Sem Evaluation of Resin/Dentin Interface Using A Resin Luting Cement

Dissertation submitted to

THE TAMILNADU DR. M.G.R.MEDICAL UNIVERSITY

in partial fulfilment for the degree of

MASTER OF DENTAL SURGERY

BRANCH III

CONSERVATIVE DENTISTRY AND ENDODONTICS

FEBRUARY 2005

CERTIFICATE

This is to certify that this dissertation titled **"SEM EVALUATION OF RESIN / DENTIN INTERFACE USING A RESIN LUTING CEMENT"** is a bonafide record of work done by **Dr. P. RAVISHANKER,** under my guidance during his Post Graduate study period between 2002-2005.

This dissertation is submitted to THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY, in partial fulfillment for the Degree of Master of Dental Surgery in Branch III Conservative Dentistry and Endodontics.

It has not been submitted (partial or full) for the award of any other degree or diploma.

Dr. P. SHANKAR, M.D.S.
Associate Professor & Guide
Department of Conservative
Dentistry & Endodontics

Dr. R. INDIRA, M.D.S.
Professor & Head of Department
Department of Conservative
Dentistry & Endodontics

Dr. S. RAMACHANDRAN, M.D.S.
Principal
Ragas Dental College and Hospital
Chennai

CONTENTS

INTRODUCTION

The increasing demand for both esthetic restorations & the ubiquity of fluoride have combined to transform the practice of restorative dentistry. Recent trends in aesthetic dentistry revolves around bonding of restorations both to enamel through micromechanical entanglement of monomers within the enamel microporosities created by acid etching & to dentin through interpenetration of monomers within the collagen interfibrillar spaces. This interpenetration of monomers is now believed to be the key for successful bonding to dentin.[33]

Dental luting cements play a significant role in successful restorative dentistry in luting inlays, onlays, posts & crowns & to provide good retention. Resin luting cements are an adaptation of dentin bonding systems used with composite resin/ceramic restorative materials.[47] Application of adhesive systems induce structural changes in the dentin surface morphology, creating a retentive interface, called the interdiffusion zone between the deep, untouched layers of dentin & the composite filling material. (Van Meerbeek et al; 1992) [44]

Previous investigations on resin luting cements by Spencer P., Swafford J, Wielzicksa, and KrugerM.B (1999) have shown incomplete adhesive penetration throughout the demineralized dentin interface with dentin bonding agents that require a separate acid treatment.[28] Incomplete resin infiltration of the demineralized dentin matrix after acid etching leaves collagen fibrils exposed at the resin/dentin interface. Such exposed collagen may be related to the premature degradation of the dentin/adhesive interface & post-operative sensitivity. (Van Strijp et al, 1992) [46]

Panavia F 2.0 is a resin based luting agent, which is provided with an acidic self- etch primer (ED Primer II). This cement is unique in that it is considered an adhesive resin cement that does not require the use of additional bonding agent. The resin cement contains the adhesive phosphoric acid monomer 10- methacryloyl decyl hydrogen phosphate (MDP), which is also present in the self-etch primer. Panavia F 2.0 is directly applied onto the prepared dentin surface after application of the self-etch primer & no additional bonding agent is required.[20, 47]

The Panavia self-etch primer produces a minimal depth of dentin demineralization of 1 –1.5   at the interface[47]. The buffer capacity of the smear layer & the smear plugs may have limited the degree of penetration of the self-etch primer bringing about shallow demineralization. (C.Prati & others 1998). [24]

However, many clinicians continue to apply a conventional acid etch prior to the application of self-etch primer[47]. Acid etching of dentin is necessary to increase the porosity of intertubular dentin for enhanced monomer infiltration. It also considerably improves the bond strength as well as the tubule aperture seal (Fusayama etal 1987; Saraceni et al, 1994). [14,17]

There are conflicting reports on the effect of acid etching on composite bond strengths in the literature with some reports showing increased bond strengths while some showed low bond strengths. Tao & Pashley (1988) concluded that 37% phosphoric acid treated dentin produced low bond strengths[35]. At the interface, the conventional acid etch demineralizes the dentin to such an extent (6-9   ) that the surface collagen morphology was significantly altered & the resin was unable to infiltrate the demineralized zone fully.[47]

The aim of this study was to qualitatively compare the depth of resin cement (Panavia F.2.0) penetration into the dentin surfaces with & without pre-treatment by acid etching and evaluation using Scanning Electron Microscopy.

REVIEW OF LITERATURE

In 1988, **Suzuki and Finger**,[32] characterized the morphology of dentin with respect to the percentage area of dentinal tubuli exposed on dentin surfaces at different distances from the pulp chamber by automatic image analysis. They concluded that when tooth defects are prepared conservatively e.g. by exclusively removing the superficial layer of a cervical erosion lesion or by careful excavation of caries only, the underlying dentin for bonding is consequently characterized by a small portion of exposed dentinal tubuli. Such a surface offers a large area of solid dentin and thus, a high potential for effective bonding of a resin restoration via a dentin-bonding agent.

In 1988, **Pashley and Tao**,[35] using SEM, examined the dentin surfaces of unerupted non-carious human third molars, receiving various treatments to permit characterization of the modifications that were produced in the smear layers and the presence or absence of resin tags. Examination of these surfaces showed that 37% phosphoric acid removes nearly the entire smear layer and opened most of dentinal tubules. Also, SEM examination revealed that the bonding occurred on the smear layer rather than on the underlying dentin. Therefore, the bond strength seems to be determined by the forces holding the smear layer together. The more the smear layer removed, lower were the resultant bond strengths.

In 1990, **Kurosaki, Kubota, Yamamoto**,[10] investigated the effect of etching on the dentin of the clinical cavity floor. Examination by SEM, revealed that etching the cavity floor, demineralized the intertubular dentin surface slightly and produced tapered cylindrical holes or ring shaped holes at the dentin tubule apertures of the turbid or transparent layer respectively. The holes were blind with solid floor of intertubular crystal deposits of the transparent layer, suggesting that etching increase permeability little. Placement of the adhesive resin on the etched cavity floor produced a resin-impregnated dentin layer and tapered, cylindrical or tubular shaped resin tags that apparently improved the bond and tubule aperture seal.

In 1991, **Watanabe et al**[49] studied the effects of dentin primers on the sensitivity of dentin by measurement of the repetitive firing observed when a class V cavity prepared in the dentin of the mandible of a rabbit was irritated by a blast of compressed air. They concluded that the combined application of an aqueous solution of 35% HEMA and a commercially available dentin bonding agent was effective for reducing the repetitive firing and for maintaining such a sedative effect. Although the resin material sealed the dentinal tubules mechanically, it was impossible to explain the mechanism by which the dentin primer application reduced firing.

In 1991, **Suzuki M** and **Kato H**[31] studied the dentin/ resin (4-META-TBB) interface by applying the Raman microprobe technique. They observed that the resin molecules penetrated 6μ into the dentin from the interface. They demonstrated the excellent infiltration ability of the 4-META monomer into dentin substrate in situ.

In 1991, **Davidson et al**,[3] measured the stress development in cement layers of various thicknesses and to demonstrate evidence of destruction of the lutes. In this study, only a slight pressure was exerted until the pre adjusted cement layer thickness was reached. When porcelain or composite inlays are cemented clinically, only low pressure can be exerted, thus lowering the risk of premature fracture of the brittle inlay. They concluded that tensile stresses develop that exceeds the cohesive strengths of the materials. The effect of thickness of the cement layer on the stress depends on the nature of the cement.

In 1991, **Hirata et al**[8] investigated the dentinal fluid movement associated with loading of restorations. The occlusal surfaces of extracted teeth were filled with amalgam as the control or various posterior composites. Their findings suggest the possibility that the dentinal fluid movement observed in vitro in resin composite cavities may cause pain in vivo. These results suggest that biting sensitivity may be caused by the movement of dentinal fluid induced by masticatory pressure to resin restorations.

In 1992, **Nakabayashi**[18] investigated the effectiveness of treating dentin with 2-HEMA prior to the application of an adhesive resin by SEM. SEM examination revealed the formation of a transitional zone of resin reinforced dentin termed the hybrid layer in those specimens receiving 10-3 pretreatment. Specimens pretreated with 10-0 did not form hybrid layer. However, if HEMA application followed the 10-0 pretreatment, “hybrids” were demonstrated on SEM. Their study indicated that HEMA applied to dentinal substrates, enhances monomer infiltration and entanglement with dentinal components & facilitates the formation of a hybrid layer.[18]

In 1992, **Van Meerbeek et al**[44] studied the morphological aspects of the resin/dentin interface with different adhesive systems. Their study clearly showed that the application of adhesive systems induced structural changes in the dentin surface morphology, creating a retentive surface called the interdiffusion zone, between the deep, untouched layers of dentin and the composite restoration. This resin/dentin interdiffusion zone offers bonding sites for copolymerization with the resin composite and might have protective potential for pulp tissues.

In 1992, **Van Strijp**[46] and others studied the effects of oral environ ment on dentin matrix collagen. They assessed the degradation of matrix collagen during an insitu period under cariogenic conditions.In order to avoid the exposure of newly accessible collagen during the insitu period, which would hamper a quantitative assessment of the collagen solubilization, the experiments were performed with completely demineralized dentin specimens. They concluded that the intra and interindividual differences in collagen loss were likely related to a variation in the composition of the microflora colonizing the demineralized specimens.

In 1992, **Nakabayashi and Ashizawa**,[16] investigated the bond of 5-META in MMA, initiated by partially oxidized tri butyl borane in the presence of polyMMA powder, to vital human dentin. In vivo dentinal substrates were pretreated for 10 to 30 secs with an aqueous solution of 10% citric acid and 3% ferric chloride. Transmission electron microscopic examination of the bonded cross sections revealed the formation of the transitional or a “hybrid” layer of resin-reinforced dentin created by the impregnation, co-mingling and envelopment of the collagen bundles and encapsulation of hydroxyapatite crystals. The in vivo adhesion was assumed to be durable, because results of microscopic examinations were comparable to those of durable bonding of the same resin to extracted bovine dentin. Vital dentin exhibited greater resistance to demineralization by the acid solution than do extracted teeth.

In 1994, **Eick et al**[4] compared the morphology of the dentin/ resin interface surface by scanning transmission electron microscopy. They concluded that the acid pretreatment of the dentin greatly influenced the wetting behavior and the wetting and penetration of the adhesive in relation to the dentin adhesive and thus could substantially affect the resultant bond strength of dentin adhesive systems.

In 1994, **Watanabe, Nakabayashi** and **Pashley**[50] demonstrated that 20% phenyl P in 30% HEMA, demineralized the dentin surface by partially dissolving mineral crystals from around collagen by TEM. When applied to smear layers, this resin system demineralized the smear layer and incorporated it into the applied resin, which penetrated a short distance into the underlying dentin; thereby creating a hybrid layer that contained the original smear layer. They concluded that this single step conditioners/primer offers several advantages over previous bonding systems by permitting a single solution to serve as both the primer and conditioner.

In 1994, **Gwinett**,[6] observed the structure of acid conditioned dentin in its wet and dry state by conventional and environmental SEM. They concluded that the loss of moisture from acid conditioned dentin through simple air-drying and drying under controlled conditions of environmental SEM, resulted in morphological alteration of the fibrous collagenous structure. The significance of this study is that while the ultra structure of dentin collagen may be chemically stabilized or relatively unaltered by acid conditioning, it is nonetheless subjected to morphological degradation and collapse during water loss. This study supports the concept of maintaining the moist state of dentin and the morphological integrity of collagen so as to facilitate optimal resin infiltration in the bonding mechanism.

In 1994, **Perdigao et al**[23] evaluated the surface morphology of dentin substrates treated with various adhesives under SEM. SEM observations failed to demonstrate consistent resin/dentin interface morphology. The interface morphology with demineralized dentin was similar for all bonding systems. The results with hypermineralized dentin suggest that the partial or total obliteration of the tubules and intertubular dentin with mineral deposits may prevent reliable bonding of resins. The mineral deposits probably prevent adequate etching and resin penetration.

In 1994, **Tay** and **Gwinett**[38] investigated the resin/dentin interface of in vivo specimens restored with All Bond 2 system by use of a total etch wet bonding technique on vital deep human dentin using SEM and TEM. Ultra structural features that were pertinent to the formation of an effective clinical seal were characterized. They suggested that the establishment of an effective seal of the patent dentinal tubules is accomplished by:

- The formation of an outer zone of a solid resin plug surrounded by a circumferential cuff of resin impregnated dentin.
- The formation of an inner zone of a hollow resin sheath with resin globules along the inner surface of the tubules closely adapting to the odontoblast process.

Their study emphasized that the removal of smear layer is considered essential in optimizing the bond strength of dentin adhesives.

In 1994, **Sano et al**[25] tested the hypothesis that the demineralized matrix of dentin contributes little to the strength of dentin. They concluded that even if the resin did not infiltrate to the entire depth of the demineralized dentin, but engaged only the top part of the exposed collagen, the unprotected unreinforced collagen could still provide approximately 30 MPa of tensile strength to that bond. That does not mean that the bond would be stable or would not degrade due to hydrolytic activities over time. The demineralized collagen fibers would provide an elastic interface between the mineralized dentin and bonded resin. If one etched dentin with 10% phosphoric acid for 30 secs, dentin would be demineralized to a depth of 6 to 10μ. If the adhesive resin infiltrated only the top 3 to 5 , then the deepest 3 to 5 μ would be unprotected collagen fibers with a modulus of elasticity of 0.25 GPa, which could stretch about 20%. On top of this would be a resin reinforced collagen layer with a modulus of elasticity of 5 GPa, then an adhesive resin layer with a modulus of elasticity of 5 GPa and over that layer, a composite with a modulus of elasticity of 6 to 25 GPa. This arrangement provides a gradient of elastic stimuli that may function to relieve stresses of polymerization contraction or occlusal function.

In 1995, **H. Sano** and **Pashley**[27] tested the hypothesis that resin infiltration of demineralized dentin can restore its tensile properties to those of mineralized dentin. They concluded that some adhesive resins, after infiltrating demineralized dentin, can restore or even exceed the ultimate tensile strength of mineralized dentin. These resins also increased the modulus of elasticity of resin-infiltrated dentin to values equal to or greater than those of resins, but far below those of demineralized dentin.

In 1996, **Maciel et al**[13] evaluated the effects of dehydration on the stiffness of the decalcified dentin matrix. They prepared small beams of dentin from mid coronal dentin of extracted human molars and placed them in 0.5M EDTA for 5 days to decalcify. The results indicated that the stiffness of decalcified human dentin matrix is very low if the specimens are wet with water. As they are dehydrated, chemically or physically in air, the stiffness increases at 3 to 6 fold at high strains. These increases in modulus were rapidly reversed by rehydration in water. Exposure to glutaraldehyde also produced an increase in stiffness that was not reversible when the specimens were placed back in water.

In 1996, **Paula R Walshaw** and **Dorothy Mc Comb**[48] investigated the resin/dentin interface by SEM. They observed that effective priming, using multiple coats to enhance resin penetration to the full depth of dentin demineralization was crucial. They also considered that adequate etching of peripheral enamel continues to be an important factor in the long-term retention of adhesive restorations.

In 1996, **Van Meerbeek et al**[45] examined the resin/dentin interface formed by two dentin adhesives using TEM. Ultra structural information from non-demineralized and demineralized sections was correlated. TEM examination proved that each of the two-dentin adhesives was able to establish a micro mechanical bond between the resin and the dentin with the formation of the hybrid layer. However, the interfacial hybridization process that took place to produce this resin bond appeared to be specifically related to the chemical composition and application modes of both systems. Although both the adhesives investigated follow the total etch concept their specific chemical formulations result in different interfacial ultra structures that are probably related to different underlying bonding mechanisms.

In 1997, **Adriana Bona Matos et al**[14] evaluated the effects of acid solutions on dentin surface and to analyze the depth of demineralization that the acid solutions cause on dentin, using different acids by SEM. The acids used were 10%, 35% and 37.5% phosphoric acid and 10% maleic acid for 15 secs, washed and dried. Dentin discs were fractured, observed on the horizontal surface and also on the fracture surface to evaluate the depth of demineralization. They concluded that acid etching of dentin, regardless of the concentration of phosphoric acid caused removal of the smear layer, exposing the aperture of the dentinal tubules. This was not observed when 10% maleic acid was used. At the fracture surface, there was an increase in demineralization of the width of dentinal tubules, to a specific depth of about 8.19μ to 11mm except for 10% maleic acid.

In 1997, **Lyons et al**[12] used a pressure chamber to compare the microleakage associated with complete metal crowns cemented to extracted teeth using zinc phosphate, glass ionomer and Panavia 21 resin cement. The results of their study showed that no microleakage was detected with the resin-cemented crowns.

In 1998, **Masahiro Yoshiyama and David Pashley**[52] observed the resin/dentin interfaces of two commercially available self etching/self priming adhesive systems by SEM. SEM showed that the thickness of the hybrid layers of both systems was about 1μ in coronal, middle and cervical root dentin; less that 0.5μ in apical root dentin. They concluded that the self etch primer produces good adhesion in coronal, cervical and mid root dentin by creating thin hybrid and transitional layers, but bonding to enamel and apical root dentin should be improved.

In 1998, **Hayakawa and Nemoto**[7] investigated the effectiveness of the self-etching primer treatment on the adhesion of resin composite to both dentin and enamel. The self etch primer tested were HEMA and 10 MDP. SEM revealed a hybrid layer of 1.5μ thickness and the smear layer on the dentin and enamel were partially dissolved by the self etch primer. They concluded that both the primers showed good adhesion and were promising materials for resin composite restorations.

In 1998, **Prati** and **Pashley**[24] evaluated the dentin/resin interfacial morphology and shear bond strength of several new and experimental dentin bonding systems by SEM. They concluded that the depth of resin-infiltrated dentin of single component and multi step DBAs was generally thinner in superficial than in deeper dentin. The thickness of the resin infiltrated dentin layer in deep dentin ranged from 3.5 μ to 7.5 mm. They observed that that there was no correlation between the thickness of the resin infiltrated dentin layer and bond strengths. However, self etching dentin bonding agents showed higher bond strengths, despite the presence of very thin resin infiltrated dentin layer and shorter but larger resin tags.[24]

In 1999, **Sano et al**[26] investigated the interfacial morphological changes and long-term durability of resin/dentin bond strengths in the oral environment produced by a self-etching primer under occlusal stress in vivo. The surfaces of the failed bonds were observed under a FE-SEM. Bond strength measurements in this study were successfully performed and were stable at 19 MPa during the one-year testing. SEM revealed degradation of the resin/dentin interface occurred over a period of one year. Long-term morphologic changes at the adhesive interface in vivo may be caused by the extraction of resinous material by water.

In 1999, **Paulette Spencer** and **James Swafford**[28] demonstrated exposed collagenous protein at the dentin/adhesive interface by a non-destructive staining technique. Light microscopic sections of the dentin/resin interfaces of each tooth were cut and stained with Goldner's trichrome. Corollary SEM examination confirmed the presence of exposed protein at the interface. They concluded that the identification of inadequacies in the dentin/adhesive bond as the first step in determining sites that may be vulnerable to premature breakdown under clinical conditions.

In 2000, **Tay et al**[36] examined the ultra structural features of the resin/sclerotic dentin interface following the application of ClearFil Liner Bond II to natural cervical wedge-shaped lesions using SEM and TEM. They observed that a hypermineralized layer devoid of intact, banded collagen was invariably present on the surface of the natural lesions. Depending upon the thickness at different locations of the lesions, the action of a self-etching primer may be limited to the surface layer alone, producing a hybridized hypermineralized surface layer. They concluded that, as the depth of demineralization of the self-etching primer is limited to around 1μ in sound dentin, it cannot etch through a hypermineralized surface layer that is thicker than 1μ in sclerotic dentin. Under such a situation, only a thin, hybridized surface hypermineralized layer is formed.

In 2000, **Franklin R Tay, H. Sano** and **David Pashley**[37] determined the depth of dentin demineralization into intact dentin using several self-etching primer systems with different pH values. They concluded that self etch primers create thin hybrid layers that incorporate the smear layer. However, the suspicion that thick smear layers may interfere with the diffusion of self etch primers into the underlying intact dentin was not confirmed.

In 2000, **Franklin R Tay, H. Sano** and **David Pashley**[39] evaluated the presence or absence of smear layers on bonds made to dentin using a self etch primer system. Their study clearly showed that, formation of true hybrid layers occurs irrespective of smear layer thickness and that both hybrid layers may function as a separate unit during loading without separation.

In 2000, **Paulette Spencer** and **others**[30] investigated the chemistry of dentin/adhesive interface by Micro Raman spectroscopy. The purpose of their study was to determine, at the molecular level, the composition of the dentin/adhesive hybrid layer interface formed under "wet" bonding conditions and to quantify the diffusion of single bottle adhesives into the wet demineralized dentin. Using intrinsic vibrational signatures of the constituents at the interface, chemical maps of the hybrid layer at 1μ spatial resolutions were taken. The Micro Raman spectroscopic results present the first direct chemical evidence of a probable phase separation in an adhesive system under wet bonding conditions.

In 2000, **Van Meerbeek et al**[43] critically evaluated current microscopy techniques that are used to image dentin/resin interfaces and more in particular, to investigate the process of hybridization and resin tag formation. With this critical appraisal on microscopy techniques available to study adhesive interfaces, it is hoped to encourage the use of high-resolution analytical tools to further elucidate the mechanisms of bonding at the ultra structural level. The final objective of such fundamental research is to establish a more durable and reliable adhesive restorative technique.

In 2000, **Nakajima et al**[15] investigated if treatment of experimentally demineralized dentin with a calcium phosphate precipitating solution could restore bond strength of self-etching adhesives to that of normal mineralized control values. They exposed the occlusal surfaces for bonding and treated them with 40% phosphoric acid. These were immersed in CPP solution for 10 min. SEM analysis was done for each dentinal slab. They concluded that the use of calcium phosphate precipitating solutions might permit higher bond strengths of the self-etching primers to demineralized dentin.

In 2000, **Maria Tanumiharja et al**[40] evaluated the dentin/resin interface of dentin adhesives by SEM. All the dentin adhesive systems showed hybrid layer formation but the thickness varied depending on the bonding system used. The self etch priming system showed the thinnest hybrid layer at 1-2μ. They concluded that the ultra morphological structures of dentin bonding systems are determined by the composition of each system.

In 2001, **Gordon J Christensen**[1] reviewed the four categories of dentin bonding agents available. The purpose of this review was to elucidate the most effective methods not only to bond restorations to dentin, but also to desensitize that dentin in the process. He observed that the main problem with the total etch system adhesives was the unpredictable postoperative sensitivity. This sensitivity probably is related to the lack of sealing of the dentinal canals, because of the very volatile alcohol or acetone containing bonding liquids. He concluded that the self-etching primer concept has proved itself both scientifically and clinically. The concept reduces clinical steps, can be placed inexpensively, provides adequate bonding to dentin and enamel and most importantly, ensures the patient’s postoperative comfort.

In 2002, **Paulette Spencer et al**[47] analyzed in vitro resin cement / dentin interface by SEM comparing the diffusion of the resin cement into dentin surfaces pretreated with a self-etching primer with or without pretreatment with conventional acid etchant (37% phosphoric acid). They observed that the resin cement penetration throughout the zone of demineralized dentin is an important step in identifying sites of exposed dentin matrix that may promote postoperative sensitivity and may leave the interface vulnerable to premature degradation. They also found that self etch primer, when used alone produced substantial resin cement penetration and left no exposed dentin matrix at the resin cement/dentin interface.

In 2003, **Lopes et al**[11] investigated whether there are differences between bonding to hyper mineralized dentin and normal dentin and if longer acid etching can improve the bond strength to this modified substrate without damaging the bond to normal dentin. Based on the results of this study, the authors concluded that:

- Hardness of sclerotic dentin is approximately 30% higher than normal dentin with equal depth showing this to be a more mineralized zone.
- The thickness of the hybrid layer formed on sclerotic dentin is less than normal dentin, thus showing this tissue to more resistant to demineralization caused by acid etching.
- Bond strength to sclerotic dentin is not as high as normal dentin.
- For normal occlusal dentin, no difference exists in bond strength when 35% phosphoric acid etchant is applied following the manufacturer’s suggested time (15secs), or when the time is extended to 30 secs.

In 2004, **Paulette Spencer** and **Yong Wang**[29] investigated the dentin/adhesive interfacial characteristics of three adhesives using a non-destructive staining technique. The depth of demineralization appeared comparable (1-2μ). This technique provided a clear representation of the depth of dentin demineralization and the extent of adhesive encapsulation of the exposed collagen at the dentin/adhesive interface. This technique also provides a mechanism for readily identifying vulnerable sites at the dentin/adhesive interface.

In 2004, **Gomes Torres et al**[41] evaluated the effects of removing dentin collagen exposed by acid etching on the micro leakage of bonded restorations with or without flowable composite application by stereomicroscopy. They concluded that the removal of dentinal collagen reduced the marginal micro leakage. The use of flowable composite did not produce significant effects.

In 2004, **Yong Wang** and **Paulette Spencer**[51] studied the effect of etching time and technique on interfacial characteristics of adhesive/dentin bond using differential staining. They observed that the thickness of the interdiffusion zone increased as a function of etching time. However, the etchant gel application methods have a significant influence on dentin demineralization.

In 2004, **Oliveira et al**[19] investigated the effect of a self-etching primer on the continuous demineralization of dentin by atomic force microscopy and SEM. This study evaluated whether dentin demineralization continues after a 20 second application of a self-etching primer and also after polymerization of the adhesive. They suggested that the etching process is stopped by three mechanisms:

- The acidic groups are first neutralized by the reaction with the calcium.

- After evaporation of the solvent, the primer’s viscosity rises, reducing the diffusion of the monomers.
- Polymerization reduces the concentration of free acidic monomers.

SUMMARY

A study was done to qualitatively compare the depth of resin cement (PANAVIA F 2.O) penetration into the dentin surfaces with and without pretreatment by acid etching and evaluation using SEM. 10 non-carious upper human premolars were selected.

Two groups of teeth (group 1 and group 2) comprising 5 teeth in each group were prepared to receive a composite inlay made by the indirect technique. Specimens in group 1 were acid etched with 37% phosphoric acid prior to cementation of the inlay. Specimens in group 2 were not acid etched prior to cementation of inlay with Panavia F 2.0. All the specimens were decoronated and sectioned mesiodistally.

Group 1

The buccal half of the specimens in this group was treated with 5% NaOCl for 5 min in order to evaluate the amount of exposed collagen.

The palatal half of the specimens in this group was treated with 5NHCl for 5 min, rinsed and then treated with 5% NaOCl for 5min in order to evaluate the degree of resin cement penetration. The specimens were then air dried.

Group 2

The buccal half of the specimens in this group was treated with 5% NaOCl for 5 min. The palatal half of the specimens in this group was treated with 5NHCl for 5 min, rinsed and then treated with 5% NaOCl for 5min. The specimens were then air dried.

The specimens in both the groups (both buccal and palatal specimens) were subjected for SEM evaluation of the resin/dentin interface.

The amount of resin cement penetration, exposed collagen and degree of dentin demineralization in both the groups were evaluated.

Results were subjected to statistical analysis (Student T-test) and compared.

CONCLUSION

The results of this study showed that acid pretreatment increased the depth of dentin demineralization (7.46μ). There was also a concurrent increase in the depth of resin cement penetration (3.13μ). However, a zone of exposed collagen was present at the resin cement/dentin interface (4.33μ) due to incomplete resin cement penetration.

On the other hand, self-etch primer when used alone resulted in shallow dentin demineralization (1.28μ) and the resin cement had penetrated the whole demineralized zone leaving no exposed collagen.

The more complete resin cement penetration associated with the self-etching primer/resin cement and dentin interface suggests that this interface may be less susceptible to immediate and/or long term complications like postoperative sensitivity and marginal leakage associated with fixed restorations.

Further research has to be done to arrive at a distinct conclusion.

BIBLIOGRAPHY

1. **Christensen.J.Gordon** Self-etching primers are here. *JADA* 132, July 2001

2. **Claus- Peter Ernst, Priv.-Doz.,Dr.med.dent.** Positioning self-etching adhesives: versus or in addition to phosphoric acid etching? *Journal of esthetic and restorative dentistry* 16(1); 2004, 57-67

3. **Davidson C.L., Van Zeghbroeckl, Feilzer A.J.** Destructive stresses in adhesive luting cements .*J Dent Res* 70(5): 1991; 880-882

4. **Eick J.D, Robinson S.J., Byerley T.J., Chappell R.P, Spencer P., and Chappelow C.C.** Scanning transmission electron microscopy/energy – dispersive spectroscopy analysis of the dentin adhesive interface using a labeled 2- hydroxyethylmethacrylate analogue. *J Dent Res* 74(6): 1995; 1246-1252

5. **Eick, J.D., Gwinett A.J., Pashley.D.H, Robinson S.J.** Current concepts of adhesion to dentin. *Crit Rev Oral Biol Med* 8(3): 1997; 306-335

6. **Gwinnett A John,** Chemically conditioned dentin: A comparison of conventional and environmental scanning electron microscopy findings. *Dental Materials* 10: 1994; 150-155

7. **Hayakawa Tohru, Kazuyo Kikutake, Kimiya Nemoto** Influence of self-etching primer treatment on the adhesion of resin composite to polished dentin and enamel. *Dental materials* 14: 1998; 99-105

8. **Hirata.K., Nakashima.M, Sekine.I., Mukouyama.Y, Kimura K.** Dentinal fluid movement associated with loading of restorations. *J Dent Res* 70(6): 1991; 975-978

9. **Hoos.C.Jeffrey.** Clinical findings using a self etch primer. *Professional resources for dentists.*

10. **Kurosaki Norimasa, Minoru Kubota, Yumiko Yamamoto, Takao Fusayama.**The effect of etching on the dentin of the clinical cavity floor. *Quintessence International* 21(2): 1990; 87-92

11. **Lopes GC, Vieira KCC, Monteiro S Jr., Caldeira de Andrada, Baratieri CM** Dentin bonding: effect of degree of mineralization and acid etching time. *Operative dentistry* 28(4): 2003; 429-439

12. **Lyons.M.Karl, Rodda.C. John, Hood.A. James.** Use of a pressure chamber to compare microleakage of three luting agents. *The international journal of prosthodontics* 10(5); 1997: 426-433

13. **Maciel K.T., Carvalho R.M., Ringle R.D., Preston C.D., Russell C.M., Pashley D.H.** The effects of acetone, ethanol, HEM, and air on the stiffness of human decalcified dentin matrix. *J Dent Res* 75(11): 1996; 1851-1858

14. **Matos Adriana Bona, Palma Regina Guenka, Saraceni Cintia Helena Coury, Matson Edmir.** Effects of acid etching on dentin surface: SEM Morphological study. *Braz Dent J 8(1): 1997; 35-41*

15. **Nakajima Masatoshi, Miwako Ogata, Naoko Harada, Junji Tagami, David H. Pashley.**ond strengths of self-etching primer adhesives to in vitro- demineralized dentin following mineralizing treatment. *Journal of adhesive dentistry* 2 (1): 2000; 29-38

16. **Nakabayashi. N, Ashizawa. M, Nakamura M,** Identification of a resin-hybrid layer in vital human dentin created in vivo: durable bonding to vital dentin. *Quintessence International* 23(2): 1992; 135-141

17. Nakabayashi Nobuo; David H Pashley. Hybridization of dental hard tissues **Quintessence Publications**

18. **Nakabayashi N, Takarada. K.** Effect of HEMA on bonding to dentin. *Dental materials* 8: 1992; 125-130

19. **Oliveira Sofia S.A., Sally.J. Marshall, Stefan Habelitz, Stuart** A.Gansky, Robert S. Wilson, GraysonW. Marshall Jr. The effect of a self-etching primer on the continuous demineralization of Dentin. ***Eur J Oral Sci* 112:2004; 376-383**

20. Panavia F2.0 Instructions for use, Kuraray, U.S.A.

21. **Pashley D.H.** Clinical correlations of dentin structure and function.*Journal of prosthetic dentistry* 66(6): 1991; 777-781

22. **Pashley D.H, Bernard Ciucchi, Hidehike Sano, Jack A. Horner.** Permeability of dentin to adhesive agents. *Quintessence International* 24(9): 1993; 618-631

23. **Perdigao J., Swift E.J Jr, Denehy G.E.,.Wefel J.S, and Donly K.J.** In vitro bond strengths and SEM evaluation of dentin bonding systems to different dentin substrates. *J Dent Res* 73(1): 1994; 44-55

24. **Prati C, Chersoni S, Mongiorgi R, Pashley D.H.** Resin-infiltrated dentin dentin layer formation of new bonding systems Operative dentistry 23: 1998; 185-194 Journal of adhesive dentistry 4(3): 2002; 181-189

25. **Sano H, Ciucchi B., Matthews W.G, and Pashley D.H.** Tensile properties of mineralized and demineralized human and bovine dentin. *J Dent Res* 73(6): 1994; 1205-1211

26. **Sano H, Yoshikawa T, Periera P.N.R., Kanemura N, Morigami M, Tagami J, and Pashley D.H.** Long term durability of dentin bonds made with a self etching primer, in vivo. *J Dent Res* 78(4): 1999; 906-911

27. **Sano H, Takatsu T., Ciucchi B., Russell C.M, Pashley D.H.** Tensile properties of resin-infiltrated demineralized human dentin. *J Dent Res* 74(4): 1995; 1093-1102

28. **Spencer Paulette, James R. Swafford.** Unprotected dentin at the dentin-adhesive interface. *Quintessence International* 30: 1999; 501-507

29. **Spencer Paulette, Yong Wang, Lawrence Katz K.** Identification of collagen encapsulation at the dentin/adhesive interface. *Journal of adhesive dentistry* 6:2004; 91-95

30. **Spencer P., Wang Y, Walker M.P., Wieliczka D.M.,and Swafford J.R.** Interfacial chemistry of the dentin/adhesive bond. *J Dent Res* 79(7): 2000; 1458-1463

31. **Suzuki M, Kato. H, and Wakumoto. S.** Vibrational analysis by Raman Spectroscopy of the interface between dental adhesive resin and dentin. *J Dent Res* 70(7): 1991; 1092-1097

32. **Suzuki T., Finger W.J.** Dentin adhesives: site of dentin vs. bonding of composite resins. *Dental materials* 4: 1988; 379-383

33. Sturdevant Clifford M, The art and science of operative dentistry Fourth edition

34. **Tam L.E., and Pilliar R.M.** Effects of dentin surface treatments on the fracture toughness & tensile bond strengths of a dentin-composite adhesive interface. *J Dent Res* 73(9): 1994; 1530-1538

35. **Tao L., Pashley D.H.** Shear bond strengths to dentin: effects of surface treatments, depth and position. *Dental materials* 4: 1988; 371-378

36. **Tay F.R., Share- Moon Kwong, Anut Itthagarun, NigelM. King, Hak-Kong Yip, Keith M. Moulding, Nakabayashi N, David H. Pashley.** Bonding of a self-etching primer to non-carious cervical sclerotic dentin: Interfacial ultrastructure and microtensile bond strength evaluation. *Journal of adhesive dentistry* 2: 2000; 9-28

37. **Tay F.R., Hidehiko Sano, Ricardo Carvalho, Edna L. Pashley, David H. Pashley.** An ultrastructural study of the influence of acidity of self-etching primers and smear layer thickness on bonding to intact dentin. *Journal of adhesive dentistry* 2: 2000; 83-98

38. **Tay F.R., Gwinett A.J., Pang K.M.,and WEI.S.H.Y.** Structural evidence of a sealed tisuue interface with a total etch wet bonding technique in vivo. *J Dent Res* 73(3): 1994; 629-636

39. **Tay F.R.,, Ricardo Carvalho Hidehiko Sano David H. Pashley** Effect of a smear layers on the bonding of a self-etching primer to dentin. *Journal of adhesive dentistry* 2: 2000, 99-116

40. **Tanumiharja Maria, Michael F. Burrow, Martin J. Tyas, Jocelyn Carpenter.** Field- emission scanning electron microscopy of resin-dentin interface morphology of seven dentin adhesive systems. *Journal of adhesive dentistry* 2(4): 2000; 259-169

41. **Torres C.R.G., Maria Amelia Maximo de Araujo, Adrian Cristina de Mello Torres.** Effects of dentin collagen removal on micro leakage of bonded restorations. *Journal of adhesive dentistry* 6: 2004; 33-42

42. **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 *Operative dentistry* 28(3): 2003; 215-235

43. **Van Meerbeek B., Marcos Vargas, Satoshi Inoue, Yasuhiro Yoshida, Jorge Perdigao, Paul Lambrechts, Guido Vanherle** Microscopy investigations. Techniques, results, limitations. *Am J Dent* 13: 2000; 3D-18D

44. **Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P, and Vanherle G.** Morphological aspects of the resin-dentin inter diffusion zone with different adhesive systems. *J Dent Res* 71(8): 1992; 1530-1540

45. **Van Meerbeek B, Conn L.J., Duke E.S., Eick J.D., Robinson S.J., Guerrerro D** Correlative transmission electron microscopy examination of non demineralized and demineralized resin-dentin interfaces formed by two dentin adhesive systems. *J Dent Res* 75(3): 1996; 879-888

46. **Van Strijp A.J.P., Klont. B.,and TenCate J.M.** Solubilization of dentin collagen matrix in situ. *J Dent Res* 71(8): 1992; 1498-1502

47. **Walker Mary.P, Yong Wang, Paulette Spencer.** Morphological and chemical characterization of the dentin/resin cement interface produced with a self-etching primer. *J Adhesive Dent* 4; 2002: 181-189

48. **Walshaw. Paula. R., Dorothy Mc Comb .** Clinical considerations for optimal dentinal bonding. *Quintessence International* 27: 1996; 619-625

49. **Watanabe T, Sano M, Itoh K, Wakumoto S.** The effect of primers on the sensitivity of dentin. *Dental materials* 7: 1991; 148-150.

50. **Watanabe I, Nakabayashi N, and Pashley D.H.** Bonding to ground dentin by a Phenyl-P self-etching primer. *J Dent Res* 73(6): 1994; 1212-1220

51. **Yong Wang, Paulette Spencer:** Effect of acid etching time and technique on interfacial characteristics of the adhesive-dentin bond using differential staining. *Eur J Oral Sci* 112: 2004; 293-299

52. **Yoshiyama Masahiro, Takashi Matsuo, Shigeyuki Ebisu, David Pashley :** Regional bond strengths of self-etching/self-priming adhesive systems. *Journal of dentistry* 26: 1998; 609-616