

## **BONDING CAPACITY OF A UNIVERSAL ADHESIVE IN DIFFERENT ETCHING MODES TO CARIES AFFECTED DENTIN PREPARED BY TWO CARIES EXCAVATION TECHNIQUES**

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### **ABSTRACT**

**Objectives:** The aim of the present study was to evaluate and compare the microtensile bond strength ( $\mu$ TBS) and resin penetration into caries affected dentin (CAD), of a universal adhesives applied into two different etching modes (i.e. self-etch or etch-and-rinse), after caries excavation with two methods, Round tungsten carbid bur CB or Carisolv chemomechanical caries removal (CMCR). **Materials and Methods:** Fourty human third molar teeth with moderate occlusal carious lesions were collected. The selected teeth were cut parallel to the occlusal surface of the tooth to expose the carious lesion. Teeth were divided into two groups (n=20) relative to the caries excavation methods i.e. Group1: Carisolv CMCR and group 2: round (CB). Each group was subdivided into two subgroups (n=10) relative to the applied adhesive mode i.e. subgroup a: the adhesive system was applied in a self-etch mode (SE), while in subgroup b: the adhesive system was used in the etch & rinse mode (E & R). Following the application of the adhesive, Filtek Z350 XT resin composite was applied incrementally onto the CAD. The bonded specimens were thermocycled for 5000 cycles. The restored teeth were sectioned longitudinally to obtain bonded beams for  $\mu$ TBS. Beams were mounted into the universal testing machine. Bond strength, for each subgroup, was calculated and statistically analyzed. After debonding the beams, the fracture surfaces were examined under stereomicroscope to detect the failure modes. One beam, from the area of CAD, from each subgroup was selected for SEM examination. **Results:** Two-way ANOVA indicated high significances for caries excavation and adhesive modes factors ( $p < .0001$ ). The highest mean value of  $\mu$ TBS was recorded with CMCR, bonded with E & R mode, followed by CB method, bonded with E & R mode, CMCR bonded with SE and CB and bonded with SE adhesive; respectively. Tukey's test displayed that there is no significant difference in the  $\mu$ TBS between the groups bonded with E & R adhesives, while CB method bonded with SE mode showed significant decrease in the  $\mu$ TBS value ( $p < 0.05$ ), with all test groups. All the tested groups showed cohesive failure mode in dentin rather than adhesive. In contrast, CB excavation method with SE mode displayed increased adhesive mode of failure rather than the cohesive and mixed modes. Removal of the caries with both methods and bonding with E & R adhesive resulted in relatively thin hybrid layer, but thicker with CMCR, with moderate distribution of resin tags. SE mode of adhesion showed a relatively thick hybrid layer with short conical shaped resin tags. The dentinal tubules were at most occluded with smear blugs and calcific precipitates. **Conclusions:** Based on the limitation of this study, the results showed that the use of Carisolv CMCR does improve the  $\mu$ TBS of universal adhesives to CAD, either in SE or E & R modes.

**KEYWORDS:** micro-tensile bond strength, caries excavation techniques, universal adhesives.

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## INTRODUCTION

Dental caries is one of the most prevalent problematic disorders, dramatically resulted into tooth loss. Histopathological studies led to recognition of two important layers of dentinal caries. The outermost layer is called the caries-infected dentin (CID), with non-remineralizable necrotic collagen matrix, while the inner layer, caries-affected dentin (CAD), with reversibly denatured collagen.<sup>1</sup>

In the past, a standard protocol of caries treatment is the complete removal of all the caries-infected/affected tissues, till reaching a sound tooth structure. At present, clinicians generally agree the fact that the main objective of caries treatment is to excavate the infected dentin layer, leaving the affected layer of dentin intact, to remineralize if the acid attack is removed.<sup>2</sup> Therefore, with developments in equipments and materials, there is a substantial shift in caries treatment from the destructive surgical model, drill and fill, to the conservative medical models, including the minimal tooth preparation.<sup>3,4</sup>

For minimal tooth preparation, various caries excavation techniques have been developed including chemomechanical caries removal (CMCR) technique.<sup>2</sup> Banerjee et.al, in a review, determined that other than the rotary cutting instruments, CMCR methods are the most efficient method for removal of dentin caries. Chemomechanical caries removal technique is an excellent example of noninvasive model, based on the hand excavation of infected dentin after softening with the aid of a chemical agent.<sup>4</sup>

Historically, CMCR was introduced in the 1970s through attempts to use different chemicals such as sodium dodecyl sulfate, ethylene diamine tetra-acetic acid (EDTA)<sup>5</sup> and collagenase.<sup>6</sup> Most of these techniques are considered time consuming with less effectiveness. Entering 1972, a solution

of 5% NaOCl was used for caries removal, but it has no selectivity as it act on both caries-affected and caries-infected dentine.<sup>7</sup> In 1976, Goldman launched GK-101, the first true sodium hypochlorite based system.<sup>8</sup> GK-101 was prepared by mixing 6% NaOCl with 0.05% N-monochloroglycine, which is a solution of sodium chloride, sodium hydroxide and glycine. Modifications of GK-101 resulted in introduction of the Caridex system, which contains N-monochloro D, L-2-aminobutyrate (NMAB, GK-101E), developed by replacing glycine by amino-butyric acid.<sup>9</sup> This system was considered the first true chemo-mechanical technique for caries removal, as light abrasion of the softened, by NMAB (GK-101E), carious dentin surface with the applicator tip was required. Several studies have indicated that the ability of both GK-101 and Caridex to remove carious dentin in permanent teeth was limited.<sup>7,10-12</sup>

Later on, in 1998, Medi Team, Sweden evolved a gel based system, Carisolv, as the latest version of NaOCl-based chemo-mechanical caries removal systems. Specially designed non-cutting hand instruments, should be used with this system to abrade the softened carious dentine surface.<sup>13</sup> Although Carisolv system has the same mode of action as Caridex, three different amino acids (glutamic acid, leucine and lysine) replaced monoaminobutyric acid, to enhance selectivity on the carious dentine.<sup>13</sup> Carisolv is marketed into two separate solutions, which are mixed just before application. Solution 1 contains three amino acids (leucine, lysine, glutamic acid) and NaOH, NaCl in purified water; while solution 2 contains 0.5% NaOCl.<sup>14</sup> The specially designed instruments, with non-cutting end, for Carisolv system improve the caries excavation efficacy and keep maximum preservation of the remaining caries-affected dentine, by excavation of caries through a scraping movement.<sup>13</sup> This movement cannot be provided by conventional

excavators as they only cut the dentine in a single direction by a scooping motion. The mode of action of CMCRs is by chlorination of the denatured collagen and cleavage by oxidation of glycine residue, which results in easy removal of friable collagen fibril. Based on that, the main advantage of this technique is its ability to specify an excavation end point, when the chemical agent applied into the cavity become clear with no turbidity.<sup>4</sup>

After caries excavation, a precise sealing of the prepared cavity is mandatory to reduce the nutrition supply to the residual bacteria.<sup>15,16</sup> Developments in dental adhesives are directed towards improving bond strength to dentin, ensuring restoration longevity and preventing caries recurrence.<sup>17</sup> In this direction, a new category of adhesive systems, with chemical adhesive potential, are widely marketed, to improve adhesion of ultra-mild adhesives to dentin. Vitrebond co-polymer is an ingredient found in several commercial dental adhesives, that can form a strong ionic bond between carboxylic groups (COO-) of the polyalkenoic acid and calcium in hydroxyapatite in dentin.<sup>18-20</sup> 10-MDP monomer was also incorporated in adhesives, as it can bond ionically with calcium in hydroxyapatite and form a strong 10-MDP-calcium salt, which is hydrolytically stable.<sup>21,22</sup> In 2012, multimode “universal” adhesives were introduced to markets, with chemical adhesion ability. These versatile adhesives could be applied in a total-etch, self-etch and selective etching modes, with manufacturers' claims that it could replace all previous strategies of adhesion.<sup>20,23</sup> In general, to overcome the permeability of adhesives, all the universal adhesives are more hydrophobic than previous self-etch adhesives. Little is known about the bonding capacity of multimode adhesives on caries-affected permanent dentin, excavated using carisolv chemomechanical caries removal technique.<sup>24-27</sup>

The goal of this study was to compare the bonding capacity of a universal adhesive, in self-etch and etch&rinse modes, bonded to dentin after caries removal by chemomechanical method (Carisolv™) and conventional method (airotor) in freshly extracted permanent molars. The null hypotheses are: 1) there is no difference between the  $\mu$ tensile bond strength of a universal adhesive, in self-etch and etch&rinse modes to caries-affected dentin, and 2) methods of caries removal have no effect on the microtensile bond strength of universal adhesives to the caries-affected dentin.

## MATERIALS AND METHODS

### Materials

One chemomechanical caries removal system, Carisolv, and an airotor conventional method, round tungsten carbide bur (CB), were employed in this study. One type of resin composite Filtek Z350 XT was used, with one type of adhesive, Scotchbond Universal adhesive with the corresponding etchant, Scotchbond etchant, for etch & rinse mode (E & R). All descriptions of the materials are represented in Table 1.

### Teeth selection

Human maxillary and mandibular third molar teeth with moderate occlusal carious lesions, scheduled for extraction because of periodontal reasons, were collected. The attached debris were cleaned using ultrasonic scaler (woodpecker dental ultrasonic DTE D1). The teeth were radiographed to confirm extension of caries upto the middle third of dentin. Forty teeth were selected and stored in distilled water, at  $4\pm 0.1^{\circ}\text{C}$ , until use in no more than two weeks of extraction. The selected teeth were cut parallel to the occlusal surface of the tooth, through the deepest part of the occlusal fissure, to remove enamel and expose the carious dentin.

Table (1) Materials used in the study

Materials	Composition	Manufacture
Carisolv™	gel composed of sodium hypochlorite 0.5% and 3 amino acids glutamic acid leucine and lysine Carisolv Instrument Kit	MediTeamDentalutveckling AB, Sweden
Scotchbond Universal Adhesive	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St. Paul, MN, USA
Scotchbond Universal Etchant	32% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminium oxide.	3M ESPE, St. Paul, MN, USA
Filtek Z350 XT Nano-composite Universal body Shade	Resin: Bis-GMA, UDMA, TEGDMA, Bis-EMA Filler: Silica particles, Zirconia, Silica/Zirconia clusters.	3M ESPE, St. Paul, MN, USA
Round carbide bur	H1SE.204.023	Komet, Dental-GbrBrasseler GmbH & Co, Lemgo, Germany

*The round bur is considered one of the variables in this study as it is the second method of caries excavation*

### Grouping of the samples

The cut teeth were divided into two main groups (n=20) relative to the caries excavation techniques i.e. Group1: Carisolv chemomechanical caries removal method (CMCR) and group2: round tungsten carbide bur (CB). Each group was further divided into two subgroups (n=10) relative to the applied adhesive mode i.e. subgroup a: the dentin adhesive was applied in a self-etch mode (SE), while in subgroup b: the adhesive system was applied in etch & rinse mode (E & R).

#### *Teeth preparation*

In group 1, caries was excavated using Carisolv system following the manufacturer's instructions. The two components of carisolv gel were mixed applied to the occlusal carious dentin, with the aid of hand instrument number 2, to dissolve the CID layer. After a waiting period of 30 seconds, to allow the chemical softening of caries, the gel becomes cloudy. Soften infected dentin was then gently excavated using Carisolv hand curette number 4. The excavated dentin surface was rinsed with a

copious amount of water, and dried with a dry air stream for visual inspection. A thin layer of caries detector dye (Kurary, Medical Inc, Tokyo, Japan) was gently placed on excavated dentin for 10 seconds to confirm complete removal of the CID. If the detector dye indicates presence of CID, the procedure was repeated until the solution remained clear without turbidity. After excavation, the surface was cleaned with a moist cotton pellet.

In group 2, A round tungsten carbide bur in a low speed handpiece was utilized to excavate the CID. The extent of excavation was determined by application of the Caries detector dye into the cavity for staining the CID. The excavated dentin was washed with water, and then air dried for further inspection of presence of caries. These procedures were repeated until complete removal of CID.

#### **Application of the restorative system**

In subgroups a: Scotchbond Universal adhesive was applied in a SE mode following the manufacturer's instruction. A thick layer of SE adhesive was applied to the dry and clean area of

CAD surface with rubbing action for 20 seconds. The adhesive was then dried with compressed air for about 5 seconds, to ensure complete vaporization of the solvent, then light cured, with a LED curing unite (LEDition, IvoclarVivadent, Germany) at a light intensity of 800 mW/cm<sup>2</sup>, for 10 seconds.

In subgroup b: Scotchbond adhesive was applied in an E & R mode following the manufacturer's instructions. Scotchbond Universal etchant gel, 32% conc., was applied on the dry and clean dentin surface for 15 seconds. The etched dentin surface was then washed with a stream of water and air dried, before application of the adhesive.

Following the application of the adhesive, a custom-made square metallic mold, with 5 mm high and 4 mm width, was used to build the composite over the bonded CAD surface. Two increments of composite each of 2±1 mm thickness were applied onto the CAD. Each increment was light activated separately for 20 seconds. After that, a blue colored permanent marker was used to paint the central square area of composite occlusally. This color coded marking allows for the selection of three central beams bonded to the CAD. All the Specimens were then stored in an incubator in a distilled water at 37±1°C for 24 hours.

### **Thermocycling**

Thermal fatigue is a method used for simulation of the thermal fluctuation that occurred in the oral cavity. It is assessed that about 10,000 thermal cycles correspond to 1 year of clinical function. The bonded specimens in each group were thermocycled (SD Mechatroniks thermocycler, Germany) at 5-55°C for 5000 cycles, with the dwell time of 20 seconds and the transfer time of 5seconds.

### **Microtensile bond strength test ( $\mu$ TBS)**

The restored teeth were longitudinally sectioned in bucco-lingual direction, then turned into 90°

clockwise rotation, for sectioning into the mesio-distal direction, along their long axis. Serial sectioning was done across the dentin/restoration interface using Isomet cutting machine. The obtained bonded sticks have 0.9±0.1 mm cross-sectional surface area. After cutting the sticks, the tooth was cut horizontally at the cemento-enamel junction to obtain beams. For estimation of the actual bond strength values, the cross-sectional area of each beam was verified with a digital calliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) to the nearest 0.01 mm. Each beam was composed of composite and dentin with adhesive at the interface. The central three beams were selected, to ensure that all the beams were bonded to the CAD surface.

For each tested subgroup, 30 beams were used for  $\mu$ TBS. Each beam was fixed with cyanoacrylate glue (Zapit; DAVA, Corona, CA, USA) in the central groove of Geraldeli's jig. The jig was utilized for mounting the beams into the universal testing machine (*Instron, MA, USA*). Tensile load was applied, at a cross-head speed of 0.5 mm/min, until bond failure was occurred. Bond strength was calculated in MegaPascal (*BluehillLite software, Instron, MA, USA*). The results were then subjected to statistical analysis, using two way ANOVA test, to be followed by post hoc Tukey test.

### **Failure mode**

After debonding the beams, the fracture surfaces were microscopically examined under stereomicroscope (Olympus, SZ61, Tokyo, Japan) at X40. The modes of failure were categorized into, adhesive failure if the fractured surface was completely present between the dentin and the bonding resin; cohesive failure if the debonding is completely present in the resin composite or in dentin; or mixed failure if the failures is present in adhesive/ cohesive modes.



### Scanning electron microscopic (SEM) Evaluation

One beam, from the area of CAD, representing each subgroup was selected for SEM examination. These beams were fixed in 2.5% glutaraldehyde in phosphate-buffered solution 0.1 M for 24 hours. The fixed beams were dehydrated in ascending grades in ethyl alcohol and hexamethyl disilazane. The specimens were sputter-gold plated, and the interfaces were examined with SEM. The entire interface was scanned, then the most representative aspect was photographed at 1.000× and 1500× initial magnification.

## RESULTS

### Microtensile bond strength ( $\mu$ TBS)

The obtained data were tabulated and subjected to Kolmogorov-Smirnov test, where values in each group showed normal distribution. Then these values were subjected to ANOVA (two-way) test, followed by Tukey post hoc test. Two-way ANOVA indicated significant differences for caries excavation and adhesive modes factors ( $p < .0001$ ) (table 2). Mean values and standard deviations of  $\mu$ TBS for the two caries excavation methods with the two

adhesive modes tested are tabulated in Tables 3. The highest mean value of  $\mu$ TBS was recorded with CMCR, bonded with E & R mode (27.061), to be followed by CB method, bonded with E & R mode (26.211) and CMCR bonded with SE (25.663); respectively. The least value of  $\mu$ TBS was recorded with the subgroup prepared with CB and bonded with SE adhesive (22.345). Tukey post hoc test displayed that, with E & R adhesive mode  $\mu$ TBS to dentin surface prepared with CMCR was higher than with CB, but with insignificant values. For CMCR method, E & R adhesive mode showed significant increase in the  $\mu$ TBS in comparison with SE mode, while CB excavation method bonded with SE mode showed significant decrease in the  $\mu$ TBS value ( $p < 0.05$ ), when compared with all test groups.

### Failure modes

Failure mode distribution, either adhesive, cohesive or mixed, for the different caries removal methods and adhesive modes were presented in table 4. Etch and rinse mode of adhesion showed cohesive failure in dentin rather than adhesive and mixed failures, regardless the excavation method. For SE adhesive, CMCR revealed equal adhesive

TABLE (2) Two-Way ANOVA test Results (Both factors significantly influenced the results)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	127.932 <sup>a</sup>	3	42.644	77.349	.000
Intercept	25644.096	1	25644.096	46514.183	.000
Method	43.431	1	43.431	78.776	.000
Adhesive	69.274	1	69.274	125.652	.000
Method * Adhesive	15.228	1	15.228	27.620	.000
Error	19.847	36	.551		
Total	25791.876	40			
Corrected Total	147.780	39			

a. R Squared = .866 (Adjusted R Squared = .855)

and cohesive failures, higher than the mixed failure. In contrast, CB excavation method with SE mode displayed increased adhesive mode of failure rather than the cohesive and mixed modes.

**Scanning electron microscopy**

SEM photo micrographs of CAD dentin after CMCR and CB caries removal are shown in Figure 1.(A-D), and bonding of a universal adhesive in the E & R and SE modes. After removal of the caries with CB and bonding with E & R adhesive, the resin/dentin interface showed a relatively thin hybrid layer

with moderate distribution of resin tags with varied lengths (original magnification 1000X). The same observations were seen in the teeth prepared with CMCR and bonded with E & R mode, except that the hybrid layer was thicker (original magnification 1500X). Resin/dentin interfaces prepared with either CB or CMCR, and bonded SE mode showed relatively thick hybrid layer with short conical shaped resin tags. The dentinal tubules were mostly obturated with smear blugs and calcific precipitates. Some areas of open tubules were also observed (original magnification 1500X).

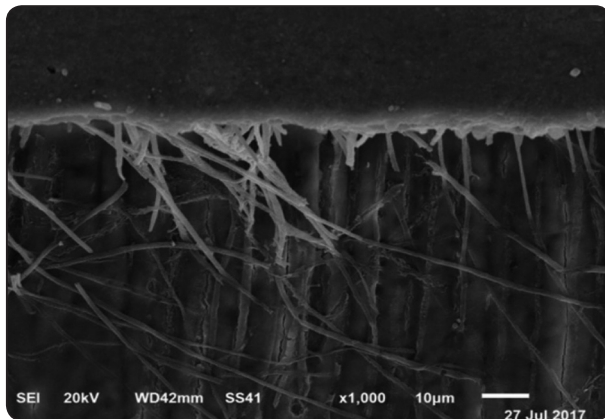
TABLE (3) Tukey post Hoc Multiple comparison test results of caries excavation methods and adhesive modes

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
CB_E&R	10	26.21 <sup>A,B</sup>	.98289	.31082	25.5079	26.9141	24.46	27.45
CB_SE	10	22.35 <sup>C</sup>	.53198	.16823	21.9644	22.7256	21.70	23.21
CMCR_E&R	10	27.06 <sup>A</sup>	.84928	.26857	26.4535	27.6685	25.73	28.11
CMCR_SE	10	25.66 <sup>B</sup>	.48468	.15327	25.3163	26.0097	24.91	26.23
Total	40	25.32	1.94659	.30778	24.6974	25.9426	21.70	28.11

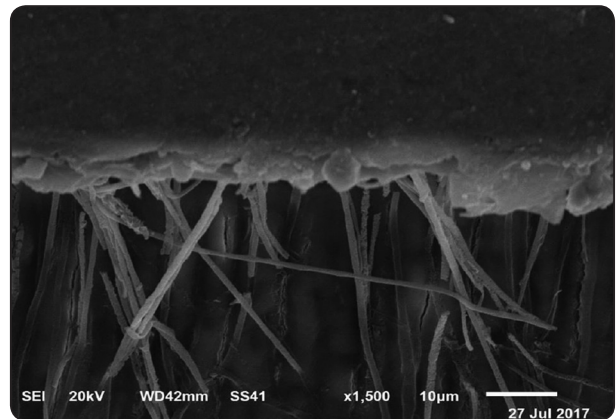
*Different superscript letters indicate significant differences between pairs, with A is the highest value. (Tukey's, P<0.05)*

TABLE (4) Percentage distribution of failure modes

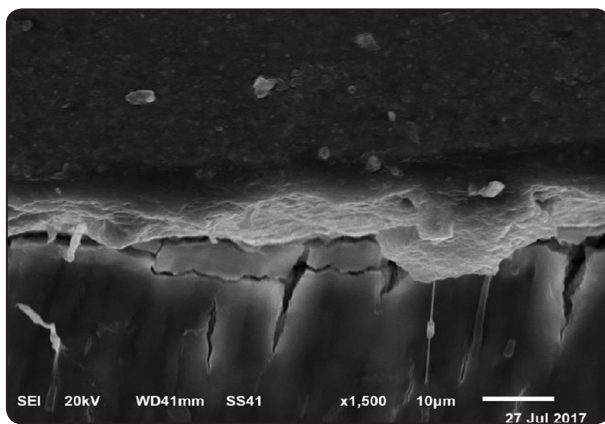
Caries excavation mode	Adhesion mode	Mode of failure			
		Adhesive	Cohesive in dentin	Cohesive in composite	Mixed
Carisolv	SE	40	40	-	20
	E & R	30	40	10	20
Carbide bur	SE	60	30	-	10
	E & R	30	40	-	30



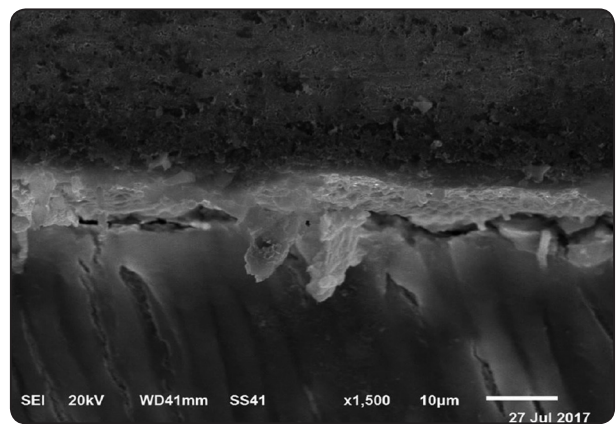
Caribide Bur caries excavation with E &amp; R adhesive mode



Carisolv caries excavation with E &amp; R adhesive mode



Caribide Bur caries excavation with SE adhesive mode



Carisolv caries excavation with SE adhesive mode

Fig. (1) Composed figure (A-D) of SEM photomicrographs of resin/CAD dentin after CMCR and CB caries removal, and bonding of a universal adhesive in the E & R and SE modes. CB with E & R mode, a relatively thin hybrid layer with moderate distribution of resin tags with varied lengths (A) (original magnification 1000X). CMCR with E & R mode, slightly thicker hybrid layer (B) (original magnification 1500X). Either CB or CMCR with SE mode, relatively thick hybrid layer with short conical shaped resin tags. Mostly occluded dentinal tubules with smear blugs and calcific precipitates. (original magnification 1500X).

## DISCUSSION

Recently, developments in the restorative field are directed towards the minimal invasive approach, to conserve healthy tooth structures. One of the most interesting approaches is the chemomechanical caries removal methods. Carisolv system is considered a promising method in this direction, as it's capability for caries removal is believed to be comparable to the conventional airtor methods.<sup>4</sup> Although several studies<sup>4,11-14,24,25,28,29</sup> approved the efficiency of the CMCR method as a conservative

method of caries removal, the influence of different adhesive strategies, with CMC, is still questionable. Bonding ability of different caries removal techniques gives a good impression about changes affect the surface characteristics of dentin, after removal with different methods.

In the present study, the obtained results displayed that the significant least values of  $\mu$ TBS were recorded with samples prepared with CB and bonded with SE adhesive. These results may be due to twofold causes, including the nature of cut CAD



and the acidic nature of the adhesive system used. With the progress of caries, most of the dentinal tubules' lumen in CAD undergo continuous mineral deposition, resulting in occlusion of the dentinal tubules with intratubular rhombohedral mineral crystals,<sup>30,31</sup> These crystals, magnesium substituted beta-tricalcium phosphate, form the whitlockite, which is less soluble than hydroxy-apatite and more acid resistant. Moreover, permeability of CAD decreases due to dentinal tubules occlusion, with the smear blugs.<sup>32</sup> The mineral deposits, whitlockite, and impaired dentin permeability interfere with the infiltration of resin monomer peripheral to the tubules as well as resin tag formation. In addition, smear layer of CAD is obviously different, in both chemical and morphological composition, from that of normal dentin, because CAD is partially demineralized<sup>33</sup>, the resulting smear layer is thicker, with increased organic contents, compared with that of normal dentin.<sup>34</sup>

Although the CB excavation is a widespread method in the dental clinics, it tends to produce overzealous excavation. A homogenous thick smear layer, with increased organic content, is left with a uniform roughness and occluded dentinal tubules with smear plugs.<sup>35</sup> In case of SE mode of adhesion, all of the abovementioned conditions in conjunction with its mild acidity, resulted in decreased  $\mu$ TBS.<sup>36</sup> The mild acidic monomer, PH=3, present in the SE adhesive failed to penetrate through the thick smear layer, developed during CB excavation, of CAD to etch the covered dentin surface and remove dentin bulges.<sup>37</sup> All the previous reasons led to adhesion to the smear layer rather than the underlying dentin, with formation of hybridized smear layer. Conversely, while smear layer has been shown to reduce bonding capacity of SE adhesives, it does not interfere with E & R adhesive capacity, as etchant acid tends to completely remove the smear layer and its bluges.<sup>36</sup> Although phosphoric acid etching might be overly aggressive to the partially demineralized CAD, this acid may solubilize the acid resistant

whitlockites within the lumens of caries-affected tubules, leading to increase the lateral penetration of the adhesive monomer.<sup>38</sup> In the present study E & R adhesive mode provided improved  $\mu$ TBS values, compared with the SE mode of adhesion, regardless the method of caries removal. When dentin/adhesive interface in this study was examined by SEM, caries removal with CB with E & R adhesion mode left a relatively thin hybrid layer with moderate distribution of resin tags with varied lengths, while removal with CMCR and E & R adhesion mode gave slightly thicker hybrid layer. That is because CB over-removes CAD to reach a relatively sound dentin. These results are partially disagreed with Wagner et al.,<sup>39</sup> who postulated that although application of universal adhesives after acid-etching does improve penetration to dentin, it does not affect their bond capacity and durability. Follak et al.,<sup>27</sup> results are conflicting with this study, as they stated that the etching mode does not influence the bonding of multi-mode adhesives to either sound or artificially CAD. They considered that the dentin condition has the only great influence on the immediate performance of multi-mode adhesives applied.

The highest values, in this study, were recorded with CMCR bonded with E & R adhesive, to be followed by CB caries excavation method with E & R mode of adhesion, and CMCR bonded with SE mode. After caries excavation with CMCR method, the chemical and micromorphological composition of dentin does not pose significant alterations. The calcium/phosphorus content and hardness value of CAD remain comparable with sound dentin.<sup>40,41</sup> Scanning Electron Microscopic analysis of the excavated dentin surface showed that NaOCl in Carisolv has a role in smear layer removal, leaving naked CAD surface, whereas, dentin surfaces excavated with CB showed a dentin surface covered with a thick smear layer.<sup>42</sup> Thereby, the depth of penetration and a thicker hybrid layer observed, are due to this rough surface with open dentinal tubules

and increased interfibrillar spaces.<sup>43</sup> This condition enhances the interaction between the resin adhesive and dentin surface. In addition, residual sodium hypochlorite, present after removal of in carisolv, may be partially ionized to solvated  $\text{Na}^+$  and  $\text{OCl}^-$  ions. At a pH of about 4, such as in the ultra-mild acidic monomer, the nonionized hypochlorous acid is formed, following the reaction:  $\text{ClO}^- + \text{H}^+ \rightleftharpoons \text{HClO}$ .<sup>44</sup> This weak acid may deproteinize smear layer, eliminating hybridized smear layer. This may enhance bond quality of dentin to the multi-mode adhesives and prevent formation of reticular nanoleakage at the adhesive/dentin interface.<sup>45</sup> It was observed that excavation of dentin with CMCR method increases its surface energy with higher wettability, and leaves the dentinal collagen intact, although damaging the odontoblastic processes does occur.<sup>46,47</sup>

Several studies<sup>24,48-50</sup> are in agreement with this study, and approved Carisolv CMCR method to improve bonding to CAD. On the contrary, Yildiz E et al.,<sup>51</sup> found that bonding to dentin surfaces excavated with CMCR method resulted in slight decrease in the  $\mu\text{TBS}$  of resin composite, regardless the adhesive used either E & R or SE. These results disagree with that of the present study. This discrepancy may be referred to the difference in the substrate, as primary teeth were used in this study while the present study used permanent teeth. In the present study cohesive failure was recorded in CAD, in all the tested groups, except surfaces excavated with CB and bonded with SE adhesive where most of the failure mode is adhesive. This result may be attributed to the decreased dentin hardness and mineral content in CAD.<sup>52-54</sup>

A contemporary family of adhesives was introduced to markets, with versatile uses, known as multi-mode or universal adhesives. Scotchbond Universal Adhesive used in this study, is an ultra-mild multi-mode self-etch adhesive, containing methacrylate-modified polyalkenoic acid

copolymer and acidic 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer. The ultra-mild adhesives have two-ways of bonding with dentin, the micromechanical interlocking, due to *in situ* polymerization of the adhesive monomers infiltrated in the hybrid layer; and the chemical adhesion, due to ionic bond between the adhesive's acidic functional monomers and calcium in residual dentin hydroxyapatite (HAp). The chemical interaction of self-etch adhesives with calcium in HAp could be explained depending on the adhesion-decalcification concept.<sup>55</sup> This concept depends on that, all the acidic monomers present in self-etch adhesives form ionic bond with calcium in HAp, through the exchange of phosphate ion from HAp into the solution.<sup>56</sup>

Polycarboxylic acid decalcifies HAp, following the dissolution rate and stability of the formed salt. In the first phase, polycarboxylic acid ionically bonds to calcium in HAp. In the second phase, according to the diffusion rate calcium/acid complex into the solution, polyacrylic acid acts by two ways. The acid may either remain bonded to HAp surface with minimal decalcification capacity, or the calcium/acid may debond with substantial decalcification reaction.<sup>56,57</sup> If the acid remains bonded, a strong ionic interaction is created between the carboxylic groups in the copolymer of methacrylate-modified polyalkenoic acid and calcium in HAp.<sup>58,59</sup> In a study comparing bond strengths of Scotchbond Multi-Purpose with and without polyalkenoic acid copolymer in their composition, It was found that the primer containing polyalkenoic acid developed a high bond strength to CAD.<sup>60</sup>

10-MDP is an acidic monomer 10-MDP that can react with calcium in HAp to form a hydrolytically stable MDP/calcium salts,<sup>61,62</sup> through the assembled nano-layered interaction.<sup>22</sup> This complex bonding becomes the adhesive interface, which is more resistant to bond degradation by aging. Therefore, with CMCR, the adhesive system applied in both SE and E & R modes of adhesion performs well in

the term of  $\mu$ TBS, because bond stability depends mainly on the chemical interaction of the adhesive with the prepared dentin surface. The question persists is, if the thickness of the hybrid layer and length of resin tags contribute to long-term bond stability of the multi-mode self-etch adhesives. It is believed that although micromechanical interaction of the adhesive with dentin surface can provide immediate improved bond ability to CAD, chemical adhesion is thought to be the keyword of improving bond durability and preventing adhesive/interface degradation.<sup>21</sup>

The null hypotheses are totally rejected as  $\mu$ tensile bond strength of a universal adhesive, to caries affected dentin, in etch and rinse mode is higher than in self-etch and etch&rinse modes. In addition, method of caries removal does affect the microtensile bond strength of universal adhesives to the caries-affected dentin.

## CONCLUSIONS

Based on the limitation of this study, the results showed that the use of Carisolv CMCR does improve the  $\mu$ TBS of universal adhesives to CAD, either in SE or E & R modes. The reactions of Carisolv treated dentin surface with different adhesive strategies and systems should be evaluated in term of microtensile bond strength test. Further investigations are required to study the micromorphology of the hybrid layer and characteristics of resin/dentin interface following chemo-mechanical caries removal method. Conventional caries excavation method is still inferior in adhesives bonding to CAD.

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