The effect of pre-etching of dentine, cut and uncut enamel on the shear bond strength of silorane-based and methacrylate-based composite resin systems

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SUMMARY

Introduction: Modern composites are exceptional physically and aesthetically, but success is dependent on effective bonding to tooth structures, possibly enhanced by phosphoric acid pre-etching.

Aims: To determine the effect pre-etching of tooth surfaces has on the shear bond strengths of silorane-based and of methacrylate-based composite resin systems.

Method: To 120 recently extracted human molars and pre-molars, composite resin stubs were bonded with Silorane System Adhesive or Scotchbond Universal, to pre-etched or to non-pre-etched tooth surfaces, using an Ultradent jig. After thermocycling, each specimen was debonded under a shear force, in an Instron Universal testing machine (crosshead speed 0.5mm/minute).

Results: Data were subjected to an analysis of variance and when significant, to pairwise comparisons. The silorane-based system achieved significantly higher mean shear bond strengths (MPa) on pre-etched surfaces of: uncut enamel (15.204 vs 9.424), cut enamel (21.352 vs 12.110) and dentine (19.787 vs 8.278). Methacrylate-based bonds to pre-etched uncut enamel were significantly stronger (28.898 vs 18.451). Pre-etching cut enamel enhanced the bond (20.548 vs 16.384, not significant). Pre-etched dentine recorded lower mean bond strengths (15.288 vs 19.645) also not significantly.

Conclusions: The effect of pre-etching on the shear bond strength to uncut enamel, cut enamel, and dentine was product-specific.

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ACRONYMS

FS: Filtek Silorane
FSX: Filtek Supreme XTE
HEMA: 2-hydroxyethyl methacrylate
SBS: shear bond strength
SEP: self-etching primer
SSA: Silorane System Adhesive
SUA: Scotchbond Universal Adhesive

INTRODUCTION

The increasing popularity of dental composites as restorative materials on posterior teeth has forced manufacturers to contend in the on-going 'Battle of the Bonds', in order to remain competitive in the constantly advancing and innovative world of dental materials. The introduction of the sixth and seventh generation bonding agents in the late 1990's enabled the dental fraternity to finally eliminate the problems of technique, and of post-operative sensitivity. These "self-etching adhesives" have further contributed to the increasing popularity of dental composites.

The major disadvantage with composites has always been polymerisation shrinkage and its associated polymerisation stress. Volumetric shrinkage, in the range of <1% up to 6%, has been reported for filled dimethacrylate-based dental composites.[1] This polymerisation shrinkage results in an associated polymerisation stress on the tooth-resin interface which may lead to: microleakage, marginal staining, tooth deformation, enamel cracks, stress-induced post-operative sensitivity and eventually failure of the restoration.[2] This is more pronounced in high 'C-factor' cavities. The 'C-factor' is defined as the ratio of bonded to unbonded areas within a restored cavity.[1,3,4] The higher the C-factor, the lower the potential for plastic deformation and relaxation of the composite resin (i.e. the less likely a restoration can withstand shrinkage).[1,4] In 2007 3M ESPE introduced Filtek Silorane, the first direct posterior composite displaying < 1% polymerisation shrinkage.[2] Even though reduced polymerisation shrinkage has been achieved with this silorane-based composite, it comes at the cost of convenience.[2] Since the curing of this resin system involves chemical mechanisms different from those of conventional methacrylate-based composites, a dedicated adhesive is needed to bond to the silorane-based composite.[2,5] Incremental layering of the composite material has always been and is still recommended as one of the main techniques useful in minimising the effect of polymerisation shrinkage in high C-factor cavities.[1] Other clinical techniques include: modifying cavity configurations; directional curing techniques; variations in curing modes (such as "Soft Start", "Ramped curing" and "Delayed curing") as well as the use of stress breaking liners.[3,5,6,7]

Contemporary dental composites display exceptional mechanical properties, low polymerisation shrinkage and pleasing aesthetics, but the success of the material is ultimately dependent on its ability to bond to tooth structure. The golden question then arises: "What is a clinically acceptable bond strength?" An answer to this question was hypothesised by Munksgaard in 1985 and Retief in 1994. They proposed that a minimum bond strength of 17 MPa to tooth structure was required for successful adhesion.[8] If the bond strength to tooth structure is less than 17 MPa, it means that the polymerisation shrinkage of the composite resin is greater than the force adhering the material to the tooth.[8] Therefore, polymerisation shrinkage causes the resin to contract toward the centre of the composite, thus pulling the restorative material away from the walls of the cavity.[8] This leads to marginal gap formation which then allows the micro-infiltration of bacteria which will eventually lead to marginal breakdown and composite failure.[8] When bond strengths to tooth structure exceed 17 MPa, the shrinkage of the composite is toward the walls of the cavity.[8] "The laws of entropy dictate that the polymerisation contraction process always tends to go in the direction of least resistance (or higher attraction)"[8] and since the composite is more attracted to the tooth surfaces than it is to itself, shrinkage occurs toward the walls of the cavity and away from the centre, resulting in no marginal gap formation.[8] Therefore, adhesive systems must have bond strengths greater than 17 MPa to be considered clinically acceptable.[8,9,10] Bond strengths to enamel and dentine should also be relatively equal.[8] If, for instance, the adhesion to enamel is much greater than the adhesion to dentine, during polymerisation the stronger force at the enamel interface will pull the composite away from the dentinal interface thus weakening it.[8]

The idea of etching enamel in order to improve bonding of restorative materials, all began in 1955 when Dr Michael Buonocore published a paper titled "A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces".[8,11] The acid etching of dentine on the other hand was initially frowned upon. In 1979 Fusayama and colleagues made a breakthrough, and published a study demonstrating that acid etching of dentine substantially improved adhesion of composite restorations, without the risk of pulpal damage.[11,12] However, etching of dentine was only widely embraced in the early 1990's.[11]

The introduction of the self-etching sixth and seventh generation bonding systems in the 20th century, eliminated the need for the initial step of acid etching. The main concern with these bonding systems, however, is their mild acidity and short tooth application time resulting in insufficient etching of enamel.[8] It is not surprising that to date many clinicians, opinion leaders and researchers still favour the "total etch" bonding systems.[8,13]

The null hypotheses to be tested were as follows: 1) Pre-etching of dentine, cut and uncut enamel does not significantly affect the shear bond strengths of the silorane-based composite resin system. 2) Pre-etching of dentine, cut and uncut enamel does not significantly affect the shear bond strengths of the methacrylate-based composite resin system. Thus, the aim of this study was to determine the effect that pre-etching of dentine, cut and uncut enamel would have on the shear bond strengths of both a silorane-based and a methacrylate-based composite resin system.

MATERIALS AND METHODS

The Silorane System Adhesive (SSA) (Lot number: N322114) and its paired low shrinkage posterior restorative composite Filtek Silorane (FS) (Lot number: N344850) manufactured by 3M ESPE (Seefeld, Germany), was one of the systems chosen for this study. SSA is classified as a two-step, mild, sixth generation type I self-etching bonding system.[2] It is a two bottle system, a self-etching primer (SEP) with a pH of about 2.7 and an adhesive bond. The acidic monomer used in the SEP is an unidentified phosphorylated methacrylate. SEP also contains a "Vitrebond copolymer" with carboxylic acid functionality.[2] Other components include: bisphenol A diglycidyl methacrylate (Bis-GMA); 2-hydroxyethyl methacrylate (HEMA); 1,6-hexanediol dimethacrylate (HDDMA); silane-treated silica filler particles and a camphorquinone-based photo-initiator.[2,14] This SEP uses both water and ethanol as solvents.[2] The functionality of Silorane Adhesive is based on methacrylate chemistry, for it contains as a main component a hydrophobic bifunctional monomer that allows for easy adaptation of Filtek Silorane composite on the cured adhesive layer.[2]

The methacrylate-based bonding agent Scotchbond Universal Adhesive (SUA) (Lot number: 455901) together with the nanocomposite Filtek Supreme XTE (FSX) (Lot number: N340262) (3M ESPE, Seefeld, Germany), was the other set of materials selected for this study. SUA is classified as a mild seventh generation self-etching bonding agent, with a pH of about 2.7.[15] It is a single solution system containing methacryloyloxydecyl dihydrogenphosphate (MDP); dimethacrylate resins; HEMA; a "Vitrebond copolymer"; filler particles; initiators and silane in an ethanol/water based solvent.[15] The paradox surrounding Scotchbond Universal, however, is that it can be used in 'self-etch', as well as in 'total etch' and 'selective etch' modes. This means that the operator can choose to pre-etch all, or selected, tooth structures with 34 % phosphoric acid before application of this single solution adhesive system.

In this *in vitro* study, 120 caries-free posterior teeth were collected from Medunsa Oral Health Centre exodontia clinic and other surrounding clinics in the northern parts of Pretoria. The sample size was determined from similar previous studies.[16,17,18] All teeth were stored in a 0,5 % solution of Chloramine T trihydrate (Merck Group, Darmstadt, Germany) during the collection period. The teeth were first cleaned, ensuring removal of any residual periodontal tissue and calculus, and mounted in Bencor stainless steel rings (Danville Engineering, San Ramon, CA, USA), using Excel self-curing acrylic (Wright Health Group, Kingsway, West Dundee, Scotland). The teeth were mounted with the bonding surfaces parallel to the base of the ring to ensure a force application perpendicular to the long axis of the resin stub.

Eighty teeth were randomly selected, de-rooted and mounted with their buccal or lingual surfaces facing upwards. Forty of the eighty teeth were ground to a flat surface, removing about 0.5 mm of the superficial layer of enamel on the buccal/lingual surfaces, using an Imptech grinder (Innovative Met Products, Boksburg, South Africa) fitted with P600 grit SiC sandpaper under water cooling. The ground enamel surface was used as the bonding site for cut enamel. The other 40 teeth were used for bonding to uncut enamel. The remaining 40 of the 120 teeth were mounted with their occlusal surfaces facing upwards, and the middle dentine was exposed by removing approximately ⅓ of the crown using the Imptech grinder fitted with P600 grit SiC sand paper under water cooling, providing suitable bonding sites. Before bonding of the composite resins was carried out, all specimens were stored in distilled water in a Memmert Laboratory oven (Memmert, Schwabach, Germany) at 37°C for at least 24 hours.

An Ultradent mould (Ultradent, Salt Lake City, UT, U.S.A.) with a central opening of 2.3798mm in diameter and 3mm in length, was used in conjunction with the Ultradent bonding jig (Ultradent, Salt Lake City, UT, U.S.A.) when bonding the composite resin stubs to the selected bonding surfaces. Bonding to pre-etched and non-pre-etched tooth substrates was performed strictly according to manufacturer's instructions.

When the SSA and FS combination was utilized for bonding to substrates that were not pre-etched, each specimen was first cleaned using an aqueous slurry of fine pumice powder, gently rinsed and dried with oil- free, dried, compressed air. This was followed by the application of the SSA-Self-Etching Primer for approximately 15 seconds. This primer was then spread over the bonding surface using a gentle stream of air and thereafter light cured for 15 seconds, using a Mini LED curing light (KaVo Dental, Biberach, Germany) at a light intensity of 500 mW/cm^2. Application of the SSA-Silorane Adhesive Bond to the prepared surface then followed. The film of adhesive was gently spread, using a stream of air and light cured for 15 seconds. Each tooth specimen was then clamped in the Ultradent bonding jig, ensuring that the prepared surface of the tooth was perpendicular to the long axis of the central opening of the mould. The central opening of the mould was then sequentially packed with two equal increments of FS composite resin material and light cured for 40 seconds per increment, using the Mini LED curing light. The Ultradent jig was then loosened and removed from the bonded specimen by gently applying downward pressure on the resin stub using a small amalgam plugger, together with a steady upward lifting force on the mould.
When bonding to pre-etched substrates, specimens were first cleaned with pumice, rinsed and dried as described earlier. Enamel and dentine surfaces were then etched with 34% phosphoric acid for 20 and 15 seconds respectively. The phosphoric acid was rinsed off and each specimen gently air dried with oil- free, dried, compressed air. The remaining steps for SEP application, adhesive application and resin stub bonding were then carried out as described above.

When SUA and FSX was utilised for bonding to substrates that were not pre-etched, each specimen was first cleaned, rinsed and air dried as described earlier, followed by the application of the single solution SUA. The solution was rubbed onto the bonding surface for approximately 20 seconds, before it was spread over the entire bonding surface using a gentle stream of air and thereafter light cured for 15 seconds. Each tooth specimen was then clamped in the Ultradent bonding jig, as described earlier. The central opening of the mould was packed with FSX composite resin material in two equal increments and light cured for 20 seconds per increment. The Ultradent jig was then loosened and the bonded specimen gently removed as described earlier.

When bonding SUA and FSX to pre-etched substrates, specimens were cleaned with pumice as described earlier, and enamel and dentine surfaces then etched with 34 % phosphoric acid for 20 and 15 seconds respectively. The phosphoric acid was rinsed off and each specimen gently air dried with oil-free, dried, compressed air. The remaining steps for SUA application and resin stub bonding as described in the paragraph above were then followed.

When bonding to pre-etched dentine using either system, caution was exercised not to desiccate dentine, but to only remove the excess water and to leave a glistening surface.

All 120 prepared, bonded specimens were stored in distilled water in a Memmert laboratory oven at 37°C for no longer than 24 hours before they were subjected to 500 cycles of thermocycling,at a speed of 1 minute per full cycle (between 5±2˚C and 55±2˚C), with a dwell time of 30 seconds per bath. This method is consistent with the ISO/TS 11405 technical specifications recommendations of 2003.[19]

Immediately after thermocycling, each specimen was secured in an Ultradent ring clamp (Ultradent, Salt Lake City, UT, U.S.A.) and subjected to debonding under a shear force in an Instron Universal testing machine (Model 3366, Instron, Norwood, MA, U.S.A.). The resin stubs were debonded, using a notched shear castle attachment head at a crosshead speed of 0.5 mm/minute. The software package Bluehill version 2 (Instron, Norwood, MA, U.S.A.), loaded on a desktop personal computer with a Microsoft Windows XP (Microsoft Corp., Redmond, WA, U.S.A.) operating system, was connected to the Instron Universal testing machine in order to record the measurements. The maximum compressive load was recorded in Newtons and the maximum compressive stress at maximum compressive load was calculated in MPa.

De-bonded specimens were examined under a light microscope at 40x magnification and the mode of fracture recorded as: I = fracture occurred at the adhesive interface; C = cohesive fracture occurred within the composite resin and M = mixed fracture involving the tooth structure and adhesive junction occurred.

All data collected in the study were captured in a Microsoft Excel 2010 (Microsoft Corp., Redmond, WA, U.S.A.) spread sheet. Data capturing was verified and validity checks were performed on the data. All statistical procedures were performed on SAS, Release 9.2 (SAS Institute, Inc., Cary, NC, USA), running under Microsoft Windows XP for a personal computer. Statistical tests were performed at a significance level of 0.05. Normality was demonstrated for each of the groups, using the Shapiro-Wilk test. The mean shear bond strengths in MPa were compared by analysis of variance (ANOVA). The comparisons in the ANOVA included adhesive systems, tooth substrates and treatments. If the p-value of the ANOVA was significant, pairwise comparisons were done using the t test (LSD) as part of ANOVA in SAS.

RESULTS

The shear bond strengths obtained when Silorane System Adhesive (SSA) and Filtek Silorane (FS) were used to bond to tooth substrates that were not pre-etched and pre-etched are presented in Table 1 and Figure 1. When uncut enamel was not pre-etched, a mean shear bond strength (SBS) of 9.42 MPa was recorded whilst pre-etching enhanced the SBS to a mean of 15.20 MPa, a significantly higher value ($p=0.0151$). Similarly, the SBS to pre-etched cut enamel is significantly higher than the bond to un-etched cut enamel (21.35 MPa vs 12.11 MPa :$p<0.0001$). When dentine was not pre-etched, a mean SBS of 8.28 MPa was recorded but pre-etching resulted in a significantly higher mean value of 19.79 MPa ($p=0.0001$).

When the bonds achieved with Scotchbond Universal Adhesive (SUA) and Filtek Supreme XTE (FSX) were tested, both uncut and cut enamel surfaces recorded higher mean values on the pre-etched samples (28.90MPa vs 18.45MPa, $p<0.0001$ and 20.55MPa vs 16.38MPa, not significant, $p=0.0595$) see Table 2 and Figure 2. When dentine was not pre-etched, a mean SBS of 19.65MPa was recorded. A lower mean value was achieved on pre-etched dentine (15.29MPa), this difference not being statistically significant ($p=0.1198$).

Thus, both the null hypotheses are rejected.

The mean shear bond strengths (MPa) achieved by both systems when bonded to tooth substrates without and with pre-etching are depicted in Figures 3 and 4. SUA and FSX showed significantly higher mean SBS than SSA and FS when bonded to uncut enamel that was not pre-etched ($p=0.0003$). SUA and FSX also showed a higher mean SBS than SSA and FS when bonded to cut enamel that was not pre-etched, but this difference is not statistically significant ($p=0.0534$). When bonded to dentine that was not pre-etched SUA and FSX showed significantly higher mean SBS than did SSA and FS ($p=0.0002$).

SUA and FSX showed significantly higher mean SBS than SSA and FS when bonded to pre-etched uncut enamel ($p<0.0001$). SUA and FSX also performed better than SSA and FS when bonded to pre-etched cut enamel, but not to a statistically significant difference ($p=0.7108$). On the other hand it was SSA and FS which recorded a higher mean SBS than SUA and FSX when bonded to pre-etched dentine, although that difference was not statistically significant ($p=0.1085$).

When SSA and FS were used for bonding to tooth substrates that were not pre-etched, an adhesive fracture rate of 100% occurred. When SSA and FS were used for bonding to pre-etched tooth substrates, an adhesive fracture rate of 96.6% occurred. Only one specimen bonded to pre-etched cut enamel showed a mixed fracture (i.e. involving both the tooth structure and the adhesive). When SUA and FSX were used for bonding to tooth substrates that were not pre-etched, an adhesive fracture rate of 96.6% occurred. Again, only one specimen bonded

Table 1: The mean shear bond strengths (MPa) and p-values of Silorane System Adhesive (SSA) and Filtek Silorane (FS), bonded to tooth substrates that were either pre-etched or not pre-etched (standard deviations are in parentheses).

	Uncut enamel	Cut enamel	Dentine
Not pre-etched	9.42 (±4.55)	12.11 (±4.56)	8.28 (±1.70)
Pre-etched	15.20 (±5.61)	21.35 (±4.43)	19.79 (±6.48)
Stat. Sig.	p=0.0151 Yes	p<0.0001 Yes	p=0.0001 Yes

Table 2: The mean shear bond strengths (MPa) and p-values of Scotchbond Universal Adhesive (SUA) and Filtek Supreme XTE (FSX), bonded to tooth substrates that were either pre-etched or not pre-etched (standard deviations are in parentheses).

	Uncut enamel	Cut enamel	Dentine
Not pre-etched	18.45 (±4.57)	16.38 (±3.38)	19.65 (±4.69)
Pre-etched	28.90 (±4.82)	20.55 (±4.75)	15.29 (±6.25)
Stat. Sig.	p<0.0001 Yes	p=0.0595 No	p=0.1198 No

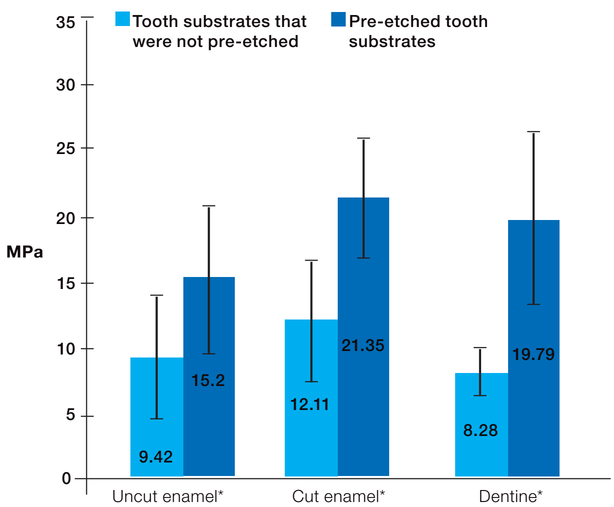

Figure 1: Bar Graph depicting the mean shear bond strengths (MPa) of Silorane System Adhesive (SSA) and Filtek Silorane (FS), bonded to tooth substrates that were either pre-etched or not pre-etched. ** Tooth substrates marked with an asterisk showed significant differences. Error bars represent standard deviations.*

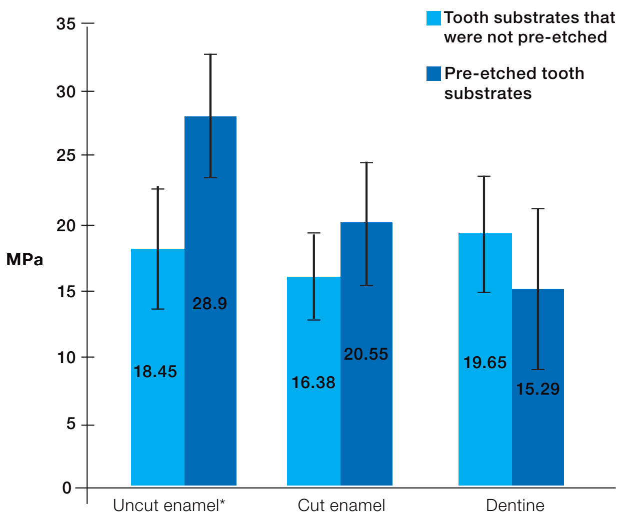

Figure 2: Bar Graph depicting the mean shear bond strengths (MPa) of Scotchbond Universal Adhesive (SUA) and Filtek Supreme XTE (FSX), bonded to tooth substrates that were either pre-etched or not pre-etched. ** Tooth substrates marked with an asterisk showed significant differences. Error bars represent standard deviations.*

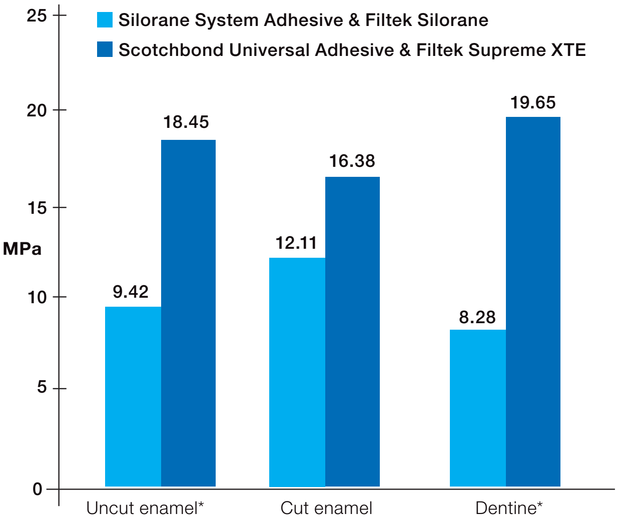

Figure 3: Bar Graph depicting the mean shear bond strengths (MPa) of both systems bonded to tooth substrates that were not pre-etched.
* *Tooth substrates marked with an asterisk showed significant differences.*

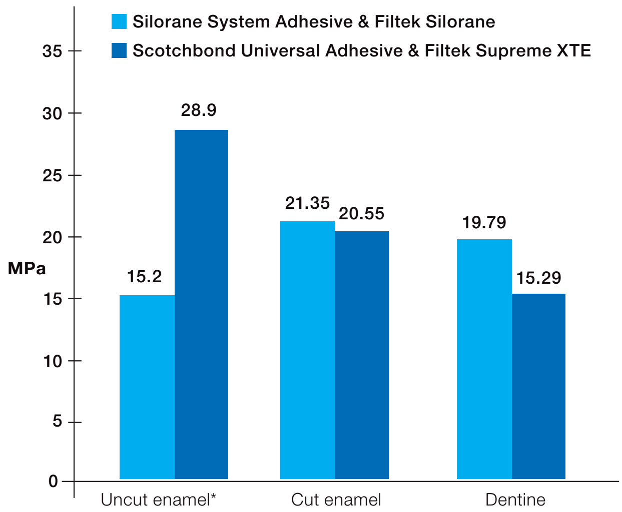

Figure 4: Bar Graph depicting the mean shear bond strengths (MPa) of both systems bonded to tooth substrates that were pre-etched.
* *Tooth substrates marked with an asterisk showed significant differences..*

to dentine that was not pre-etched experienced a mixed fracture. When SUA and FSX were used for bonding to pre-etched tooth substrates, an adhesive fracture rate of 96.6% also occurred. Once more, only one specimen bonded to pre-etched uncut enamel revealed a mixed fracture.

DISCUSSION

Despite some conflicting evidence in the literature, thermocycling is still considered to be a useful procedure to artificially age resin bonds in the endeavour to simulate in vivo conditions.[19,20] It is the opinion of Mcleod and colleagues that “studies that have not subjected specimens to thermocycling may not have imparted sufficient stress between the resin restoration and the tooth to simulate the intra oral environment” and that results obtained in the absence of thermocycling may not indicate the performance of the adhesive *in situ*.[4] The manufacturers of SSA recommend pre-etching of uncut enamel, but claim that pre-etching of cut enamel and dentine is unnecessary.[2] This study found that pre-etching of all three tooth substrates (uncut and cut enamel for 20 seconds and dentine for 15 seconds) with 34 % phosphoric acid significantly improved SBS (Table 1). The increased SBS when bonding to both uncut and cut enamel may be expected as the 34 % phosphoric acid (pH about 0.1) has strong etching potential. The mildly acidic SSA self-etching primer (pH 2.7) was unable to match the more aggresive phosphoric acid etch on both the uncut and cut enamel in order to create comparable micromechanical retention.

When enamel is etched with phosphoric acid, approximately 10μm of the surface is removed, and a morphologically porous layer, 5μm to 50μm deep, is created; the surface energy is also increased from 28mJ.m^2 to about 42mJ.m^2 thus enhancing the potential for bonding via micro- and macro- resin-tag interlocking.[7,21,22,23] Several studies have confirmed substantial increases in bond strength measurements when enamel was pre-etched with phosphoric acid prior to application of certain two-step self-etching adhesive systems.[23-27]

The significant improvement in SBS to pre-etched dentine by SSA was an unexpected finding and is also inconsistent with the manufacturers' recommendations.[2] Excessive demineralisation of the dentine by the stronger phosphoric acid should have resulted in an un-infiltrated resin-sparse dentine zone with naked collagen fibres below the hybrid layer which would in turn jeopardize bond strengths.[25,26,27] The significant improvement in SBS to pre-etched dentine is probably associated with the poor performance on un-etched dentine of the unnamed acidic monomer found in the SEP. It can only be speculated that the SEP was unable to sufficiently modify/penetrate the artificially created smear layer to demineralise the underlying dentine to create a homogenous hybrid layer. Only after the removal of the artificially created smear layer with the aid of phosphoric acid pre-etching, was the SEP able to penetrate the dentine.

Studies documenting the effect of pre-etching of tooth structures on shear bond strengths when using SSA and FS are scarce. A study in 2013 concluded that pre-etching of dentine with 37% phosphoric acid, when combined with a moist dentine surface and the use of primer agitation, improved the micro-tensile bond strengths of SSA.[28] An earlier 2008 study had, however, determined that phosphoric acid pre-etching of dentine did not significantly improve the micro-tensile bond strengths when using SSA.[29] In the same study it is worth mentioning that, none of the pre-etched specimens had spontaneously de-bonded, compared with nine out of 52 specimens that spontaneously de-bonded when the dentine was not pre-etched.[29]

As mentioned earlier, SUA can be used in 'self-etch' mode, as well as in 'total etch' and 'selective etch' modes. It is claimed that SUA performs well on cut enamel and dentine when used in self-etching mode.[15] The manufacturers do, however, recommend the pre-etching of uncut enamel before the application of SUA.[15] The results obtained when the SUA/FSX system was used are presented in Table 2. A marked improvement in the SBS to pre-etched uncut enamel and to a lesser extent to pre-etched cut enamel with the use of a self-etching adhesive system was an expected finding, and is consistent with results published by other researchers.[23, 30-34]

Taking into account the abundance of information available, it would be reasonable to conclude that most self-etching adhesive systems (the mild SEA systems in particular) do not bond as effectively to enamel as their total-etch counterparts.[23,24] The main reason for this is that mild SEA only superficially demineralises enamel, resulting in a very thin micro-retentive pattern without formation of micro- and macro- resin tags.[27] This ill-defined etching pattern and planar interface has been associated with poorer bond strengths.[23,27] When enamel is etched with phosphoric acid, the potential for bonding via micro- and macro- resin tag interlocking is enhanced. Based on the scientific evidence available, the increase in SBS to pre-etched uncut and cut enamel was expected and can be justified.

Pre-etching of dentine, however, resulted in a decrease in SBS for SUA. Even though the drop in SBS is not statistically significant, this finding was expected and is also in accordance with the findings of other researchers.[25,27,30,35,36] The lower bond strengths to pre-etched dentine can be attributed to incomplete infiltration of the demineralised collagen network and subsequent poor adaptation of the bonding resin to the collagen fibrils, thus leading to the formation of a non-homogenous, low-quality, porous hybrid layer that is prone to nanoleakage.[25,26,27,35]

Another possibility is over-etching of dentine (pre-etching followed by application of the SEA) which may have resulted in removal of the residual hydroxyapatite from the collagen mesh, which in turn could compromise the potential for chemical adhesion.[25] Lastly the collapse of unsupported collagen after phosphoric acid treatment and exposure to air could inhibit resin monomer penetration into the entire depth of demineralised dentine. This possibility is, however, unlikely as SUA contains water as a solvent which should be capable of rehydrating the desiccated collagen fibres.

In general, the mean SBS of SSA and FS bonded to tooth substrates that were not pre-etched was extremely poor, as none of these bond strengths exceeded 12.11 MPa. Furthermore these results were extremely low when compared with the findings of other researchers.[37-41] This discrepancy can be attributed to the inconsistency of *in vitro* bond testing protocols between researchers.[5,7,22,42]

A limitation of this study that must be mentioned is that testing was conducted on both molars and premolars, without any restriction on the type of posterior tooth (e.g. third molars only) or the age of the tooth/patients. Another limitation is the large intra-sample variability between the groups involving bonding to dentine. This may be indicative of random error during specimen preparation within these groups or perhaps structural differences in dentine particularly between molars and premolars. Recommendations regarding the performance of these materials in relation to dentine will therefore be reserved.

CONCLUSIONS

Within the limits of this study, the following conclusions were reached:

- When used on tooth substrates that were not pre-etched, the *in vitro* adhesion of Filtek Silorane composite, together with its dedicated sixth generation type I bonding agent Silorane System Adhesive, is poor. Pre-etching of both uncut and cut enamel with 34% phosphoric acid for 20 seconds significantly improved the mean shear bond strengths on uncut enamel by 61% and cut enamel by 76%. Pre-etching of dentine with 34% phosphoric acid for 15 seconds also significantly improved the mean shear bond strength by 139%.
- The methacrylate-based composite Filtek Supreme XTE together with the seventh generation bonding agent Scotchbond Universal Adhesive when used on tooth substrates that were not pre-etched showed acceptable *in vitro* adhesion. Pre-etching of uncut enamel with 34 % phosphoric acid for 20 seconds significantly improved the mean shear bond strength by 57%. Phosphoric acid pre-etching of cut enamel improved the mean shear bond strength of the Filtek Supreme XTE/Scotchbond Universal system by only 25%, which is not significant. In contrast to the silorane-based system, pre-etching of dentine with 34% phosphoric acid for 15 seconds when using Scotchbond Universal Adhesive resulted in a 22% drop in shear bond strength, which, however, is not statistically significant.
- Based on the results of this *in vitro* study, it would be prudent to conclude that the effect of pre-etching on SBS to uncut enamel, cut enamel, and dentine is product specific.

RECOMMENDATIONS

Within the limits of this study, the following recommendations are made:

- The *in vitro* SBS performance of the novel low shrinkage composite system Filtek Silorane and Silorane System Adhesive is of concern when used on tooth substrates without pre-etching. Additional clinical/*in vivo* studies are needed to better evaluate the success rate of this material. In a clinical situation, phosphoric acid pre-etching of uncut enamel and cut enamel before the application of the self-etching primer is recommended.
- The *in vitro* SBS performance of Scotchbond Universal Adhesive and Filtek Supreme XTE when used on tooth substrates that were not pre-etched was acceptable. However, the authors concur with the recommendations of the manufacturer, regarding phosphoric acid pre-etching of uncut enamel.
- The clinical significance of this study is highlighted when it comes to the restoration of cavities that are dependent on strong bonds to enamel, such as large Class IV incisal fractures; Class V cavities with cavosurface margins in enamel; and high C-factor cavities. In such clinical situations and when mild self-etching adhesives are used, the authors recommend selective pre-etching of enamel to improve bond strengths and to reduce marginal defects and staining.

References

1. Van Ende A, Mine A, De Munck J, Poitevin A, Van Meerbeek B. Bonding of low-shrinking composites in high C-factor cavities. J Dent 2012; 40: 295-303.
2. 3M ESPE (2007), 3M ESPE, St Paul, U.S.A. Filtek Silorane Low Shrink Posterior Restorative: 1-37.
3. Anusavice KJ. Phillips' Science of Dental Materials, 11th ed. St. Louis, Missouri (US): Saunders, Elsevier, 2003: 381-417.
4. Mcleod ME, Price RBT, Felix CM. Effect of configuration factor on shear bond strengths of self-etch adhesive systems to ground enamel and dentin. Oper Dent 2010; 35: 84-93.
5. Sakaguchi RL, Powers JM. Craig's Restorative Dental Materials, 13th ed. Philidelphia (US): Elsevier Mosby, 2012: 162-81.
6. Karthick K, Sivakumar K, Geetha Priya PR, Shankar S. Polymerisation shrinkage of composites: A review. Journal of Indian Academy of Dental Specialists 2011; 2: 32-6.
7. McCabe JF, Walls. Applied Dental Materials, 9th ed. United Kingdom: Blackwell, Oxford, 2008: 202- 44.
8. Freedman G. Contemporary Aesthetic Dentistry. St. Louis, Missouri (US): Mosby, Elsevier, 2012: 168-213.
9. Hegde MN, Bhandary S. An evaluation and comparison of shear bond strength of composite resin to dentin, using newer dentin bonding agents. J Conserv Dent 2008; 11: 71-5.
10. Kaaden C, Powers JM, Friedl K, Schmalz G. Bond strength of self-etching adhesives to dental hard tissues. Clin Oral Investig 2002; 6: 155-60.
11. Söderholm K-JM. Dental Adhesives …. How it all started and later evolved. J Adhes Dent 2007; 9: 227-30.
12. Kugel G, Ferrari M. The science of bonding: From first to sixth generation. J Am Dent Assoc 2000; 131: 20-5.
13. Alex G. Is total-etch dead? Evidence suggests otherwise. Compend Contin Educ Dent 2012; 33: 12-25.
14. Mine A, De Munck J, Van Ende A, Cardoso MV, Kuboki T, Yoshida Y, Van Meerbeek B. TEM characterisation of a silorane composite bonded to enamel/dentin. Dent Mater 2010; 26: 524-32.
15. 3M ESPE (2011), 3M ESPE, St Paul, U.S.A. Scotchbond Universal Adhesive, technical product profile: 1-40.
16. Brandt PD, de Wet FA, du Preez IC. Self-etching bonding systems: *in-vitro* shear bond strength evaluation. SADJ 2006; 61: 16-7.
17. Sethusa MP1, Seedat AK, du Preez IC, Hlongwa P. Shear bond strength comparison of RelyX Unicem with six other orthodontic resin adhesive systems. SADJ 2009; 64: 72-5.
18. Villela-Rosa AC1, Gonçalves M, Orsi IA, Miani PK. Shear bond strength of self-etch and total-etch bonding systems at different dentin depths. Braz Oral Res 2011; 25: 109-15.
19. Dental materials -Testing of adhesion to tooth structure. Technical Specifications. ISO/TS11405, Second edition, 2003.
20. Sheets JL, Wilcox CW, Barkmeier W, Nunn ME. The effect of phosphoric acid pre-etching and thermocycling on self-etching adhesive enamel bonding. J Prosthet Dent 2012; 107: 102-8.
21. Van Noort R. Introduction to Dental Materials, 2nd ed. London: Mosby, 2002: 149-67.
22. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 2003; 28: 215-35.
23. Erickson RL, Barkmeier WW, Kimmes NS. Bond strength of self-etch adhesives to pre-etched enamel. Dent Mater 2009; 25: 1187-94.
24. Erickson RL, De Gee AJ, Feilzer AJ. Effect of pre-etching enamel on fatigue of self-etch adhesive bonds. Dent Mater 2008; 24: 117-23.
25. Sabatini C. Effect of phosphoric acid etching on the shear bond strength of two self-etch adhesives. J Appl Oral Sci 2013; 21: 56-62.
26. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P,Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. J Dent 2006; 34: 77-85.
27. Van Landuyt KL, M. Peumans M, De Munck J, Lambrechts P, Van Meerbeek B. Extension of a one-step self-etch adhesive into a multi-step adhesive. Dent Mater 2006; 22: 533-44.
28. Pucci CR, de Oliveira RS, Caneppele TMF, Torres CRG, Borges AB, Tay FR. Effects of surface treatment, hydration and application method on the bond strength of a silorane adhesive and resin system to dentine. J Dent 2013; 41: 278-86.
29. Duarte, S Jr, Phark JH, Varjao F, Blatz, MB, Sadan A. (2008). Performance of different adhesives to a new low shrinkage composite, The IADR 86th General Session and Exhibition, Toronto, Canada.
30. Erhardt MC, Cavalcante LM, Pimenta LA. Influence of phosphoric acid pretreatment on self-etching bond strengths. J Esthet Restor Dent 2004; 16: 33-40.
31. Barkmeier WW, Erickson RL, Kimmes NS, Latta MA, Wilwerding TM. Effect of enamel etching time on roughness and bond strength. Oper Dent 2009; 34: 217-22.
32. Lührs A-K, Guhr S, Schilke R, Borchers L, Geurtsen W, Günay H. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. Oper Dent 2008; 33: 155-62.
33. Poggio C, Scribante A, Della Zoppa F, Colombo M, Beltrami R, Chiesa M. Shear bond strength of one-step self-etch adhesives to enamel: effect of acid pretreatment. Dental Traumatol 2013; 1-6.
34. Devarasa GM, Subba Reddy VV, Chaitra NL, Swarna, YM. Self-etching adhesive on intact enamel, with and without pre-etching. Microsc. Res. Tech. 2012; 75: 650-4.
35. Ikeda M, Kurokawa H, Sunada N, Tamura Y, Takimoto M, Murayama R, Ando S, Miyazaki M. Influence of previous acid etching on dentin bond strength of self-etch adhesives. J Oral Sci 2009; 51: 527-34.
36. Yazici AR, Karaman E, Ertan A, Özgünaltay G, Dayangaç B. Effect of different pre-treatment methods on dentin bond strength of a one-step self-etch adhesive. J Contemp Dent Pract 2009; 10: 41-8.
37. Guven Y, Tuna EB, Aktoren O. Dentin shear bond strengths of self-etch adhesives. J Dent Res 2008; 87: 403.
38. Syrek A, Kappler O, Guggenberger R, Weinmann W, Dede K, Loll H, Thalacker C. (2008), One year bond strength evolution of the silorane restorative system, The IADR 86th General Session and Exhibition, Toronto, Canada.
39. Brandt, PD, de Wet FA, du Preez IC. Bonding silorane to dentine. J Dent Res 2008; 87: 1730.
40. Boushell LW, Getz G, Swift EJ Jr, Walter R. Bond strengths of a silorane composite to various substrates. Am J Dent 2011; 24: 93-6.
41. Ghuman T, Kadam P, Cakir D, Ramp L, Burgess J. (2009), In-vitro evaluation of mechanical and physical properties of low-shrink composites, The IADR 87th General Session and Exhibition, Miami, U.S.A.
42. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: A critical literature review. Dent Mater 2010; 26: 78-93.