Appraisal of Degradation Resistance at OptiBond All.In.One /Affected Dentin Interface

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Abstract: This study was aimed to assess degradation resistance of glycerol phosphate dimethacrylate GPDM based adhesive system (OptiBond All. In.One) with normal and affected dentin substrates at different storage period. Method: A forty orthodontically extracted caries-free human permanent third molars were used in this study. Buccal surfaces of teeth were ground to expose dentin at the maximum convexity using a grinding wheel under water coolant. Dentin depth was standardized using depth grooves and periodontal probe. The prepared teeth were divided into two groups (20 for each); group I: the prepared teeth were stored in distilled water while group II: the prepared specimens were subjected to pH-cycling (Demineralization and Remineralization cycling) to produce artificially affected dentin. The selected adhesive system was applied to the dentin surface according to the manufacturer's instructions followed by a light cured composite resin restoration. All specimens were stored in distilled water at 37°C for different storage periods; one day, one, three and six months. The specimens were subjected to μ-shear bond test. The degraded dentin adhesive interface was chemically analyzed using Fourier transform infra-red spectroscopy (FTIR). Data was calculated and statistically analyzed. Result: At one day the μ-shear bond strength to normal dentin with adhesive was found to be significantly higher than to that of affected dentin. Over the storage period μ-shear bond strength of normal dentin revealed a significant reduction while it was non-significant with affected dentin. On the other hand, affected dentin revealed significant improvement of degree of hybridization after six months rather than that of normal dentin. Conclusions: 1.The degradation resistance and the adhesive performance is a material and time defendant. 2. Using contemporary self -etch phosphorous containing adhesive improved the quality of the affected dentin and its degree of hybridization.
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Key words: degradation resistance, affected dentin, self etching adhesives

1. Introduction

Durability of dentin bonding adhesive is one of the most important issues of recent adhesive materials. It seems to be dependent on adhesive's specific formulation, quality of the hybrid layer and not only on the bonding strategy. Biodegradation of the collagen matrix and/or hydrophilic resin components within the hybrid layer is related to: (1) incomplete penetration/infiltration of resin into dentin substrate; (2) heterogeneous distribution of resin monomers through the inter-diffusion zone; (3) suboptimal polymerization in presence of water; (4) alteration of organic matrix during preparatory procedures; (5) hydrolysis of polymeric components of unprotected collagen.

Dentin bond strength has been shown to decrease *in-vivo* and after water storage. **Nakonchai *et al.*, 2005** studied micro-tensile bond strengths after 24 hours' storage in 37 ^{0}C water of caries- affected dentin and intact dentin bonded to two dentin adhesives. They found that self-etching adhesive demonstrated no statistical difference in bond strength between intact and caries-affected dentin. However, the total-etching adhesive demonstrated different bond strengths for intact and caries-affected dentin. Moreover, the remaining dentin thickness of specimens with intact and caries-affected dentin was not significantly different. Also a thicker hybrid layer in intact and caries-affected dentin of specimens in the total-etching group was found. They concluded that the adhesives exhibited significantly different bond strengths in intact dentin of primary teeth. However, they exhibited similar bond strengths in caries-affected dentin

Wie *et al.*, 2008 studied the micro-shear bond strength of three current adhesives to normal and caries-affected dentin, and to examine the correlation between dentin nanoindentation hardness and bond

strength. Nanoindentation hardness of dentin and microshear bond strength of Clearfil SE Bond, Clerafil Tri-S Bond (Kuraray Medical) and Single Bond (3M ESPE) were measured on caries-affected and normal dentin. Modes of fracture for the bonding tests and the resin/dentin interfaces were observed using SEM. They found that all three adhesives showed lower bond strength to caries-affected dentin than to normal dentin. The differences between adhesives were not significant with caries-affected dentin as the bonding substrate. They concluded that dentin mechanical properties are not the only factors responsible for lower bond strength to caries-affected dentin.

Dentin type substrates should be considered as they represent the principal part in developing adhesives that provide durable function under clinical conditions. The hybrid layers formed on caries affected dentin are thicker than those formed on normal dentin. However, it has been generally accepted that there are fewer discrepancies between depths of demineralized zone and resin monomer penetration (degree of hybridization), because demineralization and resin monomer penetration occur simultaneously.

Recent developments in dentin bonding have reintroduced the concept of utilizing the smear layer as a bonding substrate, but improved formulations could etch through the smear layer and beyond, into the underlying dentin matrix. Self –etch adhesives is an approach based on the use of non-rinse acidic monomers that simultaneously condition and prime dentin. The bonding agents of these adhesives have mechanical interlocking together with chemical adhesion which is reliable and best obtained by weaker acids **(Van Landuyt *et al.*, 2006)**.

There are basically two types of self-etch adhesives; strong and mild **(Van Merbeek *et al.*, 2001)**.Strong self –etch adhesives have a very low pH (<1) and exhibit a bonding mechanism and interfacial ultra-morphology resembling that produced by etch and rinse adhesives. On the other hand, mild self etch adhesives (pH of around 2) partially dissolve the dentin surface, so that a substantial number of hydroxyapatite crystals remain within the hybrid layer. Specific carboxyl or phosphate groups of functional monomers can then chemically interact with the residual hydroxyapatite **(Van Landuyt KL *et al.*, 2007)**

The objectives of this study were to appraise quantitatively and qualitatively degradation resistance at interfaces of glycerol phosphate di-metha-acrylate GPDM (OptiBond All.In.One) with affected and normal dentin substrates. The μ-shear bond strength and FTIR chemical analysis were determined after water storage periods of one day, one month, three months, and six months.

2. Material and Methods:

A light cure self-etch glycerol phosphate dimethacrylate (GPDM) based adhesive system was selected for this study; (**OptiBond All.In.One**) together with a light cure resin composite; **Filtek Z/250**.

1-Tooth selection and mounting:

A total number of 40 caries-free human permanent third molars extracted for orthodontic treatment reasons were used in this study. Teeth were examined and thoroughly washed with water, scaled with periodontal scalar to remove blood, attached periodontal tissues, plaque or calculus. Then, they were incubated in distilled water with 0.5 % Thymol to avoid microbial growth **(Lolayekar *et al.*, 2007)**.

The selected teeth were mounted in specially constructed mold according to methodology described by **Elshamy *et al.*, 2007.** The mounted teeth were stored in distilled water until samples preparation.

2-Peparation of dentine specimen:

The enamel surface was completely removed by using diamond wheel tip (909 wheel diamond tip, Midwest Diamonds, Dentsply-professional, USA) of 4 mm in diameter and 2mm height mounted at high-speed dental hand-piece with coolant system. A 2mm depth at the maximum convexity of the external buccal surface was verified using periodontal probe to act as horizontal guide groove during enamel removal **(Barros *et al.*, 2005)**. Then, the superficial layer of dentin was exposed and trimmed by using a grinding wheel on a laboratory trimmer under water coolant, **(Tanumiharaja *et al.*, 2000)**. Finally, the exposed dentin was inspected by magnifying lenses (X6) to secure a surface free from any enamel remnants.

The dentin surfaces were fine polished using silicon carbide paper grits 240, 400 and 600 successively for 30 seconds each under water irrigation to produce a uniform standard smear layer,**(Wang and Spencer, 2004, and Wang *et al.*, 2006)**. The prepared teeth were randomly divided into two groups. In **group I**, the dentin specimens were rinsed and gently dried with an air stream and stored in distilled water. In **group II** the prepared specimens were subjected to certain regime to get an affected dentin before adhesive systems application**.**

3- The creation of an artificially affected dentin:

The specimens of **(group II)** were subjected to a pH-cycling procedure; demineralization followed by remineralization cycling according to **Marquezan**

***et al.*, 2009** to created an artificially affected dentin. The demineralization solution used consisted of 2.2 mM $CaCl_2$, 2.2 mM NaH_2PO_4, and 50 mM acetic acid adjusted to a pH of 4.8, whereas the remineralization solution consisted of 1.5 mmol/l $CaCl_2$, 0.9 mmol/l KH_2PO_4, 0.15 mmol/l KCl of pH 7. The whole tooth surface was double coated with an acid-resistant nail varnish except for the prepared buccal dentin surface window to allow the penetration of the solutions. The specimens were immersed in such a chemical demineralization solution for 8 hours then immersed for 16 hours into the remineralization solution. This procedure was carried out for 14 days at room temperature without agitation.

4- Samples preparation for micro-shear bond strength testing:

A. Adhesive systems applications:

The selected adhesive system was applied to the dentin surface according to the manufacturer's instructions. A double-sided adhesive tape with a hole of 2 mm diameter was fixed on dentin surface to be isolated except for the area to be bonded (2mm diameter)**.**

B- Resin composite application:

A transparent polyethylene tube obtained from a scalp vein infusion set, 23G **(JMS, JMS Singapore PTE, LTD, Singapore)** were cut into small irises of approximately 0.75-1mm in length using a sharp lancet. The transparent polyethylene iris (2.35mm external diameter and 0.93mm internal diameter) was used to aid in resin composite packing **(Sattabanasuk *et al.*, 2005)**. A small condenser **(Primadent, Germany)** was used to pack and adjust the resin composite material inside the polyethylene iris in slight excess from bonded dentin side. The resin composite was polymerized for 40 seconds by using light curing unit at intensity >450mW/cm^2, **(Ivoclar Vivadent, Schaan, Leichenstein)**

The polyethylene irises were then cautiously removed by lancet no.11 leaving the resin composite micro-cylinders bonded to dentin surface. All resin composite rods were checked using (X6) magnifying lens for any defect or air bubble where defected specimen was discarded, **(Sattabanasuk *et al.*, 2005).** Inside an incubator, the bonded specimens were stored in distilled water for one day, one month, three months, and six months respectively. The distilled water was changed every week.

5- Micro-shear bond strength testing:

Each bonded resin composite micro-cylinder specimen was secured to the lower part of especially designed attachment jig with four tightening bolts **(Kerr dental specialties, West Collins orange, USA)**. The attachment jig was in turn screwed into the lower fixed and the upper movable compartments of the testing machine**(Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK)** with a load cell of 5kN. An orthodontic stainless steel ligature wire (180µ) was bent into a loop which was wrapped around the bonded resin composite micro-cylinders such that it was too close to the base of the micro-cylinder **(G&H, Wire co., USA.)**. A shear load by tensile mode of force was applied via testing machine at a crosshead speed of 0.5mm/min**.** Using computer software **(Nexygen-MT; Lloyd Instruments)**, the failure load in N was divided by the bonded area (πr^2) in mm^2 and data were recorded.

6- Chemical analysis of dentin/ adhesive interface:

For qualitative and quantitative chemical analysis of the dentin / adhesive interface, the Fourier transform infra-red spectroscopy was used (FT/6300 type A. Jasco, Japan). A standardized amount of the curetted dentin/adhesive interface was mixed with a preweighed KBr to get spectra in transmission % at wavelength (4000-400 cm^{-1}). The chemical analysis of normal dentin, affected dentin and dentin adhesive system were served as references, **Numata *et al.*, 2008**. For quantitative analysis of dentin/adhesive interface, the mineral to matrix ratio (degree of hybridization) was calculated. The mineral quantity at peak (1030±10 cm^{-1}) was referred to phosphate content and matrix collagen content at peak (1645±10 cm^{-1}) was referred to amide I. The data were analyzed by using base line technique for the infra- red spectral vibration according to **Bohic *et al.*, 1998, Shalaby , 2010**.

7- Statistical analysis:

The obtained data were analyzed using one way of variance ANOVA followed by Duncan's Multiple Range Test at level of significance $P \leq 0.5$.

3. Results:

3.1. Micro-shear bond strength:

3.1.1. Effect of dentin type:

The statistical analysis for the effect of dentin type (whether normal or affected) on microshear bond strength (MPa) of the Optibond adhesive system and storage period is tabulated in table (1) and represented in figure (1).

In normal dentin / OptiBond All.In.One at one day storage period, the microshear bond strength recorded value was (37.225 ± 5.718 MPa), however it was recorded (27.750 ± 3.328 MPa) for affected dentin / OptiBond All.In.One. The difference between the two values was statistically significant.

The recorded results for normal dentin / OptiBond All.In.One and affected dentin / OptiBond All.In.One was statistically significant different at one month storage period, the recorded values were (22.200 ± 1.364 and 26.575 ± 2.216 MPa) respectively. Meanwhile, after three and six months storage periods, there was non-significant difference between normal/ OptiBond All.In.One and affected dentin/ OptiBond All.In.One, which recorded (22.675 ± 1.212 and 22.425 ± 0.838 MPa) and (21.225 ± 0.699 and 25.725 ± 4.463 MPa) respectively.

3.1.2. Effect of storage period:

The statistical analysis for the effect of storage periods (one day, one month, three months, and six months) on microshear bond strength (MPa) regardless of dentin type and adhesive system are tabulated in table (1) and represented in figure (1).

At one day storage period, the recorded value for microshear bond strength was (37.225 ± 5.718 MPa), however at one month storage period the microshear bond strength value of normal dentin / OptiBond All.In.One revealed significant decrease in bond strength from (37.225 ± 5.718 MPa) to be (22.200 ± 1.364 MPa). Meanwhile, at three and six months storage periods non-significant decrease in bond strength values were observed; (22.675 ± 1.212 and 21.225 ± 0.699 MPa) respectively, when compared to one month storage period.

On the other hand the effect of storage period on affected dentin/OptiBond All.In.One revealed that ,At one day storage period the microshear bond strength value was (27.825±3.488 MPa), however at one month storage period the microshear bond strength value of affected dentin/OptiBond All.In.One revealed no significant difference in bond strength value (26.575±2.216 MPa).Meanwhile, at three and six months storage periods non-significant difference in bond strength values were observed; (22.425±0.838 and 25.725±4.463 MPa) respectively, when compared to one month storage period.

Table (1): Descriptive statistics and test of significance for the effect of the dentin type and storage period on micro-shear bond strength (MPa).

Adhesive system	Storage period	Normal dentin		Affected dentin		P- Value
		Mean ± S.D.	dt	Mean ± S.D.	dt	
OptiBond All.In.One	1 d	37.225 ± 5.718	a	27.750 ± 3.328	A	0.029 *
	1 m	22.200 ± 1.364	b	26.575 ± 2.216	AB	0.015 *
	3 m	22.675 ± 1.212	b	22.425 ± 0.838	B	0.746 NS
	6 m	21.225 ± 0.699	b	25.725 ± 4.463	AB	0.093 NS

S.D. = standard deviation, P= Probability level, NS= Insignificant (p>0.05), *= Significant at ≤ 0.05**= Significant at ≤ 0.01, dt= Duncan's Multiple Range Test for the effect of storage period. Means with the same letter within each column and treatment are not significantly different at p=0.05

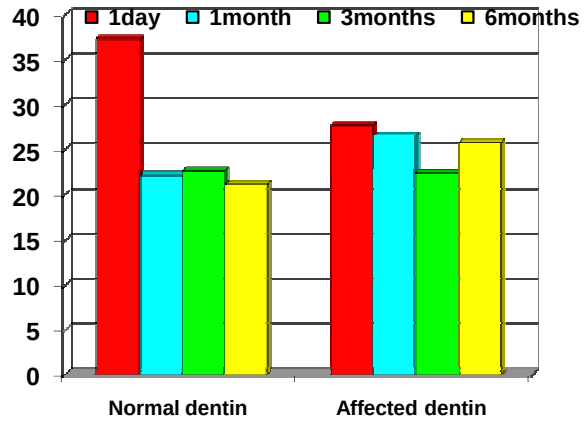

Fig.(1): A histogram of the mean micro-shear bond strength (MPa) at different storage periods within each dentin group and Optibond All in one system.

3.2. Chemical analysis of dentin / adhesive interface:

The qualitative and quantitative analysis of FTIR spectra for normal dentin and affected dentin, adhesive systems, and dentin adhesive interface are represented in **figures (2, 3, 4 and 5)** in different storage periods.

3.2.1. Molecular structure of normal and affected dentin:

Figure (2) showed the spectral characteristic bands for normal and affected dentin molecular structure. Analysis of the molecular structure of normal dentin revealed the presence of well defined bands at 565 and 603 cm^{-1} which are characteristic for symmetric stretching vibration of PO_4^{-3} (υ_4) while bands at 1032 cm^{-1} are characteristic for a symmetric stretching vibration of PO_4^{-3} (υ_3). Hpo_4^{-2} found in mature hydroxyl apatite is identified from band at 1145 cm^{-1}. The symmetric stretching vibration bands for CO_3^{-2} group were located at 873, 1420 and 1457 cm^{-1}. The band at 1455cm^{-1} is characteristic for CH_2CH_3 group. The symmetric stretching vibration bands for Amide I group was located at 1654 cm^{-1}while the symmetric stretching vibration bands for Amide II group was located at 1544 cm^{-1}

On the other hand analysis of the molecular structure of affected dentin spectra revealed the similar composition of normal dentin but with slight left shifting in Amide I group 1645 cm^{-1} (organic region) .

3.2.2. Adhesive system (Opti-Bond All.In.One):

Figure (3) showed the characteristic spectral bands for chemical composition. The data revealed the presence of well-defined bands at 565 cm^{-1} which are characteristic for symmetric stretching vibration of PO_4^{-3} (υ_4). While the more intense bands occur at 1724 cm^{-1} (carbonyl),1461 cm^{-1} (CH_2CH_3), and 830 cm^{-1} (C–C–O). These bands are associated also with methacrylate monomers in adhesive liquid. The strong wide band for H_2O was observed at 3291 cm^{-1} .The phenyl group is identified at band 1608 cm^{-1}.

3.2.3. Molecular structure of dentin /adhesive interface:

The FTIR analysis of normal and affected dentin with OptiBond All.In.One adhesive systems interfaces at different storage periods are represented in **figures (4 and 5)**.

Normal dentin/ OptiBond All.In.One:

The resultant spectra of the different groups were compared with natural dentin at spectral bands of PO_4^{-3}, HPO_4^{-2}, CO_3 and -CH3, Amide I, Amide II chemical groups and represented in **figure (4)**.

One day:

The band at 1032 cm^{-1} for symmetric stretching vibration of PO_4^{-3} (υ_3) was absent. The band at 1654 $cm^{-1}$for symmetric stretching vibration of Amide I group was shifted to the right side and captured at 1640 cm^{-1} (inorganic side). Also the band at 1544 $cm^{-1}$for symmetric stretching vibration of Amide II group was shifted to the left side and captured at1553 cm^{-1} (organic side).

One month:

The molecular structure revealed the same molecular structure of normal dentin.

Three months:

The molecular structure revealed the same molecular structure of normal dentin but the band at 1032 cm^{-1} for symmetric stretching vibration of PO_4^{-3} (υ_3) was absent. The band at 1654cm^{-1} for symmetric stretching vibration of Amide I group was slightly shifted and captured at 1649 cm^{-1} (organic side) .

Six months:

The band of symmetric stretching vibration of PO_4^{-3} (υ_4) was similar as that of normal dentin. The band at 1035 cm-1 for symmetric stretching vibration of PO_4^{-3} (υ_3) was slightly shifted to 1041cm^{-1} (organic side). The band at 1654 cm^{-1} for symmetric stretching vibration of Amide I group was shifted 1663 cm^{-1} (organic side).

B. Affected dentin/ OptiBond All.In.One:

The resultant spectra of the different groups were compared with affected dentin spectral bands for PO_4^{-3}, HPO_4^{-2}, CO_3 and -CH3, Amide I, Amide II chemical groups and represented in **figure (5)**.

One day:

The molecular structure revealed the same molecular structure as that of affected dentin but the band at 1035 cm^{-1} for symmetric stretching vibration of PO_4^{-3} (υ_3) was absent. Also the band at 1645 $cm^{-1}$for symmetric stretching vibration of Amide I group was shifted to 1667 cm^{-1} (organic side).

One month:

The band of symmetric stretching vibration of PO_4^{-3} (υ_4) was similar as that of affected dentin but the band at 1030 cm^{-1} for symmetric stretching vibration of PO_4^{-3} (υ_3) was absent. The band at 1645 cm^{-1} for symmetric stretching vibration of Amide I group was shifted to 1659 cm^{-1} (organic side).

Three months:

The band of symmetric stretching vibration of $PO4^{-3}$ (υ_4) was similar as that of affected dentin but the band at 1645 cm^{-1} for symmetric stretching vibration of Amide I group was shifted 1661 cm^{-1} (organic side). The band at 1543 cm^{-1} for symmetric stretching vibration of Amide II group was also shifted 1555 cm^{-1} (inorganic side) .

Six months:

The band at 1543 cm $^{-1}$ for symmetric stretching vibration of Amide II group was slightly shifted to 1552 cm^{-1} (organic side).

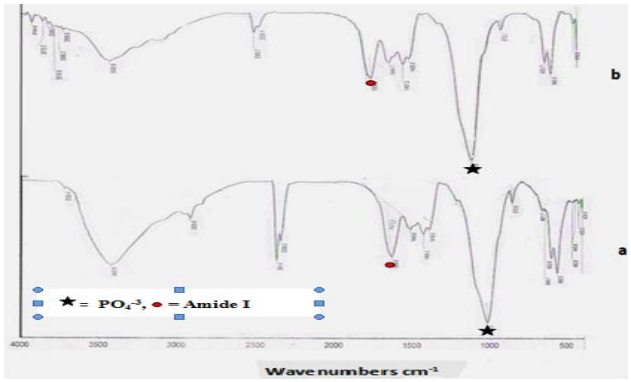

Fig. (2): FTIR spectra representing molecular structure of: a. Normal dentin b. Affected dentin

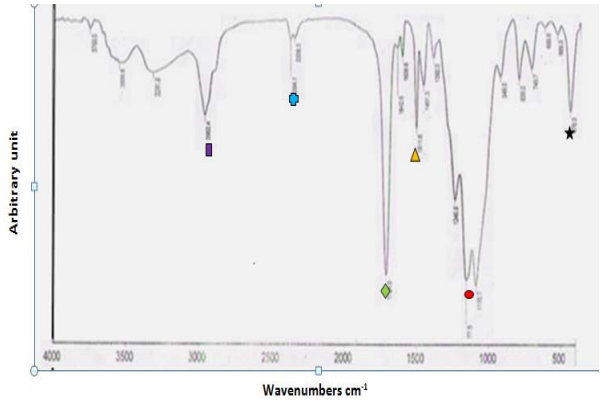

Fig. (3): FTIR spectra representing chemical composition of OptiBond All.In.One.

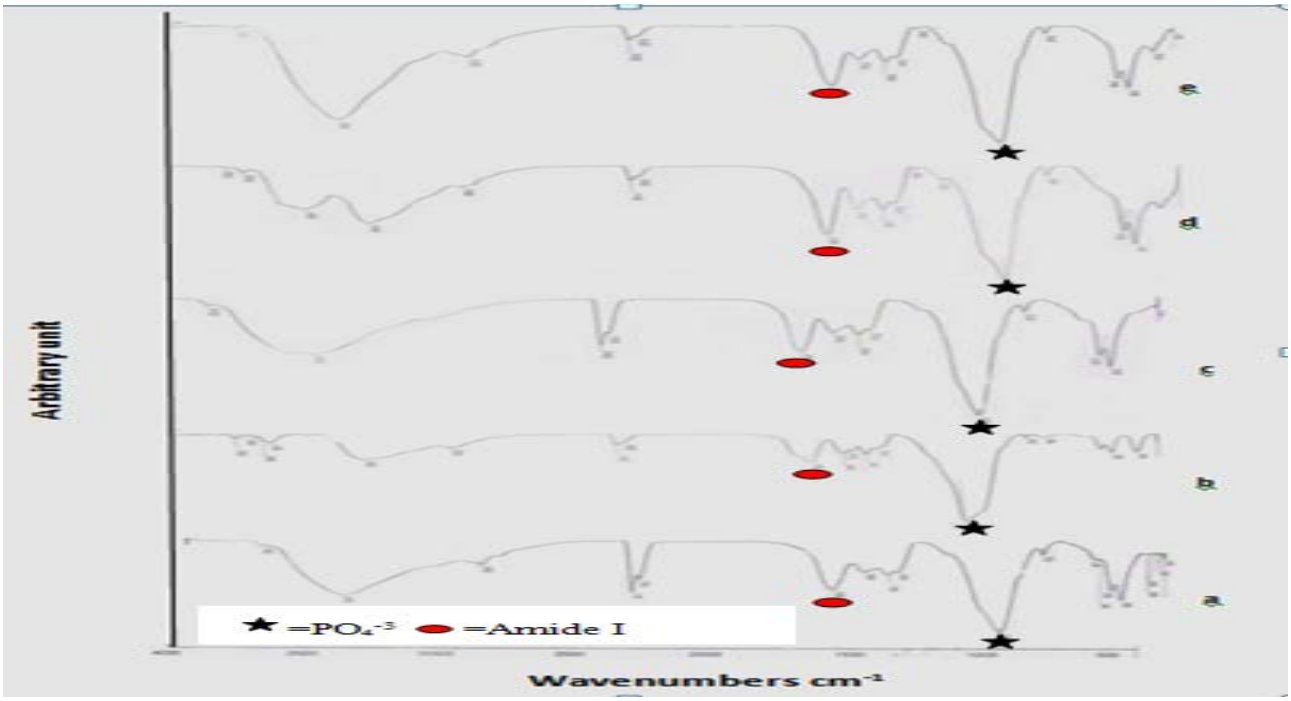

Fig. (4) : FTIR spectra representing molecular structure of a. Normal dentin b. Normal dentin/OptiBond 1 day c. Normal dentin/OptiBond 1 month d. Normal dentin/OptiBond 3 months e. Normal dentin/OptiBond 6 months.

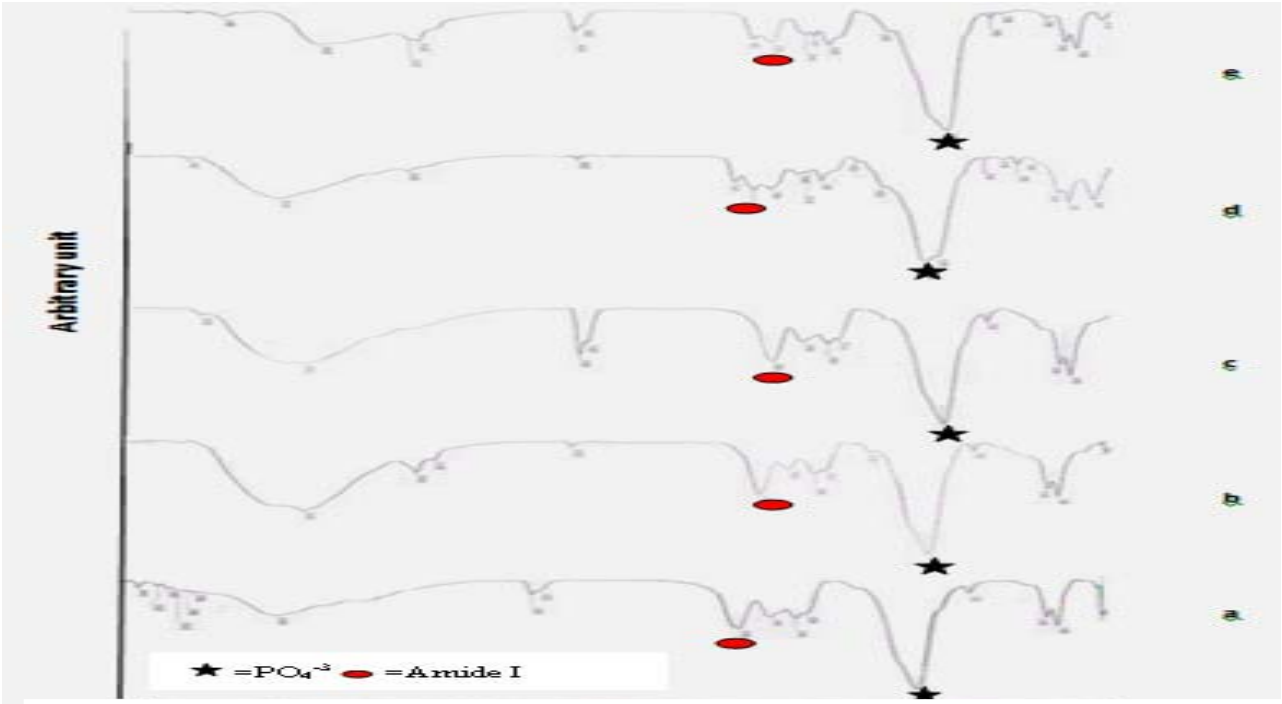

Fig. (5): FTIR spectra representing molecular structure of a. Affected dentin b. Affected dentin/OptiBond 1 day c. Affected dentin/OptiBond 1 month d. Affected dentin/OptiBond 3 months e. Affected dentin/OptiBond 6 months.

3.3. Quantitative analysis of mineral:

Mineral/collagen content ratio (Degree of hybridization):

The statistical analysis for the effect of dentin type on degree of hybridization within each adhesive system and storage period is tabulated **in table (2)** and is shown in **figure (6)**.

The data revealed significant increase in mineral / collagen content ratio in normal dentin (3.137± 0.141) as control group compared with affected dentin (2.199±0.047).

a. Effect of dentin type:

The statistical analysis revealed significant decrease in the degree of hybridization of normal dentine / OptiBond All.In.One interface at one day, one month storage periods (2.847± 0.026 and 2.773± 0.035) respectively compared with affected dentin/OptiBond All.In.One interface (3.461± 0.031 and 3.266± 0.01), however, analysis of normal dentine / OptiBond All.In.One interface revealed significant increase in the degree of hybridization at three months storage period, (3.065± 0.013) compared to affected dentin/ OptiBond All.In.One interface (1.963±0.032), whereas at six months storage period , there was a significant decrease in the degree of hybridization of normal dentine / OptiBond All.In.One interface (2.236±0.022) compared to affected dentin/OptiBond All.In.One interface (2.451± 0.021).

b. Effect of storage period:

The statistical analysis for degree of hybridization at Opti-Bond All.In.One /normal dentin interface revealed significantly decrease in degree of hybridization at one day and one month storage periods (2.847±0.026 and 2.773±0.035) respectively. Also, at three month storage period there was a slight significant decrease in degree of hybridization (3.065±0.013), however the lowest value was recorded at six months storage period (2.236±0.022).

On the other hand, The statistical analysis of quantitative analysis of degree of hybridization of affected dentin / OptiBond All.In.One interface revealed significant increase compared with normal dentin in the degree of hybridization at storage periods one day, one month (3.461± 0.031, and 3.266± 0.117) respectively. However, the lowest degree of hybridization was recorded at three months storage period, where there was a significant decrease in the amount of PO_4/amide I ratio, (1.963± 0.032) while at six months the degree of hybridization significantly increased (2.451±.0021).

Table (2): Descriptive statistics and test of significance for the effect of dentin type on the degree of hybridization within the adhesive system and different storage periods

Adhesive system	Storage period	Normal dentin			Affected dentin			
		Mean	± S.D.	dt	Mean	± S.D.	dt	P- Value
Control (natural dentin)		3.137	0.141	a	2.199	0.047	D	0.001 ***
OptiBond All.In.One	1 d	2.847	0.026	b	3.461	0.031	A	0.001 ***
	1 m	2.773	0.035	b	3.266	0.117	B	0.002 **
	3 m	3.065	0.013	a	1.963	0.032	E	0.001 ***
	6 m	2.236	0.022	c	2.451	0.021	C	0.001 ***

S.D. = standard deviation. P= Probability level, NS= Insignificant (p>0.05),*= Significant at ≤ 0.05 **= Significant at ≤ 0.01, dt= Duncan's Multiple Range Test for the effect of storage period, Means with the same letter within each column and treatment are not significantly different at p=0.05

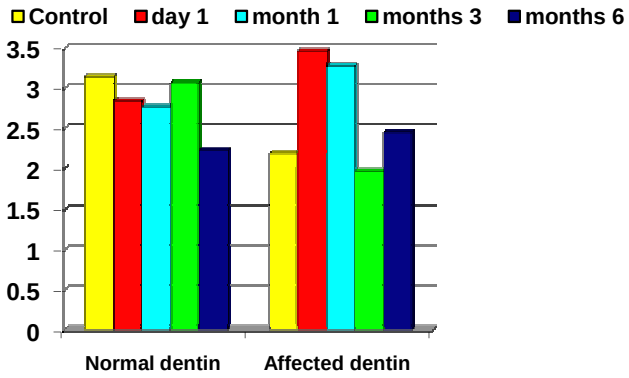

Fig. (6): A histogram of the degree of hybridization of both dentin types at different storage periods

4. Discussion:

Much of our understanding of dentin bonding strength has been based on investigations performed on sound, normal dentin on contrary to actual clinical condition. The dentists frequently bond to caries-affected dentin that generally encountered in clinical practice to replace carious tissues. Bonding to dentin depends not only on adhesive systems but also on dentin substrates.

The molecular structure analysis of the in-vitro prepared affected dentin using FTIR revealed the same spatial structure of normal dentin with a slight left shifting toward organic side which might be attributed to an increase in amount of organic contents and this was proved by significant reduction in the degree of mineral to matrix ratio (2.199±0.047) for affected dentin while normal dentin was (3.137±0.141), **(Table 2)**.

The micro-shear bond strength of the affected dentin / Optibond All.In.One interface is less than normal dentin/ OptiBond All.In.One at one day storage period and this might be attributed to the imperfect nature of affected dentin. Whereas the microshear bond strength at one month storage

period significantly increased with affected dentin/ OptiBond All.In.One interface (26.575±2.216 MPa), than normal dentin/ OptiBond All.In.One interface (22.200±1.364 MPa) (**Table 1).** In addition to that, molecular structure revealed the splitting of 1032 cm^{-1} into 1039 cm^{-1} and 1098 cm^{-1} which indicated the increase in inorganic amount. This might be due to significant increase in amount of mineral content with affected dentin/ OptiBond All.In.One interface than normal dentin/ OptiBond interface. Moreover, the degree of hybridization increased significantly in affected dentin/ OptiBond All.In.One interface (3.266±0.117) than normal dentin/ OptiBond All.In.One interface (2.773±0.035). Presumably, a thicker hybrid layer was formed due to the fact that caries affected dentin is already partially demineralized and offers a more porous substrate for acid etching than normal dentin **(Nakajima *et al.*, 2011).**

Microshear bond strength of affected dentin/ OptiBond All.In.One interface was slightly decreased at one month and three months storage periods then increased to be non-significant with one month storage period. This might be attributed to the improvement in affected dentin quality as the degree of hybridization was increased by time on contrary to normal dentin.

The mode of action of the adhesive used in this study might interpret for the improvement as the functional monomer contain di-hydrogen-phosphate group, which can dissociate in water to form two protons led to partial demineralization. Partial demineralization leaves some hydroxyapatite crystals around collagen within a submicron hybrid layer (**Van Meerbeek *et al.*, 2003).** Such residual hydroxyapatite crystals may serve as a template for additional chemical interaction with the adhesive's functional monomer. In addition, these monomers are capable of forming strong ionic bonds with calcium due to the low dissolution rate of the resulting (Calcium) Ca-salt in its own solution **(Yoshida *et al.*, 2004)**. Consequently, the chemical interaction may result in bonds that better resist hydrolytic breakdown.

It was worthy noticed that the quality of affected dentin enhanced by time to mimic the natural substrate which in turn would improve the micro-shear bond strength. In agreement with biomimetic hypothesis stated by **Liu *et al.*, 2011,** the modulus of elasticity of hybrid layer increased to be in close proximity to the normal dentin allowing elastic distribution of stresses.

Conclusion:

1. The resistance to degradation and the adhesive performance is a material and time defendant.
2- The storage time has a negative effect on the adhesive bond strength to normal dentin; in contraire to, the affected dentin which was enhanced.
3. Using contemporary self etch phosphorous containing adhesive improved the quality of the affected dentin and its degree of hybridization.

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