ORIGINAL ARTICLE

THE EFFECT OF DIFFERENT SURFACE TREATMENTS ON SHEAR BOND STRENGTH OF A LOW SHRINKAGE COMPOSITE TO ENAMEL

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ABSTRACT

This study evaluated the effect of different surface treatment protocols on Shear bond strength of a low shrinkage Silorane-based composite to enamel. Twenty four extracted molars were used. The convex enamel surfaces were reduced by silicone carbide paper to obtain a flat enamel surface. The teeth were distributed into four groups according to the treatment protocols. Etch and rinse adhesive system (Excite, Ivoclar Vivadent, Schaan, Liechtenstein) with methacrylate-based composite (Tetric N Ceram, Ivoclar Vivadent Schaan, Liechtenstein) was used as a control group. Three adhesive application modalities were used for teeth restored with silorane-based composite (Filtek P90 / P90 Adhesive System, 3M ESPE St. Paul, MN, USA) as follows: according to the manufacturer's instructions (self-etching), pre-etching with phosphoric acid and with double application of the self-etching primer. The specimens were thermocycled and shear bond strength was determined in Instron Universal Testing Machine at a crosshead speed of 0.5mm / min. No significant difference was found between etch & rinse group and phosphoric acid pre- etched Filtek P90 group; both were significantly higher than that of Filtek P90 self-etching and the double self-etch primer application. The double self-etch primer application Filtek P90 group showed the lowest strength when compared to other groups. It can be concluded that pre-etching with phosphoric acid results in better bond performance of silorane based composite specific self-etching adhesive system to enamel.

Key Words: *Enamel bond strength, adhesives; low-shrinkage, Silorane based-composite, selective etching.*

INTRODUCTION

The past two decades have seen rapid progress to improve dental composite material, adhesive bonding systems and technologies as well as simplifying the clinical application to dentin and enamel. The major shortcoming of the composite resin material is shrinkage due to polymerization, which is an important factor in shrinkage stress development.[1] This stress can cause detrimental tooth changes such as marginal gaps which could lead to post-operative sensitivity, microleakage, and secondary caries.[2,3] The well-known shortcomings of composites have produced a continuous effort on the part of clinicians and researchers to provide several restorative techniques and materials to reduce stress development.[2,4,5] Guggenberger and Weinmann[6] have proposed an oxirane-based resin formulation to overcome the disadvantages of polymerization shrinkage of resin based composites (RBCs). A resin chemistry has been developed from the reaction of oxiranes and siloxane molecules and termed 'silorane'.

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Dentin bonding agents provide micromechanical retention for the resin-based composite restorations leading to an improved bond strength between the resin and the tooth structure and minimizing the microleakage across dentin-resin interface as well as distributing the occlusal stress evenly within the cavity walls.[7] One of the biggest areas of development has been to simplify the bonding procedure by the use of self-etching adhesives. Recently, bonding to tooth structure with self-etching adhesive systems has gained popularity because they contain specific acid monomers to condition and prime the tooth substrates simultaneously reducing the application procedure and technique sensitivity. Moreover, it was reported that some functional monomers in self-etching adhesives could interact with the hydroxyapatite in the partially demineralised structure which, hypothetically, can improve the bond strength.[8,9,10,11] When restoring teeth with silorane-based composites, the manufacturers recommend a two-step self-etching adhesive system (P90 Adhesive System, 3M ESPE) that was developed exclusively for this purpose. P90 System Adhesive Self-Etch Primer is a hydrophilic mixture of phosphorylated methacrylate, acid copolymers, methacrylate-based monomers of Bisphenol A-Glycidyl dimethacrylate (Bis-GMA) and Hydroxyethyl-methacrylate (HEMA) dissolved in water and ethanol solvent system. The high

content of HEMA in the primer will make it susceptible to water sorption. The specific adhesive of the system (P90 System Adhesive Bond) is methacrylate-based with hydrophobic monomers. Its function is to bond the primer and the hydrophobic monomer of composite.[12]

Unlike the obvious position of self-etching adhesive in dentin bonding, bonding to enamel is a controversial issue. Some studies indicated that contemporary self-etching adhesives could be used successfully when bonding to enamel[13,14,15], whereas others reported that the self-etch approach is adequate for dentin but revealed some limitations in bonding to enamel.[16,17,18,19] Bond strength in particular was found to be lower when compared to that of etch and rinse adhesive systems. Therefore, preliminary phosphoric acid etching was recommended to improve the adhesion to enamel.[19,20,21]

The aim of this study was to evaluate the effect of different surface treatment protocols on the shear bond strength of low shrinkage Silorane-based composite to enamel. The null hypothesis tested was that there is no significant difference between bond strength of Silorane-based self-etch adhesive system (P90 Adhesive System) to enamel when compared to etch and rinse adhesive system irrespective of the application protocol used.

METHODOLOGY

The study was conducted at the Department of Restorative Dental Sciences of King Saud University College of Dentistry. Twenty four freshly extracted teeth were collected from maxillofacial surgery clinic and used in the study. The teeth were cleaned and kept refrigerated in distilled water and 0.02% thymol. The crowns were sectioned mesio-distally using a diamond saw (Isomet 2000, Buehler 41 Waukegan Road, Lake Bluff, IL 60044, USA) and the teeth were embedded in customized teflon moulds filled with self-cure acrylic resin (Orthoresin, Dentsply, UK). The convex enamel surfaces were reduced to 0.5 mm by using 240 and 400 grit silicone carbide paper (Automata, Jeanwirtz, GMBH, West Germany) under water cooling to obtain a flat enamel surface. The mounted teeth were randomly distributed into four groups each containing 12 samples. Table 1 shows different restorative materials used in this study.

A split Teflon mold, 5 mm in diameter and 2mm long, was placed over each tooth, perpendicular to the polished surface. A metal ring was used to secure the mold and the appropriate material was placed.

In Group 1, etch and rinse adhesive system was used. 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc, UT, USA) was applied for 15 seconds, rinsed for 10 seconds, and excess water was removed with air blast for three seconds. The Excite F adhesive (Ivoclar, Vivadent, Schaan, Liechtenstein)was then agitated on the prepared surface for 10 seconds, dispersed to a thin layer with a weak steam of air and light cured for 10 seconds. Tetric –N- ceram (Ivoclar Vivadent, Schaan, Liechtenstein) composite was placed after that using the Teflon mold and light cured for 20 seconds following the manufacturers' instructions.

The direct restorative filling used in Group 2 was the low-shrinkage silorane-based composite Filtek P90(3M, ESPE, St. Paul, MN, USA) with its specific self-etching adhesive system following the manufacturers' instructions. P90 System Adhesive Self-Etch Primer was applied, agitated on the prepared surface for 15 seconds then light cured for 10 seconds. P90 System Adhesive Bond was then applied, dispersed to a thin layer with a weak steam of air and light cured for 10 seconds. Filtek P90 composite was placed after that using the Teflon mold and light cured for 20 seconds following the manufacturers' instructions.

In Group 3, the prepared surface was first etched by 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc, UT, USA) for 15 seconds, then rinsed for 10 seconds and excess water was then removed with air blast for three seconds before the application of Filtek P90 adhesive and composite (3M, ESPE, St. Paul, MN, USA) in the same manner of Group 2.

In Group 4, Double application of the P90 System Adhesive Self-Etch Primer (3M, ESPE, St. Paul, MN, USA) was done. The first layer of the Self-Etch Primer was agitated on the prepared surface for 15 seconds then light cured for 10 seconds. A second layer of the Self-Etch Primer was then agitated for 15 seconds then light cured for 10 seconds. P90 System Adhesive Bond was applied after that, dispersed to a thin layer with a weak steam of air and light cured for 10 seconds. Filtek P90 composite was placed after that using the Teflon mold and light cured for 20 seconds.

All the specimens were light cured using Elipar Highlight (3M, ESPE, St. Paul, MN, USA) Intensity of the light was monitored periodically with a radiometer (Demetron/Kerr, Danburg, CT, USA) to ensure a minimum value of 500 mW/cm.^{2} For all the specimens, the curing light was held two mm away from the restoration and each layer was cured according to the manufacturers' instructions. The specimens were stored in distilled water at 37°C for 24 hours. They were thermocycled for 5000 cycles at a temperature differential of 5C to 55 C with 30 second dwell intervals and 5 second transit time between baths (Huber THE 1100/1200, SD Mechatronik GmbH, Feldkirchen-Westerham). The specimens were mounted with the treated surfaces parallel to the shearing rod of the Instron Universal Testing Machine (Instron Cor. 8500 Canton, Massachusetts, USA) and sheared to failure at a cross head speed of 0.5mm/min and the results recorded in Mega Pascals (MPa). The testing was carried out at room temperature of 23°C and relative humidity of 50%.

One way analysis of variance (ANOVA) was used to detect any significant differences ($p \leq 0.05$) in bond strengths among the groups. Post hoc comparisons were made using the Tukey HSD test. The failed surfaces were examined under a light microscope (Travelling Mic., By TITAN Measuring Microscope, Buffalo, N.Y.) at a magnification of x10 and the mode of failure of the specimens were recorded according to the following categories;

1. Adhesive failure at the enamel – restoration interface (no composite on enamel surface).

2. Cohesive failure in the composite if remnants of the composite remained on enamel.
3. Mixed failure in the enamel and composite

Percentages were used to explain the mode of failure across the four groups.

RESULTS

The shear bond strengths of the four groups showed statistically significant difference (Tables 2 & 3). The shear bond strength of the etch & rinse group and phosphoric acid pre- etched Filtek P90 groups was significantly higher than that of Filtek P90self-etching group and double self-etching primer application group. No significant difference was found between etch & rinse and phosphoric acid pre- etched Filtek P90 groups. The double self-etch primer application Filtek P90 group showed the lowest strength when compared to other groups (Fig 1).

TABLE 1: RESTORATIVE MATERIALS USED IN THIS STUDY

Materials & Treatments	Category	Batch No. & Shade	Composition
Ultra-Etch, Ultra-dentProducts Inc, UT, USA)	Etchant	H053	35% phosphoric acid
Excite F, Ivoclar Vivadent, Schaan, Liechtenstein	Ad1esive bond	P24171	Light cured adhesive, fluoride releasing, single component total etch adhesive. phosphonic acid acrylate, HEMA, dimethacrylate, highly dispersed silicone dixoide, initiators, stabilizers and potassium fluoride alcohol.
Tetric-N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein	Nanohybrid composite	P30262, A2	Dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments. 55-57% volume. 40 nm - 3000 nm
Filtek P90, 3M ESPE, , St. Paul, MN, USA	Low Shrinkage Posterior Restorative	N297243, A2	Silorane resin, Initiating system: camphorquinone, Yttrium fluoride, Stabilizers, Pigments, Quartz filler, 55 % volume, 0.1-2 µm
P90 System Adhesive Self-Etch Primer, 3M ESPE, St. Paul, MN, USA	Self-Etch Primer	N225306	Phosphorylated methacrylates , Vitrebond™ copolymer, BisGMA, HEMA, Water, Ethanol , Silane-treated silica filler, Initiators, Stabilizers
P90 System Adhesive Bond, 3M ESPE	Adhesive Bond	N225307	Hydrophobic dimethacrylate, Phosphorylated methacrylates, TEGDMA, Silane-treated silica filler, Initiators, Stabilizers

TABLE 2: SHEAR BOND STRENGTH MEANS AND STANDARD DEVIATIONS FOR THE FOUR GROUPS

Materials & Treatments	N	Mean (Mpa)	Std. Deviation	Std. Error	95% Confidence Interval for Mean	
					Lower Bound	Upper Bound
Tetric N Ceram, Etch & Rinse	12	13.436583	4.1360526	1.1939755	10.808661	16.064506
Filtek P90, Self-etching	12	6.940250	2.2106061	.6381470	5.535698	8.344802
Filtek P90, Phosphoric acid pre-etched	12	11.751500	2.3411450	.6758303	10.264007	13.238993
Filtek P90, Double self-etch	12	1.601250	.8766192	.2530582	1.044273	2.158227
Total	48	8.432396	5.3197089	.7678338	6.887713	9.977078

TABLE 3: RESULTS FOR BONFERRONI MULTIPLE COMPARISONS SHOWING HOMOGENEOUS SUBSETS OF MEAN FRACTURE STRENGTH FOR THE FOUR TREATMENT GROUPS

Materials & Treatments	Subsets for alpha (p ≤0.05)		
	1	2	3
Tetric N Ceram, Etch & Rinse	13.436583		
Filtek P90, Phosphoric acid pre-etched	11.751500		
Filtek P90, Self-etching		6.940250	
Filtek P90, double self-etch			1.601250

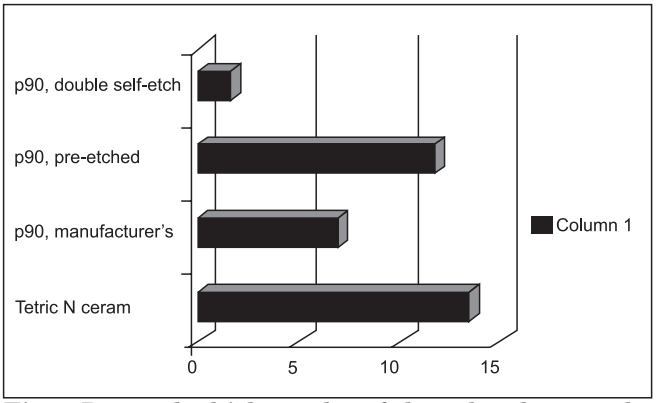

Fig 1: Box and whiskers plot of shear bond strengths among the groups

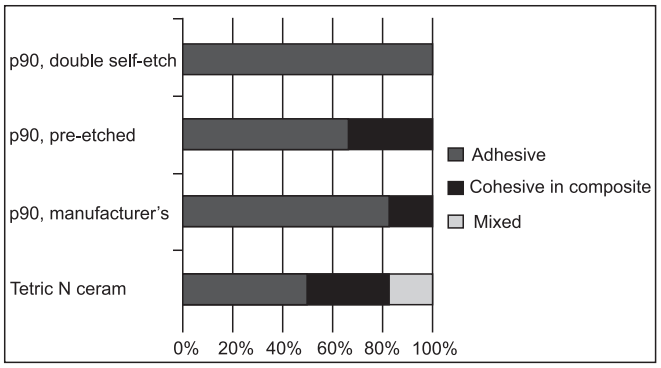

Fig 2: Stacked bar showing the distribution of the failure modes for all groups

With regard to the mode of failure, no premature failures were found. The prevalence of adhesive mode of failure of the etch & rinse group was 50%, cohesive in composite was 33.3% and mixed was 16.7%. In Filtek P90 Self-etching group, adhesive mode of failure was 83.3% and 16.7% of cohesive in composite and for the phosphoric acid pre-etched Filtek P90 group was 66.7%

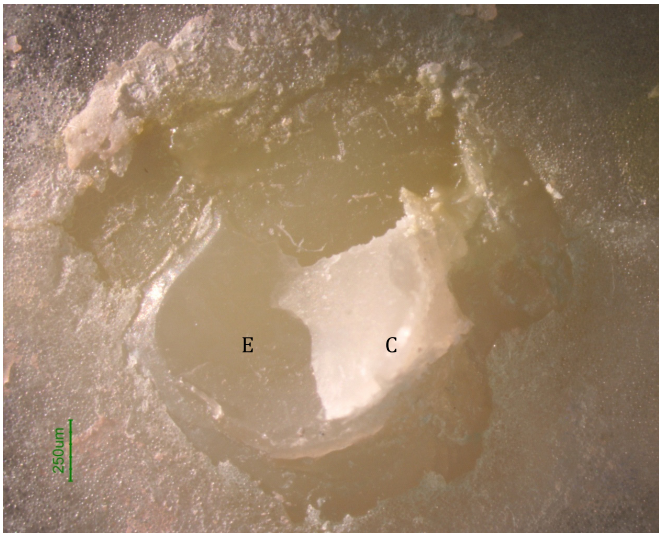

Fig 3: Mixed failure on the enamel (E) and composite (C)

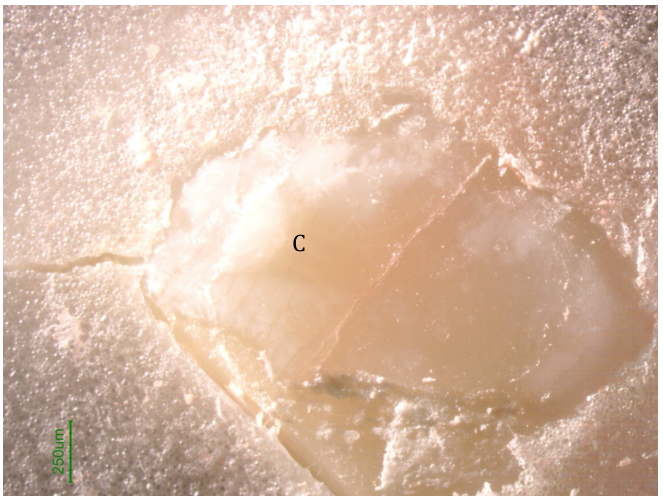

Fig 4: Cohesive failure in the composite: remnants of composite on enamel (C)

Fig 5: Adhesive failure at enamel-adhesive interface (no composite on enamel)

of adhesive and 33.3% of cohesive in composite, whereas the double self-etch primer application Filtek p90 group had 100% adhesive mode of failure (Fig 2). Fig 3, 4 & 5 show different modes of failure under microscopic examination.

DISCUSSION

The results of this study showed a significant difference between bond strength of Silorane-based composite self-etch adhesive system to enamel when compared to etch and rinse adhesive system and to the different application protocols tested, therefore, the null hypothesis was rejected. The results of this study also showed that etch and rinse adhesive system revealed higher bond strength when compared to the specific self-etch adhesive system (P90 Adhesive System) used with silorane-based composite Filtek P90. Given the fact that enamel is composed mainly of minerals and contains little organic compounds, proteins and water, the ability of resin to bond reliably to the etched enamel surface by micromechanical mean is well accepted. Micro-mechanical retention results from the formation of resin tags that fill the micro-porosities ranging in depth from 5-50 µm produced by phosphoric acid as it causes selective dissolution of either enamel prism cores or boundaries.[16,22,23,24]

The self-etch P90 Adhesive System demonstrated lower bond strength when compared to etch and rinse adhesive system even when the surface was agitated with the etching primer in attempt to increase the effectiveness of the acidic monomers. The less than desirable results in this investigation of the two-step self-etching adhesive system used with Silorane-based composite, may have been due to the mild acidity of the monomer (pH 2.7).[12] At this pH level, the adhesive is not capable of intensely etching the enamel and thus provides a lower degree of demineralization and further infiltration of the enamel surface when compared to phosphoric acid conditioning which in turn compromises the adhesion to enamel. This result is in agreement with several previous studies which reported a reduction in enamel bonding efficacy as a major disadvantage of the self-etch protocol.[25,26,27] On the other hand, a study done by Abdallah et al[28] revealed that some self-etching adhesive systems showed bond strength values comparable to that of etch-and-rinse system when the micro-shear bond strengths to ground enamel was evaluated. It is believed that enamel adhesion is a two-fold mechanism; one is micromechanical interlocking and the other is chemical interaction between functional monomers of adhesives and enamel, the later mode, however, is dependent on the type of adhesive system. This is in agreement with Rosa et al[26] who reported that the different formulations among adhesive systems might play role on the material performance. Therefore, not all self-etch adhesives will bond to enamel as effectively as etch and rinse adhesives.

Based on the results obtained, etching of the surface with phosphoric prior to Self-etching adhesive system improved the bond strength and showed no significant difference when compared to the total etch adhesive system as it helps to increase the impregnation of the monomers. Several previous studies recommended to combine etch and rinse treatment at enamel with mild self-etch approach.[20,26,29,30,31] This combined adhesive protocol was applied clinically with success in a long-term clinical trial by Peumans et al.[15]

Double application of P90 System Adhesive Self-Etch Primer on the enamel surface resulted in the least bond strength when compared to other groups. The specific adhesive system of silorane-based composite differs from the majority of two-step self-etching systems. It requires separate light activation steps for the primer and the adhesive whereas the primer and adhesive in other two-step self-etching systems are polymerized simultaneously in a one-time light-curing step. The individual light curing of the primer and the adhesive of this system is beneficial as it provides stability of the composite-tooth substrate bond and long-term durability of the hybrid layer.[32] Mine et al 2010[32] examined the ultra-structure of the hybrid layer formed between the enamel/dentin and the adhesive system of a silorane based composite and observed nano-leakage along the adhesive interface of the silorane system, between the primer and adhesive, and within the primer itself, indicating the presence of porosities. The authors explained that the water-based primer containing the hydrophilic HEMA component, which makes evaporation of excess solvent (water) with air drying very difficult, resulting in failure at the primer/adhesive interface. Therefore, it was speculated that double application of P90 System Adhesive Self-Etch Primer might result in a thick adhesive interface with more porosities within the primer, between the first and second layer of the primer and between the primer and adhesive leading to poor bond strength displayed by this group in the present study when compared to other groups. Moreover, the first layer of the primer may create a barrier after curing that prevents further etching of the surface when the second layer of the primer was applied. Its believed that prolonged application of the P90 System Adhesive Self Etch Primer might be more beneficial to the bond strength than double application.

The most prevalent failure in our study was cohesive in Tetric N Ceram when etch and rinse adhesive system was used or when the surface was etched with phosphoric acid prior to the application of P90 System Adhesive self-etching primer in Feltik P90 composite, this is in agreement with previous studies.[31,33] Cohesive failures may indicate a higher mechanical strength of the adhesive.[31,33] Mittal[34] reported that separation of the adhering phases is a way to measure adhesion experimentally. Separation in the bulk of the weaker adhering phase is called cohesive failure and related to the cohesive strength of that bulk phase.[34] In another word, cohesive failure within the composite occurs when the bond strength between the enamel and composite is stronger than cohesive strength of the restorative material. Therefore, the material fails before the restoration debonds. The two-step self-etch P90 Adhesive System used revealed less cohesive failure and more frequent adhesive failures while double application of P90 System Adhesive Self-Etch Primer group showed adhesive failures in all specimens. The increase in adhesive failures maybe due the reduced etching ef-

fect on the enamel surface, thus reducing surface area available for adhesion as reported by Pires et al.[31]

This in vitro study had some limitations. A conventional shear bond strength test was used, where the load was not being distributed uniformly to the adhesive interface. Moreover, the adhesive interface is subjected to different stress loading other than compressive and to temperature changes in the oral cavity, which may affect the behavior of the material intraorally.

CONCLUSION

- Etch and rinse adhesive system had the highest bond strength to ground enamel.
- Pre-etching with phosphoric acid resulted in better bond performance of silorane-based composite specific self-etch adhesive system to enamel.
- Double application of silorane-based composite specific Self-Etch Primer in attempt to over-etch enamel surface and improve the bond resulted in low strength and should be avoided clinically.

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