CAM surface treatment and the type of adhesive on the shear bond strength (SBS) of CAD/CAM composite to dentin. The null hypothesis tested was, neither CAD/CAM surface treatment, nor the type of adhesive had significant effect on the SBS of CAD/CAM composite to dentin.

MATERIALS AND METHODS

In total, 60-freshly extracted human molar teeth were collected according to the ethical regulations, Faculty of Dentistry, Ain Shams University. The teeth were cleaned under running water, and soft tissues were removed using hand scaler (Martin GmbH, Germany). The collected teeth were stored in distilled water at 4°C for no longer than 3 months from extraction. The occlusal enamel of all molar teeth was removed to obtain a flat dentin surface using low speed abrasive discs (Okodent, Tautenhain, Germany). Each tooth was then vertically sectioned in the bucco-lingual direction into mesial and distal halves using the same discs under copious water irrigation. A total of 120 halves were received, to be used for The SBS testing.

Custom-made circular polyurethane mold, with 2cm thickness and 20cm in diameter were fabricated.

The mold contained five circular holes of 2cm depth and 2cm diameter. The molds were placed on adhesive sticky tapes and positioned on a glass slab. A separating medium was applied to the walls of each mold hole using a brush, to facilitate removal of the embedding material. Dentin specimens were placed at the center of mold holes with their occlusal dentin facing the glass slab and secured in their positions using the adhesive tape. Fast-set self-cured acrylic resin (Acrostone, Acrostone dental factory, Egypt) was poured inside each hole and the mold was immersed in cold tap water to minimize the temperature rise during the curing of the acrylic resin material. After complete setting of the acrylic resin, each acrylic cylinder with its dentin specimen was removed from the mold. The dentin surfaces were wet ground over #180 grit (SiC) paper to obtain a flat dentin surface. Dentin surfaces were then wet ground using #600 SiC abrasive papers for 30 seconds to create a standardized smear layer. The specimens were ultrasonically cleaned in distilled water for 2 minutes to remove any remaining silicon carbide dusts.

Specimen grouping

Dentin specimens were divided into 12 groups (n=10/group) according to: 1- CAD/CAM surface treatment, 3 groups (sandblasting, sandblasting/ultrasonic, and sandblasting/acid etching) and 2: Type of adhesive, 4 groups (dual-cured/etch-and-rinse, dual-cured/self-etching, self-cured/etch-and-rinse and self-cured/self-etching). Material/Description (Lot #), composition and manufacturer are presented in Table 1.

TABLE (1) Materials/Description (Lot #), composition and manufacturer

Materials/ Description (Lot #)	Composition	Manufacturers
One Coat 7 Universal Adhesive (H73416). One Coat 7.0 DC Activator (l03075).	**Adhesive**: methacrylate acid, other photoinitiators, water, 10-MDP, polyacrylic methacrylates, ethanol. **DC activator**: Ethanol, water, activator	Coltene, Whaledent AG, Switzerland
Parabond: Self-cured self-conditioning adhesive. Non rinse conditioner (j09453). Adhesive A (j17232) and Adhesive B (j17027).	**Non rinse conditione**r: Water, Acryloamido sulfonic acid, Methacrylate **Adhesive A:** Methacrylate, Maleic acid, Benzoyl peroxide, GDMA. **Adhesive B:** Ethanol, Water, Initiators.	
Brillant Crios: CAD-CAM resin composite blocks, Shade: A3 LT (I16275)	**Inorganic portion (71 wt%):** amorphous SiO2 (<20 nm), barium glass (<1 µm). **Polymers (29 wt%):** Bis-GMA, Bis-EMA, TEG-DMA.	
DuoCem: Conventional Resin cement, Shade: Dentin (l82566)	**Base/catalyst**: Bis-GMA 10-<15%, TEGDMA 5-<10%, zinc oxide coated 1-<5%, dibenzoyl peroxide <1%, sodium fluoride <1%.	
Ultra-Etch/Phosphoric acid gel (E046)	35% phosphoric acid.	Ultradent Products INC. South Jordan, Utah, USA
10-MDP: methacryloyloxydecyldihydrogen phosphate, Bis-GMA: Bis phenol A glycidyl methacrylate, TEGDMA: triethylene glycol dimethacrylate and UDMA: urethane dimethacrylate, Bis-EMA: Bisphenol ethoxylated methacrylate.		

CAD/CAM composite block preparation

Three low translucent CAD/CAM resin composite blocks (size 14) were sectioned into 120 rectangular rods of 2mm x 2mm x 4mm using a slow speed disc equipped with a water-cooled diamond blade (IsoMet 4000Buehler, Lake Bluff, IL, USA). The decided bonding surfaces of all CAD/CAM rods were sandblasted using 50µm Al_2O_3 (Air prophy unit, Artspa industrial company, China) perpendicular to the surface from 10mm for 10 seconds at 1.5 bar according to manufacture instructions. The sandblasted surfaces were then: 1. Thoroughly rinsed using air/water spray for 10 seconds and air-dried for 10 seconds. 2. Ultrasonically cleaned (Eumax®, Hong Kong Model number: UD80SH-2.6L) in distilled water for 5 minutes and air-dried for 10 seconds, or 3. Acid etching using 35% phosphoric acid for 20 seconds, rinsed with air/water spray for 10 seconds and air dried for 10 seconds.

After the application of the different CAD/CAM rods surface treatments, One Coat 7 Universal adhesive was rubbed over the treated CAD/CAM rod surfaces for 20 seconds using a disposable micro-brush, air dried for 5 seconds and light cured for 20 seconds using LED light curing unit (Woodpecker LED F, China) with light intensity of 1400mW/cm^2. The intensity of the LED light curing unit was periodically checked after each 10 specimens using the device build-in radiometer.

Dentin surface treatment

For the dual-cured adhesive in the etch-and-rinse mode, dentin surfaces were etched using 35% phosphoric acid for 15 seconds, rinsed with air/water spray for 20 seconds, air-dried for 2 seconds and excess water, if present was removed, using lint-free tissue leaving the dentin visibly moist according to manufacturer instructions. The bottle of the One Coat 7/Universal adhesive was shaken, and a drop of the adhesive was dispensed into a well. The adhesive was applied on the dentin surface and rubbed for 20 seconds using micro-brush and air-dried for 5 seconds using oil/water free compressed air. No light curing was performed at this step. One drop of the adhesive and one drop of the DC activator were mixed in the well using disposable brush until homogenous mix was reached according to manufacturer instructions. The adhesive mix was applied on dentin, rubbed for 20 seconds, and air-dried for 5 seconds. The adhesive was light cured for 10 seconds using the LED light curing unit before the application of resin cement.

Regarding the dual-cured adhesive in the self-etching mode, acid etching step was omitted, and the adhesive was applied on dentin and light cured as described in the etch-and-rinse mode.

For the self-cured adhesive in the etch-and-rinse mode, the dentin surfaces were acid etched, rinsed, and air-dried as described for the etch-and-rinse of the dual-cured adhesive. One drop of adhesive A and one drop of adhesive B were dispensed into the plastic well supplied by the manufacturer. The adhesive was mixed thoroughly until homogenous mix was reached using a micro-brush supplied by the manufacturer. The adhesive was applied on dentin surface, rubbed for 30 seconds, and air-dried for 2 seconds. The adhesive was not light cured.

Concerning the self-cured adhesive in self-etching mode, the non-rinsing conditioner was applied on dentin surface, scrubbed for 30 seconds, and air-dried for 2 seconds according to manufacturer instructions. The mixed adhesive A and adhesive B was applied as described in the etch-and-rinse mode. The adhesive was not light cured.

CAD/CAM rod cementation

After each respective dentin and CAD/CAM rod surface treatment, resin cement was injected over the dentin surface using the auto-mix tip supplied by the manufacturer. CAD/CAM resin composite rod was seated over the resin cement and a load of

500 grams was applied over the CAD/CAM rod. The load was kept in its position, and the excess cement was carefully removed from around the resin composite rod using the micro-brush. The resin cement was light cured for 20 seconds from each rod surface, with a total light curing time of 80 seconds using the LED light curing unit. After light curing, the load was kept in its position for 10 minutes. All specimens were stored in distilled water at room temperature for 48 hours before SBS testing.

Shear bond strength testing

Each dentin specimen with its acrylic block was fixed using a specially fabricated fixing device. The device consisted of upper and lower metallic plates, with the width of the upper plate was approximately half of the width of the lower plate. The two plates were fixed to each other using 2 metallic screws. The lower plate had a center concave area to accommodate each acrylic block with its dentin specimen. Each dentin specimen was secured in its position using the metallic screws. Each dentin surface was carefully positioned in the device provided that the surface of the dentin specimen was in the same level of the upper plate, to ensure proper load application at the interface. The device with dentin specimen was placed on the lower plate of the Universal Testing Machine (LRX Plus, Lloyd instruments, Leicester, UK). A uni-beveled metallic attachment was fixed to the upper jig of the machine. The attachment was positioned as close as possible to CAD-CAM/resin cement/dentin interface[18]. A compression load was applied, and the test was run at a crosshead speed of 0.5 mm/min until failure. Shear bond strength was calculated in MPa by dividing the load recorded (Newton) over the bonded surface area (mm^2).

Fracture-pattern analysis

Fractured dentin and rod surfaces of all specimens were examined using a stereomicroscope (SMZ 745T, Nikon, Japan) at 8x magnification. Each failed bonded area was captured using the camera supplied by the microscope (WAT221S, Japan). The failure modes were assessed as follows:

1. Adhesive failure, where the failure occurred at the CAD/CAM resin composite rod/resin cement and resin cement/dentin interface.
2. Mixed failure, where the failure occurred at resin dentin interface with part of cement or CAD/CAM resin composite rod attached to dentin surface.
3. Cohesive failure, failure that occurred either in dentin or in CAD/CAM resin composite rod.

Statistical analysis

Statistical analysis was performed using SPSS program (Version 20 for Window's). Two-way ANOVA was used to evaluate the effect of adhesive type, CAD/CAM surface treatment, and their interactions on shear bond strength. One-way ANOVA followed by Tukey HSD post hoc test were performed to evaluate the effect of different adhesive type within each CAD/CAM surface treatment, and to evaluate the effect of CAD/CAM surface treatment within each adhesive type. Significant level was set at $P=0.05$.

RESULTS

Table 2 showed that regardless of the different adhesives, sandblasting (SB) showed the highest statistically significant SBS ($P<0.05$) compared to sandblasting/ultrasonic (SB/US) and sandblasting/acid etching (SB/AE). There was no statistically significant SBS difference between SB/US and SB/AE ($P>0.05$). Regardless of the surface treatment, there was no statistically significant difference in SBS ($P>0.05$) between self-cured/etch-and-rinse (SC/E&R), self-cured/self-etching (SC/SE) and dual-cured/self-etching (DC/SE). There was no statistically significant difference in SBS between dual-cured/etch-and-rinse (DC/E&R), DC/SE and SC/SE ($P>0.05$). On the other hand, SC/E&R showed statistically significant difference compared to DC/E&R ($P<0.05$).

Two-way ANOVA showed that factors "surface treatment", "type of adhesive", and "surface treatment x type of adhesive" had significant effect on SBS (*P<0.0001*, *P=0.002* and *P<0.0001*, respectively).

Failure mode analysis

Mixed failure mode was the predominate type of failure, which represented 59.2%, followed by the adhesive failure, which represented 36.6%. Cohesive failure represented 4.2% from the total failure modes. The percentage distributions of each failure mode within each adhesive type and surface treatments are presented in Figure 1. Representative stereomicroscopic photos for adhesive, mixed and cohesive failures are presented in figures 2-4.

TABLE (2): Means ± Standard Deviations for shear bond strength in MPa of different surface treatment and adhesives system.

	SB	SB/US	SB/AE	Total adhesive
DC/E&R	21.2 ± 5.4	10.0 ± 3.2	8.5 ± 2.0	13.2 ± 6.8^{C}
DC/SE	18.3 ± 4.4	10.6 ± 3.6	13.1 ± 4.3	14.0 ± 5.2^{BC}
SC/E&R	16.6 ± 4.8	16.8 ± 4.9	17.8 ± 2.8	17.0 ± 4.2^{AB}
SC/SE	15.8 ± 4.3	14.5 ± 3.2	12.9 ± 3.0	14.4 ± 3.6^{BC}
Total treatment	18.0 ± 5.1^{a}	12.8 ± 4.6^{b}	13.1 ± 4.5^{b}	

Means with same small superscript letters for total treatment are not statistically significant at (p=0.05).

Means with same capital superscript letters for total adhesive are not statistically significant at (p=0.05).

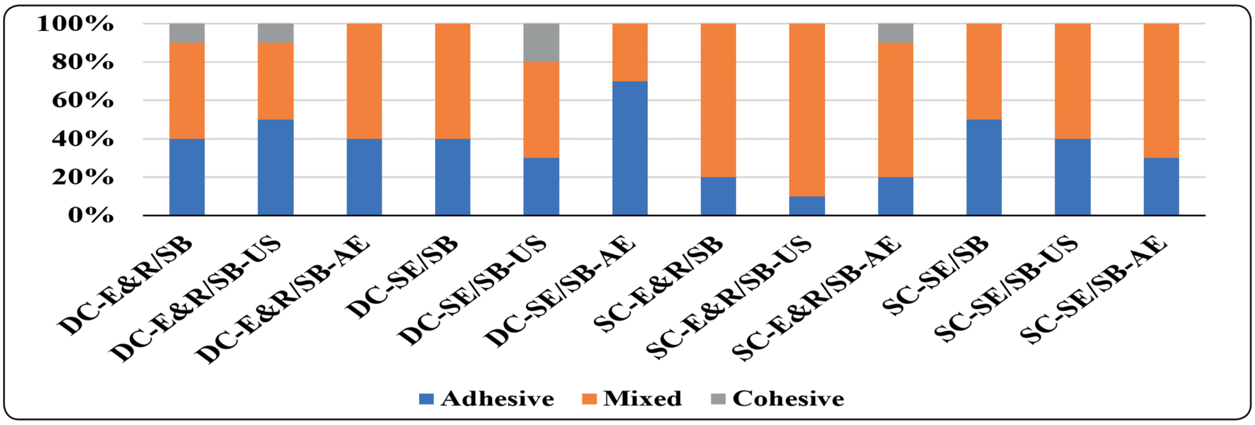

Fig. (1) Bar chart representing the percentage of adhesive, mixed, and cohesive failures of all tested groups.

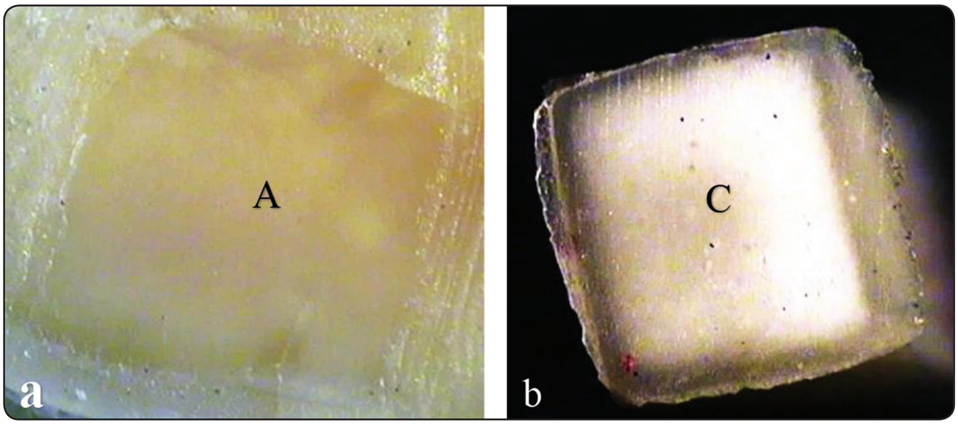

Fig. (2) a and b: Representative stereo-microscopic images for DC-SE&SB-AE group. a: Dentin side, and b: discovered the adhesive type of failure. A: Adhesive, and C: Resin cement.

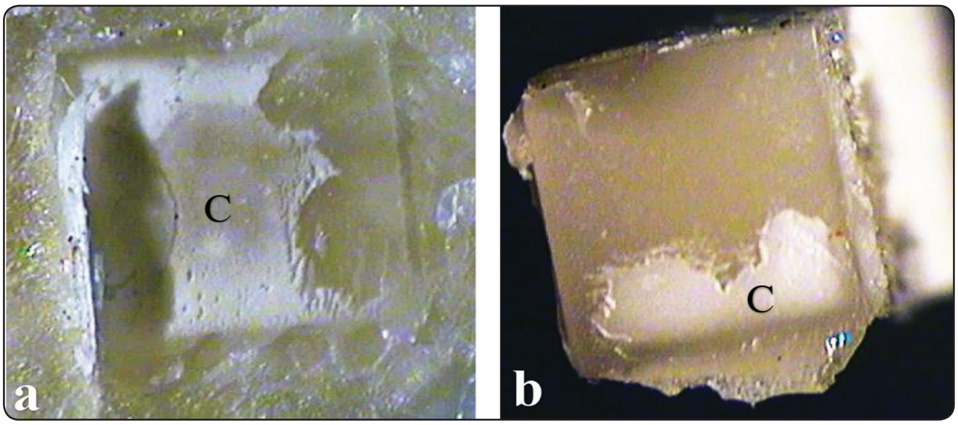

Fig. (3) a and b: Representative stereo-microscopic images for group SC-E&R/SB group. a: Dentin side, and b: CAD/CAM rod side. Mixed type of failure was detected, adhesive failure between resin cement and both dentin and CAD/CAM rod associated with cohesive failure in resin cement. C: Resin cement.

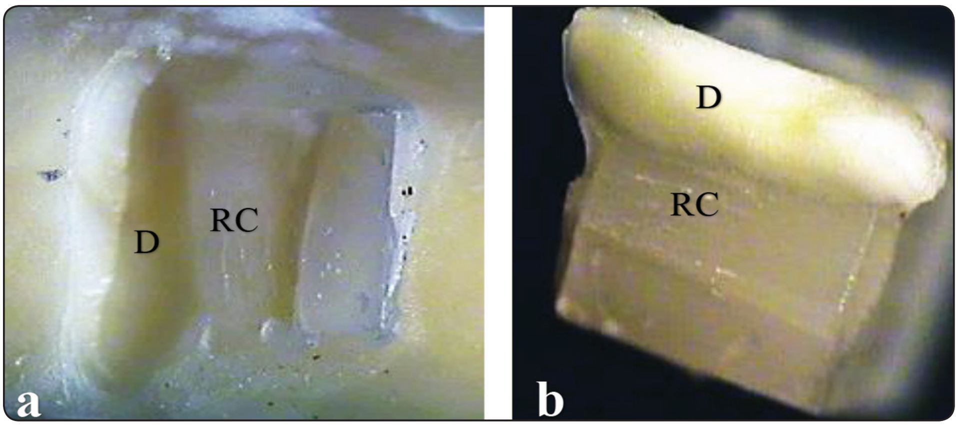

Fig. (4) a and b: Representative stereo-microscopic images for DC-E&R/SB group. a: Dentin side showed cohesive failure in both dentin, and CAD/CAM rod. b: CAD/CAM rod side. D: Dentin and RC: CAD/CAM rod.

DISCUSSION

Different surface treatments have been introduced to improve the bonding between CAD/CAM resin composite blocks and resin cement[19]. Manufacturer's instructions indicate the use of sandblasting as a main surface treatment, in order to clean the fitting surface of the restoration and create rough surface to enhance bonding. In general, sandblasting can be performed either in dental clinic after the try-in step and just before the employment of bonding procedures, or in the lab, from which the restorations are delivered with their fitting surfaces are already air-abraded. In the first situation, the sandblasting is performed after checking of restoration fitness and is followed by ultrasonic cleaning to remove the Al_2O_3 loose particles as recommended by some manufacturers. This surface treatment option did not improve bond strength of the conventional resin cements[2]. In the second situation, the cleaning of the restoration with acid etching to remove saliva contamination was reported[2,20]. There was controversial issues regarding the use of acid etching after sandblasting on the bond strength of the tested CAD/CAM materials, which either showed a negative effect[20], or had no effect on the bond strength[2], which depended on the material tested.

Dental manufactures have been lunched the simplified self-etching adhesives, in order to make the clinical procedure steps simpler and to shorten the clinical application time[21]. A new class of adhesives termed "universal adhesives", or "multi-mode adhesives" have been developed to be used with either direct or indirect restorations and can be applied in etch-and-rinse, self-etching and or selective enamel etching modes depending on the clinical situation and the operator's preference[22]. There is no general agreement on the appropriate universal adhesive application mode for dentin[23]. It was reported that only ultra-mild universal adhesive in etch-and-rinse approach showed promising results regarding the dentin bond strength, while there was no evidence that mild universal adhesives had superior bond strength in the etch-and-rinse mode compared to the self-etching mode[5].

Due to the wide spread use of dual-or self -cured resin cement with indirect restorations, dual-cured adhesives have been introduced in the market to overcome the incompatibility problem between simplified adhesives and dual-cured resin cements to establish optimal resin cements polymerization[24]. The bond strength was dropped, when simplified light-cured adhesives were used with dual-cured or self-cured resin cement[25]. Hence, manufactures introduced self-curing activators that are either incorporated in the formulation of the universal adhesives or presented as a separate bottle to be mixed with the light cured adhesives[13]. Unfortunately, this chemical incompatibility is not always reversed by adding the self-cured activator[26].

Self-cured adhesives were developed, in order to obtain secure polymerization of resin cement in regions where, the curing light was hardly reached the resin cement[27,28], which could lead to the decrease in cement's degree of conversion and consequently compromised the longevity of the restoration[29]. It was found that a chemically-cured adhesive yielded significantly greater bond strength compared to the dual-cured ones, even with the decreasing in the amount of light energy reaching the resin cement[27].

From the results of this study, both surface treatments and adhesives strategies had a significant effect in the bond strength of CAD/CAM composite to dentin. Thus, the null hypothesis must be rejected. Regardless of the adhesive used, SB showed the significant higher SBS, compared to SB/US and SB/AE. Sandblasting with 50-µm Al_2O_3 particles increased the roughness of the resin composite surface with removal of the resin matrix[1,30]. Consequently, this could lead to the exposure of filler particles, which enhanced the proper salinization of the surface. In the light of improving the bond between CAD/CAM resin composite and resin cements, SB was employed in an attempt to increase surface energy[31], surface roughness[32], surface area[1], and the creation of an irregular surface of the bonded surfaces[21,34]. This could lead to the formation of micromechanical undercuts, and in turn improving the bond between CAD/CAM material and resin cement[33].

Ultrasonic cleaning was used, as recommended by some manufacturers, in order to remove loose Al_2O_3 particles and to clean the surface after the try-in step after being sandblasted[2]. This step is recommended by the most CAD/CAM resin composite blocks manufacturers. Acid etching was used to clean the resin composite surface after being contaminated with saliva during the try-in step[2,3]. However, SB/US and SB/AE showed lower SBS compared to SB only. It was reported that ultrasonic cleaning of the sandblasted CAD/CAM surface removed large AL_2O_3 particles leaving the smaller ones, which might be the reason for the reduction in the bond strength of the conventional resin cement[2]. Ultrasonic cleaning could lead to the formation of smoother surface than that expected from sandblasting only[2]. Furthermore, ultrasonic cleaning contaminated the sandblasted surface of CAD/CAM resin composite with water[35,2]. The short air-drying time in this study might not be able to remove all remaining water from the CAD/CAM surface after ultrasonic cleaning step. Water could diffuse and hide between the AL_2O_3 particles that might be hard to be removed in short air-drying time. The water content of One Coat 7 Universal adhesive was reported to range from 5%-10%[7]. When the adhesive was applied on the ultrasonic cleaned CAD/CAM surface, the remaining water on the surface might dilute adhesive monomer and reduce its effect and consequently, led to significantly lower bond strength.

Regarding the acid etching cleaning method, it was shown that acid etching removed both large and small Al_2O_3 particles, which led to the creation of relatively smooth CAD/CAM surfaces compared to the sandblasted surfaces without acid etching[2]. The presence of relatively smooth surfaces could eliminate the micromechanical undercuts, upon which the adhesive could be mechanically interlocked. It was reported that MDP, which is a phosphate ester monomer, containing adhesives improved the bond strength of CAD/CAM materials through the adsorption of phosphoric acid by the Al_2O_3 particles[35]. The application of phosphoric acid etching over the sandblasted CAD/CAM surface might be chemically maintained on the surface of the Al_2O_3 particles, and upon rinsing it might form a thin film and might not be removed. This might hinder the action of other phosphate containing monomer, as MDP monomer in the One Coat 7 Universal adhesive, from optimally bond to the sandblasted CAD/CAM surface.

Despite the difference in CAD/CAM resin composite and in resin cement used and the difference in bond strength testing methodology, the results of this study were in agreement with the study of Kawaguchi, et al.[2] they concluded that once the surface of the CAD/CAM resin composite was sandblasted, ultrasonic or acid etching cleaning steps should not be performed. The sandblasted surface was the surface of choice for improving the bonding of conventional resin cement to CAD/CAM resin composite material[2]. On the other hand, the results of this study was in disagreement with the results of Tinastepe, et al.[4] they reported that the application of phosphoric acid on an air-abraded surfaces did not affect bond strength of resin composite to the resin nano-ceramics CAD/CAM surfaces. They showed that acid etching did not change the surface topography of the air-abraded surface, with no influence on the bond strength. In the study of Tinastepe, et al.[4] they aged the CAD/CAM surfaces before being air-abraded and they used silica particles to roughen the CAD/CAM surfaces. The difference in the type of air-abraded particles and the methodology employed for the CAD/CAM surfaces could be the reasons of such inconsistency.

Regarding the effect of adhesive strategies, the results of this study showed that there was no statistically significant difference in shear bond strength between etch-and-rinse and self-etching modes in the dual-cured adhesive. In this study, One Coat 7.0 Universal adhesive was used with its DC Activator. The DC activator contains ethanol and water[26], which could lead to the dilution of adhesive's resin monomers[26,11 ,14]. It was reported that the high increase in the percentage of solvent/ water contents could be expected upon mixing of the "self-curing activator" with the original liquid of the universal adhesive, with the disruption in the component's proportion of the adhesive[36]. Due to the expected high solvent/water content of the adhesive, the evaporation of solvent might not be perfectly performed, and consequently negatively affected adhesive's polymerization[36]. Furthermore, mixing the 2-step etch-and-rinse adhesive with its DC activator significantly decreased its bond strength, compared to the bond strength of the same adhesive without the use of its DC activator[14]. The results of this study came in agreement with the study of El-Sayed, et al.[7] They showed that when, One Coat 7 Universal adhesive was used to bond dual-cured core build-up resin composite to dentin, there was no statistically significant difference between etch-and-rinse and self-etching modes. The authors explained their results by the presence of remaining water on dentin when, the adhesive was used in the etch-and-rinse "wet-bonding" technique, as the water left on the dentin might furtherly dilute the adhesive's resin monomers. This was not the condition when, the same adhesive was not used in conjunction with the DC activator, i.e. the light curing protocol, which showed a significant higher bond strength of the etch-and-rinse mode compared to the self-etching mode[7]. In the light curing protocol, the relatively high pH of the One Coat 7 Universal adhesive (pH = 2.8), which made this adhesive with low aggressiveness on dentin. This might result in inadequate conditioned and primed the dentin substrate[7]. In the etch-and-rinse mode, especially in wet-bonding technique, the expected increase in water content of the adhesive, either from the use of the DC activator or from water left on dentin surface, might lead to increase solvent/water content, and dilute the adhesive's resin monomers. This in turn could lead to the decrease in the bond strength.

Regarding the self-cured adhesive, there was no statistically significant difference in shear bond strength between etch-and-rinse and self-etching modes. In the self-etching mode, Para bond was used with its non-rinse conditioner, which is composed of acrylamidosulfonic acid and water[37]. Acrylamidosulfonic acid is a strong acid having pH= 0.9-1.3[37,38]. The low pH could be a reason to increase the demineralization depth in dentin similar to that occurred when, phosphoric acid (pH<1) was used[39]. The presence of water in the formulation acted as an ionizing media for the action of the sulphonic acid group[40]. It was reported that self-etch adhesives containing acrylamidosulfonic acid and/or maleic acid, could decalcify the mineral part of dentin resulted in demineralization pattern same as the etch-and-rinse adhesives do[41]. This might explain why Para bond adhesive behaved similarly when used in both etch-and-rinse and self-etching modes.

In the self-etching mode, there was no statistically significant difference between the dual-cured universal adhesive and the self-cured adhesive. In the study of Jain, et al.[37] although all adhesives tested were applied in the self-etching mode, the universal adhesives yielded significant higher bond strength compared to the self-cured adhesive, which came in disagreement with the results of this study. In the study of Jain, et al.[37] they applied the universal adhesives in the light-curing protocol, which contradicted the protocol applied in this study (dual-curing protocol). Despite of the precise calculation of the water content in the final mix of adhesive/DC activator by dental manufacturers[7], increasing the water content in the final mix of the universal adhesive with its DC activator might be expected. Increasing the water content within the adhesive could lead to the dilution of the acidic monomer, resulting in the impairment of the demineralization effect of the adhesive, thus negatively affected its dentin bond strength[26]. Furthermore, the lack of hybrid layer formation with short and less dense resin tags formation was noticed in universal adhesives utilized the self-etching mode[26]. On the other hand, the non-rinse conditioner of the Para bond adhesive contains sulphonic acid, which was capable to dissolve the smear layer and demineralize the dentin as phosphoric acid did[40]. Removal of smear layer and mineral dissolution of dentin led to the formation of micro-porosities with adhesive monomers' infiltration[42]. It could be hypothesized that the use of strong acid, as sulphonic acid, in the non-rinse conditioner, with the presence of maleic acid in bottle A of the Para bond adhesive might result in the formation of hybrid layer with long and more dense resin tags[37]. This might be the reason of the non-significant SBS results between Para bond and One Coat 7 Universal adhesives, when both applied in the self-etching mode.

The dual-cured universal adhesive revealed a statistically significant lower bond strength compared to the self-cured adhesive in the etch-and-rinse mode. It was reported that adhesives that contained 10-MDP had inferior bond strength to acid etched dentin[43], as they showed weak bonding affinity to hydroxyapatite-depleted collagen[23]. In addition, in the etch-and-rinse mode, the One-Coat 7 Universal showed adhesive type of failure, which expressed 43.33% and mixed failure was 50%, while in Para bond, the adhesive type of failure was 16.67% and mixed failure was 80%. The lower percentage of adhesive failure, with the higher percentage of mixed failure of the de-bonded surfaces of Para bond adhesive might add further explanation to its significant higher bond strength compared to the One-Coat 7 universal adhesive in the etch and rinse mode.

One of the limitations of this study was that acid etching was used without saliva contamination of the sandblasted CAD/CAM surfaces. Nevertheless, it could be of clinical value to study the effect of saliva contamination and the different CAD/CAM composite's surface treatments on the bond strength of resin cements to enamel and dentin.

Based on the results of this study, the use of the dual-cured universal adhesive in its simplified one-step form (self-etching mode) could be of clinical advantage over its two-step form (etch-and-rinse mode). Self-etching mode eliminates the technique sensitivity associated with the rinsing step, make the clinical application steps simpler and the chair-side time can be reduced. Self-cured adhesive could be a good alternative to the dual-cured adhesive either in self-etching or etch-and rinse modes yet using of such adhesive in its simplified form (self-etching) could be of clinical significance.

CONCLUSION

Within the limitations of this study, the following conclusions could be suggested:

1. Sandblasting without any further surface treatments was enough to adjust the SBS of the CAD/CAM composite to dentin.
2. After sandblasting, both ultrasonic and acid etching surface treatments failed to restore the SBS. In this situation, their use will only lead to unnecessary increase in the clinical chair time.
3. No matter what application mode was applied; it was apparent that the use of both dual-cured universal and self-cured adhesive in their simplified forms (self-etching approach) was preferrable.

4. When using etch-and-rinse mode, the self-cured adhesive is more suitable than the dual-cured universal one.

Conflict of interest

The authors declare that they have no financial interest in the materials used in this study. This study was a part of Lina S. Marmoush's Master thesis in the partial fulfillment of the requirements of the Master Degree in Operative Dentistry, Faculty of Dentistry, Ain Shams university.

REFERENCES

1. Higashi M, Matsumoto M, Kawaguchi A, Miura J, Minamino T, Kabetani T, Takeshige F, Mine A YH. Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 1: Effects of sandblasting and silanization. Dent Mater J 2016;35:21-28.
2. Kawaguchi A, Matsumoto M, Higashi M, Miura J, Minamino T, Kabetani T, Takeshige F, Mine A YH. Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 2: Effect of ultrasonic and acid cleaning. Dent Mater J 2016;35:29-36.
3. Kawaguchi-Uemura A, Mine A, Matsumoto M, Tajiri Y, Higashi M, Kabetani T, Hagino R, Imai D, Minamino T, Miura J YH. Adhesion procedure for CAD/CAM resin crown bonding: Reduction of bond strengths due to artificial saliva contamination. J Prosthodont Res 2018;62:177-183.
4. Tinastepe N, Turkes E, Kazazoglu E. Comparative approach to analyse the effects of different surface treatments on CAD/CAM resin nanoceramics–resin composite repair bond strength. Biotechnol Biotechnol Equip 2018;32:142-149.
5. Da Rosa WL, Piva E, Da Silva A. Bond strength of universal adhesives: A systematic review and meta-analysis. J Dent 2015;43:765-776.
6. Salem AA, Nassif MS, El-Korashy DI. Durability of bond strength to dentin using two universal adhesives in different etching modes. Brazilian J Oral Sci 2021;20:1-15.
7. Elsayed AE, Kamel MA, El-askary FS. Adhesion strategy and curing mode of a universal adhesive influence the bonding of dual-cured core build-up resin composite to dentin. J Adhes Sci Technol 2021;35:52-62.
8. Oz FD, Ergin E, Canatan S. Twenty-four-month clinical performance of different universal adhesives in etch-and-rinse, selective etching and self-etch application modes in NCCL - a randomized controlled clinical trial. J Appl Oral Sci 2019;27:1-10.
9. Ritter AV, Ghaname E, Pimenta LAF. Dentin and enamel bond strengths of dual-cure composite luting agents used with dual-cure dental adhesives. J Dent 2009;37:59-64.
10. Sanares AM, Itthagarun A, King NM, Tay FR PD. Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites. Dent Mater J 2001;17:542-556.
11. Michaud PL, MacKenzie A. Compatibility between dental adhesive systems and dual-polymerizing composite resins. J Prosthet Dent 2016;116:597-602.
12. Tay FR, Pashley DH, Yiu CK, Sanares AM,, Wei S. Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part I . Single-step self-etching adhesive. J Adhes Dent 2003;5:27-40.
13. Moharam LM, Botros SA, El-Askary FS, Özcan M. Effect of polymerization protocol on the degree of conversion of photo- and dual-polymerized self-etch adhesives. J Adhes Sci Technol 2016;30:262-274.
14. Rathke A, Balz U, Muche R, Haller B. Effects of self-curing activator and curing protocol on the bond strength of composite core buildups. J Adhes Dent 2012;14:39-46.
15. Arrais CAG, Giannini M, Rueggeberg FA. Effect of sodium sulfinate salts on the polymerization characteristics of dual-cured resin cement systems exposed to attenuated light-activation. J Dent 2009;37:219-227.
16. Hayashi M, Ebisu S. Key factors in achieving firm adhesion in post-core restorations. Jpn Dent Sci Rev 2008;44:22-28.
17. Faria-e-Silva AL, Casselli DS, Lima GS, Ogliari FA, Piva E ML. Kinetics of conversion of two dual-cured adhesive systems. J Endod 2008;34:1115-1118.
18. Lopes FC, Palma-Dibb RG, Campi LB, Roselino RF, Gomes ÉA, Canevese VA, Pereira GK, Spazzin AO, De S-NM. Surface topography and bond strength of CAD – CAM milled zirconia ceramic luted onto human dentin : effect of surface treatments before and after sintering. Appl Adhes Sci 2018;6:1-11.

19. Yu H, Özcan M, Yoshida K, Cheng H ST. Bonding to industrial indirect composite blocks: A systematic review and meta-analysis. Dent Mater 2020;36:119-134.

20. Ishii R, Tsujimoto A, Takamizawa T, Tsubota K, Suzuki T, Shimamura Y MM. Influence of surface treatment of contaminated zirconia on surface free energy and resin cement bonding. Dent Mater J 2015;34:91-97.

21. Sofan E, Sofan A, Palaia G, Tenore G, Romeo U MG. Classification review of dental adhesive systems: From the IV generation to the universal type. Ann Stomatol 2017;8:1-17.

22. Vinagrea A, Ralhoa A, Ramosa N, Messiasb A RJ. Bonding performance of a universal adhesive : effect of hydrophobic resin coating and long-term water storage. Rev Port Estomatol Med Dent Cir Maxilofac 2019;60:96-103.

23. Elkaffas AA, Hamama HHH, Mahmoud SH. Do universal adhesives promote bonding to dentin? A systematic review and meta-analysis. Restor Dent Endod 2018;43:1-13.

24. Chen LAn pd., Suh I. Effect of hydrophilicity on the compatibility between a dual-curing resin cement and one-bottle simlified adhesives. J Adhes Dent 2013;15:325-331.

25. Michaud PL, Brown M. Effect of universal adhesive etching modes on bond strength to dual-polymerizing composite resins. J Prosthet Dent 2018;119:657-662.

26. Gutiérrez MF, Sutil E, Malaquias P, De Paris Matos T, De Souza LM, Reis A, Perdigão J LA. Effect of self-curing activators and curing protocols on adhesive properties of universal adhesives bonded to dual-cured composites. Dent Mater 2017;33:775-787.

27. Abe K, Asano T, Aida M, Nishiyama N KO. Effect of the amount of light energy transmitted through CAD / CAM resin block on bonding performance of four types of resin cement adhesive systems. Dent Mater J 2020;39:792-802.

28. De Menezes MJ, Arrais CA GM. Influence of light-activated and auto- and dual-polymerizing adhesive systems on bond strength of indirect composite resin to dentin. J Prosthet Dent 2006;96:115-121.

29. Myers ML, Caughman WF RF. Effect of restoration composition, shade, and thickness on the cure of a photoactivated resin cement. J Prosthodont 1994;3:149-157.

30. Swift Jr EJ, Brodeur C, Cvitko E PJ. Treatment of composite surfaces for indirect bonding. Dent Mater 1992;8:193-196.

31. Şişmanoğlu S, Turunç-Oğuzman R. Microshear bond strength of contemporary self-adhesive resin cements to CAD/CAM restorative materials: Effect of surface treatment and aging. J Adhes Sci Technol 2020;34:2484-2498.

32. Elsaka SE. Bond strength of novel CAD/CAM restorative materials to self-adhesive resin cement : The effect of surface treatments. J Adhes Dent 2014;16:531-540.

33. D'Arcangelo C, Vanini L. Effect of three surface treatment on the adhesive properties indirect resin composite restoration. J Adhest Dent 2007;9:319-326.

34. Kimyai S, Oskoee SS, Mohammadi N, Rikhtegaran S, Bahari M, Oskoee PA VH. Effect of different mechanical and chemical surface treatments on the repaired bond strength of an indirect composite resin. Lasers Med Sci 2015;30:653-659.

35. Nishigawa G, Maruo Y, Irie M, Maeda N, Yoshihara K, Nagaoka N, Matsumoto T MS. Various effects of sandblasting of dental restorative materials. PLoS One 2016;11:e0147077.

36. Nunes TG, Erhardt MC, Toledano M OR. One-step self-etching adhesive polymerization : Influence of a self-curing activator. J Dent 2009;37:616-621.

37. Jain G, Rajkumar B, Boruah LC BR. Comparative evaluation of shear bond strength of a dual-cure core build-up composite bonded to dentin using three different self-etch dentin bonding systems - An in-vitro study. J Dent Spec 2020;6:160-165.

38. Moszner N, Salz U ZJ. Chemical aspects of self-etching enamel-dentin adhesives: A systematic review. Dent Mater 2005;21:895-910.

39. South WUD, Jordan S. Safety data sheet. 2017:1-8.

40. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P VMB. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials 2007;28:3757-3785.

41. Poggio C, Beltrami R, Scribante A, Colombo M CM. Shear bond strength of one-step self-etch adhesives: pH influence. Dent Res J 2015;12:209-214.

42. Silva MH, Junior S, Carneiro KG, Lobato MF, Silva PD, De Góes M. Adhesive systems : Important aspects related to their composition and clinical use. J Appl oral Sci. 2010;18:207-214.

43. Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M T-MA. State of the art etch-and-rinse adhesives. Dent Mater 2011;27:1-16.