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Original Article

Effect of saliva contamination on the shear bond strength of two adhesive systems to dentin: An In vitro study

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Abstract

Aims and objectives: To evaluate the effect of saliva contamination on the shear bond strength of one bottle (single bond) and self etch primer (Clearfil SE Bond) adhesive systems to dentin and to determine the effect of contaminant removing treatments on the recovery of shear bond strengths.

Materials and methods*:* Eighty human molars were embedded in rectangular aluminium moulds with self-curing acrylic resin. The occlusal dentin surface was exposed by wet grinding with No. 800 silicon carbide abrasive paper. The specimens were randomly divided into eight groups. Group 1 to Group 4 were treated with Single Bond (3M ESPE) and Group 5 to Group 8 were treated with Clearfill SE bond (Kuraray Co, Ltd, Osaka, Japan). For Single Bond, the grinding surface was treated with 32% phosphoric acid and divided into four groups: Group1 control (uncontaminated), Group 2 (salivary contamination, completely dried), Group 3 (salivary contamination, rinse and blot dried) and Group 4 (salivary contamination, re-etching for 10 seconds, rinse and blot dried). For SE bond, the following surface treatments were done: Group 5 control (uncontaminated), Group 6 (primer, salivary contamination, dried), Group 7 (primer, salivary contamination, wash and dried), Group 8 (after procedure of group 6, re-application of primer). Each bonding agent was applied and light cured for 10 seconds. The teeth were then positioned and stabilised in a stabilizing device which holds a Teflon block having a circular hole of 3mm diameter and 2 mm thickness. Hybrid composite resin Filtek Z-250 (3M ESPE) was condensed into Teflon mold (3x2 mm) and light cured for 20 seconds to form resin cylinders. All the specimens were stored in distilled water at 37 °c for 24 hours and shear bond strength testing was done in an Instron Universal Testing Machine (5500R) at a cross head speed of 0.5mm/min. The shear bond strength data for the two adhesives was subjected to 1-way ANOVA while comparison of means between groups was done by t-test.

Results: In the Single Bond groups,Group 2 showed statistically significant lower shear bond strength than Group 1 (control),3 and 4($p<0.05$). In the clearfill SE groups, group 6 and group 7 showed lower shear bond strength than Group 5 (control)and group8($p<0.05$).

Conclusion: When using Single Bond total etch adhesive and when the etched surface is contaminated by saliva, blotting the surface and applying the primer can recover the bond strength. Complete drying of the salivary contaminated surface should be avoided. In the Clearfil SE Bond groups, the re-priming treatment resulted in the recovery of shear bond strength.

Key words: Adhesion, Contamination, Total etch, Self etch, Shear bond strength

Introduction

Materials that bond to enamel and dentin have revolutionized the field of dentistry whether a sealant or a composite material is being placed or orthodontic bracket being attached, the stability of the bond is paramount to clinical success[1].Therefore, improving adhesive restorative materials has been the object of considerable research in recent years[2].

Clinically there are many factors that affect the adhesion and retention of resin containing restorative materials.

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Moisture such as gingival fluid, blood, hand piece oil and in particular, saliva can affect the quality of the bond leading to micro leakage at the interface[3]. As a result, loss of the restoration, recurrent caries, post-operative sensitivity and discoloration may occur[4].

With increased demand and use of aesthetic restorations, contamination control has become an important topic since dental adhesives and composites are very vulnerable to contamination. Therefore proper isolation and control of contamination are essentially required during bonding procedure[5]. Controlling Saliva contamination in the field of operation is difficult in adhesive dentistry, especially in those cases where cavity margins extend below the gingival tissues or when indirect restorations are seated or in newly erupted molars or when patients have problem in mouth opening[6,7,8].

Shear bond strength has become a popular in vitro measure of the effectiveness of dentin bonding agents under different conditions. Several studies have reported that saliva contamination of etched enamel caused a significant decrease in bond strength between the resins and enamel surface[9,10,11]. The salivary proteins prevented monomers from penetrating the pores in enamel and reduced the bond strength. The organic pellicle coating masked the underlying enamel pores, decreased resin accessibility and impaired mechanical adhesion[12]. In as little as 1 second, salivary exposure will compromise bonding of resin to enamel[9,12].

Dentin bonding is extremely complex when compared to enamel bonding because dentin is a more heterogeneous substrate with much higher organic and water content than enamel[13]. The micromechanical resin adhesion to dentin differs fundamentally from the relatively simple interlocking of bonding agents with enamel. Consequently studies related to the bonding efficacy of saliva contaminated dentin bonding agents are controversial[14].

Recently one bottle and self etching primer adhesive systems have been introduced in an effort to simplify the bonding procedure[4,15]. One bottle system combines primer with the adhesive resin into a single component while the etchant is applied separately. Self etching primer system combines a weak phosphoric acid and primer in one bottle and an adhesive in a second bottle and eliminates the rinsing and drying steps. There by reducing the possibility of over wetting or over drying, both of which can negatively influence adhesion[16].

Even though these contemporary dentin adhesive systems are easier to use and less technique sensitive, salivary contamination may still occur during bonding procedures, resulting in reduced bond strength and only few studies are available on the effect of treatment methods of saliva contaminated dentin surfaces for the recovery of bond strength of these contemporary adhesive systems.

Aims and objectives

1. To evaluate the effect of saliva contamination on the shear bond strengths of one bottle (Single Bond, *3M ESPE*) and self etch primer (Clearfill SE Bond, *Kuraray*) adhesive systems to dentin.
2. To determine and compare the effect of contaminant removing treatments on the recovery of shear bond strengths of the two bonding systems.

Materials and methods

Eighty caries free extracted permanent human molars were collected and were thoroughly scrutinized to rule out the presence of cracks, fluorosis, restorations, fractures and any hard tissue defects and excluded from the study. The selected teeth were cleaned of soft tissue debris, calculus and stored in distilled water in a glass beaker till further use.

The teeth were embedded in rectangular aluminium blocks with auto polymerizing resin, such that the occlusal surfaces were parallel to the surface of acrylic resin. The occlusal surfaces were ground flat to expose dentin. The bonding area was demarcated by placing a piece of vinyl tape having a 3mm diameter hole over the prepared dentin surface yielding a standard area for bonding. The prepared teeth were stored in distilled water until preparation of the specimens for bonding.

Prior to bonding, fresh whole saliva was collected from a single female donor in sterile beaker and was used immediately.

All the teeth were randomly divided into eight groups of ten teeth each. Group 1 to Group 4 were treated with Single Bond adhesive and Group 5 to Group 8 were treated with Clearfill SE Bond adhesive (Table1) and the procedure was as follows:

Group 1 to Group 4: Single Bond groups (Fig 1)

Group 1 : Uncontaminated, control group

Group 2 : Dentin contaminated with saliva after etching that was then air dried

Group 3 : Dentin contaminated with saliva after etching that was then rinsed with water

Group 4 : Dentin contaminated with saliva after etching that was then re-etched

Group 5 to Group 8: Clearfill SE Bond groups (Fig. 2)

Group 5 : Uncontaminated, control group

Group 6 : Dentin contaminated with saliva after the application of self etch primer that was then air dried

Group 7 : Dentin contaminated with saliva after the application of self etch primer that was then rinsed with water

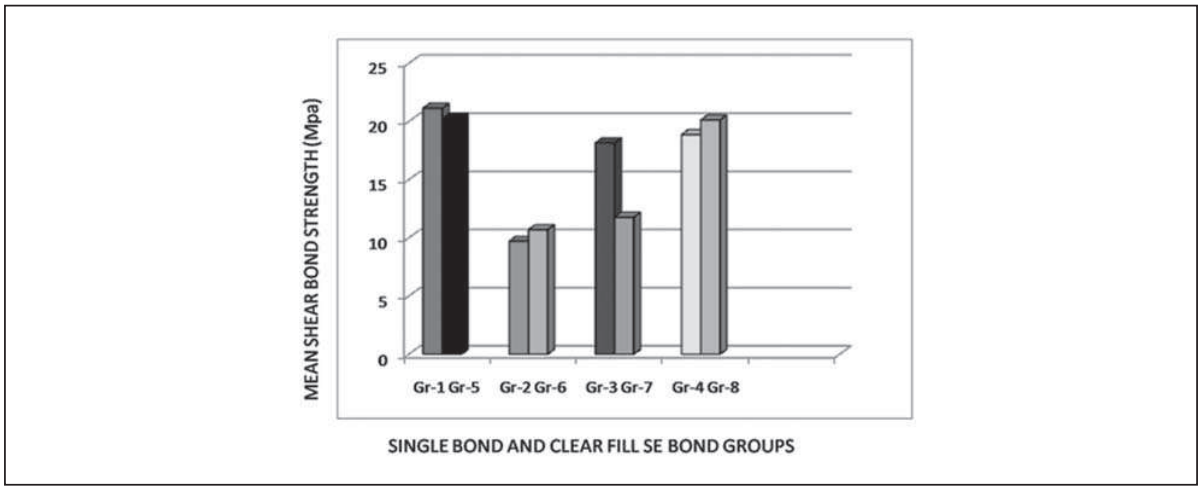
Group 8 : Dentin contaminated with saliva after self etch primer application that was then re-primed

All the teeth to be tested in the control groups were bonded according to manufacturer's directions. All the teeth in the experimental groups were conditioned as above stated and subsequent bonding agents were applied according to manufacturer's directions.

A Teflon mould was used to bond cylinders of all groups with a hybrid composite resin measuring 3mm internal diameter and 2mm height (Fig 3). The prepared specimens were stored in distilled water at 37^0C for 24 hours and shear bond strength testing was done in an Instron Universal Testing Machine (5500R) at a cross head speed of 0.5mm / min. A wire loop method of shear testing was employed to debond the 3mm x 2mm cylinders of composite restorative material (Fig 4 and 5).

Results

The maximum load in Newton's required to debond the cylinders of the above restorative materials was recorded to calculate the stress at fracture i.e., the shear bond strength in MPa. Data were summarized as Mean ± SD. The mean shear bond strength for each group is shown in Table 2. The shear bond strength data for the two adhesives was subjected to one way ANOVA (Table 3and 4) while comparison of means between groups was done using't' test. The analysis of results with 't' test was done at 5% (0.05) level of significance. (Table 5, Graph 1).



Graph 1: Comparison of mean shear bond strengths of Single Bond and Clear fill SE Bond groups

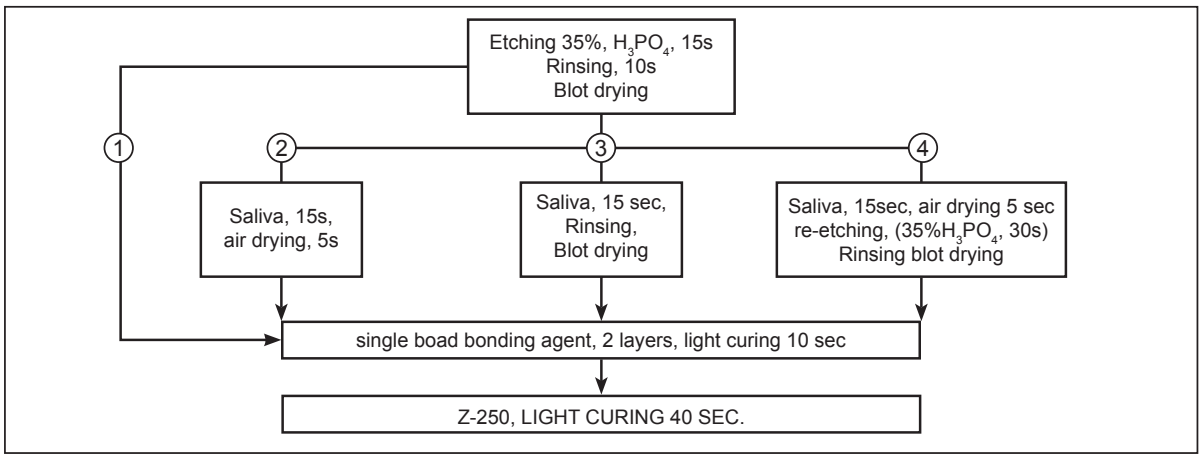


Fig 1: Bonding procedure for single bond

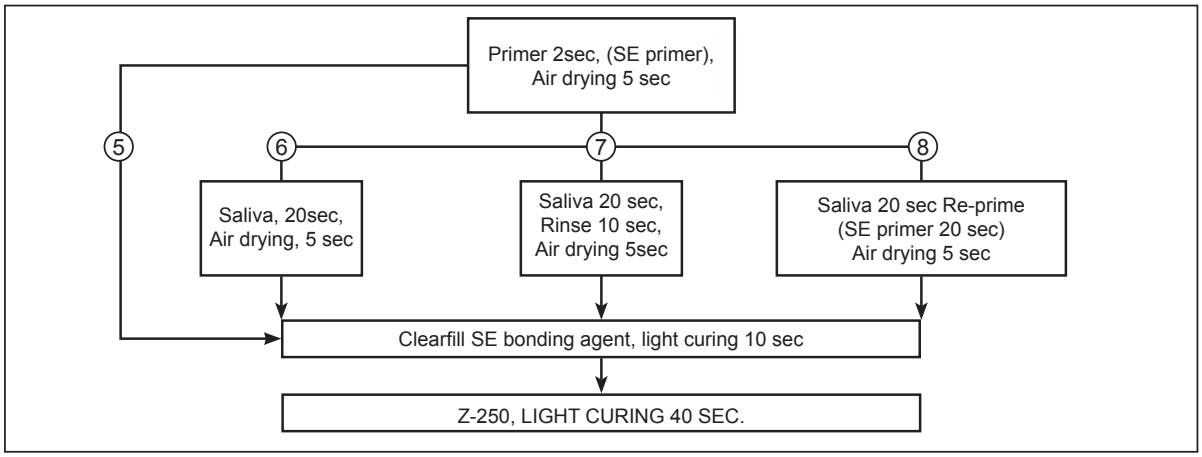


Fig 2: Bonding procedure for ClearFill SE bond

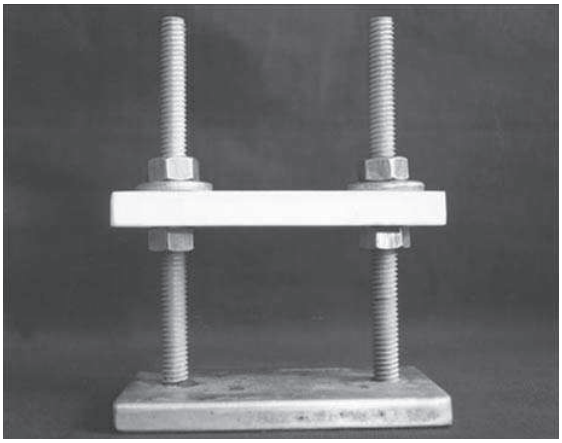


Fig 3: Teflon mould with Stabilising device

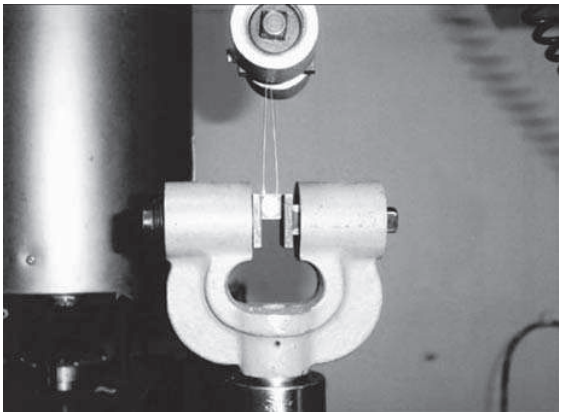


Fig 4: Specimen mounted in the Jig

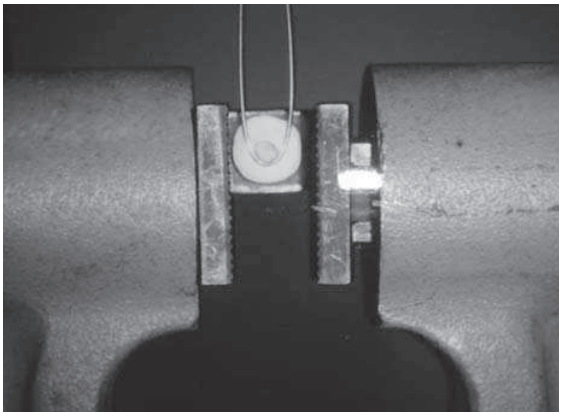


Fig 5: Stainless steel wire loop engaging the composite cylinder close to tooth interface

Table 1: Materials used in the study

Material	Type	Manufacturer
Scotchbond multipurpose	35% phosphoric acid	3MESPE, GERMANY
Adper Single bond	Fifth generation bonding agent	3MESPE, GERMANY
Clearfill SE bond	Sixth generation bonding agent	Kuraray, Japan
Filtek Z-250	Hybrid composite resin	3M ESPE, GERMANY
DPI- RR	Self cure Acrylic resin	DPI, INDIA

Table 2: Mean, Standard Deviation

Group	Sample No.	Mean (MPa)	Standard Deviation
Group 1	10	21.143	3.456
Group 2	10	9.71	1.047
Group 3	10	18.16	3.85
Group 4	10	18.87	3.70
Group 5	10	20.29	2.95
Group 6	10	10.70	1.91
Group 7	10	11.77	2.01
Group 8	10	20.13	2.97

Table 3: ANOVA – One Way ANOVA of Single Bond Groups

Source of Variation	Sum of Squares	d.f	Mean squares	F	Significance
Between groups	751.5	3	250.5	23.94	0.000
Error	376.5	36	10. 6		
Total	**1128.1**	**39**			

Table 4: ANOVA – One Way ANOVA of Clearfill SE Bond Groups

Source of Variation	Sum of Squares	d.f	Mean squares	F	Significance
Between groups	792.4	3	264.1	42.07	0.001
Error	225.9	36	6.277		
Total	1018.5	39			

($P < 0.001$ i.e., there is highly significant difference between the groups; d.f – degrees of freedom)

Table 5: Comparison of Single Bond and Clearfill SE Bond groups

Between groups	Degrees of freedom	'T' value	'P' value	Inference
Group 1 – Group 2	18	10.01	< 0.001	Significant
Group 1 – Group 3	18	1.815	0.063	Not Significant
Group 1 – Group 4	18	1.418	0.0966	Not Significant
Group 2 – Group 3	18	-6.64	< 0.001	Significant
Group 2 – Group 4	18	-7.52	< 0.001	Significant
Group 3 – Group 4	18	-0.41	0.3433	Not Significant
Group 5 – Group 6	18	8.60	< 0.001	Significant
Group 5 – Group 7	18	7.539	< 0.001	Significant
Group 5 – Group 8	18	0.12	0.452	Not Significant
Group 6 – Group 7	18	1.22	0.1191	Not Significant
Group 6 – Group 8	18	- 8.43	< 0.001	Significant
Group 7 – Group 8	18	- 7.39	< 0.001	Significant

Discussion

Controlling Saliva contamination in the field of operation is difficult in adhesive dentistry, especially in those cases where cavity margins extend below the gingival tissues or when indirect restorations are seated or in newly erupted molars or when patients have problem in mouth opening[6,7,8]. Dentin bonding systems are sensitive to contamination by saliva, blood and excess water[17]. Therefore adhesive systems capable of tolerating contamination are highly desirable.

The laboratory parameter most often measured in dentin adhesion is shear bond strength[18]. In the present study, the effect of saliva contamination on the shear bond strengths of one bottle (Single Bond) and self-etch primer (Clearfill SE Bond) adhesive systems to dentin were investigated.

A wire loop method was employed to determine the shear bond strength. The wire was engaged as close as possible to the interface to minimize the tensile component of force[19].

Natural saliva was chosen as the contaminant, because artificial saliva may confound the results[5,9,15].

Shear bond testing done at a cross head speed of 0.5 mm / min, though it could be done at various cross head speeds ranging from 0.5mm/min to 5mm/min as at higher speed the shear bond strength of the adhesive interface could not essentially be evaluated[20].

In Single Bond groups

When etched dentin was contaminated with saliva and air dried completely (Group 2), the bond strength decreased significantly. This result was in accordance with the study[6] in which the decreased bond strength values can be explained by the facts that the water filled collagen layer will collapse and that a dried protein film will be adsorbed onto the dentin surface, both phenomena prevent the penetration of the adhesive into the exposed collagen meshwork limiting the formation of a sound hybrid layer.

Rinsing (Group 3) a restored the bond strengths to that of the control group. Rinsing of contaminated etched dentin was apparently able to remove a sufficient amount of protein to let the adhesive monomer to infiltrate the exposed collagen. This result was in agreement with earlier studies done[4,15].

When the contaminated etched dentin was re-etched for 10 seconds (Group 4) the bond strengths were 18.87 MPa. The strengths were restored to that of the control group. 10 second additional phosphoric acid etching has enough acidity to remove saliva, plasma and other contaminants. These results were in accordance to the studies[3]. however the results of the present study demonstrated that re-etching did not restore the mean shear bond strength values close to the control[21]. The difference in results can be attributed to the fact that artificial saliva was used in their study and re-etching was performed for 15 seconds. Re-etching for this long time results in a too thick demineralised dentin and collapse of collagen mesh work preventing penetration of adhesive monomers well into the demineralised dentin

In Clearfill SE Bond groups

When saliva contamination occurred after the application of primer and the saliva was air dried (Group 6) the mean shear bond strengths decreased significantly matching earlier study[7]. This can be explained by the fact that the collagen layer will collapse and that a dried protein film will be adsorbed onto the dentin surface, both phenomena prevent the penetration of the adhesive into the exposed collagen meshwork limiting the formation of a sound hybrid layer.

In the present study when the primed contaminated dentin was rinsed to wash the saliva (Group 7) the bond strengths were not restored to control. This was in consistence to studies[22, 23]. A hydrophilic monomer such as HEMA in the self etching primer would be rinsed away by water easily from the demineralised dentin, which might result in collapse of the collagen when the dentin surface was air dried after rinsing. The collapsed collagen limited the size of the space between the fibres and the adhesive agent was not able to penetrate effectively.

When the primed contaminated dentin was re-primed for 20 seconds (Group 8) the values were restored close to the control. These findings were consistent with previous study[16] where the bond strengths were recovered owing to the fact that the hybrid layer reformed after the removal of the unstable contaminated primer layer.

In the present study there was no significant difference in shear bond strength values for single bond and Clearfill SE Bond to uncontaminated dentin. This can be justified by the reasoning that in the contemporary self etch primers the traditional acids have largely been replaced with acidic resin monomers such as 10 – Methacryloyloxy decyl dihydrogen phosphate (10-MDP) whose acidity is less than that of 32-37% phosphoric acid but is sufficient to etch through smear layers into the underlying dentin. Also Yoshida E, Hashimoto M, Kaga M (2004) stated that 10-MDP has been shown to exhibit high chemical interaction capacity to hydroxyapatite resulting in good performance of Clearfill SE comparable to the one bottle adhesive systems[24].

Conclusion

- The highest shear bond strengths were recorded in the uncontaminated, control groups of the single bond and clearfill SE Bond adhesives.
- Saliva contamination after etching significantly reduced the bond strengths of single bond adhesive when the saliva was completely dried.
- Saliva contamination after the application of self etch primer significantly reduced the bond strength of clearfill SE bond.
- Rinsing after saliva contamination restored the bond strengths of Single Bond to that of control while rinsing did not restore the bond strengths of clearfill SE Bond.
- Re-etching and Re-priming restored the bond strengths to that of the control group in Single Bond and Clearfill SE Bond adhesives.

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