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DENTIN BOND EFFICACY OF COMPOSITE RESIN RESTORATIONS USING RESIN INFILTRANT

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ABSTRACT

Aim: To evaluate the bond efficacy of direct resin composite restorations bonded by three different dentin adhesives an etch-and-rinse, self-etch and resin infiltrant (ICON) using microtensile bond strength testing and Analyze their failure mode.

Methodology: Thirty molar teeth were selected and thoroughly cleaned. Each tooth was vertically embedded into self-curing acrylic resin up to the level of the cervical line. The occlusal enamel of teeth was removed perpendicular to the long axis of teeth to expose flat dentin surface at a standardized depth. The teeth were divided into three main groups of 10 teeth each; using Etch and rinse adhesive system control group A1 (All-bond Universal, Bisco), self-etch adhesive system group A2 (All-bond Universal, Bisco) and resin infiltrant group A3 (Icon). Direct resin composite nano-hybrid universal composite (Filtek™ Z250 XT, 3M ESPE) cores were build up on the flat dentin surfaces treated with different adhesive systems. The restored teeth were subjected to thermo-cycling at 5°C and 55°C for 2000cycle. Then each tooth was mounted on the cutting machine, sectioned into a series of 1 mm thick. The sticks were stressed to failure under tension using Universal Testing Machine to record the microtensile bond strength. The fractured sticks were evaluated for bond failure mode under a stereomicroscope at 40X magnification to detect the mode of failure. The collected data were submitted to ANOVA and Tukey's test.

Results: Etch-and-rinse (A1) control group showed the highest significant mean Microtensile bond strength (MPa) followed by Self-etch adhesive (A2) followed by Resin infiltrant (ICON) (A3) at $p \leq 0.001$. The result of the stereomicroscopic examination revealed adhesive and cohesive failure mode in composite. The predominant mode of failure using resin infiltrant group A3 (Icon) and self-etch adhesive system group A2 group was adhesive failure with a higher percentage in resin infiltrant A3 (Icon) group 100%. On the other hand cohesive in composite is the predominant mode of failure using the Etch and rinse adhesive system group A1 80%.

Conclusion: The use of resin infiltrant (Icon) as an adhesive material impaired efficacy of the microtensile bond strength of direct resin composite to sound dentin .

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INTRODUCTION

The evolution of dentistry with new technology and techniques are applied for preservation of tooth structure [1]. Resin infiltration is a relatively new treatment material appeared in dentistry 2008 [2], and acts through a micro infiltration technology [3], postponing as long as possible the transition from initial demineralization to actual cavitations [4], giving back the characteristics similar to the adjacent sound enamel [5].

The concept of caries infiltration resinous material is to fill the gap between prevention and restoration [6]. It was first developed at the charite Berlin as a micro-invasive technique for management of initial caries lesion with marketing name ICON (DMG America Company, Englewood, NJ) [7]. The principle of resin infiltration Icon is depending on the high penetration capability into the given porous lesion through capillary forces [8].

The in-vitro study which conducted by Paris S et al [9] to assess the penetration co-efficient of the ICON® resin material in comparison to a commercially available adhesive has shown that the mean maximum penetration depth and penetration percentage was significantly higher for the infiltrant (ICON®) compared to the adhesive. Also Meyer et al [10] tested and compared the efficacy of three different etching gels in removing the surface layer. The etching gels that used were 37% phosphoric acid and two experimental HCl gels for varying applications times. The study demonstrated that 15% hydrochloric acid was more effective than 37% phosphoric acid in surface erosion and creating porosity to enable the infiltration .All those data were limited to the evaluation of resin infiltrant on enamel lesion only. Until the effect of resin infiltration in exposed dentin surface was tested by Paris et al [11], They assessed and evaluated the effect of caries infiltration technique on the infiltration patterns of proximal caries lesions differing in ICDAS (International Caries Detection and Assessment System) classification. They concluded that the resin infiltration penetrated deeply in all the demineralized parts, but no significant difference in percentage infiltration of demineralized enamel of various ICDAS codes, they also stated that Caries Infiltrant was a low viscosity resin capable of penetrating several μm into dentin.

Several in vitro studies were conducted on cavitated and non-cavitated proximal and smooth surface lesions to test and compare the infiltration pattern between available adhesive systems with Icon and measure shear bond strength on sound and demineralized enamel [12, 13, 14]. Also, clinical trials on non-cavitated proximal carious lesions extended up to the outer third of dentin, using caries infiltration in combination with non-operative procedure compared with non-operative measurement alone [15,16]. Of all the studies that had been done with resin materials' infiltration capacity and ability of those materials in prevention of secondary demineralization (secondary caries), some showed promising results [17, 18], and some concluded that further research was required [19, 20, 21]. A question arisen can we use Resin infiltrant as adhesive system?

Therefore this study was carried out to evaluate the effect of low viscosity resin (caries infiltrant) on dentin substrate compared with Etch and Rinse and Self-Etch adhesive systems on microtensile bond strength of direct resin composite.

MATERIALS AND METHODS

Materials which are used in the present study have been illustrated in table (1)

Sample preparation

Thirty molar teeth were selected for this study. All collected teeth were extracted for therapeutic reasons from patients of age group (35-45 years). The selected teeth were free of caries, cracks and showed no apparent hypoplastic defects. The selected teeth were thoroughly cleaned from calculus, tissue deposits, polished with pumice and rotating brush at conventional speed. The teeth were

TABLE (1) Materials used in the study.

Material	Content	Manufacture
Resin infiltrant (ICON)	Tetra-ethylene glycol di-methacrylate(TEGDMA), HEMA additive, Initiator Icon etch 15% hydrochloric acid gel Icon dry: solution ethanol 100%	DMG (America 242 south dean street, Engle wood, NJ 07631 www, dmg.america.com) Icon Etch, DMG, Hamburg, Germany). (Icon Dry, DMG, Hamburg, Germany).
All-bond Universal	Phosphate monomer MDP, Bis-GMA, HEMA, ethanol, nanofiller, initiator	Bisco; Schaumburg, IL www.bisco.com 1-800-247-
FiltekTM Z250XT (Nano hybrid composite)	Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA resin. Fillers were a combination of surface modified zirconia/silica and 20nm surface modified silica particles. The inorganic filler loading is 81.8% by weight (67.8% by volume) with a particle size of nm for silica and 0.1-10 microns for zirconia/silica.	3M ESPE, Dental products, lot#20030509 St.Paul, M N55144-1000 USA
Etching gel	37% phosphoric acid, 5% xanthium gum, and water,benzalkoium chloride.	(Etch-37 TM w/BAC) Pentron Clinical Technologies, LLC.

stored in saline solution at room temperature until the time of their use.

A specially fabricated cylindrical, split Teflon mould of 19mm height, 22mm external diameter and 17mm internal diameter was used for the fabrication of acrylic resin blocks. Each tooth was vertically embedded into self-curing acrylic resin* up to the level of the cervical line with their occlusal surface being parallel to the acrylic resin base. [22].

The occlusal enamel of teeth were removed perpendicular to the long axis of teeth, parallel to the acrylic resin base to expose flat dentin surface at a standardized depth. The occlusal tables were ground with a rotary grinding milling machine using #180-grit silicon carbide paper** under continuous water coolant to create a uniform thickness of smear layer. [22].

The teeth were divided into three main groups of 10 teeth each; according to the 3 materials used namely; Etch and rinse adhesive system***, self-etch adhesive system[2] and resin infiltrant****.

Experimental groups

Group A1 control (Etch & rinse adhesive)

The flat dentin occlusal surfaces were etched using 37% semi-gel phosphoric acid with Benzalkonium Chloride***** for 15 seconds, rinsed for 10 seconds, and blotted dry with absorbent sponge pellet leaving the dentin surface visibly moist. Two consecutive coats of adhesive system (All-bond Universal) were applied using a fully saturated brush tip and gently air-thinned for 5 seconds leaving a shiny surface and then polymerized with a light-emitting diode (LED) light curing unit for 20 seconds according to the manufacturer's instructions

* Acrostone Dental Factor, England
** Gamberini s.r.1, Via Della Bastia, Caslecchio Di Reno, Italy
*** All-bond Universal, Bisco
**** ICON
***** Etch-37 TM w/BAC

Group A2 (Self-etch adhesive)

The All-bond Universal was applied according to manufacturer instructions. Two coats of adhesive were applied, air dried and light cured for 10 s using light-emitting diode Light Curing unit.*

Group A3 (ICON) application

The flat dentin surfaces were etched with 15% hydrochloric acid gel* for 2 min and then rinsed with water for 30 s. The surface was dried with ethanol**, applied for 30 s. Then, the low viscosity Infiltrant resin*** was applied on the surface for 3 min with a sponge applicator. The Infiltrant was light-cured for 40 s. After light curing, the Infiltrant was applied again for 1 min and light-cured for 40 s

Direct nano-hybrid universal resin composite**** cores were build up on the flat dentin surfaces treated with different adhesive systems and resin infiltrant. This was done using Teflon mold (5mm x 5mm in diameter and thickness 4mm). Resin composite build up was done with insertion of two increments 2mm in thickness on dentin surface, then light cured for 20 s for each increment using (light-emitting diode (LED) Curing Light with irradiance of approximately 700 mW/cm^2*****

Thermo-cycling

The restored teeth were subjected to thermo-cycling in three water baths with different temperatures. The specimens were immersed at 5°C followed by 55°C for 20 seconds each, with an intermediary bath 37°C. The teeth subjected to 2000 cycles. [23]#

Microtensile bond strength testing

Each specimen was mounted on the cutting machine**, and sectioned into a series of 1 mm thick slabs under water coolant. The sectioning was performed using a diamond disc of 4" diameter x 0.3 mm thickness x 0.5"***. By rotating the tooth 90° and sectioning it lengthwise to obtain sticks of 1.0 mm^2 cross-section area (Fig.1). The two central sticks from each specimen were selected and their thickness was checked using a caliper [24].

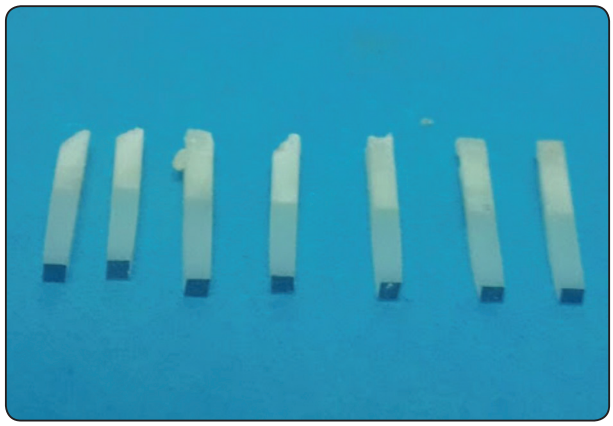

Fig. (1) A series of 1 mm2 thick sticks

A specially designed apparatus was used for the standardized serial sectioning of restored teeth in both buccolingual and mesiodistal directions to produce sticks of standardized cross sectional area. The sticks were stressed to failure under tension using Universal Testing Machine (Fig.2). The microtensile bond strength for each specimen was calculated in Mega Pascal (MPa) by dividing

* Elipar LED Curing Light; 3M/ESPE
** Icon Etch, DMG, Hamburg, Germany
*** Icon Dry, DMG, Hamburg, Germany
**** Icon Infiltrant, DMG, Hamburg, Germany
***** FiltekTM Z250 XT, 3M ESPE, Shade A3
Elipar LED Curing Light; 3MESPE
SD Mechatronik thermocycler THE-1100 Germany
** Bronwill, E. McGrath Inc
*** IPDB40305, MTI Corporation, Richmond, USA

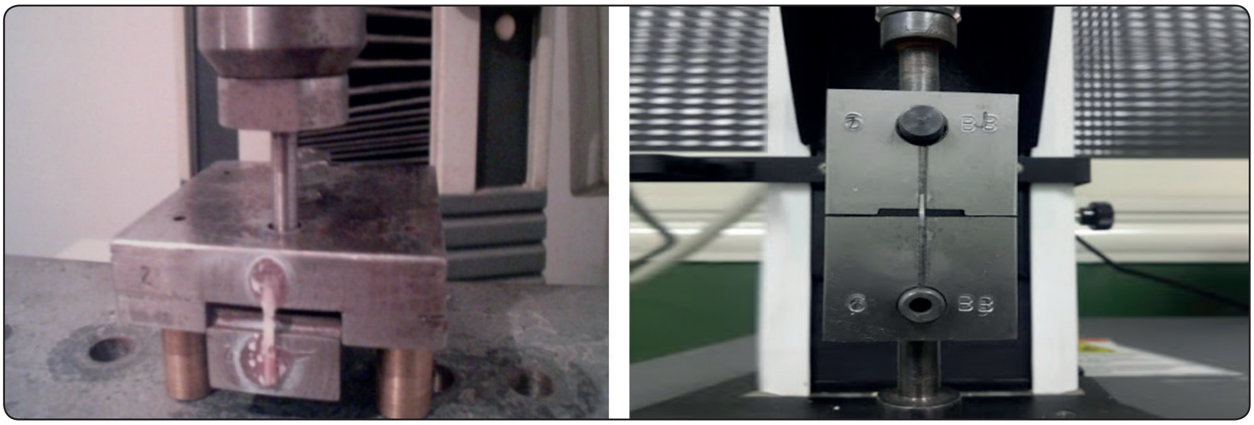

Fig. (2) Universal Testing Machine

the maximum force of fracture in Newton by the specimen's cross sectional area in mm^2 (F/A).

Mode of failure

The fractured sticks were evaluated under a stereomicroscope* at 40X magnification to detect the mode of failure**.** Failure modes were classified whether Failure Adhesive, cohesive or mixed [(25).] **Adhesive:** Failure on the adhesive/resin composite interface in more than 75% of the analyzed area.

Mixed: Failure without predominance.

Cohesive on the resin composite: Failure predominant on the resin composite in more than 75% of the analyzed area.

Statistical analysis

Data were presented as mean and standard deviation (SD) values. Regression model with one-way Analysis of Variance (ANOVA) was used in testing significance for the effect of different adhesive systems on mean microtensile bond strength. *Tukey's post-hoc test* was used for pair-wise comparison between the mean values when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Kruskal Wallis test used to compare between different tested groups followed by Mann Whitney U-test for pairwise comparison for failure mode. Significant level set at $p<0.05$. Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 24 for Windows

RESULTS

Microtensile bond strength (MPa)

The data in table 2 and figure (3) showed that, Etch-and-rinse group (A1) group have the highest significant mean Microtensile bond strength (MPa) at $p \leq 0.001$, followed by Self-etch adhesive group (A2) while Resin infiltrant (ICON) group (A3) recorded the lowest mean value.

Fractographic analysis

Frequency and Percentage (%) of the failure mode for different tested groups presented in table (3) and figure (4) showed that, Etch-and-rinse (A1) group have a significant difference compared to other groups for failure Mode at $p=0.03$. Etch-and-rinse (A1) group have the highest % of cohesive failure Mode 70%. While Resin infiltrant (A3) group have the highest % of adhesive failure Mode equal 100%

Fig 5, 6, 7, 8 and 9 showed photomicrographs of different Failure Modes for different tested groups.

* Olympus 220670; Tokoyo, Japan

TABLE (2) Mean and standard deviation (SD) for Microtensile bond strength (MPa) for different tested groups.

		Group			p-value
		Etch-and-rinse (A1)	Self-etch adhesive (A2)	Resin infiltrant (ICON) (A3)	
Microtensile Bond Strength (MPa)	Mean	25.86[a]	13.18[b]	2.04[c]	≤0.001*
	SD	8.42	4.32	0.97	

Means with different letter indicating a significance difference at p<0.005

****=significant, NS= Non-Significant***

Range for all groups (minimum and maximum bond strength) within table

TABLE (3) Frequency and Percentage (%) of Failure Mode for different tested groups.

		Group						p-value
		Etch-and-rinse (A1)		Self-etch adhesive (A2)		Resin infiltrant (ICON) (A3)		
		N	%	N	%	N	%	
Fracture mode	Cohesive (Composite)	7	70.0%	2	20.0%	0	0.0%	0.03*
	Adhesive	3	30.0%	8	80.0%	10	100.0%	
	Cohesive (Tooth)	0	0.0%	0	0.0%	0	0.0%	

****= Significant, NS=Non-significant***

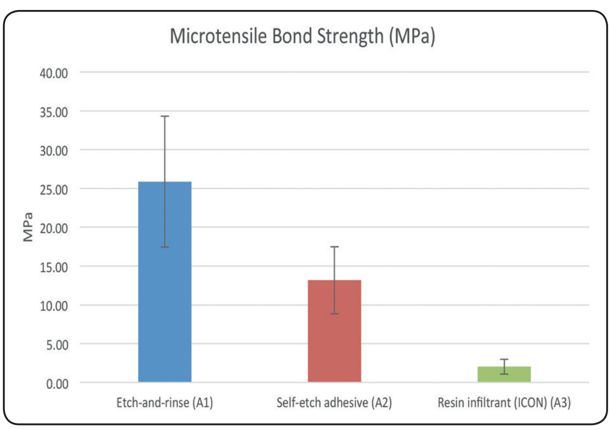

Fig. (3) Bar chart showing the mean Micro- tensile bond strength (MPa) for different tested groups.

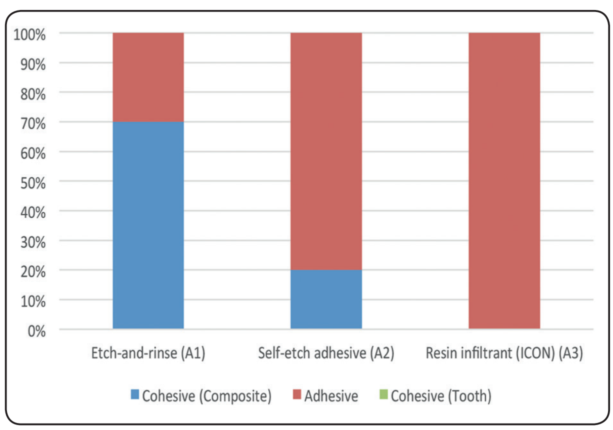

Fig. (4) Histogram showing the percentage of failure mode for different tested groups.

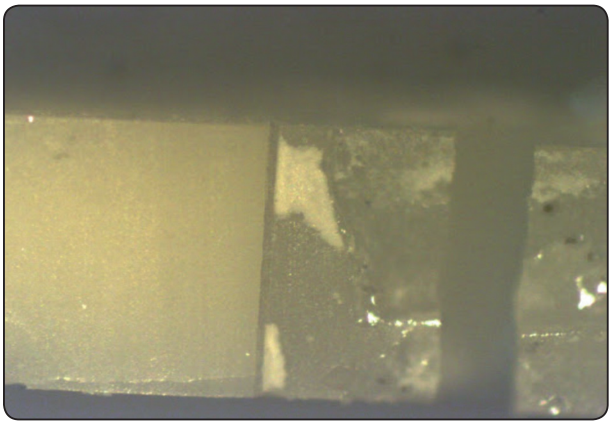

Fig. (5) Cohesive failure in composite in etch & rinse group

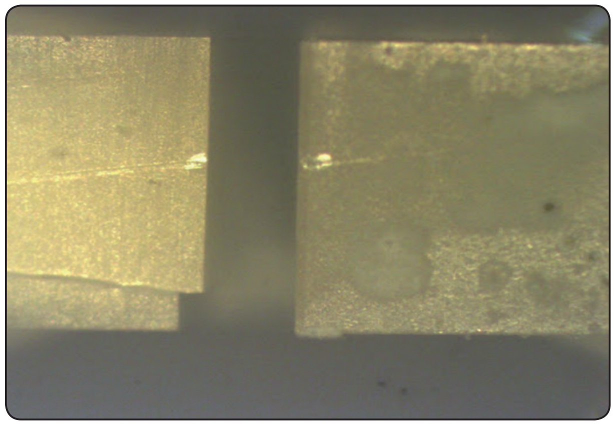

Fig. (6) Adhesive failure in etch & rinse group

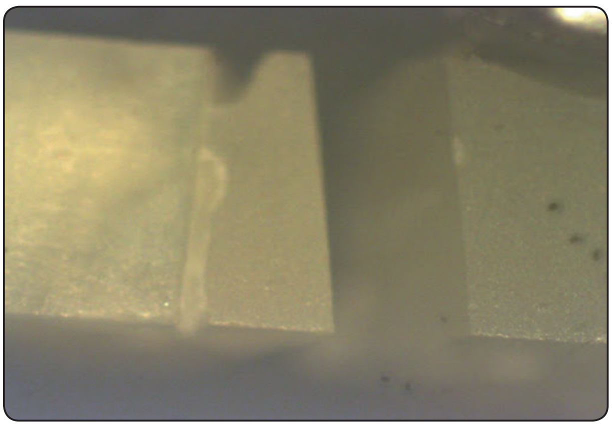

Fig. (7) Cohesive failure in composite in self-etch group

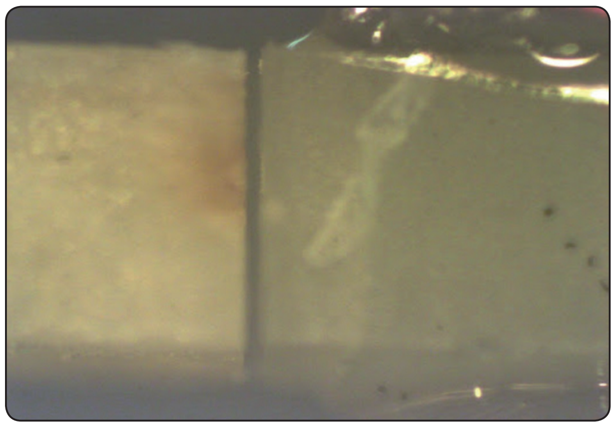

Fig. (8) Adhesive failure in self-etch group

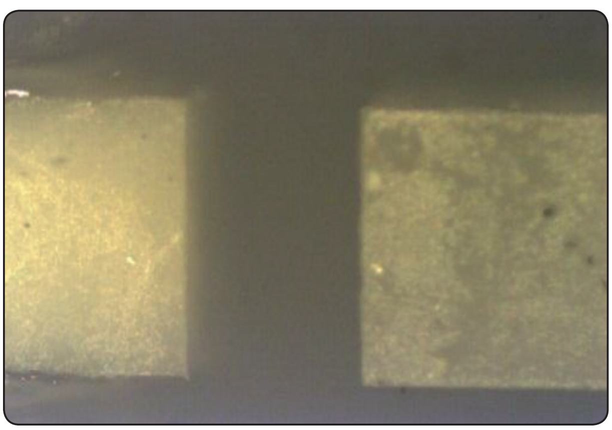

Fig. (9) Adhesive failure in resin infiltrant group

DISCUSSION

For many years, the application of resin to the tooth substrate, etched with phosphoric acid produced a well-sealed interface that is sufficiently strong to retain most of resin composite restorations [26]. However, more attempts to penetrate tooth substrate with adhesives or sealants through a micro infiltration technology [27], create a new concept in penetrating initial caries lesion with resins which has been established after many pilot studies at the University Of Kiel, Germany. They experimented various mixtures of resin materials such as TEGDMA, BisGMA and ethanol with various mixing ratios for many years [28]. They found that Low-viscosity resins were resulting in higher penetration coefficient and more rapid infiltration [29]. The newer product produce after all those studies was called ICON®, which consists of an Infiltrant composed of tetra-ethylene glycol dimethacrylate, additives and initiator, an acid conditioner to etch the enamel surface made of 15% hydrochloric acid, and ethanol [30] .

As it's a well-known that adhesion is the core of modern dentistry and the resin tag formation is the key factor of it [31]. The most effective adhesives that are used in most studies are the Etch and Rinse Adhesives, which provide excellent bond strength when compared to others. So, the fifth-generation dentin bonding agents are considered to be the 'accepted standard' against other adhesives [32]. In the current study we compared three different dentin adhesives an etch & rinse, self-etch using same adhesive system (All bond universal) and resin infiltrant (ICON) in their bond efficacy to direct resin composite restorations using microtensile bond strength testing and Analyze their fracture mode.

The results in this *in vitro* study showed resin infiltrant adhesion after thermocycling had the lowest significance difference micro- tensile bond strength when compared to E&R and SE groups. This result was in disagreement with Wiegand et al [33] and_Katkade et al [34] where they found that Icon resin infiltrant exhibits a very low viscosity, high surface tension to allow complete penetration of the resin into the lesion body, with penetration depth of 15% HCL etching more than twice (58 μm) that of phosphoric acid [35]. Also the present of ethanol in (ICON Dry) and in All bond universal used in etch&rinse and in S.E groups improve the efficacy of penetration of the hydrophobic infiltrant to get a well-defined, resin-infiltrated layer [36,37]. Recent studies have shown that it is possible to bond hydrophobic resin monomers to acid-etched dentin with a new technique called "ethanol wet bonding". The technique involves slowly replacing water within the demineralized collagen matrix with ascending concentrations of ethanol, allowing the latter to penetrate the collagen matrix without causing additional shrinkage of the interfibrillar spaces thus preventing phase separation of hydrophobic resin monomers [38, 39]. This disagreement could be due to that the resin infiltrant is formed mainly of TEGDMA a low viscous monomer easy to solute and deteriorate; it does not form a strong polymer into the dentin surface so thermocycling affect it. Xie C et al [40]testing the infiltration capability of resin infiltrant into micro-structure of dentin after application of primer and compare bond strength after and before thermocycling using different self-etch adhesives. They found a significant difference in bond strength after and before thermocycling for all groups; also found that the bond strength and the bonding durability of infiltrating resin were similar to that of self-etch Bond adhesive. This result was in disagreement with our result which may contributed to their using to primer in their study before application of resin infiltrant.

Paris et al. [41] attempted to test the infiltration pattern in cavitated and non-cavitated proximal lesions. It was concluded that under in vitro conditions, the infiltrant penetrates in most parts of the demineralized enamel but is not capable of filling up cavities and therefore the efficacy of the resin infiltration technique, particularly in lesions with larger cavitations, might be impaired [42,43,44].

On the other hand group A1and group A2 due to presence of specific functional monomers (10 MDP) in the E&R and self-etch adhesives which might interact chemically with the hydroxyapatite of the tooth, resulting in additional chemical bonding while in term might enhance bonding performance to dentin [45]. Group A2 of self-etch showed lowering in bond strength than control group this results are in accordance with earlier reports, which found that single-phase self-etching resins do not produce as good bond as etch-and-rinse adhesives [46]. Studies of such products have demonstrated the presence of water-rich phases that are detrimental to strength and stability [47].

The result of the stereomicroscopic examination revealed adhesive and cohesive failure mode in composite. The predominant mode of failure in ICON and SE groups was adhesive failure with a higher percentage in ICON group 100% followed by SE group 80%. Therefore, this area seems to be the weak link of the bond in resin infiltrant ICON as the high amount of TEGDMA in the resin infiltrant might increase the susceptibility to degradation compared to resins containing less TEGDMA. Moreover, exhibit inhomogeneity, probably as a result of polymerization shrinkage and polymerization stress of the resin, which might increase the risk of adhesive failure and affect bonding strength [48, 49]. However, the results of the current study concluded that the adhesion of the TEGDMA-based resin infiltrant affected in a way that the shear bond strength is significantly reduced compared to the adhesives containing less TEGDMA [50]. Also the predominant mode of failure in SE groups was adhesive failure values compared to the Etch & Rinse adhesives which showed cohesive failure in composite is the predominant mode of failure. This could be more logical due to that etching efficacy of Self Etch on dentin was significantly less intensive compared to phosphoric acid resulting in a less deep demineralization and more irregular etching pattern than conventional etching. [51, 52]

CONCLUSION

The use of a resin infiltrant alone as an adhesive material impaired efficacy of micro tensile bond and interfere negatively on the bond strength of direct resin composite to sound dentin. Suggestion of using ICON infiltrant resin with the adhesives either two step or one step self-etch adhesive systems

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