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The effect of storage on shear bond strength of three composite resins to dentin

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ABSTRACT

The aim of this study was to determine the effect of storage time on shear bond strength of three composite resin, two utilize total etch technique and one utilize self etching bonding agent.

One hundred twenty maxillary and mandibular molars were used in this study. The teeth were prepared by cutting the occlusal enamel with diamond bur to expose dentin and ground wet with silicone carbide papers. The specimens were then divided into three groups and stored in distilled water.

Scotchbond Multipurpose Plus was used in the first group and a stainless steel round mould, with a central hole of 4 mm in diameter and 2 mm in height was used to build up the composite Z100. The composite resin was inserted in two increments, each one was light cured for 40 seconds. This gro-up is divided into four subgroups each of ten according to the storage time (one day, fifteen days, three months and six months) then stored in distilled water accordingly.

The same procedure was repeated in the second and third groups. The composite used in the second group was Tetric with Excite bonding agent. In the third group, Definite composite and its bonding agent Etch and Prime was used.

The bond strength was measured and the data were statistically analyzed. Z100 composite had the greatest shear bond strength followed by Tetric and Definite. One day and fifteen days storage time for all types of composite had greater shear bond strength than the three months and six months storage time.

In conclusion there was a gradual decrease in bond strength with increased storage time up to six months irrespective of the type of composite.

Key Words: Composite resin, dental bonding, storage time.

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INTRODUCTION

Dental composite materials constitute an important group of materials in modern restorative dentistry.[1] Acceptable clinical longevity of composite materials has been shown although problems due to fractures, increased surface roughness and microleakage have been reported.[2] Failures due to deteriorated mechanical properties and wear may be explained by the influence of moisture from the oral environment on the composite and bonding materials, leading to degradation and erosion.[3–5]

Clinically, marginal deterioration of composite restorations remains however problematic in the long term and still forms the major reason to replace adhesive restorations.[6, 7] Consequently, the long–term stability of bonding to dentin, remains questionable.[8, 9] A factor known to promote degradation is long–term water exposure.[10–13]

The moisture in the oral environment may cause chemical degradation of the composite resin due to hydrolysis or enzymatic hydrolysis.[14] Enzymes in the saliva have been shown to take part in the chemical degradation process (i.e. enzymatic hydrolysis) of the resin matrix.[15] The presence of water is of crucial importance for the deterioration of composite resin materials. Thus, water sorption, which is a diffusion–controlled process in the resin matrix, may lead to its degradation and debonding of the filler–matrix interlayer.[16, 17] The result can be deteriorated mechanical properties.[3] The degradation as well as the leakage processes have been shown to be dependent on time.[16, 18]

Current dentin adhesives employ two different means to achieve the goal of micromechanical retention between resin and dentin. The first mean is the total etch bonding system and the second is self etching primer system.

Whenever dentin is cut, considerable quantities of cutting debris cover the surface of dentin to form the smear layer.[19] The decision whether the smear layer should be partially/completely removed or not remains under discussion. While this layer might harbor bacteria,[20] it is permeable to bacterial products and has a low cohesive force holding its particles together,[21] its total removal decreases the resistance to fluid movement across the dentin, increasing its permeability and the probability of post–operative sensitivity.[19]

In the total etch bonding system an acidic conditioner, generally 30% to 40% phosphoric acid, used to prepare the dentin surface to receive the bonding components.[22] When dentin is etched with such phosphoric acid concentrations, the smear layer is completely removed and the surface of the dentin is morphologically changed due to the dissolution of hydroxyapatite crystals, which leads to a wide opening of the dentinal tubules and the exposure of a layer of mineral depleted collagen fibrils.[23–25]

Although the interaction of the etching agents with dentin is limited by the buffering effect of the mineral and organic phases,[26] there is often a discrepancy between the depth of dentin demineralization versus monomer penetration.[23–25, 27] The remaining unprotected mineral–depleted collagen layer at the base of the hybrid layer permits nanoleakage,[28,29] pulp injury and flexural movements of the restoration, which may lead to bonding failure. It has been suggested that the exposed collagen web is susceptible to hydrolytic degradation over a long period leading to the reduction of bond strength.[10, 30, 31]

Self–etching primers which contain non–rinsing, acidic, polymerizable monomers dissolve the smear layer, or incorporate it into the bonding interface, as it demineralizes the surface and engulfs the collagen fibrils and hydroxyapatite crystals.[32] Dentin demineralization and monomer infiltration occur simultaneously, preventing collagen from collapsing and avoiding the exposure of an unprotected collagen network.[25]

However, some studies have shown a potential disadvantage in incorporating the smear layer into the hybrid layer.[33–35] Although the smear layer is reinforced by impregnated resin, bonding defects may be produced.[35] Since such defects may decrease the resistance and stability of the hybridized smear layer,[34] its removal by incorporating a separate etching step may be necessary to obtain reliable, strong resin–dentin bond.[35]

Little is known about the longevity of total–etch bonding systems compared to that of self–etching primer systems. The aim of this study was to determine the effect of storage time on shear bond strength of three composite resins, two utilize total etch technique and one utilize self–etching bonding agent.

MATERIALS AND METHODS

One hundred and twenty maxillary and mandibular molars were cleaned with rubber cup and non fluoridated flour of pumice and stored in distilled water. The teeth were mounted in upright position in plastic ring of 2.5 cm in diameter in such away that the crown portion of the tooth was protruded. A soft mixture of cold cure acrylic resin was poured around the tooth, after setting the mold was transferred into a container with distilled water. The teeth were prepared by cutting the occlusal enamel with diamond bur (Black Diamond Inc, USA); then dentin was ground wet with 400 grit silicone carbide paper followed by 600 grit silicone carbide paper to expose the underlying superficial dentin. The dentin surfaces were washed well with tap water and dried with air. Then the teeth were inspected to ensure that there was no enamel or pulpal exposure at the bonding site. The specimens were then di-vided into three groups each of forty and stored in distilled water.

Final finishing with 600 grit silicone carbide paper was done before bonding. Three types of composite resins with their adhesive systems, two total etch and one self etch, were used in this study. Scotchbond Multipurpose Plus (SBMP) was used in accordance with manufacturers' instructions. To build up the composite restoration, a stainless steel round mould, 2 mm in height with a central hole of 4 mm in diameter, was positioned over the specimen and the composite resin was inserted and light cured for 40 seconds with a light curing unit Degulux (Degussa Hulls Hanau, Germany). This group is then divided into four subgroups each of ten according to the storage time (one day, fifteen days, three months and six months) then stored in distilled water accordingly.

The same procedure was repeated in the second and third groups, but the composite used was Tetric for the second group with the bonding agent Excite and Definite for the third group with the bonding agent Etch and Prime.

The bond strength was measured with universal compression machine (Electronic Compression Apparatus, Soil test Co Inc, USA). The specimen was tested at a cross–head speed of 0.5 mm/min. The results were recorded in megapascal (MPa) and the data were statistically represented as descriptive statistics and analyzed using analysis of variance (ANOVA) followed by Duncan's Multiple Range Test at 0.05 level of significance.

The debonded specimens were examined with reflecting microscope (Corlzesis, Germany) at ×50 magnification to determine the type of failure.

RESULTS

Table (1) shows the mean shear bond strength for each storage time of the three composites. In one day and three months storage times there are statistical differences between the three types of composite. Z100 composite showed the highest shear bond strength followed by Tetric then Definite. In fifteen days storage time Z100 showed a statistical difference with the other two types of composite, but there is no statistical difference between Tetric and Definite resin composites. The six months storage time showed no statistical differences between Z100 and Tetric, while there is a statistical difference between Definite and the other two types of composite.

Table (1) also shows the mean shear bond strength for each storage time of the three composites. For Z100 there is a statistical difference between one day storage time and the other storage times, while there is no statistical difference between the fifteen days and the three month storage times. The six months storage time showed the lowest shear bond strength. For Tetric there is a statistical difference between the storage times, one day showed the highest shear bond strength followed by fifteen days, three months and six month. For Definite there is a statistical difference between one day storage, fifteen days storage time and the other storage times; while there is no statistical difference between the three months and the six months storage time.

Table (2) shows a gradual reduction in shear bond strength as the storage time was increased. The percentage of reduction in shear bond strength for Z100 composite was 9.6 % reduction in shear bond strength after fifteen days of storage, 15.4 % reduction in shear bond strength after three months of storage and 27.2 % reduction in shear bond strength after six months of storage. Tetric composite showed 9.2 % reduction after fifteen days of storage, 13.7 % reduction after three months and 18.9 % reduction after six months of storage. While Definite showed 6.5 % reduction after fifteen days of storage, 11 % reduction after three months of storage and 16 % reduction in storage time after six months.

Table (1): Mean shear bond strength (MPa) for each storage time of the three composites and for the storage times of each composite

Composite	1 Day	15 Days	3 Months	6 Months
Z100	21.25 ± 3.17 [A, a]	19.21 ± 2.44 [A, b]	18.20 ± 1.69 [A, b]	15.49 ± 3.96 [A, c]
Tetric	18.27 ± 2.00 [B, a]	16.59 ± 1.63 [B, b]	15.77 ± 1.47 [B, c]	14.82 ± 1.59 [A, d]
Definite	15.92 ± 2.34 [C, a]	14.89 ± 1.70 [B, b]	14.18 ± 1.04 [C, c]	13.38 ± 1.39 [B, c]

Means with the same letters were statistically not significant (p>0.05).
Duncan's grouping with capital letters represented statistical differences for each storage time of the three composites.
Duncan's grouping with small letters represented statistical differences for the storage times of each composite.

Table (2): Percentage reduction in shear bond strength for the storage times of each composite

Composite	After 15 Days	After 3 Months	After 6 Months
Z100	9.6%	15.4%	27.2%
Tetric	9.2%	13.7%	18.9%
Definite	6.5%	11%	16%

The mean shear bond strength and standard deviations for the three types of composite are shown in Table (3). Z100 composite had the greatest bond strength followed by Tetric then Definite. Analysis of variance and Duncan's Multiple Range Test in Table (4) shows a significant difference in shear bond strength between the three types of composite.

Table (3): Descriptive statistics of shear bond strength for the three types of composite

Type of Composite	No.	Mean	SE	SD	Minimum	Maximum
Z100	40	18.5365	0.4721	2.9858	14.20	27.20
Tetric	40	15.9200	0.3408	2.1552	12.90	21.70
Definite	40	14.2550	0.2582	1.6329	11.50	18.30

SE: Standard error, SD: Standard deviation.

Table (4): Analysis of variance and Duncan's Multiple Range Test for the three types of composite

Source	df	SS	MS	F–value	*p*–value
Between groups	2	366.827	183.413	34.689	0.000
Within groups	117	618.627	5.287		
Total	119	985.454			

df: Degree of freedom, SS: Sum of squares, MS: Mean square.

Composite	No.	Mean ± SD	Duncan's Grouping
Z100	40	18.5365 ± 2.9858	A
Tetric	40	15.9200 ± 2.1552	B
Definite	40	14.2550 ± 1.6329	C

Means with the same letters were statistically not significant (p>0.05).

The mean bond strength and standard deviations for the storage time are shown in Table (5). One day storage time for all types of composite had the greatest shear bond strength while the six months storage time had the lowest bond strength. Analysis of variance and Duncan's Multiple Range Test in Table (6) shows a significant difference in shear bond strength between one day and 15 days on one hand and the other two groups on the other hand. In addition, no significant difference between the one day and the 15 days storage time. However, there is a significant difference between the three months and the six months storage time. The Figure shows the shear bond strength of the three composites in relation with the storage time.

Table (5): Descriptive statistics of the storage time

Storage Time	**No.**	**Mean**	**SE**	**SD**	**Minimum**	**Maximum**
1 Day	30	18.367	0.6489	3.5543	12.10	27.20
15 Days	30	16.8067	0.4915	2.6921	12.10	23.10
3 Months	30	16.0333	0.3974	2.1764	12.20	20.20
6 Months	30	14.5633	0.2855	1.5634	11.50	17.70

SE: Standard error, SD: Standard deviation.

Table (6): Analysis of variance and Duncan's Multiple Range Test for the storage time

Source	**df**	**SS**	**MS**	**F–value**	***p*–value**
Between groups	3	200.648	66.833	9.886	0.000
Within groups	116	784.805	6.766		
Total	119	985.453			

df: Degree of freedom, SS: Sum of squares, MS: Mean square.

Storage Time	**No.**	**Mean $\pm$ SD**	**Duncan's Grouping**
1 Day	30	18.3670 $\pm$ 3.5543	A
15 Days	30	16.8067 $\pm$ 2.6921	A
3 Months	30	16.0333 $\pm$ 2.1764	B
6 Months	30	14.5633 $\pm$ 1.5634	C

Means with the same letters were statistically not significant (p>0.05).

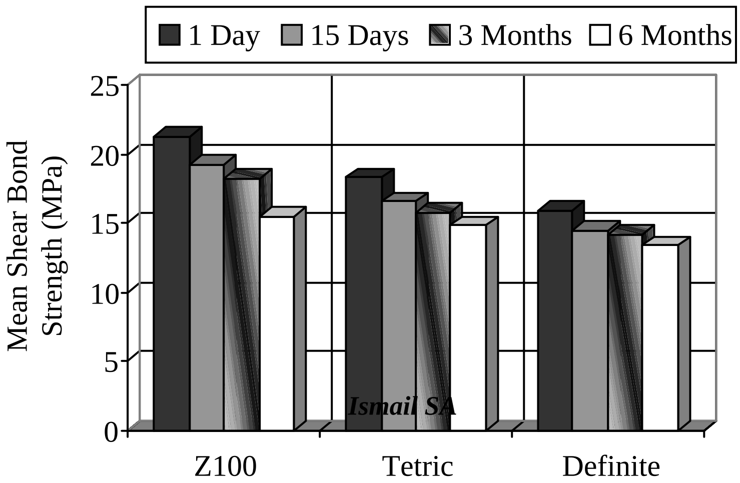

Figure: Mean shear bond strength for the tested composites at all storage times

The mode of failure is shown in Table (7), Z100 composite had 23 adhesive fractures, 13 mixed fractures and 4 dentin fractures. Tetric composite had 24 adhesive fractures, 14 mixed fractures and 2 dentin fracture. Definite had 34 adhesive fractures, 6 mixed fractures and no dentin fracture.

Table (7): Failure type found after shear bond testing of the composite at each storage time

	1 Day			15 Days			3 Months			6 Months			Total		
Composite	**A**	**M**	**D**	**A**	**M**	**D**	**A**	**M**	**D**	**A**	**M**	**D**	**A**	**M**	**D**
Z100	5	3	2	6	3	1	5	4	1	7	3	0	23	13	4
Tetric	4	5	1	5	4	1	7	3	0	8	2	0	24	14	2
Definite	7	3	0	8	2	0	9	1	0	10	0	0	34	6	0

A: Adhesive; M: Mixed (adhesive and cohesive); D: Fracture of dentin.

DISCUSSION

The three composite materials which were investigated showed significant variations in the shear bond strength over time for water storage, although it seemed to be material dependent. The self–etch compos-ite (Definite) showed the lowest shear bo-nd strength.

A potential disadvantage of self–etching primer systems is the incorporation of the smear layer within the bonding interface. Although reinforced by impregnated resin, the hybridized smear layer represents a weak zone since it can result in bonding defects.[34, 35] Also, a separation of the hybridized smear layer from the true hybrid layer is possible, since the connection between these two layers is established by only resinous material that diffused around the globular particle aggregates that form the substructure of the smear.[33] In addition, some self–etching primers promote greater water sorption, resulting in considerable water penetration into the adhesive layer.[36]

Sano *et al.*[12] showed an increased porosity over time at the top of the hybrid layer and within the adhesive resin, probably due to water extraction of resinous material, especially from interfibrillar spaces. Li *et al.*[37] found an increased nanoleakage after 12 months storage period, even for the self–etching primer system, showing that the interface created by this class of adhesive systems may also be subjected to hydrolytic attack over time.

In the present study, there is a decrease in shear bond strength of the three composite resins over time. This finding is in agreement with the results of several studies; among these are the followings:

Long–term studies have shown a gradual reduction in bond strength over time.[10, 38] This finding has been explained by the complete resin impregnation of the collagen network exposed after the dentin surface treatment with strong acidic agents such as phosphoric acid, leaving a zone of unprotected collagen at the base of the hybrid layer,[9, 39] considered a defective zone.

Other studies using total–etch bonding systems have suggested that a demineralized and non–protected dentin zone remains below the hybridized layer, due to incomplete resin infiltration.[31, 40] Some long–term *in vitro* studies,[34, 38] using water as storage medium, have demonstrated that the decrease in bond strength over time was not uniform for all materials tested. That is, the rate of decreasing bond strength was dependent on the adhesive system applied. Similarly, the bond strength of adhesive systems in some *in vivo* studies tended to decrease over time, and alterations in the hybrid layer were observed.[33]

Several studies have shown reductions of bond strength after resin–dentin bonded specimens were stored in water for long time.[11, 12, 39] It has been suggested that water absorption into the demineralized dentin might cause hydrolytic degradation[10] of the collagen fibrils at this zone, leading to a decrease in bond strength. Hence, the presence of the demineralized dentin was thought to affect the integrity of the resin–dentin bonds for both the short and long periods.

On the other hand, some researchers showed disagreement with the results of this study. Mendouca *et al*.[41] showed in a study that the microtensile bond strength of Dyract, Freedom and F2000 composites at one day storage and six months storage is not influenced by storage in water, the reason behind this finding is unclear.

Eliades *et al*.[42] showed an increase in bond strength of Dyract composite after storage. The researcher claims that the material may have undergone a slow strengthening effect due to the acid reaction in the presence of water, and did not explain why this happen with this material.

In the present study, there is a gradual reduction in shear bond strength as the storage time was increased. The percentage of loss in shear bond strength for Definite was less than that of Tetric and Definite. The reason behind this finding was unclear.

CONCLUSION

The results from this study showed that Z100 composite had the highest shear bond strength followed by Tetric then Definite. There is a gradual decrease in shear bond strength over time. One day storage time had the highest shear bond strength and the lowest shear bond strength was determined at six months of storage time.

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