

## CHAPTER V

### DISCUSSION

Bonding effectiveness of self-etching adhesives has been improved, especially the mild self-etching adhesives (De Munck *et al.*, 2005b; Yoshida *et al.*, 2004), which could be acceptable alternatives to the clinically proven three step etch-and-rinse adhesives (Brandt *et al.*, 2006; De Munck *et al.*, 2005b; Peumans *et al.*, 2005a). However, mild self-etching adhesives has brought some compromising problems, such as high incidence of margin deterioration (Akimoto *et al.*, 2007; Chuang *et al.*, 2006). *In vitro* durability studies regarding two-step self-etching adhesives showed that they could create stable bonds of resin restorations similar to those of etch-and-rinse adhesives, however, more rapid progressive marginal degradation has been identified (Miyazaki *et al.*, 2000; Owens *et al.*, 2006; Perdigao *et al.*, 2005).

Since enamel bonding is primarily based on micromechanical interlocking of a low-viscosity resin into micro-porosities, the extent and depth of etching pattern should logically influence bonding performance of an adhesive (Nakabayashi and Pashley, 1998). Mild self-etching adhesives adhere weakly to enamel due to the inability of mild acidic conditioner to dissolve minerals in enamel. Etching pattern of enamel using these adhesives appears to be less retentive than that produced by phosphoric acid etching (Perdigao and Geraldeli, 2003). Mild self-etching primer being applied simultaneously onto enamel and dentin, only shallowly demineralized enamel resulting in a very thin micro-retentive pattern without formation of distinct macro- and microresin tags. This ill-defined etching pattern has also been found to be associated with low bond strengths, however, cohesive strength of the adhesive layer may be more important than etching potential of an enamel adhesive (Kanehira *et al.*, 2006; Pashley and Tay, 2001; Perdigao *et al.*, 1997). Because effective bond in enamel is critical for good marginal seal of the restorations, applying the enamel etching step should be considered in case of restorations that rely mainly on enamel bonding.

In this study, we examined whether an additional preceding etching step with phosphoric acid provides any supplementary effect on sealing ability of the two

commercially available self-etching adhesive using a microleakage test. Most of the previous studies were developed to assess microtensile bond strength to enamel by adding a prior acid etching step before application of self-etching adhesive, which found that microtensile bond strength was significantly improve (Torii *et al.*, 2002; Van Landuyt *et al.*, 2006a; Van Landuyt *et al.*, 2006b). There are some studies reporting that higher bond strengths were observed for Clearfil SE Bond after phosphoric acid pretreatment on enamel, however, the results were not statistically different from those obtained when the adhesive was applied according to the manufacturers' instruction (Rotta *et al.*, 2007). Although no difference in clinical performance was recorded in clinical studies, more marginal defects at the enamel side were noticed when enamel was not beforehand etched with phosphoric acid but these defects were small and of clinically negligible relevance (Abdalla and Garcia-Godoy, 2007; Peumans *et al.*, 2007; Van Meerbeek *et al.*, 2005a).

When the two different adhesive protocols were compared in this study, it was found that Clearfil SE Bond exhibited lower microleakage value when beforehand enamel was etched with phosphoric acid, but there was no statistical difference. This result can be explained by the presence of acidic but non-polymerizable hydrolytic adhesive components (Carvalho *et al.*, 2005), creating potential sites for the degradation of the bonded created by these self-etching adhesives. Furthermore, the chemical reaction between MDP and hydroxyapatite may form a low soluble calcium salt (Yoshida *et al.*, 2004) that limits resin infiltration capacity in enamel etching pattern.

The concept of self-etching adhesives is based on the use of polymerizable acidic monomers that simultaneously condition and prime dentin and enamel. Water is present as an essential component to enable ionization of the acidic monomers and demineralization of dental hard tissues (Tay *et al.*, 2004). Ideally, all solvents and water should be completely eliminated from the adhesive before light-curing. With increased concentration of hydrophilic and ionic resin monomers in self-etching adhesives, water may be incompletely removed and remain trapped at the bonded interface and this may compromise the overall bonding and the mechanical properties of the cured resin (Carvalho *et al.*, 2003; Paul *et al.*, 1999). Our group would like to propose another way to improve bonding effectiveness of Clearfil SE Bond by beforehand etching with

phosphoric acid on enamel alone and using self-etching primer to condition only dentin, then applying hydrophobic bonding agent all over the cavity. However, the proposed protocol would be similar to that commonly used in three-step etch-and-rinse system, although the only difference would be that dentin is not double etched.

Two self-etching adhesives tested were Clearfil SE Bond and Clearfil S<sup>3</sup> Bond, which were from the same manufacturer containing the same functional monomers with similar compositions; however, Clearfil SE Bond revealed superior sealing than Clearfil S<sup>3</sup> Bond at both enamel and dentin margins. This result may be due to different components between two self-etching adhesive. Clearfil SE Bond is a two-step self-etching system for which primer is a hydrophilic aqueous solution that should be air-dried to remove water and solvent before adhesive is applied. Clearfil SE Bond adhesive contains a hydrophobic resin with no water or solvent while for Clearfil S<sup>3</sup> Bond, as for the one-step self etching systems, is a complex mixture of both hydrophobic and hydrophilic ingredients, with a significant amount of water and solvent included in the adhesive components. As the solvent and functional monomers usually make up almost 50% of the adhesive, the concentration of hydrophobic monomers is drastically reduced. Since the mechanical strength of the adhesive is mainly provided by the polymerization of cross-linking monomers, relatively less hydrophobic monomers are available on the tooth surface after application of the one-step self etching systems, which impairs the bond strength (Pashley and Tay, 2001). One-step adhesives bond less effectively to enamel/dentin than do their multi-step versions (Peumans *et al.*, 2005a). Additionally, Clearfil S<sup>3</sup> Bond has a higher pH milder acidity, compared to Clearfil SE Bond. However, the sealing ability of Clearfil S<sup>3</sup> Bond was improved by beforehand enamel etch, because micro-retentive enamel surface produced by phosphoric acid provide more effective enamel bond. Application of multiple coats of self-etching adhesive or application of hydrophobic resin over polymerized self-etching adhesive may provide better bonding effectiveness of Clearfil S<sup>3</sup> Bond.

Comparison of the marginal qualities between enamel and dentin margins within each adhesive revealed a significantly higher percentage of effective margins in enamel. The findings of the present study confirmed that enamel was less critical substrate for bonding compared to dentin. Many studies have shown that bonding of

restorative material to enamel is adequate to resist contraction stress (Gamborgi *et al.*, 2007). Enamel is based mainly on homogeneous characteristics of the substrate, irrespective of its depth and location, except for some aprismatic enamel at the outer surface (Nakabayashi and Pashley, 1998). Adhesion to dentin demonstrated less perfect margins compared to the enamel cavity segments. Dentin shows a wider biologic variability than enamel, which makes it much more difficult to create a good adhesion capable of resisting the negative effects of polymerization shrinkage and subsequent thermal and mechanical stress factors (De Munck *et al.*, 2003b; Santini *et al.*, 2004; Silveira de Araujo *et al.*, 2006).

The depth of demineralized intact dentin relates to the concentration of the acidic monomer and the application time of primer (Ferrari *et al.*, 1997). The overall acidity of a primer mixture determines its self-etching potential. In Optibond FL groups, when dentin had been etched with phosphoric acid prior to the application of Optibond primer, an increasing in microleakage was observed. This must be attributed to double etching with phosphoric acid and Optibond primer, which pH of the latter is about 1.5 (Wang and Spencer, 2005). As the major function of primer solutions in three-step etch-and-rinse adhesive is to ensure sufficient wetting of the exposed collagen fibrils (Nakabayashi and Saimi, 1996), a primer application time 15 seconds with agitation should be respected to allow sufficient monomers to interdiffuse to the complete depth of surface demineralization. The results of this study suggested that there was incomplete resin penetration throughout the demineralized dentin when conventional acid etch treatment is used in combination with the Optibond primer. The continued demineralization of the dentin tubules eliminated any structural support for the resin tags, causing the dentin portion to lose its structural integrity. Deeper demineralization may prevent proper resin infiltration, hence, compromising the bond (Jacques and Hebling, 2005).

When resin polymerization shrinkage is restricted by adhesion to the cavity walls, stresses build up at the bonded interface. A detached margin will cause the composite to move away and toward those margins which are still intact. For a well-bonded composite restoration, the shrinkage vectors are oriented toward the bonded margins. If bonding at the gingival margin is poor, the vector orientation is away from the

light direction and again toward the remaining bonded areas (Versluis *et al.*, 1998). During polymerization, the volume of resin monomers is reduced, which creates sufficient shrinkage stresses to debond the material from dentin, where debonding is more prone to occur, thereby, decreasing retention and increasing leakage (Tay *et al.*, 2005).

Enamel leakage from outer surface was observed in the majority of specimens. The polymerization shrinkage generates contraction stress especially at tooth/resin interfaces. In the presence of high bond strength, the shrinkage stress might cause fracture of the tooth substrate and/or resin composite at the margin. Basically, enamel is weak in tension. The tensile phase of deformation, therefore, causes prisms to be disrupted (Gjorgievska *et al.*, 2008). After restoration, enamel microcracks were found in composite restorations and increased over time. In clinical tooth preparation with diamond burs, sharp diamond particles indent and scratch the enamel, causing material removal. Such operations may produce subsurface damage in enamel (Xu *et al.*, 1997). These observations proved that enamel microcracks were related both to resin contraction and cavity preparation procedures.

The silver nitrate method of measuring microleakage is an acceptable technique although diffusion of silver nitrate through dentin might have confounding effect on leakage tests. That is a severe test because the silver ion is extremely small (0.059 nm)(Youngson *et al.*, 1998), when compare with the size of a typical bacterium (0.5-1.0  $\mu\text{m}$ ) and the solution has an acidic pH of 4.2 resulting in increased porosity due to a light etching effect by the mildly acidic solution (Tay *et al.*, 2004). However, as the organic developer molecule has the larger diameter, it is the penetration of this chemical which is being observed. It is therefore unlikely that observations are of the full extent of silver ion penetration (Douglas *et al.*, 1989). Thus, the exact location of the leakage should be carefully determined.

The main disadvantage of microleakage evaluation is that it is a qualitative method, which can be made semi-quantitative by the application of a nonparametric scale. The degree of microleakage between a restoration and the cavity wall is difficult to quantify objectively. The nominal values are usually very low, so that the actual leakage path is sometimes unclear. Generally, the results obtained in each study group

differ only slightly, rendering interpretation of the results difficult and reducing the sensitivity of the test. Compared with bond strength tests, the effect of artificial aging methods such as water storage and thermocycling, on microleakage has been found to be minimal (Gale and Darvell, 1999). Evaluation of penetration was scored from three buccolingual sections from class V cavity to avoid underestimated evaluation from single section. Microleakage may be more extreme at the most mesial and distal surfaces and these end surfaces should be scored so that an accurate microleakage value could be assigned to composite restorations (Mixson *et al.*, 1991). However, it was found that reading leakage score from mesial and distal sections was difficult due to limitation of anatomical shape of the teeth.