EGYPTIAN DENTAL JOURNAL

Vol. 64, 2433:2443, July, 2018

I.S.S.N 0070-9484

Fixed Prosthodontics, Dental materials, Conservative Dentistry and Endodontics

www.eda-egypt.org • Codex : 08/1807

INFLUENCE OF IMMEDIATE DENTIN SEALING TECHNIQUE ON THE INTERFACIAL QUALITY OF THE INDIRECT CERAMIC RESTORATION

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ABSTRACT

Purpose: To examine the microtensile bond strength and the micromorphology of ceramic restorations bonded to sealed dentin with different protocols.

Methods: Fifty rectangular cavities were prepared in the occlusal dentin surfaces. They were randomly divided into five groups(n=10) according to the dentin sealing protocols: Group DDS: delayed dentin sealing(control group); IDS1: immediate dentin sealing with removal of O_2 inhibited layer; IDS2: same as IDS1 but with dentin finishing immediately before the final cementation; IDS3: immediate sealing without removal of O_2 inhibited layer; IDS4: same as IDS3 but with final dentin finishing. Temporization was performed using a provisional material (Tempfil Inlay, Kerr) for two weeks. Lithium disilicate glass-ceramic blocks (IPS e.max Press: Ivoclar, Vivadent, Liechtenstein, Schaan) were machine milled to fit inside the prepared cavities and they were cemented in place using resin cement (Rely X Unicem, 3M ESPE). Half of the restored teeth from each group was thermocycled (SD Mechatronicks THE 100 thermocycler, Germany) 5000 cycles 5-55˚C with 30 seconds dwell time. Specimens were prepared for microtensile bond strength test and micromorphological analysis with Environmental scanning electron microscope (ESEM). Data was statistically analyzed using ANOVA and Tuckey's post hoc test(P=0.05). Bond reliability was checked using the Weibull analysis.

Results: Superior bond strength values (MPa) were evident in DDS group (30.56) and IDS2 (30.21) and the lowest value was in IDS3 (21.25). Weibull test confirmed that IDS2 revealed the most reliable bond even after thermocycling. All bond strength values decreased significantly after thermocycling. SEM results supported the bond strength findings.

Conclusions: Immediate dentin sealing with removal of oxygen inhibited layer together with finishing just before final cementation are the keys for reliable interfacial bond between the dentin and ceramic restorations.

KEYWORDS: Indirect-Ceramics-Dentin-Sealing-Bond strength-micromorphology

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INTRODUCTION

Successful restoration of the defected hard tooth structure dictates presence of a long lasting adhesive joint between the restoration and the prepared cavity walls especially the dentin wall. Biomimetic concept stated that the lost tooth structure should be restored biologically, mechanically and esthetically using a naturally imitating restorative materials. [1] These materials, either directly or indirectly fabricated, gained a great popularity due to the enormous advancements in the field of bondodontics and restorative materials. It is worth mentioning that ceramic is the best material that suits with the requirements of the biomimetic approach. [2,3]

Cavity preparation for indirect restoration to receive ceramic or even indirect resin composite restoration dictates eliminating of any undercuts. Therefore, the so provisional restoration suffers weak retention inside the prepared cavity. As a consequences of that situation; contamination of cavity walls and post-operative hypersensitivity occur. [4] Here, management of the tooth substrate during the provisionalization time has an important role in the success of the final restoration. It is well known that the freshly cut dentin surface is the optimal surface to bond with; however, it is not achievable in the case of indirect restoration. Management of such problem can be established by proper sealing of the freshly cut dentin by a bonding agent in a step termed immediate dentin sealing. [5-9]

Different variables have been studied to evaluate their influence on the outcome of this step including the dentin depth, type of bonding agent, interaction with provisional restoration, type of impression material and many other variables. [10] Although, the optimal technique for performing the dentin sealing is still controversial; some operators remove the oxygen inhibited layer from the cured bonding agent while others do not, some perform finishing for the sealed dentin immediately before cementation of the final restoration while others do not. [6,7,9,11-17] Therefore this study was conducted to evaluate the interfacial quality of the ceramic restorations and dentin sealed with different protocols. The null hypothesis stated that there was no significant difference in the microtensile bond strength and micromorphology of the ceramic restoration bonded to dentin sealed with either removal of oxygen inhibited layer or finishing before cementation or both.

MATERIALS AND METHODS

Materials, compositions and manufacturers are summarized in **Table 1**. A total of fifty extracted crack and caries-free human mandibular molars were selected for this study. All soft tissue remnants were removed from the extracted molars; they were scaled with hand instrument and stored in distilled water containing 0.2% sodium azide at room temperature for not more than 3 months, to keep them hydrated and prevent cracking during milling. [18,19] Teeth were vertically mounted inside acrylic blocks 2mm above the cervical line to facilitate their handling.

Specimen preparation:

Rectangular cavities (4 X 5 X 2 mm) were prepared in the mid coronal dentin after removal of the occlusal enamel using a model trimmer. The cavities dimensions were confirmed by a digital caliper (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan). The prepared specimens were divided into five groups (10 molars each) according to the dentin sealing time either delayed dentin sealing (DDS); IDS1: immediate dentin sealing with removal of O_2 inhibited layer; IDS2: same as IDS1 but with dentin finishing immediately before the final cementation; IDS3: immediate sealing without removal of O_2 inhibited layer; IDS4: same as IDS3 but with final dentin finishing. Half of each group (5 each) was thermocycled and the other half was not.

Delayed Dentin Sealing (DDS (Control group)):

Cavities in this group were covered with a provisional restoration material (Tempfil Inlay;

Kerr) and immersed in saline solution 48 hours. Following that delay, the provisional restoration was removed with an excavator and dentin was cleaned using airborne-particle abrasion (CoJet, 3M ESPE), the adhesive (Single Bond Universal adhesive, 3M ESPE) was applied, which was applied for 15 seconds with gentle agitation using a fully saturated applicator and gently air thinned for 5 seconds to evaporate solvent, then adhesive was light cured for 10 seconds using LED curing unit (Elipar S10, 3M, ESPE) at a light intensity of 1000 mW/cm2. Air-blocking barrier (glycerin Gel) was applied to polymerize the oxygen-inhibition layer with 10 seconds of additional light curing.

Immediate Dentin Sealing (IDS):

In the first group (IDS 1); using the same adhesive cavities were immediately bonded in the same manner used for delayed dentin sealing group and then isolated with petroleum gel to avoid any bonding with the subsequently applied provisional restoration. After 48 hours, the provisional restoration was removed and the ceramic block was cemented in its place. **In the second group (IDS 2);** same procedures were done as in IDS1 but after removal of the provisional restoration the sealed dentin was cleaned by airborne-particle abrasion (CoJet,3M ESPE) before final cementation. **In the third group (IDS 3);** polymerization of the adhesive was done without the application of an air-blocking barrier (glycerin jelly) and after removal of the provisional restoration the sealed dentin surfaces did not being finished before cementation of the ceramic block. **In the fourth group (IDS4);** same procedures were done as in IDS3 but with finishing of the sealed dentin before cementation.

Fabrication and preparation of ceramic blocks:

Lithium disilicate glass-ceramic restorations (IPS e.max Press: Ivoclar, Vivadent, Liechtenstein, Schaan) were used in this study. Dimension was standardized (4x5x4mm) were obtained by sectioning the blocks using water -cooled slow speed saw. The cut surfaces of the blocks were etched using <5% hydrofluoric acid gel (IPS Ceramic etching gel (IPS Ceramic Refill) Ivoclar, Vivadent, Liechtenstein,Schaan) for 20 seconds according to the manufacturer instructions and washed thoroughly with air/water spray for 30 seconds. They were then dried using compressed air and primed using a silane coupling agent (ESPE-Sil, 3M ESPE) for 60 seconds, then air dried before cementation. One coat of the used adhesive (Single Bond Universal adhesive, 3M ESPE) was applied and light cured for 10 seconds.

Cementation of the ceramic blocks

Self-adhesive resin cement (Rely X Unicem, 3M, ESPE) was gently dispensed directly into the prepared cavities and the ceramic blocks were seated in their corresponding places. A static load (1Kg for 5 minutes) was applied during block cementation using a specially designed cementation device. It consisted of two horizontal plates connected together with two rigid vertical rods. The upper plate had a central hole through which passed a vertical cylindrical steel bar with a flat disc-shaped lower end to rest on the specimen and a flat horizontal upper disc-shaped end to carry the load during the cementation procedure. Excess cement was removed immediately with a micro-brush and the exposed margins were covered with glycerin gel to ensure complete polymerization. The margins were cured with a LED curing unit for 20 seconds from the occlusal, lingual, mesial and distal directions each respectively.

Thermocycling of the specimens:

Half of the specimens (n=5) in each group were thermocycled using SD Mechatronicks THE 100 thermocycler (Germany) for 5000 cycles (5-55^{o} C) with 30 seconds dwell time.

Microtensile bond strength testing:

Specimens were sectioned along the buccolingual and mesiodistal planes using a diamond disk

(MTI Corporation, Richmond, CA, USA) in a low speed micro-slicing machine (Isomet, Buehler, Lake Bluff, IL, USA) under water-cooling, to produce beam-shaped specimens (bonding areas approximately 1 mm2). Centralized 3 to 4 specimens were taken from each cavity and mean value was calculated for each tooth. The beam specimens were attached with cyanoacrylate gel (Zapit; Dental Ventures of America, Corona, CA, USA) to the testing customized microtensile jig. This jig is designed to transmit tensile forces to the specimen purely without any torque and designed to fit the μTBS Instron Universal testing machine (Bisco Inc. Schaumburg, IL, USA). The tensile load was applied at a cross-head speed of 0.5/minute until specimen failure occurred. At this point the failure load in Newton was recorded. The bond strength was calculated as the ratio between the failure load and the beam area that was checked with a digital caliper before testing.

Micromorphological analysis:

Representative beam from each group were prepared to be investigated under Environmental scanning electron microscope (ESEM, Philips 505, Eindoven, Netherlands). They were pretreated using 6N HCl for 30s followed by 5% NaOCl for 60s before washing with distilled water then dehydration for 30s in 70% ethanol. Photomicrographs were taken at magnification 500X to evaluate qualitatively the dentin-ceramic interface.

Statistical analysis:

Data are explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. Microtensile bond strength (MPa) showed normal distribution so Two Way-ANOVA test was used to study the effect of different sealing protocols and Thermocycling on mean Microtensile bond strength (MPa). Tukey's post hoc test was used for analysis between the different groups.The significance level was set at $P \leq 0.05$.Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 24 for Windows. Weibull analysis was done to estimate the bond reliability with 95% confidence level using Excel 2010 for windows (Microsoft, Redmond,

TABLE (1): Specifications of the materials used in this study

Material	Description	Composition	Manufacturer
Single Bond Universal	Universal adhesive	10 MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond TM Copolymer, filler, Ethanol, Water, Initiator and Silane	3M ESPE Dental products, St.paul,MN, USA
ESPE-Sil	Silane	Ethyl alcohol, methacryloxypyrol, trimethoxysilane	
Cojet Sand	Air abrasion particles	Aluminium trioxide particles coated with silica, particle size 30μm	
Rely X Unicem	Resin cement	Multifunctional, phosphoric acid modified methacrylate monomers (70 wt% fillers)	
IPS E.max Press	Lithium disilicate glass-ceramic blocks	97% SiO2, Al2O3, P2O5, K2O, Na2O, CaO, F, 3% TiO2, and pigments, water, alcohol, chloride	Ivoclar, Vivadent, Liechtenstein, Schaan
IPS Empress etching gel	Ceramic etching gel	<5% hydrofluoric acid	
Glycerine Gel	Glycerine	Glycerine Gel	Johnson & JohnsonFrance

WA,USA).[20-22] The Weibull characteristic strength (α_0) and the Weibull modulus (β) were calculated, according to ISO Standard 6872:2008, with the following equation:

$$Pf=1-\exp [- (\alpha/\alpha_0)^{\beta}]$$

where: Pf is the probability of failure between 0 and 1. It was defined by the relation Pf=K/(N+1), K is the rank of the strength from the mimimum to the maximum value and N is the total number of specimens, α is the maximum strength in MPa, α_0 is the Weibull characteristic strength in MPa (to which the 63% of the specimens fail), and β is the Weibull modulus which shows the slope of data distribution and infancies the failure with respect to the maximum strength α. [23] These parameters and 5% probability of failure were calculated for each group.

RESULTS

Microtensile bond strength results

Immediately after the restorative phase, IDS1 and IDS3 showed lower mean bond strength (MPa) compared to DDS, IDS2 and IDS 4 at $p \leq 0.001$. After thermocycling; IDS 1 and IDS 3 showed the lowest mean bond strength values (MPa) and on the other hand DDS and IDS2 revealed the highest values while IDS4 was in between at $p \leq 0.001$*. In addition all thermocycled subgroups revealed a statistically lower values than the non thermocycled in each group at $p \leq 0.001$* . **Table (2)**

Weibull results revealed that DDS, IDS2 and IDS4 showed the highest Characteristic strength (α_0) for immediate evaluation followed by IDS 1 followed by IDS 3. IDS2 group showed also the highest Weibull modulus (β) and the 5% probability of failure. After thermocycling ; DDS and IDS 2 showed the highest Characteristic strength (α_0) followed by IDS 4 followed by IDS 1 and IDS3. Also the IDS2 showed the highest Weibull modulus (β) and the 5% probability of failure. **Table (3)and Figures (1,2)**

Environmental scanning electron microscope results

ESEM photomicrographs are presented and described in figure 3(a-j). They revealed qualitatively the bond deterioration after thermocycling. Relatively thin cement and hybrid layer was observed in IDS2 and IDS4 groups in which refreshing of the sealed dentin was done before the final cementation.

TABLE (2) Mean and standard deviation (SD) and level of significance for the microtensile bond strength values (MPa) for the different group

	Groups										p-value
	DDS		IDS 1		IDS 2		IDS 3		IDS 4		
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Immediate	30.56[a]	1.55	23.98[b]	1.33	30.21[a]	1.31	21.25[b]	1.80	28.80[a]	1.52	≤0.001*
Thermocycled	24.08[a]	2.21	18.99[c]	1.66	25.28[a]	1.07	18.80[c]	1.42	21.92[b]	1.90	≤0.001*
p-value	≤0.001*		≤0.001*		≤0.001*		≤0.001*		≤0.001*		

Means with the same letter within each row are not significantly different at p=0.05. *= Significant

TABLE (3) Weibull modulus (β), Characteristic strength (α) in MPa and 5% probability of failure.

Groups		Weibull α			Weibull β			5% probability of failure
		α	CI Lower	CI Upper	β	CI Lower	CI Upper	
Immediate	DDS	31.30[a]	30.35	32.27	19.68	13.99	33.14	20.89
	IDS 1	24.58[c]	23.81	25.37	20.76	14.15	38.97	16.02
	IDS 2	30.83[a]	30.10	31.57	25.21	17.65	44.11	23.20
	IDS 3	22.05[d]	20.99	23.15	12.80	8.95	22.50	16.05
	IDS 4	29.50[a]	28.60	30.42	21.22	14.43	40.11	19.42
Thermocycling	DDS	24.89[b]	24.04	25.78	16.94	11.64	31.07	20.89
	IDS 1	19.70[e]	18.87	20.58	14.36	9.73	27.43	16.02
	IDS 2	25.76[b]	25.20	26.33	28.38	19.44	52.56	23.20
	IDS 3	19.43[e]	18.62	20.26	15.55	10.52	29.85	16.05
	IDS 4	22.95[d]	22.12	23.81	17.80	12.06	33.97	19.42

Same letter within column indicates insignificant difference based on the 95% CI.

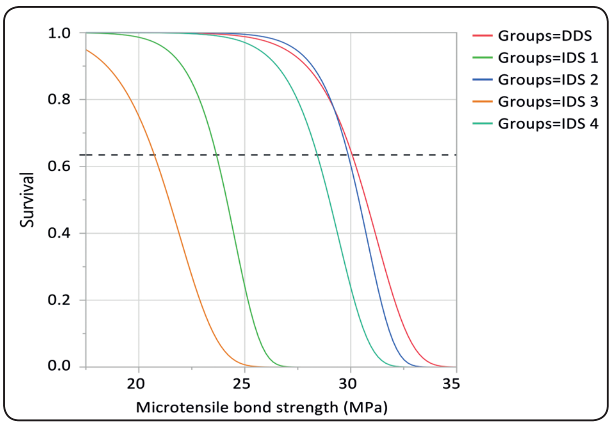

Fig. (1) Weibull analysis Survival probability for different tested groups at immediate evaluation.

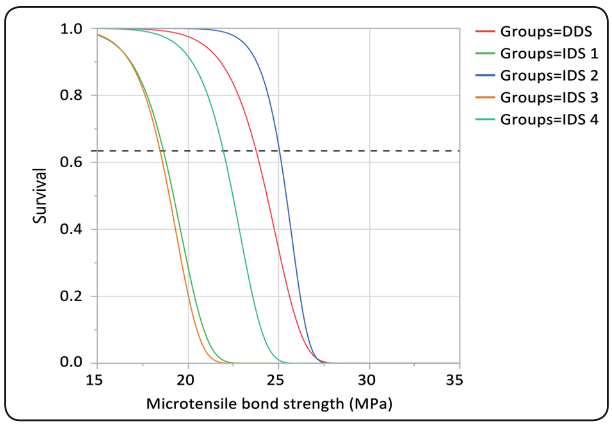

Fig. (2) Weibull analysis Survival probability for different tested groups at 6 Months evaluation

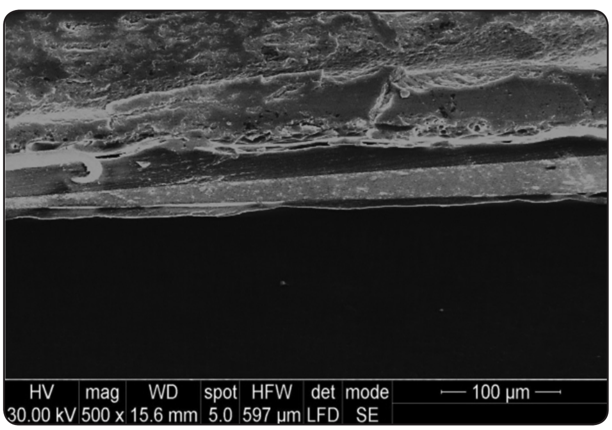

Fig. (3a) ESEM photomicrograph (500X) for Delayed dentin sealing (DDS) showing absence of gap formation and variable cement line thickness

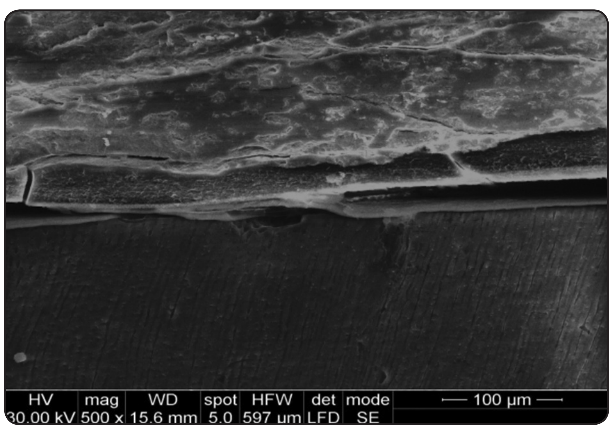

Fig. (3b) ESEM photomicrograph (500X) for DDS group after thermocycling showing presence of gap in some areas between the dentin and the cement line.

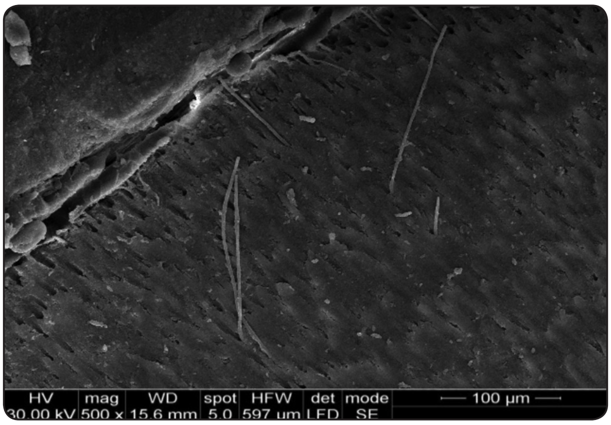

Fig. (3c) ESEM photomicrograph (500X) for Immediate dentin sealing with removal of O2 inhibited layer and without finishing before cementation (IDS1) showing interruption of the interface with relatively thick cement line and presence of few long and very thin resin tags.

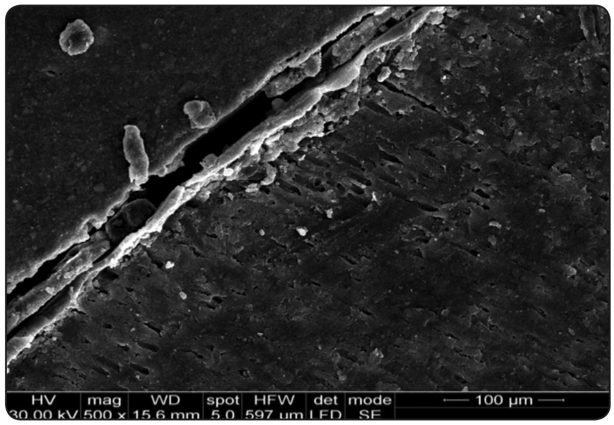

Fig. (3d) ESEM photomicrograph (500X) for IDS1 group after thermocycling showing degradation of cement and interruption of the interface with hybrid layer adapted to the dentin surface

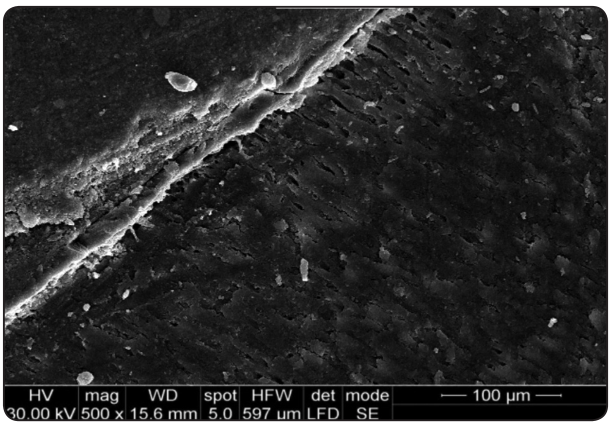

Fig. (3e) ESEM photomicrograph (500X) for Immediate dentin sealing with removal of O2 inhibited layer and finishing before cementation (IDS2) showing intact interface with a relatively thin cement line and hybrid layer

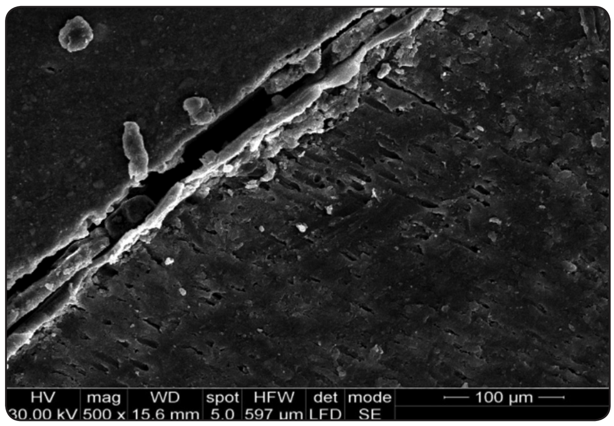

Fig. (3f) ESEM photomicrograph (500X) for IDS2 group after thermocycling showing presence of gap in some parts of the interface with evidence of few long and very thin resin tags.

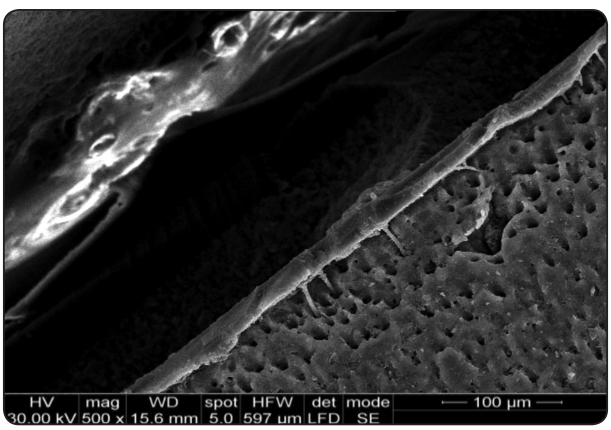

Fig. (3g) ESEM photomicrograph (500X) for Immediate dentin sealing without removal of O2 inhibited layer or finishing before cementation (IDS3) showing presence of definite gap between the relatively thick hybrid layer and the cement.

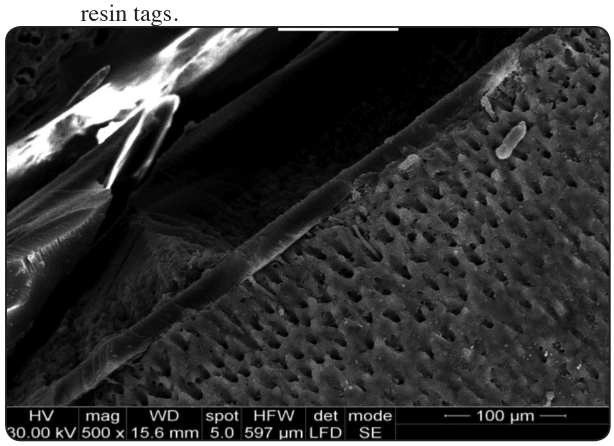

Fig. (3h) ESEM photomicrograph (500X) for IDS3 group after thermocycling showing showing presence of definite gap between the relatively thick hybrid layer and the cement.

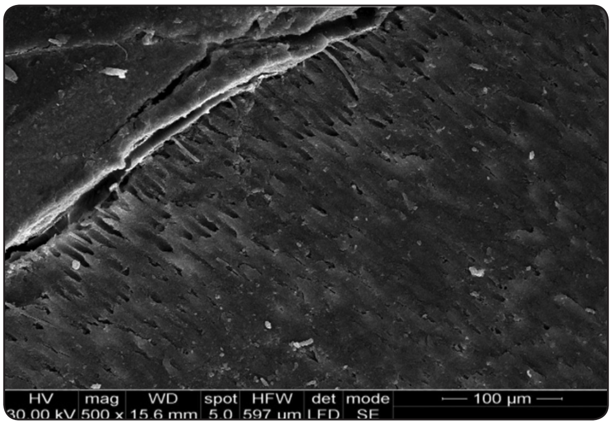

Fig. (3i) ESEM photomicrograph (500X) for Immediate dentin sealing without removal of O2 inhibited layer and with finishing before cementation (IDS4) showing intact interface with evidence of few resin tags

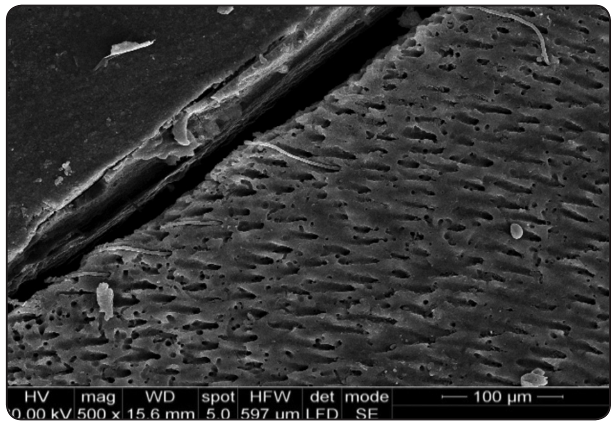

Fig. (3j) ESEM photomicrograph (500X) for IDS4 group after thermocycling showing presence of definite gap at the interface without any evidence for hybrid layer presence.

DISCUSSION

This study was conducted to evaluate the influence of immediate dentin sealing on the microtensile bond strength and interfacial quality of the indirect ceramic restoration with dentin. Also, it is induced to assess two different modalities in establishing proper dentin sealing. These modalities were the importance of removal of oxygen inhibited layer and finishing of sealed dentin immediately before cementation of the final restoration. Study groups were; delayed dentin sealing (DDS), Immediate dentin sealing with removal of O2 inhibited layer and without pre-cementation finishing (IDS1), Immediate dentin sealing with removal of O2 inhibited layer and pre-cementation finishing (IDS2) , Immediate dentin sealing without removal of O2 inhibited layer or pre-cementation finishing (IDS3) and Immediate dentin sealing without removal of O2 inhibited layer and with pre-cementation finishing (IDS4). Regarding the results of the present study demonstrated in table (1), it was found that some groups of the immediate dentin sealing (IDS2, IDS4) exhibited statistically similar mean microtensile bond strength values with those of the delayed dentin sealing (DDS). This result was nearly maintained after thermocycling except in the (IDS4) group which exhibited a statistically lower mean value than DDS and IDS2 groups. IDS1 and IDS3 showed inferior results in the immediate and thermocycling groups. It is worth mentioning that thermocycling deteriorated the bond strength in all groups. The results of the Weibull test confirm this mentioned results but with prioritization of the IDS2 group results before and after thermocycling regarding the Weibull modulus (β), Characteristic strength(α) in MPa and 5% probability of failure **(Table 3 and Figures 1 and 2)**. Based on these findings the null hypothesis was rejected as there was a statistically significant difference between the immediate dentin sealing groups. The rationale behind using the immediate sealing technique can be summarized in the following points. **First**, the most favorable substrate for bonding is the freshly cut dentin which is available only at the time of cavity preparation before impression taking. Many studies showed marked decrease in the bond strength due to dentin contamination during provisionalization period.[4,24,25] **Second,** higher bond strength values can be achieved due to pre-polymerization of the dentin bonding agent (DBA). [26, 27] However, this pre-polymerization can generate major problem in restoration seating when it is done at the time of cementation. On the other hand keeping the DBA unpolymerized during insertion of the final restoration will lead to movement of the dentinal fluids with its dilution and blocking the retentive areas that are created to be impregnated with DBA. [28,29] Also, collapse of the demineralized dentin occurs from the pressure induced from the cementation process with subsequent weak bond strength. So it is indicated to prepolymerize the DBA immediately after cavity preparation. **Third**, application of DBA at the time of cavity preparation without subsequent application of resin composite or occlusal loading will help the bonding agent to develop its strength without any threatening stresses. [30] **Fourth,** immediate dentin sealing minimizing the incidence of post-operative hypersensitivity and dentin contamination during provisionalization. Shifting to the technique for immediate dentin sealing it is found that a lot of variables can control the outcome of this technique. These variables include type of bonding agent, film thickness, cement type, interaction with the impression materials including the importance of O2 inhibited layer removal, type of provisional restoration used, time for provisionalization and cavity conditioning protocols before final cementation. [10] In this study two variables are tested which were the O2 inhibited layer removal and using the airborne particle abrasion for conditioning. This layer resulted from oxygen diffusing from atmosphere into curing resin forming soft, sticky superficial layer on freshly polymerized resin, referred to as oxygen inhibited layer. It was claimed that removal of the O2 inhibited layer might decrease the possibility for dentin contamination and improve the interfacial characteristics of the final restoration. This layer may also inhibit the polymerization of vinyl polysiloxane impression material which may interfere later on with the adaptation accuracy of the final restoration with the prepared cavity. [11] However, it was found that its removal in this study did not play a significant role in enhancement of the interface. This finding might be due to the methodology followed in this paper as ceramic restoration was in the form of standard block prepared according to predetermined dimensions without impression taking. Also its presence or absence did not affect the dentin substrate as it was shown in the scanning electron microscope photomicrographs and this is in agreement with **Tsujimoto et al in 2017**. [16] The other tested variable was the importance of pre-cementation conditioning and it has a significant effect on improvement the interface quality and this is in agreement with Magne etal in 2005 [6],2007[7] and 2011[31]. Airborne particle adhesive with aluminum oxide and coated silica cleaned the sealed surface and eliminated any dentin contamination before final cementation providing favorable dentin substrate to be optimally bonded with the ceramic restoration. This finding was supported with the SEM which showed more homogenous interface in the specimens which received pre-cementation finishing. **(Figures 3e,3i)** Gap formation is obvious in **figure 3g** representing IDS3 group in which neither O2 inhibited layer was removed nor the finishing was done, IDS3 showed the lowest bond strength values between the immediate groups. This result is in disagreement with **Rocca etal in 2012**[14] who stated the possibility for dentin re-exposure after conditioning and so they recommended the soft air abrasion instead of air born particle abrasion. Augmentation of O2 inhibited layer removal and pre-cementation finishing demonstrated the best of the modalities to be done with immediate dentin sealing as from the Weibull test result. The information provided by the test is more accurate than the other used statistical evaluation as it considered the materials structure reliability and deboning assembly. [32] Also the 5% survival probability is of high clinical relevance as it describes the expected strength at which the samples will fail. [33] These results were maintained after thermocycling with a significant reduction in the bond strength values in all groups whether thermocycled or not and this deterioration are shown in the SEM photomicrographs **(Figures 3b,3d,3f,3h,3j).**

Immediate dentin sealing is a promising approach to seal the dentin before impression taking and to improve bond strength with the final restoration. However, it needs more researches in order to recommend its use in our daily practice.

CONCLUSIONS

Under the limitations of the present study it could be concluded that:

1. Removal of oxygen inhibited layer did not play a significant role in enhancement of immediate dentin sealing outcome.
2. Finishing of sealed dentin before final cementation with air borne particle abrasion is highly recommended with immediate dentin sealing.
3. Thermocycling is a deteriorating factor that challenges the dentin ceramic restoration interface.

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