The Effect of Thermocycling on Shear Bond Strength of Two Types of Self–Etch Primers

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ABSTRACT

Aims: To determine the shear bond strength of two types of self–etch primer (Transbond plus 3M Unitek, USA made and Clearfil, Japan made) when used to bond metal orthodontic brackets to enamel surface after thermocycling test and water storage for two months and to check the failure site after debonding using adhesive remnant index. **Materials and methods:** sixty extracted upper first premolars for orthodontic reason were used in this study; 30 teeth were bonded with light curing Transbond plus 3M Unitek and the other 30 teeth were bonded with light curing Clearfil self–etch primers. For both groups, Dentaurum stainless steel orthodontic brackets were bonded to enamel surface with Transbond XT light curing composite. Then 10 samples from each groups were tested for shear bond strength after 24 hours, the other 10 samples from each groups were tested after 500 manual thermocycles between 5°C &55°C, the third 10 samples of each groups were tested after 500 thermocycles and 2 months water storage at room temperature. The adhesive remnant index was tested under 10X magnification lens. **Results:** Both materials demonstrated a very good shear bond strength before thermocycling (14.5825 MPa for 3M Unitek & 14.3966 MPa for Clearfil groups). After 500 thermocycles, there were no significant changes in shear bond strength for both materials (15.0567 MPa for 3M Unitek & 13.997 MPa for Clearfil groups) and this is clinically acceptable. After 500 thermocycles and two months water storage the shear bond strength of the 3M/Unitek reduced progressively below the acceptable clinical value (3.469 MPa), whereas the shear bond strength of Clearfil group still above the acceptable level (10.607 MPa). The tendency of bond failure at the enamel–adhesive interface was increased after thermocycling and water storage. **Conclusions:** this study was done in vitro and further in vivo investigations are needed to evaluate these 2 materials.
Key words: Thermocycling, Water storage, Self etch primers, Shear bond strength.

INTRODUCTION

Thermocycling simulates the temperatur dynamics in the oral environment, with direct bonding; thermocycling reduces the bond strength of orthodontic adhesives [1].

Orthodontic adhesives are routinely exposed to temperature variations in oral cavity. Air temperature, humidity, and air velocity when breathing can also alter resting mouth temperature [2]. Although these variations are erratic and hard to anticipate when testing, it is important to determine whether they introduce stresses in the adhesive that might influence its bond strength. Therefore, Bishara *et al.*,[3] have suggested that thermal cycling should be part of the testing protocol of new adhesives.

In vivo, Transbond specimens debonded after 4 weeks had significantly lower bond strength (9.78 Mpa) than did the controls (14.34 Mpa)[4]. A significant decrease in bond strength after thermocycling have been observed using brackets bonded with three different glass ionomer cements and an autopolymerizing composite resin, with the resin showing the greatest decrease[5]. Other study found an 11.1% and 26.5% reduction in bond strength after 200 and 20,000 thermocycles, respectively, for a resin–modified glass ionomer and a 5.7% and 17.9% reduction for a composite resin[6]. Whereas other study found an 80% decrease in

bond strength of a cyanoacrylate orthodontic adhesive after thermocycling[3].

Although phosphoric acid etching still seems to be the most frequently used method for enamel preparation[7,8], enamel damage from debonding still is a major clinical problem. In contrast, self–etching primers show much less etching ability because of their relatively higher PH as compared with phosphoric acid etching [9,10], thus minimizing the potential for iatrogenic damage to enamel.

Researchers have tested alternative enamel conditioners, such as maleic acid primers, to determine if they can attain clinically useful bond strength while decreasing the depth of enamel dissolution and decreasing the number of steps during the bonding procedure[11]. Conventional bonding systems used 3 steps to prepare enamel surfaces: an enamel conditioner, a primer solution, and an adhesive resin[12], so that the time saved by the use of a self–etching primer is more than that spent in the preparation of the adhesive before bonding[13].

A study used computerized three dimentional scanner to measure enamel loss caused by orthodontic bonding and debonding after phosphoric acid etching and reported an average loss of enamel of 7.4μm[14]. Using field emission scanning electron microscopy observed more dissolution of the enamel surface resulting from phosphoric acid etching (5–50μm) than from self–etching primer treatment[15,16].

The shear bond strength of orthodontic brackets to enamel have been compared using 37% phosphoric acid, 10% maleic acid, or an acidic primer solution containing the acidic phenyl–p and a hydrophilic primer consisting of hydroxy methyl methacrylate and dimethacrylate. That study demonstrated that acidic primer systems used to bond orthodontic brackets to tooth enamel could provide acceptable shear strength[11].

The aims of this study are to determine the shear bond strength of two types of self–etch primers (Transbond Plus 3M Unitek, USA made & Clearfil, Japan made) when used to bond metal orthodontic brackets to enamel after thermocycling test and water storage for two months, and to measure the failure site after debonding in the three conditions using adhesive remnant index (ARI).

MATERIALS AND METHODS

In this study two types of self–etch primers are used (Clearfil SE bond, made in Japan and Transbond™ plus 3M/Unitek, made in USA). The compositions of these two materials are presented in Table (1).

To exclude the possible differences in bond strength caused by the orthodontic adhesive, all brackets were bonded with the same material (Light–cure highly filled orthodontic adhesive Transbond XT 3M/ Unitek, Monrovia, Calif, made in USA)[17].

Table (1): Composition of self–etching adhesive systems used in present study.

Test Material	Composition
Transbond Plus 3M Unitek	Bonding agent A:MAC–10, photoinitiator, methacryloyloxyalkyl acid phosphate, BisGMA, TEGDMA. Bonding agent B:methyl methacrylate, HEMA, water, F–deliverable micro filler, photoinitiator.
Clearfil SE Bond	Primer:MDP, HEMA, hydrophilic dimethacrylate, di–camphoroquinoneN, N–diethanol–p–toluidine, water. Adhesive:10–MDP, Bis–GMA, HEMA, silanated colloidal silica, hydrophilic dimethacrylate, di–camphoroquinoneN, N–diethanol–p–toluidine.

MAC: methacrylate; BisGMA: isphenol–glycidil methacrylate; TEGDMA: triethyleneglycol dimethacrylate; HEMA: 2–hydroxyethyl methacrylate; MDP: methacryloxymethacrylate.

Sixty freshly extracted human maxillary first premolar for orthodontic reason were collected and stored in a solution of 0.1% thymol. The criteria for tooth selection included intact buccal enamel, not subjected to any pretreatment chemical agents, no cracks, and no caries[18]. The teeth were poured in metal rings using cold–cure acrylic resin and numbered randomly, then the teeth were polished with non–fluoridated pumice using prophylactic rubber cup for 10 seconds[13,17,19].

A stainless steel metal brackets (Dentaurum type) of maxillary first premolar with an average base surface area of 10.5 mm^2 were used in this study. Then the teeth were randomly divided in to two groups, each group consist of 30 teeth, and bonded as follows (Table 2):

The first group was bonded using Clearfil SE bond; The primer was applied for 20 seconds then dried with mild air flow (without water), then the bond was applied and air flow gently and cured with Halogen bulb light–cure device (Dentsply type) for 10 seconds. The second group was bonded using Transbond™ plus 3M/Unitek etching primer; The two components were squeezed together and mixed well before application on the enamel surface for 3–5 seconds and then gently evaporated with air for 5 seconds.

For both groups the orthodontic brackets bonded to tooth surface using Transbond XT composite under finger pressure, the access composite was removed with sharp scaler and light cured for 20 seconds (10 seconds on each side)[18]. Group of 5 teeth was bonded at a time to simulate the bonding of each quadrant in the oral cavity.

The specimens were stored in water at $37\pm2^{\circ}C$ for 24 hours to discriminate between those materials that can and those that cannot withstand a wet environment. After 24 hours, 10 samples of Clearfil SE group & 10 samples of Transbond™ plus 3M/Unitek were debonded at room temperature, whereas the others were thermocycled between 5°C and 55°C for 500 complete cycles.The thermocycling was done manually following the recommendation of the international Organization for Standardization[20], the exposure to each bath was 20 seconds, and the transfer time between the two baths was 5–10 seconds. Debonding was also performed at room temperature for 10 samples of Clearfil SE group & 10 samples of Transbond™ plus 3M/Unitek, the remaining 10 samples of each group were stored in distilled water at room temperature for 2 months before debonding.

Table (2): Sample distribution.

	Transbond™ Plus 3M/ Unitek (No. of Specimens)	Clearfil SE Bond (No. of Specimens)
Debonding After 24 Hours	10	10
Debonding After 500 Thermocycles	10	10
Debonding After 500 Thermocycles and 2 Months Water Storage	10	10
Total Sample	30	30
	60	

The debonding was done on the unconfined shear testing machine. A knife–edge shearing blade was secured on the cross head with the direction of force parallel to facial surface and the bracket interface. The shearing blade struck flush against the edge of the base without touching the enamel. Bond strength was measured at a crosshead speed of 1mm/ minute[17,21]. The force required to dislodge the bracket was recorded in Newton and converted to Megapascals with the following equation:

Shear force (MPa)= debonding force (N)/ surface area of bracket base(mm^2), so that 1MPa=1N/mm^2. After debonding, the

teeth and brackets were examined with 10X lens magnifications. The debonding condition of each specimen was scored using the adhesive remnant index (ARI)[22]. The ARI scores ranged from 0–3, ie, score 0= no adhesive remained on the enamel; 1= less than half of the adhesive remained on the tooth surface; 2= more than half of the adhesive remained on the tooth surface; 3= all the adhesive remained on the tooth surface with a distinct impression of the bracket base.

Student T–test was performed between Transbond™ plus 3M/Unitek and Clearfil groups before thermocycling, after thermocycling, and after thermocycling and water storage to determine the difference in shear bond strength of the 2 materials. It is considered significant when $p \leq 0.05$. One–way analysis of variance (ANOVA) and Duncan's multiple range analysis tests were used for both materials before thermocycling, after thermocycling, and after thermocycling and water storage to show the difference between the 2 materials in the three phases. It is considered significant when $p \leq 0.05$.

To assess the difference in the failure site of bracket between the 2 materials in the three phases, the two sample Kolmogorov–Smirnov test and Kruskal–Wallis test were used in this study. It is considered significant when $p \leq 0.05$.

RESULTS

Shear Bond Strength:

The descriptive statistics for the shear bond strength of both 3M/Unitek and clearfil groups in the three conditions (before thermocycling, after thermocycling, and after both thermocycling and storage) are presented in Table (3).

Table (3): Descriptive statistics of shear bond strength for 3M Unitek and Clearfil before thermocycling, after thermocycling and after both thermocycling and 2 months water storage.

	Material	No.	Mean*	± SD	SE Mean
Before Thermocycling	3M/Unitek	10	14.58250	4.337538	1.371650
	Clearfil	10	14.39660	3.043000	0.962281
After Thermocycling	3M/Unitek	10	15.05670	2.521552	0.797385
	Clearfil	10	13.99700	2.060792	0.651680
After Thermocycling and Water Storage	3M/Unitek	10	3.46900	1.178346	0.372626
	Clearfil	10	10.60700	4.617207	1.460089

* The mean in MPa (N/mm²); No: number; SD: standerd deviation; SE: standerd error.

In Table (4) the comparative statistical *t*–test between 3M/Unitek samples and Clearfil samples indicated that there were a non significant differences in shear bond strength at level of 95% confidence interval of the difference before thermocycling groups and after 500 thermocycles, whereas after 500 thermocycles and 2 months water storage this test shows a high significant difference between 3M/Unitek and Clearfil groups because of a great reduction in shear bond strength for 3M/Unitek group. The 3M/Unitek group when tested in the three phases (before thermocycling, after thermocycling, and after both thermocycling & storage) using one way analysis of variance and Duncan's multiple range analysis test (Tables 5, 6) shows that there were non significant difference in shear bond strength before thermocycling and after 500 thermocycle, but the groups that tested after 500 thermocycle and 2 months water storage were significantly have lower shear bond strength. The same results were shown in Tables (7, 8) for the Clearfil groups.

Table (4): Comparison of shear bond strength of 3M/Unitek and Clearfil before thermo-cycling, after thermocycling and after both thermocycling and 2 months water storage.

	T–test for Equality of Means			95% Confidence Interval	
	T–value	**df**	***p*–value**	**Lower**	**Upper**
Before Thermocycling	0.111	18	0.913	–3.334263	3.706063
After Thermocycling	1.029	18	0.317	–1.103851	3.223251
After Thermocycling and Water Storage	–4.737	18	0.000	–10.303853	–3.972147

df: degree of freedom.

Table (5): One way analysis of variance of 3M/Unitek self–etch primer.

	Sum of Squares	**df**	**Mean Square**	**F–value**	***p*–value**
Factor	860.032	2	430.016	48.569	0.000
Error	239.049	27	8.854		
Total	1099.080	29			

df: degree of freedom.

Table (6): Duncan's Multiple Range Test of 3M/Unitek self–etch primer.

Groups	**No.**	**Mean* ± SD**	**Duncan's Group-ing****
After Thermocycling and Water Storage	10	3.46900 ± 1.178346	A
Before Thermocycling	10	14.58250 ± 4.337538	B
After Thermocycling	10	15.05670 ± 2.521552	B

* The mean in MPa (N/mm^2); No: number; SD: standerd deviation ; **Means with same letter were statistically not significant ($p > 0.05$).

Table (7): One way analysis of variance of Clearfil self–etch primer.

	Sum of Squares	**df**	**Mean Square**	**F–value**	***p*–value**
Factor	86.709	2	43.355	3.735	0.037
Error	313.428	27	11.608		
Total	400.137	29			

df: degree of freedom.

Table (8): Duncan's Multiple Range Test of Clearfil self–etch primer.

Groups	**No.**	**Mean* ± SD**	**Duncan's Group-ing****
After Thermocycling and Water Storage	10	10.60700 ± 4.617207	A
After Thermocycling	10	13.99700 ± 2.060792	B
Before Thermocycling	10	14.39660 ± 3.043000	B

* The mean in MPa (N/mm^2); No: number; SD: standerd deviation **Means with same letter were statistically not significant ($p > 0.05$).

Adhesive Remnant Index and Failure Site:

The adhesive remnant index between orthodontic brackets and enamel surface were presented in Table (9). The two–sample Colmogorove–Smirnov test was used to show the difference in failure site for the 2 materials before thermocycling, after thermocycling, and after both thermocycling & water storage phases which indicated that there were non significant difference between them (Table 10).

The adhesive remnant index were compared between 3M/Unitek and Clearfil groups using Kruskal–wallis test (Table 11) showed that the adhesive remnant index in 3M/Unitek samples after thermocycling and water storage was significantly differ from the remaining samples (In all 10 samples the failure occure at the enamel–adhesive interface).

Table (9): Adhesive Remnant Index frequency distribution.

Group	Score	Before Thermocycling	After Thermocycling	After Thermocycling & Water Storage
3M/Unitek	0	5	8	10
	1	3	1	0
	2	2	1	0
	3	0	0	0
Clearfil*	0	6	7	9
	1	4	2	1
	2	0	0	0
	3	0	0	0

*Fracture enamel in one sample of Clearfil group after thermocycling have been excluded from the study.

Table (10): Statistical analysis between 3M/Unitek and Clearfil materials before thermocycling, after thermocycling and after both thermocycling and 2 months water storage (Kolmogorov–Smirnov Test).

Group	Z value	D(absolute)	*p*–value
Before thermocycling	0.44	0.200	0.988*
After thermocycling	0.218	0.100	1.000*
After thermocycling and storage	0.224	0.100	1.000*

* not significant ($p > 0.05$).

Table (11): Statistical analysis among before thermocycling, after thermocycling and after both thermocycling and 2 months water storage for the two materials (Kruskal–Wallis Test).

Group	χ^2	df	*p*–value
3M/Unitek	6.623	2	0.036 (S)
Clearil	2.398	2	0.301 (NS)

df: degree of freedom; S: significant difference; NS: no signifinat difference.

DISCUSSION

Bond strength should not only be high enough to resist the forces during the course of orthodontic treatment but also low enough to allow the removal of the bracket without any complications at the end of orthodontic treatment.

In this study the shear bond strength of 3 M/Unitek and Clearfil before thermocycling and after 500 thermocycle was not significantly changed. This result is supported by Hasegawa *et al.*,[23] who reported that subjecting specimens to 500 cycles might not affect bond strength, de-

pending on the adhesive system used. Other study shows in its results a reduction in shear bond strength of self–etch primer but after 10.000 thermocycles[24].

The other factor concern in the biodegradation of the adhesive and composite as a result of immersion in water or exposure to oral fluids leading to decrease in bond strength values over time. So the reduction in shear bond strength after thermocycling and water storage for 2 months was significant in this study. These results are also supported by Murray and Hobson[25] who demonstrated a significant loss of bond strength in brackets bonded with Transbond after they were immersed in water in vitro or exposed to oral environment for as long as 12 weeks.

The reduction in shear bond strength for both Clearfil and 3M/unitek groups after thermocycling and water storage may possibly be explained by the absorption of water and the alternating stressing of the system resulting from the large mismatch of the thermal expansion coefficient of different surfaces [26]; of the adhesives (α=20–55 ppm/°C) with those of the stainless steel bracket (α=16 ppm/°C) and enamel (α=16 ppm/°C).

It is logical to assume that water storage was another major contributing to the low bond strength achieved with the self–etch primer and adhesive system in our study. Water diffusion into the bonding interface causes the resinous components to swell and become plasticized [27]. During the thermal cycling test, hot water might accelerate the hydrolysis of the resin and extract poorly polymerized resin oligomers[28].

The adhesive remnant index scores indicated that brackets bonded with either material showed a similar range of bond failure mode in each phase. There is an increase in the risk of adhesive failure occur at enamel–adhesive after thermocycling and water storage, this may be advantageous to leave enamel surface clean after debonding and not need bur cleaning[19,29], however this result may be of controversy. As an example, bracket failure at the bracket/adhesive interface is advantageous because it leaves the enamel surface relatively intact. However, considerable chair time is needed to remove the residual adhesive, with the added possibility for damaging the enamel surface during the cleaning process. Conversely, when brackets fail at the enamel/adhesive interface, less residual adhesive remain, but the enamel surface can be damaged when failure occurs in this mode.

CONCLUSIONS

The use of single step self–etch systems, in terms of simplifying clinical procedures, might have benefits even after exposure to a number of thermal cycles simulating conditions in oral environment. However, it should be born in mind that these results were obtained under in vitro conditions.

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