

Sem Evaluation of Nanoleakage in Class-V Restorations with Flowable Resin Composite

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CERTIFICATE

This is to certify that this dissertation titled "**SEM EVALUATION OF NANOLEAKAGE IN CLASS-V RESTORATIONS WITH FLOWABLE RESIN COMPOSITE AS A LINER**" is a bonafide record of work done by **Dr. S. ARUTSELVAN** under my guidance during his postgraduate 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.

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INTRODUCTION

The advent of adhesives has caused a dramatic change in restorative dentistry. Resin composites are widely used for restorations including cervical lesions, as they are mercury free, esthetic and bond to tooth structure with the use of bonding systems. Adhesive dentistry could be expressed as the relationship between bond and stress. If the bonds can withstand the stress, the restorative technique will be successful. Unfortunately, the coefficient of linear thermal expansion of resin composites is three to four times more than that of tooth structure. In addition to the differences in coefficients of thermal expansion, the shrinkage of composites during curing induces stress at the tooth/restoration interface and generally results in gap formation. Therefore polymerization shrinkage and the coefficient of thermal expansion of these restorative materials have been suggested as major causes of micro leakage.

Restoring cervical lesions with resin composites has always been a problem, especially where no enamel is present in the gingival margin for bonding. The higher organic component, tubular structure, fluid pressure and the lower surface energy of dentin make bonding to dentin more difficult than to enamel.

Poor adhesion between dentin and restorative material predisposes gap formation. Marginal gap formation leads to leakage, which may be responsible for secondary caries, marginal discoloration, pulpal inflammation and hypersensitivity.²⁵

Materials like silver amalgam, silicate cement and GIC were used previously for Class-V restorations. Resin composite came as an alternative filling material for restoring Class-V cavities. Dentin bonding agents have been developed to improve the marginal seal of resin composite restorations at the

composite/tooth interface. Flowable resin composite was introduced in late 1996. Its modulus of elasticity is low, thereby, it has the ability to undergo plastic deformation to flex and absorb polymerization shrinkage stress.⁴ It has less filler content and the coefficient of thermal expansion of flowable resin composites is close to that of tooth structure.^{4,10}

The use of a flowable resin composite as a liner is another recently recommended technique for overcoming the shortcomings of resin composites. Flowable resin composite when used as an initial thin layer under composites, functions as a stress breaker.⁴³ Researchers have used flowable composites as lining materials and have obtained favourable results in reducing micro leakage.^{28,19} Micro leakage may allow the penetration of bacterial products and dentinal oral fluids along the interface, which may result in hydrolytic breakdown of either the adhesive resin or collagen within the hybrid layer thereby compromising the stability of the resin-dentin bond.²⁰

While numerous micro leakage studies have been performed, **Sano et al** described a leakage pattern by observing under Scanning Electron Microscope and Transmission Electron Microscope, the presence of silver within the hybrid layer in the absence of gap formation between the composite resin and the tooth. They termed this process “Nanoleakage” to distinguish it from micro leakage that occurs through gaps. Apparently, silver nitrate can diffuse from the margins of the restoration deep into the hybrid layer via interconnected defects, porosities, and flaws that exist between the hybrid layer and the underlying demineralized dentin. Nanoleakage tests can provide useful information on the sealing ability of restorations.³²⁻³⁴

The aim of this study was to evaluate the Nanoleakage in Class-V restorations using flowable resin composites under Field - Emission Scanning Electron Microscope using back scattered electron mode. The hypothesis tested was that the amount of nanoleakage would vary with different curing modes – Bulk cure, Dual cure and Co-cure.

REVIEW OF LITERATURE

Brannstrom et al⁷ in 1982 reported that partial removal of smear layer is more acceptable as resin infiltrates through the smear layer and into the underlying dentin. They stated that this might prevent leakage, as there is no discontinuity between dentin and the resin, i.e., there is no discernible interface between dentin and the bonding resin.

Feilzer et al¹³ in their study in 1988 investigated the development of free-curing contraction and the final shrinkage values in a series of composites and two glass ionomer cements as a first step in a study of stress development in direct restorations that are bonded to cavity walls. They suggested that although the composites showed much higher shrinkage rates, it was too early to determine whether shrinkage rates and ultimate shrinkage values were acceptable. They also concluded that much depends on the flow characteristics during the early setting of these materials and the total stress build up in the final setting, to be resisted by the dentin bonding agents.

Kemp-Scholte CM & Davidson CL¹⁷ in 1990 tested the marginal sealing capacity of some adhesive restorative systems and combinations of these systems with various lining materials as a mechanism of compensation for the curing contraction stress. They concluded that flexible linings applied between the bonding agent and the bulk restorative appeared to preserve the marginal integrity of Class-V restorations during curing. Measurements of the polymerization contraction stress showed 20% - 50% relief due to various flexible intermediate materials. Apparently, reduction of the total stiffness by application of an intermediate layer can render the total restoration sufficiently flexible to compensate for that part of the stress that would otherwise exceed the bond strength.

Prati et al²⁹ in 1991 evaluated the shear bond strength and microleakage of Class-V non-retentive restorations with ten different bonding systems and some tested with different composite resins. They found out that both test procedures resulted in significant differences between the various materials. They concluded that the bond strength failed to completely prevent microleakage at the dentinal wall and that the new generation of dentin bonding systems offer a significant improvement over previous materials.

Puckett et al³⁰ in 1992 compared the microleakage behaviour of three hybrid composites / bonding agent systems using bulk and incremental filling techniques utilizing a glass-ionomer liner in freshly extracted bovine incisors and ⁴⁵Ca radioisotope and autoradiography. They concluded that microleakage was significant regardless of placement technique in all the systems.

Van Meerbeek et al⁴⁴ 1993 used nanoindentation for assessing the hardness and Young's modulus of the successive layers of a resin-dentin bonding area to confirm the elastic wall concept. They found out that the hardness of the resin-dentin interdiffusion zone was significantly lower than that of unaltered dentin. They also observed a gradient of moduli of elasticity from the rather stiff dentin over a more elastic resin-dentin inter diffusion zone and adhesive resin layer to the restorative composite. They concluded that gradient was more substantial in those systems that produced relatively thick adhesive layers or supplementally provided a filled low-viscosity resin as an intermediate layer between the adhesive resin and the bulk restorative composite. Such an elastic bonding area might have a strain capacity sufficient to relieve stresses between the shrinking composite restoration and the rigid dentin substrate, thereby improving the conservation of the dentin bond and, as a consequence, the marginal integrity and retention of the restoration.

Sano et al³² in 1994 investigated marginal microleakage in Class-V cervical cavities of bovine anterior teeth by examining the resin / dentin interface using a Cryo-SEM and a silver ion penetration method. Despite of excellent marginal

adaptation of the material to cavity walls and absence of gap formation, they observed leakage of silver ions between the resin and decalcified tooth structure. They suggested that this resulted from the subsurface porosity of demineralized dentin that would not occur in cavities that were not purposely acid-etched. They concluded that creation of the hybrid zone during acid-etching of dentin may leave a leakage pathway beneath intact resin restorations capable of staining.

Tay et al⁴⁰ in 1995 observed that the presence of silver staining within the hybrid layer or bonding resin is always accompanied by a discrete line of silver grains along the interface, regardless of the presence or absence of interfacial gaps. They postulated that the weak link along the resin-hybrid layer interface acts as an initial site whereby movement of extraneous substances like water molecules can occur.

Sano et al³³ in 1995 conducted a study where the penetration of silver nitrate solution was examined in the interface between dentin and five different bonding agents used to restore Class-V cavities. They found that, even in the absence of gap formation, several leakage patterns occurred. They reported that the leakage was seen within the de-mineralized dentin indicating that adhesive systems do not completely permeate the de-mineralized dentin, but leave a hybrid layer with large amounts of porosity which may allow dentinal / oral fluid to slowly diffuse along the interface and hydrolyse the adhesive resin and collagen. They concluded that nanoleakage can occur at gingival margins of gap-free composite restorations.

Sano et al³⁴ in 1995 in their study examined the nanoleakage of the hybrid layer in two different bonding systems using silver nitrate staining technique. They found that silver nitrate penetrated most hybrid layers to varying degrees under SEM examination. However, they suggested the use of TEM to determine the exact distribution of the silver within the hybrid layer.

Munro et al²³ in 1996 measured the effect rebonding, with and without etching restoration margins, had on the microleakage of Class-V composite restorations. They concluded that rebonding the gingival margins of Class-V composite restorations significantly reduced microleakage under laboratory conditions.

Pashley DH and Carvalho RM²⁵ in 1997 reviewed the structure of dentin as it pertains to adhesive bonding and to describe the importance of resin permeation into dentinal tubules and into spaces created between collagen fibrils by acid-etching during resin bonding. They concluded that acid-etching of dentin produced profound changes in the chemical composition and physical properties of the matrix which can influence the quality of resin-dentin bonds, their strength and perhaps their durability.

Ferdianakis K¹⁴ in a study in 1998 attempted to determine the more effective material and technique for reducing microleakage in composite restorations, using three hybrid composite resins - two paste and one flowable. He concluded that the paste composite leaked significantly more than flowable composite. In addition, by using flowable materials, the voids, which contributed to the weakened surface and microleakage occurrence, are expected to be reduced.

Payne JH²⁸ in 1999 compared the effect of a flowable resin composite with an injectable glass ionomer on microleakage at the cavosurface margin of the proximal box of Class-II restorations in permanent teeth in-vitro. He concluded that the use of a flowable resin composite accompanied by a bonding agent in the proximal box of a Class-II restoration will significantly reduce the microleakage at the cavosurface margin when compared with an injectable glass ionomer with or without a bonding agent.

Unterbrink GL and Liebenberg⁴³ in 1999 reported that the contribution of a single-component adhesive as a dentin primer with a highly radiopaque flowable resin composite as a filled adhesive may help to overcome difficulties in radiographic diagnosis, shrinkage stress and modulus kinetics.

Bertolotti RL and Laamanen H⁶ in 1999 suggested in their study that the flowable composite, as a completely cured and well-adapted first increment, will resist disturbance by the polymerization shrinkage of the overlying composite.

S. J. Paul et al²⁷ in 1999 conducted a study to evaluate the influence of increasing etching times on the microtensile bond strength of a bonding agent and the leakage of silver ions within the hybrid layer. Excessive etching of the dentin revealed an increase in nanoleakage that raised concern about the long-term hydrolytic stability of resin and collagen fibrils in the resin-infiltrated dentin.

Heping Li, Michael F. Burrow, Martin .J. Tyas²⁰ in 2000 performed a study to evaluate the effects of load cycling on nanoleakage of three self-etching priming systems and a single bottle system. They inferred that load cycling did not influence nanoleakage of the selected bonding agents and that current dentin bonding systems were unable to achieve a perfect seal at the resin-dentin interface i.e., they were unable to prevent nanoleakage.

Tung et al⁴² in 2000 evaluated microleakage in Class-II cavities restored with the condensable resin composite with or without a liner and also to evaluate one of two different dentin bonding agents. They demonstrated that the use of flowable resin composite beneath a condensable resin composite significantly reduced microleakage and that the use of flowable composite as a liner or extended base deserved further consideration. They concluded that while two of the dentin bonding systems were equally effective in their ability to limit microleakage, the other system (control) did not significantly reduce cervical marginal leakage underneath a posterior resin composite.

K.K. Choi, R.J Condon, J.L. Ferracane⁹ in 2000 conducted a study to measure the contraction stress of composite with varied adhesive layer and to relate the effect of adhesive thickness to marginal leakage in Class-V cavities placed in bovine teeth. The results showed that contraction stress leakage of

composite restorations may be influenced by external stress produced during chewing as well as by stress produced by polymerization contraction and difference in the thermal expansion characteristics of the material and the tooth.

R. Frankenberger et al¹⁵ in 2000 determined in their study that application errors of dentin bonding systems caused dramatic decrease in adhesive performance.

P. Spencer, Y. Wang, M.P Walker et al³⁵ in 2000 performed a direct and quantitative measurement of the adhesive concentration throughout the hybrid layer in 'wet' bonding conditions under Micro-Raman Spectroscopy. They used intrinsic vibrational signatures of the constituents at the interface and generated chemical maps of the hybrid layer at 1 μm spatial resolution. They quantified adhesive diffusion by comparing spectral features from the chemical maps of the hybrid layer. They found a drop in adhesive monomer penetration in the hybrid layer as it approaches the floor of the demineralized dentin indicating that they are vulnerable to hydrolytic breakdown and susceptible to penetration by bacterial enzymes or other toxic substances.

H. Li, M.F. Burrow and M.J. Tyas²¹ in 2000 performed a study to evaluate the nanoleakage patterns of four dentin-bonding systems. They noted the uptake of silver particles at the base of the hybrid layer as well as within the hybrid layer and the adhesive resin. They suggested that the composition of each adhesive system may have had a role in forming different leakage patterns and that the current dentin bonding systems in use failed to achieve a perfect seal at the resin-dentin interface, which may influence the durability of the bond to dentin.

Leevailoj et al¹⁹ in 2001 compared marginal microleakage in Class-II cavities restored with packable composites alone to packable composites used in conjunction with flowable liner. The study also compared marginal microleakage in Class-II cavities of packable resin composites to one conventional microhybrid composite with and without a flowable liner. They

evaluated the microleakage of occlusal and gingival margins of Class-II cavities using ^{45}Ca radioisotope and autoradiographs. They concluded that when used as liners in Class-II cavities, flowable composites reduced, but did not eliminate, microleakage of the tested packable and microhybrid resin composites at gingival margins apical to the CEJ.

Shu-Fen Chuang et al¹⁰ in a simulated study in 2001 determined the influence of flowable composite linings on marginal microleakage and interval voids in Class-II composite restorations performed by practitioners with two different levels of experience. They concluded that flowable composite linings made by the experienced practitioner effectively reduced the voids of Class-II restorations in the gingival wall interface and cervical portion as well as the total void volume. Marginal sealing, however, was not influenced by the experience of the practitioners.

Belvedere PC⁵ in 2001 stated flowable composite as a low-viscosity resin-based composite having physical properties equal to heavy-viscosity products. They are excellent strengthening composites in areas where the etchant has demineralized the enamel and dentin structures because the filler particles will be drawn into the etched areas of tooth structure. Because the first placed resins were not individually polymerized, the low viscosity of the dentin primer-adhesive now being covered with the hybrid filler particles in the flowable forces these together to create one mass. If the dentin primer-adhesive was polymerized individually, the next application would not necessarily become part of this sealing resin system. By placing all of the resin-based composites into the preparation in bulk, the different components must mix together and become one mass.

N.R. Patricia et al²⁶ in 2001 conducted a study combining the microtensile bond strength and nanoleakage test in order to analyze the relationship of nanoleakage and bond strength. The results rejected the null hypotheses that there was correlation between nanoleakage and resin-dentin bond strength. They

inferred that nanoleakage occurred at the dentin interface of both total-etch and self-etching primer adhesive systems. They concurred that deeper the demineralized dentin and thicker the hybrid layer, the greater was the potential for nanoleakage of silver ions within partially or fully demineralized dentin, the hybrid layer or within partially polymerized adhesive resin. Self-etching systems that do not etch as deeply as total etch systems may have as much or more silver uptake / unit volume of hybrid layer than more aggressive etching system.

H.Li, M.F. Burrow, M.J. Tyas et al²² in 2001 evaluated the durability of dentin bonding over time by investigating the nanoleakage of dentin bonding systems over 24 hrs, 3 months, 6 months and 2 months. The results showed that systems using phosphoric acid as the etchant had a line of silver deposition at the base of the hybrid leakage that increased slightly during the 12-month storage period. This indicated that they may be subject to hydrolytic attack over time.

F.R. Tay, D.H. Pashley and M. Yoshiyama⁴¹ in 2002 conducted a study where the resin-dentin interfaces bonded with four single-step, self-etch adhesives were examined for nanoleakage by conventional (pH 4.2) and basic ammoniacal (pH 9.5) silver nitrate under TEM. They observed that all adhesives exhibited a reticular mode of nanoleakage within hybrid layers when conventional silver nitrate was used. With ammoniacal silver nitrate, they observed an additional spotted pattern of nanoleakage within adhesive and hybrid layers. They concluded that while reticular mode of nanoleakage in self-etch adhesives probably represented sites of incomplete water removal leading to regional suboptimal polymerization, the spotted pattern identified with the use of ammoniacal silver nitrate probably represented potentially permeable regions in the adhesive and hybrid layers that result from the interaction of the basic diamine silver ions with acidic / hydrophilic resin components.

Edward. J. Swift and John Kanca³⁹ in 2002 focused their studies on the management of dentin for effective bonding of the restoration. They suggested that when adhesive systems were applied to moist etched dentin surfaces, primers containing acetone and alcohol were effective after 5 secs of dwell time prior to drying while the primers containing water applied to dry etched surfaces were to be allowed to dwell for upto 30 secs prior to drying.

C.K.Y. Yiu, F.R. Tay et al⁴⁷ in 2002 analyzed the unknown mechanism responsible for sodium hypochlorite-induced reduction in dentin bond strength and its reversal with reducing agents. They examined the relationship between nanoleakage and reversal of compromised bonding to oxidized dentin under TEM using two single-bottle dentin adhesives. They found that acid-etched dentin was completely depleted of demineralized collagen matrix when sodium hypochlorite was used. They concluded that for both adhesives, tensile bond strengths of acid-etched dentin were significantly reduced after sodium hypochlorite treatment, but were reversed when sodium ascorbate was used. After sodium hypochlorite application, reticular nanoleakage patterns in hybrid layers were replaced by vertical, shag-carpet-like patterns along the demineralization front. This type of nanoleakage was completely eliminated after sodium ascorbate treatment with the materials tested. Residual sodium hypochlorite within the porosities of mineralized dentin may result in incomplete resin polymerization, and hence may have significant consequences on the long-term stability of the bond strength.

Hashimoto et al¹⁶ in 2003 evaluated the effect of multiple consecutive coatings of two total-etch adhesive resins on dentin under TEM by measuring both microtensile bond strength and nanoleakage. They concluded that the method of multiple consecutive coatings during dentin bonding improved the bond strength of the adhesive resins and reduced nanoleakage, due to an increase in resin-infiltration of the hybrid layer.

S. Guzman-Armstrong et al² in 2003 conducted a study to evaluate the correlation between microtensile dentin bond strength and silver ion penetration in total-etch 3 step and one self-etch 2 – step system. No correlation between silver penetration area and bond strength was demonstrated.

Attar N, Turgat MD and Güngör³ in 2004 investigated the effect of flowable resin composite on gingival microleakage of microhybrid and packable resin composite restorations in Class-V cavities of human premolars. They concluded that the use of a flowable composite or compomer as the first gingival increment of Class-II restorations either with microhybrid or packable composites decreased gingival microleakage regardless of the material used.

SUMMARY

This in-vitro study was done to evaluate nanoleakage in Class-V restorations with flowable resin composite. The materials used were Filtek™ Flow and Z100™ (3M) hybrid, which served as the control. Class-V cavities prepared (buccal and lingual) in human mandibular premolars were acid-etched and bonded (SingleBond™ 3M). Restoration was done with hybrid composite in Group-I and bulk cured. Group-II was restored with flowable composite and bulk cured. In Group-III, a thin layer of flowable composite was lined and cured initially. This was followed by restoration with hybrid composite and curing (Dual-Cure). In Group-IV, flowable and hybrid composites were co-cured after they were restored as above.

After 24 hrs of storage in water at 37⁰C, the restorations were finished and polished. The root apex of each tooth was sealed with flowable composite. The entire tooth except for the Class-V restorations and 1 mm of the tooth

surface surrounding the restorations was coated with nail varnish. The samples were immersed in 50% w/v solution of silver nitrate for 24 hrs in total darkness.

The samples were washed, photodeveloped and exposed to fluorescent light for 8 hrs. They were then longitudinally sectioned through the centre of the restorations, subjected to ultrasonic cleaning, desiccated, platinum sputtered and observed in a field emission SEM using back scattered electron mode. The degree of silver penetration along the resin-dentin interface was observed and calculated as a percentage of the total preparation wall length to obtain the leakage scores. Results were analyzed by ONEWAY (Descriptive and ANOVA) and Bonferroni tests.

CONCLUSION

The results of this in-vitro study revealed the following **ranking of Nanoleakage** in Class – V composite restorations with different curing modes:

- **Group-III** (Dual-cure) revealed *least leakage* due to low modulus of elasticity of flowable composite
- **Group-IV** (Co-cure) showed *highest leakage* due to contraction forces created by polymerization shrinkage of the hybrid composite
- **Group-I** and **Group-II** fell *in between Group-III* and **Group-IV**.

From this in-vitro study, it can be inferred that none of the composite restorative materials tested were consistently capable of preventing nanoleakage despite gap free margins at enamel and dentin/cementum restoration interfaces, confirming the hypothesis that variation in the amount of nanoleakage exists, with different curing modes.

This study suggests that the different leakage patterns observed are due to the difference in constituents of organic matrix and filler content in each type of composite. Further, the leakage is also influenced by the ability of the adhesive

to infiltrate into demineralized dentin. Low modulus of elasticity of flowable resin composite may allow it to be used as a liner. It should be cured initially, followed by restoration with hybrid composite, to minimize nanoleakage.

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