

**AN *IN VITRO* STUDY OF THE MICROLEAKAGE OF A
COMPOMER (POLYACID MODIFIED RESIN
COMPOSITE) BONDED TO ENAMEL AND DENTINE
WITH DIFFERENT BONDING SYSTEMS AND THE
EFFECT OF SALIVA CONTAMINATION THERE OF.**

by

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Thesis presented in partial fulfilment of the requirements of the degree of

Master of Science in Dental Sciences

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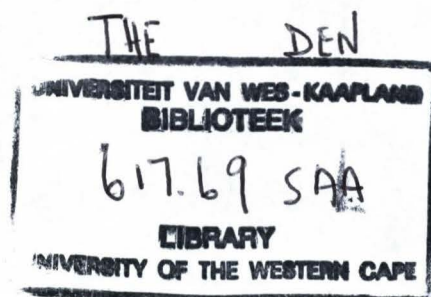
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March 2002



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DECLARATION

I, Charlene Margaret Saayman, hereby declare that the work contained in this thesis is my own original work and that I have not previously in its entirety or in part submitted it at any university for a degree.

CM Saayman

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6th Day of December of 2001

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ABSTRACT

Restorative systems classified as polyacid modified composite resins, or compomers, have appeared on the market. An example of this is Dyract AP. Dyract AP must be used with the Prime & Bond NT bonding system. Prime & Bond NT can be applied without any form of prior etching, or it can be applied after application of Non Rinse Conditioner, or it can be applied after etching with 36% phosphoric acid.

The purpose of the study was to determine the qualitative microleakage of Dyract AP and its bonding systems, as well as the influence of saliva contamination there of. Freshly extracted, non-carious, human premolars were randomly divided into 8 groups of 18 teeth each. Apisectomies coated with Polivar varnish and restored with amalgam were performed on all teeth. Class V type cavities of 3 mm diameter and 1,5 mm depth were prepared on the CEJ junction on the buccal side of all teeth. Dyract AP restorations were placed using the bonding procedures indicated: Group 1: P&B NT (Prime & Bond NT); group 2: **acid** (36% phosphoric acid) + P&B NT; group 3: **NRC** (Non Rinse Conditioner) + P&B NT; group 4: P&B NT + **Saliva**; group 5: **acid** + **Saliva** + P&B NT; group 6: **acid** + P&B NT + **Saliva**; group 7: **NRC** + **Saliva** + P&B NT; group 8: **NRC** + P&B NT + **Saliva**. Restorations were finished with Sof-Lex discs. After 24 hours storage in distilled water the teeth were removed and coated with two layers of nail varnish, except for 1 mm around the restorations. The teeth were then thermocycled in a 0.5% basic fuchsin solution for 500 complete cycles between 8°C and 50°C, with a dwell time of 15 seconds. After being cleaned and

embedded in resin, the teeth were sectioned in 600 µm thick sections and examined under a microscope at X60 magnification. The microleakage was evaluated on a scale of 0 to 4 on both the enamel and dentine/ cementum margins.

The data was statistically analyzed using Kruskal-Wallis one way ANOVA on ranks (significance at $p < 0.05$). Enamel data: Groups 1 and 4 differed significantly from all the groups except group 7. Group 7 differed significantly from all the groups except groups 1 and 4. Groups 2,3,5,6 and 8 only differed significantly from groups 1,4 and 7. Dentine/cementum data: Groups 6 and 8 differed significantly from groups 1,2,3,4,5 and 7. Group 1 differed significantly from groups 2,3,5,6,7,8; Group 2 from 1,4,6,8; group 3 from 1,4,6,7,8; group 4 from 2,3,6,8; group 5 from 1,6,8; group 7 from 1,3,6 and 8.

Microleakage was not eliminated at either the enamel or the dentine/cementum margins, but it was statistically significantly reduced at the enamel margins, by etching with NRC or phosphoric acid. Saliva contamination did not have a significant effect on the enamel margins when accompanied with 36% phosphoric acid etching. On the dentine/cementum side the least microleakage was obtained in the absence of etching. However, less microleakage was found if the dentine had been etched, and contamination took place between etching and Prime & Bond NT application, than when the contamination took place after curing of the Prime & Bond NT.

OPSOMMING

Herstelsisteme wat geklassifiseer word as polisuur gemodifiseerde komposiet harse, of kompomere, het op die mark verskyn. 'n Voorbeeld hiervan is Dyract AP. Dyract AP moet gebruik word saam met die Prime & Bond NT bindingsteem na aanwending van die Non Rinse Conditioner, of Prime & Bond NT kan gebruik word met of sonder etsing met 36% fosforsuur.

Die doel van die studie was om die kwalitatiewe mikrolekkasie van Dyract AP en die gepaartgaande bindingsisteme te bepaal, asook die invloed van speekselkontaminasie daarop. Vars getrekte, nie karieseuse, menslike premolare is lukraak ingedeel in 8 groepe van 18 tande elk: Apisektomies, wat bedek is met Polivar vernis en herstel is met amalgaam, is uitgevoer op al die tande. Klas V tipe kawiteite wat 3 mm in deursnit en 1,5 mm in diepte is, is voorberei op die glasuursementale aansluiting van die bukkale kant van al die tande. Dyract AP herstellings is geplaas met die betrokke bindingsprosedures soos aangedui: Groep 1: P&B NT (Prime & Bond NT); groep 2: **suur** (36% fosforsuur) + P&B NT; groep 3: **NRC** (Non Rinse Conditioner) + P&B NT; groep 4: P&B NT + **speeksel**; groep 5: **suur** + **speeksel** + P&B NT; groep 6: **suur** + P&B NT + **speeksel**; groep 7: **NRC** + **speeksel** + P&B NT; groep 8: **NRC** + P&B NT + **speeksel**. Herstellings is afgewerk met Sof-Lex skyfies. Nadat die tande vir 24 uur in gedistilleerde water gestoor is, is hul verwyder en met twee lae naellak toegeverf, behalwe vir die 1 mm naasliggend aan die herstellings. Die tande is daarna blootgestel aan 500 termiese siklusse tussen 8°C en 50°C met 'n rus tyd van 15

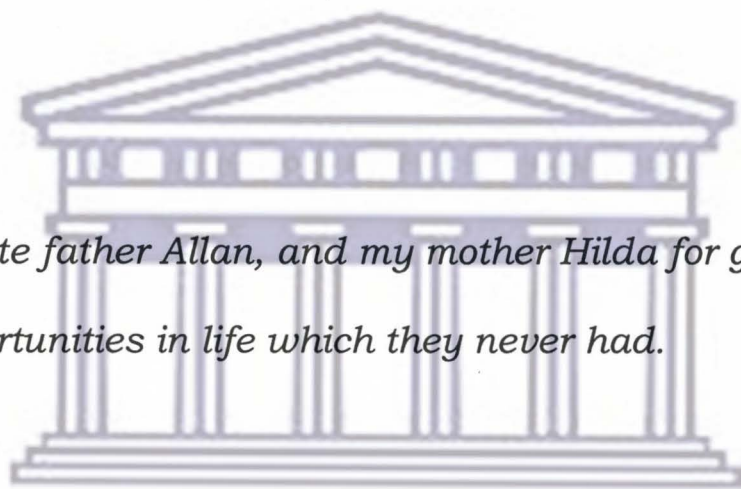
sekondes in 'n basiese fuchsienoplossing. Nadat die tande skoongemaak is en ingebed is in hars, is snitte van 600 μm dikte van die tande gesny en ondersoek onder 'n mikroskoop teen X60 vergroting. Die mikrolekkasie op beide die glasuur en dentien/sementum randte is op 'n skaal van 0 to 4 evalueer.

Die data is statisties ontleed deur gebruik te maak van die Kruskal Wallis ANOVA analise (kritieke afsnypunt van $p < 0,05$). Glasuur data: Groepe 1 en 4 verskil beduidend van al die groepe behalwe groep 7. Groep 7 verskil beduidend van al die groepe behalwe groepe 1 en 4. Groepe 2,3,5,6 en 8 verskil slegs beduidend van groepe 1,4 en 7. Dentien/sementum data: Groepe 6 and 8 verskil beduidend van groepe 1,2,3,4,5 en 7. Groep 1 verskil beduidend van groepe 2,3,5,6,7,8; Groep 2 van 1,4,6,8; groep 3 van 1,4,6,7,8; groep 4 van 2,3,6,8; groep 5 van 1,6,8; groep 7 van 1,3,6 en 8.

Mikrolekkasie is nie geëlimineer op die glasuur of die dentien/sementum kawiteitswande nie. Mikrolekkasie is statisties beduidend verminder aan die glasuurkant, indien daar vooraf geëts (NRC of fosforsuur) is. Speekselkontaminasie het nie 'n statisties beduidende invloed gehad op die glasuurkant nie, indien daar vooraf met 36% fosforsuur geëts is. Die minste mikrolekkasie aan die dentien/sementum kant is gevind indien daar glad nie geëts is nie. Indien die dentien geëts is, het kontaminasie tussen etsing en Prime & Bond NT aanwending, egter minder mikrolekkasie tot gevolg gehad as wanneer die kontaminasie na die kuring van die Prime & Bond NT plaasgevind het.

DEDICATION

*To my late father Allan, and my mother Hilda for giving me all
the opportunities in life which they never had.*



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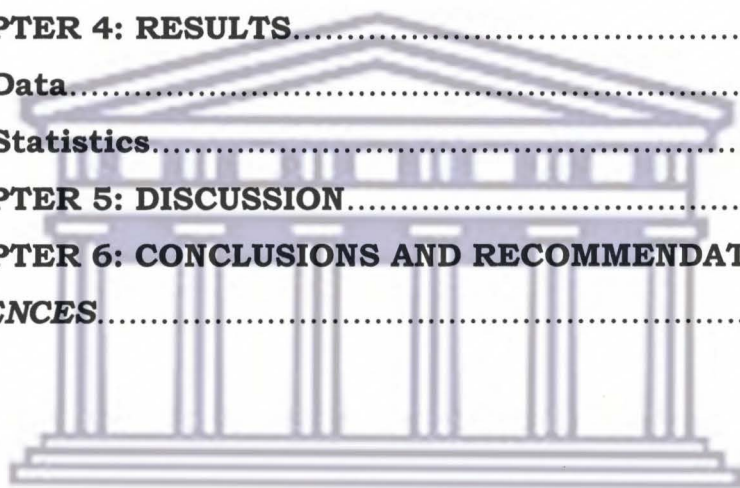
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CHAPTER 1

LITERATURE REVIEW

Recently several new restorative systems classified as polyacid modified composite resins (McLean, Nicholson & Wilson, 1994), or often referred to as compomers (Vaikuntam, 1997), appeared on the market.

Compomers contain both polymerizable groups of a composite and acidic groups of a glass ionomer. The compomers possess some of the aesthetic advantages of composite materials as well as fluoride leaching properties (Ferrari, Vichi, Mannocci & Davidson, 1998). An example of the compomers is Dyract AP. Research has been done on the microleakage of bonded Dyract (Chersoni, Lorenzi, Ferrieri & Prati, 1997; Freedman & Goldstep, 1997; Friedl, Schmalz, Hiller & Mortazavi, 1997; Hse & Wei, 1997), the forerunner of Dyract AP. However, limited independent research results could be found on the microleakage of Dyract AP using this new generation bonding system (Estafan, Pines, Erakin & Fuerst, 1999; Evancusky & Meiers, 2000; Kugel, Perry, Hoang E, Hoang T & Ferrari, 1998).

Most studies on the microleakage of Dyract AP were done in vitro and without saliva contamination (Dietrich, Kraemer, Losche & Roulet, 2000; Moodley, Grobler, Rossouw, Oberholzer & Patel, 2000).

Very few studies have been done on the influence of saliva contamination on microleakage (Dietrich, Kraemer, Losche, Wernecke & Roulet, 2000; Evancusky & Meiers, 2000) and none have made use of Prime & Bond NT as bonding agent.

Although very few independent studies have been published on the microleakage of the above mentioned products, or the influence of saliva contamination, when used according to specifications, the instructions for the use of Dyract AP does not specifically advise the use of rubber dam. However, the different systems should be properly evaluated to determine the influence of saliva contamination on microleakage before any recommendations could be made.

1.1 ADHESION TO ENAMEL AND DENTINE

1.1.1 ENAMEL

Composition

According to mass, 96%-97% of enamel consists of inorganic material, less than 1% of organic material and the rest consists of water. According to volume, 86% of it consists of inorganic material, 2% of organic material and the remaining 12% of water. The inorganic component consists mainly of hydroxyapatite while the organic component consists of two proteins. The one protein is a soluble glycoprotein, while the other is more insoluble (Jansen van Rensburg, 1981).

History of adhesion to enamel

Adhesion to enamel has been achieved since 1955 with the introduction of acid etching by Buonocore. He demonstrated that by etching enamel with ortho-phosphoric acid, adhesion of acrylic restorative resin to enamel could be achieved. The procedure introduced by Buonocore typically involved phosphoric acid conditioning of the enamel surface and application of a bonding resin (Buonocore, 1955). Phosphoric acids in the range of 32% to 40% have been most popular for enamel acid conditioning. Initially a sixty second acid-conditioning time was used (Silverstone, 1975), but Barkmeier, Shaffer and Gwinnett (1986) as well as Glasspoole and Erickson (1986) showed that a reduced conditioning time of fifteen

seconds yielded bond strengths equivalent to a 60-second time etch procedure.

Art-Smart, Retief and Russell (1993) reported bond strengths of between 8 and 15 MPa when enamel was etched with 10% phosphoric acid for 15 seconds and 8 to 17 MPa when the etching time was increased to 30 seconds. According to Nordenvall, Brännström and Malmgren (1980), as well as Barkmeier *et al.* (1986), scanning electron microscopy (SEM) studies have shown a similar etch pattern on enamel that is conditioned for either 15 or 60 seconds. The more concentrated the acid, the greater the degree of demineralization of the intertubular and intratubular dentine (Titley, Smith, Chernecky, Maric & Chan, 1995). Etching enamel with 35%-40% phosphoric acid usually prevents marginal leakage around composite restorations (Sidhu & Henderson, 1992; Swift & Le Valley, 1992). When enamel is conditioned, the inorganic component is removed, and microporosities are created (Buonocore, 1955). Enamel etching with phosphoric acid creates an etch pattern characterized by a deep uniform demineralization area (Ferrari, Mannocci, Kugel & García-Godoy, 1999).

It is assumed that the retentive ability of etched enamel for resin composite is a function of the increase in surface area and the increase in the wettability of etched enamel (Gwinnett, 1971; Retief, 1973). The inorganic composition of enamel results in acid etching producing interprismatic and prismatic dissolution, creating irregularities in which resin can flow and, after polymerization, create a mechanical interlocking (Gwinnett, 1976). To obtain micro-mechanical bonding of restorative materials, phosphoric acid is commonly used to etch enamel and remove the smear layer (Retief, 1978).

Several studies, especially those using scanning electron microscopy (SEM) revealed a range of structural changes when enamel was etched with phosphoric acid. These changes include (partly quoted from NRC Technical Manual, 1998):

- dissolution of the superficial enamel layer of about 10 μ m (Retief, 1973)
- peripheral regions of prisms are dissolved creating a picket fence prism profile and correspondingly wedge-shaped channels of about 25 μ m depth.
- prism cores are dissolved leaving crater-like depressions
- prismless enamel of deciduous teeth and in the cervical and fissure areas of permanent teeth are superficially dissolved, but surface roughness is much less pronounced.

Demineralization of enamel depends on the pH of the acid and the etching time (Ferrari, Mannocci, Vichi & Davidson, 1997; Perdigão, Lopes, Lambrechts, Leitao, Van Meerbeek & Vanherle, 1997).

A low viscosity resin, usually unfilled, is then able to penetrate into the microspaces of the etched enamel surface and interlocks mechanically upon polymerization. According to Retief (1978), mechanical bonding occurs when bonding agent is mechanically interlocked into micro-undercuts on enamel.

In 1963 Buonocore stated that a dry enamel surface is necessary to achieve good adhesion. Contamination of etched enamel prior to application of bonding agent will prevent proper bonding, because the micropores become occluded (Silverstone, 1984). A uniform frosty appearance after drying an enamel surface has been widely accepted to indicate that the enamel has been adequately etched. Several studies have shown less enamel and dentin demineralization when phosphoric acid was not used or when self-etching primers were applied (Ferrari *et al.*, 1997a; Perdigão *et al.*, 1997). With self-etching

primers it is difficult to judge adequate enamel etching (Miyazaki, Hirohata, Takagaki, Onose & Moore, 1999).

Phosphoric acid has a lower pH than maleic acid, and is therefore a stronger etching agent than maleic acid, is more soluble and can create a more microporous surface on normal enamel (Windholz, 1988). Owens (1997) reported that 10% maleic acid gave the least microleakage when it was applied for 30 or 60 seconds to enamel, while a 15 second application time gave the most microleakage. Swift & Cloe (1993) reported bond strengths of 25 MPa when etching was performed with 35% phosphoric acid for 15 seconds to enamel. With 10% maleic acid or 10% phosphoric acid, etched for 15 seconds, the bond strength declined to 13,2 MPa.

Beveling of enamel

Beveling of enamel margins results in a greater surface area of prepared enamel available for bonding. The beveling of margins followed by etching virtually eliminates microleakage (Saunders & Muirhead, 1992). However beveling of the enamel cavity margins failed to affect the clinical results of composite restorations in long-term clinical trials by Wilson NH, Wilson MA, Wastell & Smith (1991).

1.1.2 DENTINE

Requirements of dentine bonding

A polymerized layer of a dentine bonding system should be non-permeable to oral fluids, seal dentinal tubules, be long lasting and durable and protect the pulp (Titley *et al.*, 1995).

Composition

The organic content of dentine is much higher than that of enamel. It forms 18% of the mass. 93% of the organic content of dentine consists of collagen, 0,9% of citric acid, 0,2% of an insoluble protein, 0,2% of mucopolysaccharides and 0,2% of lipids. The inorganic

component comprises 70% of the mass and the rest (2%) consists of water. The inorganic component mainly consists of hydroxyapatite molecules. Calcium phosphates are also present (Jansen van Rensburg, 1981; Lee & Lin, 1997). The high organic content and tubular structure of dentine as well as the odontoblastic processes and outward flow of fluid makes dentine bonding difficult to achieve (Vargas, Cobb & Armstrong, 1997).

Dentine – Smear layer

Mechanically prepared dentine surfaces are covered by a film of denatured protein and ground hydroxyapatite, which is referred to as the smear layer (Eick, Wilko, Anderson & Sorensen, 1970; Pashley, 1984). The smear layer is approximately 1-5 μm thick and extends into the dentine tubules to form plugs (Boyde, Switsur & Steward, 1963) therefore decreasing the permeability of the dentine surface (Pashley, 1984). According to Tao and Pashley (1988) the smear layer is bonded to the dentine by a force estimated to be 5-6 MPa. In spite of this weak attachment, the smear layer cannot be easily scrubbed or washed away with water.

Dentine – early bonding agents

Due to its different nature, the etching of and adhesion to dentine has proven to be a challenging situation (Heymann & Bayne, 1993). Early dentine bonding systems attempted to chemically bond to dentine by ionic bonds to calcium (Eliades, Caputo & Vougiouklakis, 1985). There is however little evidence for the formation of chemical bonds between resins and dentine (Spencer, Byerly, Eick & Witt, 1992; Van Meerbeek, Moubacher, Celis, Roos, Bream, Lambrechts & Vanherle, 1993).

Dentine bonding and the smear layer

Early bonding agents were applied directly on smear layers and gave low bond strengths (5MPa) seeing that they did not permeate well in

dentine. It was established with SEM evaluation that the 5MPa bond strength was actually a measure of the cohesive forces holding the smear layer together (Pashley, 1991a).

Treatment of the smear layer

In order to increase bond strength of resin to dentine, one either has to remove the smear layer, or develop agents that can penetrate through the smear layer into the dentine matrix (Pashley & Carvalho, 1997).

Removal of the smear layer provides a demineralized dentine surface with a collagen rich meshwork for monomer diffusion. Nakabayashi, Kojima and Masuhara (1982) proposed that 10% citric acid be used to remove the smear layer and smear plugs in order to expose the collagen fibril network.

A **preserved** smear layer could have monomers or organic or inorganic components of the dentine incorporated into it.

A **partly dissolved/modified** smear layer results in a thin resin-impregnated dentine layer and a resin impregnated smear plug (Watson & Barlett, 1994).

Dentine etching

1.1.2.1 Conventional etching of dentine

Dentine etching was first thought to cause pulpal inflammation (Retief, Mandras, Russell & Derys, 1992; Stanley, Going & Chuancey, 1975). Fusayama (1987) believed that inflammation was caused by bacterial penetration through the dentine into the pulp. He therefore proposed total etching of dentine to open dentinal tubules and remove the smear layer of debris caused by cutting on the dentinal surface. This was done in order to promote more complete mechanical bonding of material to tooth structure.

Application of acid to dentine, results in the removal of the smear layer and smear plugs (Vargas, Cobb & Denehy, 1997b). This opens the dentine tubules, causes the demineralization of peritubular and intertubular dentine and exposes a collagen-rich transition zone (Titley *et al.*, 1995). The demineralization could be to a depth of approximately 2-7 μ m (Nakabayashi *et al.*, 1982). Removal of the smear layer causes a dramatic increase in surface permeability, in direct access to the pulp chamber and in wetness (Bowen, Eick & Henderson, 1984; Gwinnett, 1984). The demineralization also results in loss of support (Pashley, Horner & Brewer, 1992) and an inevitable degree of collapse of the collagen network, resulting in small loss of surface porosity after the initial dramatic increase (Titley *et al.*, 1995). The open spaces between the collagen fibers that had been occupied by hydroxyapatite crystals shrink due to the collapse of the unsupported collagen mesh. Protein collagen is susceptible to hydrolysis (Kato & Nakabayashi, 1996). Van Meerbeek, Inokoshi, Braem, Lambrechts and Vanherle (1992) found that the collagen matrix bulges out laterally after collapsing. This results in a constriction of the tubule orifices. Demineralization also results in the contamination of the surface with dentinal fluid, leading to a decrease in bond strength (Tao & Pashley 1988). The concentration of H₃PO₄ in a dentine etchant influences the permeability of demineralized dentine and therefore the resin bond to that substrate (Kato & Nakabayashi, 1996).

Abate, Bertacchini, Polack and Macchi (1997) reported that the use of phosphoric acid etching before primer and bonding agent placement, produced a better bond between a compomer and enamel, but was not particularly needed on dentin and cementum. In contrast Tate, You and Powers (1999) found tensile bond strengths to dentine to be higher overall when using phosphoric acid conditioning with primers and moist bonding surfaces. Ferrari *et al.*, (1999) demonstrated by scanning electron microscopy that resin tags penetrated further into

dentine etched by 37% phosphoric acid than into conditioned dentine where the smear layer was retained.

1.1.2.2. Total etch technique

To simplify the bonding procedure and to reduce application time, a single procedure was proposed to simultaneously etch the enamel and the dentine (Ferrari, Goracci & García-Godoy, 1997). This was termed the total etch technique (Kanca, 1992a). Phosphoric acid was the first acid to be used for this technique (Ferrari *et al.*, 1997b). Due to its higher inorganic content in comparison to dentine enamel must be etched for a longer time than dentine. To achieve this, weaker concentrations of acid have been used, or the exposure time of the dentine to the etchant has been shortened. All-Bond (Bisco) makes use of 10% phosphoric acid, while Scotchbond Multi Purpose (3M) makes use of 15% maleic acid. However, these lower concentration acids are also applied for a shorter time, usually, 15 seconds (Ferrari *et al.*, 1997a; Perdigão *et al.*, 1997).

1.1.3. THE ROLE OF WATER IN THE BONDING PROCESS

Working mechanism in dentine

Acid etching results in dentine displaying hydrophobic characteristics and does not increase its surface energy to facilitate spreading of adhesive resins (Attal, Asmussen & Degrange, 1994). Rinsing with water after etching removes the dissolved minerals and leaves a demineralized dentine surface covered with water (Pashley & Carvalho, 1997). The water fills the spaces around the collagen fibrils and the resins must diffuse through this water to form a hybrid layer (Nakabayashi *et al.*, 1982). Titley *et al.* (1995) found that hydrophilic unfilled resin monomers were unable to completely infiltrate the intertubular layer, as there was evidence of gaps, particularly at the normal dentine demineralized zone interface. A region of unprotected collagen fibers may exist if the resin does not completely infiltrate and surround the decalcified collagen fibers (Miyazaki, Sato, Onose &

Moore, 1998). Several researchers have speculated that incomplete infiltration of the demineralized collagen network could result in a weak zone within the hybrid layer, closest to the intact dentine, and could make it susceptible to long-term degradation (Nakabayashi, Nakamura & Yasuda, 1991; Pashley *et al.*, 1992).

Bound water exists inside the collagen fibrils in demineralized dentine (Nomura, Hiltner, Lando & Baer, 1997). If the water around the collagen fibrils can be completely removed and replaced by resin monomer, the bound water inside the collagen fibrils may move via osmosis and form a layer of water around the fibrils, preventing adequate contact with the resin. This could influence the degree of polymerization of the resin within the hybrid layer (Jacobsen, Ma & Söderholm, 1994). Santini and Mitchell (1998) reported that it is the degree of collapse of collagen that cannot be assessed clinically which is of importance in microleakage. They also reported that the resin hybrid layer interface represents the intrinsic weakness in most cases.

Air-drying of dentine results in the water that was supporting the collagen network, to evaporate. This is a result of a decrease in surface tension forces. Shrinkage or collapse of the collagen fibrils takes place (Pashley & Carvalho, 1997). Due to the water evaporation and reduced spaces between the collapsed collagen network, it in turn results in a reduction of the permeability of intertubular dentine to adhesive resins (Pashley, Sano, Oucchi, Yoshiyama & Carvalho, 1995).

Dehydration in air also increases the modulus of elasticity of collagen (Maciel, Carvalho, Ringle, Preston, Russell & Pashley, 1996). Dried, collapsed collagen needs water to lower the modulus of elasticity enough so that it can re-expand (Gwinnett, 1994). Water acts as a plasticizer for collagen (Jameson, Hood & Tidmarsh, 1993; Nomura *et*

al., 1997). If water is not available and a non-aqueous resin primer is applied on the dentine surface, the resin will not infiltrate effectively into the demineralized zone (Pashley & Carvalho, 1997). If the collagen fibril matrix of the demineralized dentine collapses due to air-drying, it may reduce the permeability to as little as 10% of its theoretical maximum value (Pashley, Ciucchi, Suno & Homer, 1993).

Kanca (1992a) demonstrated that bonding to moist dentine delivered higher bond strengths than bonding to dry dentine. Other researchers also reported similar results (Abdalla & Davidson, 1998; Gwinnett, 1992a; Kanca, 1992b; Swift & Triolo, 1992). Tay, Gwinnett and Wei (1996a) reported on what they referred to as the "overwet phenomenon". According to them, moist dentine is desirable for bonding, but large amounts of water on the dentine surface could result in poor bonding. They reported that whenever water was trapped at the orifice of a dentinal tubule, no resin tags were observed in that particular tubule. Several resin globules were usually present in the tubule lumen. The lack of resin tags as well as the globules could result in weaker bonds. Tay, Gwinnett and Wei (1996b) reported that the displacement of moisture across the dentine surface could result in areas where tubules were completely filled with water and the excess being pooled on the dentine surface. Entrapment of the displaced water, and dispersion of excess resin from the intertubular network into the pool of displaced water, could account for the formation of blister-like spaces and resin globules along the surface of the hybrid.

Water will rapidly re-expand a collapsed collagen network, but too much water would also dilute the monomer concentration. A critical amount of water is therefore necessary on the dentine surface to prevent water from evaporating, and the resultant collapse of the collagen fibrils. At the same time however, it should be small enough to be replaced by the resin monomer and not compete with it (Pashley

& Carvalho, 1997). If incomplete penetration occurs, water can infiltrate these spaces, producing hydrolysis of exposed collagen peptides not protected by hydroxyapatite or resin and microleakage could take place (Nakabayashi, Ashizawa & Nakamura, 1992; Sano, Shono, Takatsu & Hosoda, 1994). When collagen is protected by hydroxyapatite, peptide enzymes are unable to break down the collagen peptides (Van Strijp, Klont & Ten Cate, 1992).

If dentine is not air dried, the adhesive still has to physically replace the water in order to infiltrate into the spaces between the collagen fibrils. In stead of air drying dentine, one could use water-miscible organic solvents to chemically remove the water in the demineralized zone by diffusion (Pashley & Carvalho, 1997).

Solvents used to chemically remove water

Acetone- or alcohol-containing dentine adhesives are particularly capable of infiltrating the spaces (Kanca & Sandrik, 1998; Perdigão, Swift, Heymann & Malek, 1998). The alcohol or acetone solvent acts as a “water-chaser”, and removes the water molecules. The water diffuses from the wet dentine into the acetone, while the acetone diffuses into the demineralized dentine matrix. The acetone is in excess relative to the water, and replaces it. The primer monomers are dissolved in acetone and diffuse into the spaces previously occupied by water, increasing the depth of infiltration into the collagen network (Pashley & Carvalho, 1997). This process also increases the modulus of elasticity of collagen, but seeing that it is a more gradual process than in the case of air-drying of dentine, there is much less shrinkage or collapse of the collagen network (Carvalho, Yoshiyama, Brewer & Pashley, 1996). Wet bonding therefore prevents collagen fibers from collapsing (Kanca, 1992a; Saunders & Saunders, 1996).

Kanca (1992b) reported that acetone acts as a water chaser and also reduces the surface tension of the water and allows spreading of the mixture. He also reported that when acetone-based primers were applied to moist dentine the boiling point of acetone is increased and that of water reduced. The acetone and water then evaporates, leaving the resin behind. Resin tags of greater length and numbers are then formed. The tags also show high surface details indicating intimate contact with internal structure of dentine tubules. Tags formed on air dried dentine were fewer and shorter.

Earlier dentine bonding agents were highly hydrophobic and ineffective in the presence of water, therefore water was considered as a contaminant (Pashley & Carvalho, 1997). Bonding agents were modified and reformulated to be more hydrophilic. This resulted in an increase in bond strengths to dentine (Barkmeier & Cooley, 1992).

Eick, Robinson, Cobb, Chappell & Spencer (1992) reported that the addition of HEMA (hydroxy ethylmethacrylate) to bonding agents made them more hydrophilic. This enabled them to spread uniformly on hydrophilic dentine surfaces and penetrate deeper into the smear layer, thus improving the bond strength.

Hydrophilic primer monomers can fill-in the interfibrillar spaces of the collagen when they are maintained (Kanca & Sandrik, 1998; Tay, Gwinnett & Wei, 1996c). The addition of HEMA could prevent the collapse of the demineralized collagenous dentine surface (Sugizaki, 1991; Titley *et al.*, 1995). Abdalla *et al* (1998) reported that Prime & Bond 2.1 produced resin tags of greater length and number when the material was applied to wet dentine rather than to air-dried dentine.

Tate *et al.* (1999) reported that bonding did not occur without a primer/bonding agent, regardless of the use of phosphoric acid etching or surface conditioning. The highest tensile bond strengths

were attained when phosphoric acid conditioning, primer/bonding agent and moist bonding surfaces were used. He found that acetone-based primers were more sensitive to increases in surface moisture and produced lower compomer-dentine bond strengths with phosphoric acid etching and wet surfaces than water-based primers did. The resin monomer therefore attempts to diffuse through and displace the same amount of water as that which was initially necessary to preserve the spaces between the collagen fibrils. Pashley and Carvalho (1997) reported that water presence is constantly renewed by transdental permeability, therefore several layers of primer could be necessary to ensure proper infiltration into the demineralized zone and dentinal tubules.

2.1.4. PRIMERS

Working mechanism

An intermediary primary agent allows a hydrophobic bonding agent to bond to the wet surface of dentine (Berg, 1998). Primers possess both hydrophilic and hydrophobic groups and therefore improve the wettability of acid-etched dentine (Pashley & Carvalho, 1997).

Nakabayashi (1991) reported that a primer is essential to achieve good wetting and penetration through a demineralized area. The hydrophilic part has an affinity for both the organic and inorganic component of dentine, and the hydrophobic part has an affinity for adhesive resin (Gordan, Vargas, Cobb & Denehy, 1998).

Hydrophilic primer application raises collapsed collagen and keeps the porosity's open (Pashley *et al.*, 1992; Van Meerbeek *et al.*, 1992). Primers containing acetone as a solvent possess an affinity for water and are very compatible with the moist characteristics of acid-conditioned dentine (De Goes, Pachane & Garcia Godoy, 1997). This facilitates deeper penetration of adhesive resins into the dentine (Kanca, 1992b). Nakabayashi *et al.* (1991) reported that the goal of acidic primers is to obtain infiltration of resin monomer through the

smear layer while simultaneously demineralizing and infiltrating the dentine to form the hybrid layer.

The use of HEMA promotes infiltration of monomers into dentine due to its hydrophilic nature thereby preventing the collapse of demineralized dentine (Eick, Robinson, Chappell, Cobb & Spencer, 1993; Sugizaki, 1991). Van Meerbeek *et al.* (1992) and level Sugizaki (1991) demonstrated that HEMA has the ability to raise the unsupported and /or collapsed collagen network to almost its original dentine surface. Primer systems with 35-50% HEMA in water provide the maximum bond strength (Nakabayashi & Takarada, 1992). The original surface is however not restored seeing that the resin tags form a constriction as they pass through the interdiffusion zone, before they enter the dentinal tubules. Titley *et al.* (1995) demonstrated that resin infiltration of the demineralized zone does not extend completely to the normal dentine interface.

Self-etching/self-priming systems

Self-etching primer systems combine the tooth surface etching and priming steps to simultaneously treat enamel and dentine with an acidic monomer (Ferrari, Cagidiaco, Kugel & Davidson, 1996; Gordan, Vargas, Cobb & Denehy, 1997; Ikemura, Kouro & Endo, 1996). A self-etching primer is an aqueous mixture of acidic functional monomers and other constituents (Miyazaki *et al.*, 1998). It dissolves the smear layer and demineralizes the dentine (Barkmeier, Los & Triolo, 1995; Ikemura *et al.*, 1996), and can permeate through water-filled channels between the constituent particles of the smear layer. This self-etching/self-priming solution is not rinsed from the surface and is therefore incorporated into the hybrid layer once resin has permeated through the smear layer into the top of the underlying solid dentine (Chigira, Yukitani and Hasegawa *et al.*, 1994; Watanabe, Nakabayashi & Pashley, 1994).

Self-etching primer systems are applied directly to the cavity without the need for an acid-etching step (Cardoso, Placido, Francci & Perdigão, 1999). Eick, Miller, Robinson, Bowles, Gutshall and Chappelow (1996) reported that the dentine was wetted but not penetrated with a self-etching primer system, resulting in low bond strength. Self-etching primers generally produce a shallower depth of demineralization than systems with a separate etching agent. When self-etching self-priming agents are applied to dentine covered with a smear layer, they dissolve the mineral phase from the smear layer and etch approximately 0.5-1.5 μm into the sound underlying dentine. These systems form thin hybrid layers (1-2 μm) (Sano, Takatsu, Ciucchi, Horner, Matthews & Pashley, 1995; Yoshiyama, Carvalho, Sano, Homer, Brewer & Pashley, 1996a).

“One-bottle” or self-priming adhesives systems

Three step bonding systems are considered too time consuming and have been replaced by “one-bottle” systems (Cardoso *et al.*, 1999). The latest generations of adhesive systems use priming solutions and adhesive resins combined in a single bottle, and recommend etching the enamel and dentine simultaneously (Ferrari *et al.*, 1997b; Swift, Wilder, May & Waddell, 1997).

Self-priming adhesives in "one-bottle" systems are hydrophilic solutions that are very effective in wetting the dentine surface (Miyazaki *et al.*, 1998). The liquid penetrates the etched dentine surface and hardens after evaporation of the solvent and light exposure (Cadroy, Boj & García-Godoy, 1997; Ferrari & Davidson, 1996).

Self etch/prime bonding systems

Modern dentine bonding systems use high concentrations of phosphoric acid (30-38%) to etch enamel and dentine. Acidic monomers in self-etching adhesives infiltrate the collagen fibers as

they decalcify the inorganic components. The acidic part is neutralized by calcium and phosphate ions released during demineralization, thus being self-limiting (Gordan *et al.*, 1997). This creates hybrid layers composed of collagen and polymerized monomers (Inai, Kanemura, Tagami, Watanabe, Marshall & Marshall (1998).

1.1.5. DENTINE BONDING

Hybrid Layer

Dentine bonding thus takes place either to a smear layer, a modified smear layer, or to a cleansed and conditioned dentinal surface. The wetting process and the depth of adhesive penetration into the dentine surface, plays a major role in determining the dentine-adhesive bond strength. Little is known about the impact of collagen denaturation caused by conditioning of dentine on the final dentine-adhesive bond strength (Lee & Lin, 1997). When surface dentine is demineralized, a reduction in bond strength may take place (Okamoto, Heeley & Dogon, 1991).

After the smear layer has been removed, the hydrophilic priming agent can infiltrate into the layer of collagen fibers. The hydrophilic resin monomers of the bonding agent infiltrates the open collagen network of the dentine to form a zone of resin impregnated dentin (Eick, Gwinnett, Pashley & Robinson, 1997; Van Meerbeek, Dhem, Goret-Nicaise, Lambrechts & Vanherle, 1993). Nakabayashi *et al.* (1982) infiltrated resin monomers into the collagen fibril network. This provided micromechanical retention of the resin to the dentine after polymerization. They called this resin infiltrated surface layer a **hybrid layer**. The hybrid layer therefore consists of collagen fibrils and partially dissolved hydroxyapatite encased by polymerized resin (Van Meerbeek *et al.*, 1993b).

The morphology and role of the hybrid layer and resin tags formed in bonding to dentine, depends on several factors. Included in these are, the bonding agent used, the orientation of the dentine tubules, the presence and density of branches of dentine tubules, the different clinical conditions and the dentine depth (Ferrari *et al.*, 1997b).

Bonding - Inter as well as intratubular resin penetration

Intertubular as well as intratubular resin penetration is important in achieving dentine bonding. Resins must penetrate in the open tubules and seal them to prevent dentine sensitivity and pulpal irritation from leakage through dentinal tubules to the pulp (Pashley & Carvalho, 1997).

Age and depth influence on RIDL (Resin infiltrated dentine layer) or Hybrid layer

Ageing causes an increased type I collagen crosslink density in dentine microstructure, which may decrease penetration of primer and adhesive in dentine (Prati, Chersoni, Mongiorgi & Pashley, 1998). Superficial dentine contains fewer tubules than deep dentine, therefore permeation of intertubular dentine will provide the most bond strength in superficial dentine, while intratubular infiltration of resin will provide the most bond strength in deep dentine (Pashley & Carvalho, 1997).

Prati, Chersoni, Mongiorgi, Montanari & Pashley (1999) found that the morphology of the resin-infiltrated dentine layer varied as follows. The RIDL was thicker in deep, young dentine than in superficial dentine. The RIDL in sclerotic and old dentine was generally thinner than in normal dentine and the resin tags also appeared to be shorter and fewer. Tagami, Nakajima, Shono, Takatsu & Hosoda, 1993; Yoshiyama, Sano and Ebisu *et al.*, 1996) also gave similar reports. Although sclerotic dentine generally exhibited a thinner RIDL, it did vary to a large extent (Prati *et al.*, 1999). The resin tags were longer

and more numerous in young normal dentine than in old sclerotic dentine. Pashley (1991b) reported that superficial dentine has fewer, smaller tubules and few lateral branches.

Prati *et al.* (1998) found that in deep dentine, nearly all of the volume of the hybrid layers were occupied by resin tags and by criss-crossed extensions of peritubular and intratubular dentine. This probably plays the most important role in adhesion of resin to deep dentine. A similar network was not seen in superficial dentine because of the paucity of tubules and lateral branches in that area. The depth of infiltrated dentine was generally thinner in superficial than in deeper dentine. Generally, the greater the thickness of the RIDL the more numerous the irregularities and more likely the presence of noninfiltrated dentine.

Burrow, Takakura, Nakajima, Inai, Tagami and Takatsu (1994) concluded that the quality of the resin-impregnated layer is more important than its thickness, the age of the tooth or the dentine depth.

Lateral branches

Tay *et al.* (1996b) reported that the base of the RIDL is filled with resin monomer via radial diffusion from the tubules via lateral branches. Prati *et al.* (1999) found only a few resin lateral branches in sclerotic dentine, which could reduce the diffusion of bonding resin into dentine. Chappell, Cobb, Spencer and Eick (1994); Ferrari and Davidson (1996) and Ferrari *et al.* (1997b), demonstrated lateral branches deep in the dentine beyond the hybrid layer, while Mjör & Nordhal (1996) hypothesized the important role of the bonding mechanism of adhesive lateral branches.

1.1.6. BOND STRENGTH IN GENERAL

Adhesives based on an acetone solution of HEMA, results in significantly higher bond strengths than adhesives based on acetone solutions of a more hydrophobic monomer such as UDMA (urethane dimethacrylate (Finger & Fritz, 1997).

Gwinnett (1992a) commented that the resin infiltration into the collagen layer does not contribute quantitatively to the bond strength. High tensile bond strength can be achieved even though the resin-impregnated layer is very thin (Finger, Inoue & Asmussen, 1994). The quality, rather than the quantity (thickness) of the resin-impregnated layer, is more important than the creation of high bond strengths to dentine (Burrow *et al.*, 1994).

Yoshiyama, Carvalho, Sano, Homer, Brewer and Pashley (1996) also reported that there does not seem to be any relationship between the thickness of the hybrid layer and the bond strength. Prati *et al.* (1998) also did not support the hypotheses that thicker RIDL produces higher bond strengths.

The resin infiltration or hybridization of the dentine tissue can contribute approximately one third of the shear bond strength of the total etch system (Gwinnett, 1993).

The presence of moisture, particularly from the pulp could be an important factor influencing high bond strengths (Burrow *et al.*, 1994). Bond strength was not compromised with the remoistening of acid-conditioned, air-dried dentine (Gwinnett, 1994; Perdigão *et al.*, 1998), but decreased when bonding took place to an excessively wet dentin surface (Tate *et al.*, 1999). Lucena-Martin, Gonzalez-Rodriquez, Ferrer-Luque, Robles-Gijon and Navajas (1999) reported a significant decrease in bond strength after 1 month of water storage.

1.2 FROM COMPOSITE TO COMPOMER

In recent years, several new restorative materials have been developed as an alternative to dental amalgam. This is partly due to the awareness of the safety of dental amalgam (Hse, Leung & Wei 1999), as well as the demand for products with adhesive, esthetic and caries protective properties (Yap, Teoh, Hastings & Lu, 1997). Among these materials are resin composites, glass ionomers, resin-modified glass ionomers and the polyacid-modified resin composites or compomers (Hse *et al.*, 1999).

A brief review of these four groups of materials follows in order to understand the reasons for the development of compomers as well as their properties and reactions. It will also clearly demonstrate why compomers are more closely related to composites than to glass ionomers or resin-modified glass ionomers.

1.2.1 RESIN COMPOSITE

Resin composites and glass ionomer cements have been the materials of choice for the restoration of anterior teeth since the early 1970's (Wilson & Prosser, 1982). Dr Bowen's invention of resin composites in the late 1960's revolutionized the field of modern day dentistry (Cortes, García-Godoy & Boj, 1993). Previously anterior teeth were restored with silicate cement, acrylic, or other esthetically less acceptable restorations. Composite resins or resin composites are the most esthetically desirably of the four groups discussed further (Berg, 1998).

Composites have two main constituents namely the matrix and the filler. They are polymer-ceramic materials in which methacrylate and dimethacrylate monomers polymerize to form the matrix, and glasses, ceramics or glass-ceramics are incorporated as spherical fillers (Berg, 1998; Ruse, 1999; Tyas 1998).

Among the most commonly used dimethacrylate monomers are bis-glycidyl dimethacrylate (BIS-GMA) or urethane dimethacrylate (UDMA) or triethyleneglycol dimethacrylate (TEGDMA). The filler particles are coated with silane-coupling agents to allow the filler particles to bond to the hydrophobic resin matrix. The quality and extent of the silane coating affects the properties of the composite (Berg, 1998, Ruse, 1999; Tyas 1998). Resin composites also contain pigments and stabilizers. A photoinitiator and radiopaquing agent may also be added if the filler itself is not radiopaque (Berg, 1998).

Several procedures are performed in order to allow resin composites to adhere to tooth structure. Phosphoric acid is commonly used to etch enamel and remove the smear layer (Retief, 1978). The inorganic composition of enamel results in acid etching producing interprismatic and prismatic dissolution, creating irregularities in which resin can flow and, after polymerization, result in mechanical interlocking (Gwinnett, 1976).

Mechanical interlocking is initiated by flowing a water-tolerant primer over the dentine surface. The primer permeates the spaces in the networked structure of the collagen, which was previously created by acid etching. Resin composite is bonded to the tooth structure by means of an intermediary bonding agent. The use of the intermediary priming agent allows a hydrophobic bonding agent to bond to the wet dentine surface. The bonding agent in turn also bonds to the hydrophobic resin composite (Berg, 1998). In the fifth-generation bonding systems the primer and bonding agents are combined in the same bottle (Ferrari *et al.*, 1997b; Swift *et al.*, 1997).

Setting of resin composite takes place by polymerization initiated either by self-curing or light curing (Tyas 1998). Currently available resin composites undergo polymerization shrinkage when they are polymerized (Yap, Wang, Siow & Gan, 2000). Composites are

clinically durable, display excellent esthetics, are available in variety of shades and opacities and their colour stability has improved in recent years (Berg, 1998). The overall bond strength of resin composites to dentine is a combination of the individual bond strengths provided by surface adhesion, resin tag formation and hybrid layer formation (Gwinnett, 1993; Pashley & Carvalho, 1997).

1.2.2 GLASS IONOMERS

Wilson and Kent introduced self-cure glass ionomer cements in 1969, as hybrids of dental silicate and zinc polycarboxylate cements (Desai & Tyas, 1996; Wilson & Kent, 1972). According to certain researchers the term "glass ionomer cement" (GIC) is reserved for materials consisting of a basic glass and acidic polymer that sets by means of an acid-base reaction between the two components in the presence of water (Mitra, 1991).

A glass ionomer (GI) restorative material, is a tooth coloured direct adhesive restorative material (Safar, Davis & Overton, 1999). It has the ability to chemically bond to enamel and dentine with insignificant heat formation or shrinkage, and has a similar coefficient of thermal expansion to tooth structure (Hse *et al.*, 1999), particularly to dentine. GI's are compatible with the pulp and periodontal tissues (Berg, 1998) and release and absorb fluoride ions and also adhere to non-precious metals and plastics (Hotz, McClean, Sced & Wilson, 1977). Glass ionomers allow less microleakage than resin composites (El-Kalla & García-Godoy, 1998), are more stable in water than are other dental cements (Bertacchini, Abate, Blank, Baglieto & Macchi, 1999), and is indicated for Class V restorations in high caries risk patients and for subgingival restorations (Peutzfeldt, 1996).

The disadvantages of glass ionomers include esthetic problems, poor handling properties, a long setting time, dehydration during initial

setting, sensitivity to moisture and contamination, poor finishing qualities due to rough surfaces and low resistance to compression and tension (Cortes, Garcia, Perez L & Perez D, 1998; Hse *et al.*, 1999).

By definition a glass ionomer is a salt which is formed by the reaction between a polyalkenoic acid and an aluminium-containing glass. Aluminium, a constituent element in the glass, is critical for the glass ionomer reaction to occur (Berg, 1998). As mentioned before glass ionomers set via an acid base reaction in an aqueous environment (Ruse, 1999). The acid (liquid) component is a homopolymer or copolymer of alkenoic acid. Usually it is a mixture of polyacrylic acid and itaconic acid copolymer in water. The base (powder) component is a calcium alumino-silicate glass containing fluoride (Peutzfeldt, 1996). Tartaric acid (10%) is incorporated into the liquid of GIC's to prolong the working time and increase the setting rate (Hse *et al.*, 1999).

In the acid/base reaction, the acidic polymer attacks the aluminosilicate glass particles (Mount, 1991). Calcium, aluminium, sodium, fluoride and silicate ions are released from the acid-soluble glass. A silicagel layer rich in fluoride surrounds the unreacted glass particles within the cement and improves its strength. Water sorption-desorption facilitates an ion exchange between hydroxyl (OH^-) and fluoride (F^-) ions. As a result fluoride can be released from a set GI into the surrounding environment (Ruse, 1999). The fluoride in the glass material is released over time (Berg, 1998).

Calcium and aluminium, from the ion-leachable glass, crosslink the acrylic-maleic-itaconic copolymer chains with ionic bonds, leading to the setting of GI's. The carboxylic groups of the polyalkenoic chains (of the acid) can chelate the calcium of the hydroxyapatite on the tooth surface to bond the cement to mineralized hard-tooth tissue

(Ruse, 1999). Polyalkenoic chains also displace phosphate ions from the tooth surface. An ion-enriched layer is developed between the material and the tooth structure by the calcium and phosphate ions so that the glass ionomer can firmly adhere to both enamel and dentine without signs of marginal leakage. (Hse *et al.*, 1999; Swift, Pawlus & Vargas, 1995). Mount (1991) proposed that, the ion-rich layer, forms the primary mechanism bonding the glass-ionomer to enamel and dentine.

Conventional glass ionomer cements adhere physiochemically to tooth structure, therefore they were initially placed directly into the cut cavity (Cacciafesta, Jost-Brinkmann, Sussenberger & Miethke, 1998). Since then, pretreating the cavity walls with acid solutions (e.g. polyacrylic acid) became routine before restoring a glass ionomer in order to remove the smear layer which covers the cut dental surfaces (Pereira, Yamada, Tei & Tagami, 1997). This improves clinical retention and shear bond strength (Tao & Pashley, 1988). GIC bonds to enamel with strengths of around 5 MPa and to dentine with strengths of around 3 MPa (Peutzfeldt, 1996).

Polyacrylic acids are weak, but their high molecular mass and chain entanglement makes it difficult for them to penetrate through the dentinal tubules. As a result dentine is an excellent buffer and the polyacrylic acids are precipitated by the calcium ions in the tubules (Hse *et al.*, 1999).

A 10 year evaluation of cervical lesions restored with traditional glass ionomers or with a composite system revealed that for glass ionomers the retention rate was 76% at 10 years, whereas the retention rate of the composite system was much lower namely 17% (Ferrari *et al.*, 1998). The strength of traditional glass ionomers was increased with the introduction of high powder-to-liquid ration glass ionomer materials. These materials have a "condensable" feel. They were

originally developed to be used in areas of the world where atraumatic restorative treatment (ART) is used. Electricity and thus light curing is often unavailable in these areas (Berg, 1998).

1.2.3 RESIN MODIFIED GLASS IONOMERS

Introduction

The physical properties of conventional GIC's deteriorate due to moisture contamination and dehydration during setting. To overcome these problems as well as poor aesthetics, low mechanical properties, and delayed setting reaction, Mathis and Ferracane (1989) introduced a group of materials known as resin-modified glass ionomer cements (RMGIC's).

History and composition

Resin-modified glass ionomer (RMGI) restorative materials are hybrids of conventional glass-ionomer cements and visible light-activated composite resins (Swift *et al.*, 1995). They were first introduced as light-hardened Type I resin-modified glass ionomer liner/base materials (Attin, Buchalla, Kielbassa & Helwig, 1995). In 1987 Vitrabond (3M Dental Products, USA) was introduced as a base and liner (Mitra, 1991) and in 1992 Croll and Killian introduced Type II resin-modified glass ionomers for restoration of Class I, III and V cavities (Attin *et al.*, 1995). The resin component cures on command by means of light-initiated curing (Berg, 1998). RMGI's consist roughly of 20% light cured resin and up to 80% glass ionomer cement (Vaikuntam, 1997). Depending on the powder : liquid ratio of the mixture, 4-5% of the final cement mass can be regarded as extra resins (Lim, Neo & Yap, 1999). According to Sidhu, Sherriff and Watson (1999), a true RMGIC is a two-part system which is characterized by an acid-base reaction critical to its cure, a diffusion-based adhesion between the tooth surface and the cement, and continual fluoride release.

RMGIC's are marketed in a liquid-powder form which when mixed can be light-cured to a maximum of 2mm. Renewed mixing is therefore necessary when the material is used in deeper cavities (Andersson-Wenckert, Folkesson & van Dijken, 1997). RMGIC's contain the same acid (liquid) and base (powder) as GIC (Berg, 1998). The powder component is primarily composed of a radiopaque fluoroaluminosilicate glass (Mitra, 1991). The glass can be silanized to allow adherence within the resin matrix. Also included are the necessary initiators for the self-cured resin reaction (Berg, 1998). The liquid component is an aqueous solution of a modified polyalkenoic acid with pendant light sensitive side-chains that can cross-link and thus polymerize (Tvas, 1998), as well as a hydrophilic methacrylate resin such as (HEMA) or Bis-GMA, and water (Bertacchini *et al.*, 1999; Sidhu & Watson, 1995). Camphorquinone is often used as the photoinitiator (Van Dijken, 1996).

Two subgroups of RMGIC's can be recognized. In one group, e.g. Fuji II LC (GC Newport Pagnell, UK), polymerizable monomers like HEMA are blended with the polyalkenoic acid liquid. In the other group e.g. Vitremer (3M Dental Products, St Paul USA) - the above mentioned process takes place and the polyalkenoic acid itself is also modified by the attachment of polymerizable methacrylate side groups (Gladys, Van Meerbeek, Braem, Lambrechts & Vanherle, 1997). The unsaturated carbon-carbon bonds on the polyalkanoate backbone enables the covalent crosslinking of the matrix via free radical polymerization reactions (chemically or light activated). A covalently crosslinked matrix significantly improves the mechanical properties of the set cements (Ruse, 1999).

Pretreatment

Conventional GIC's can adhere physiochemically to tooth structure without any prior surface treatment (Pashley, 1984). The smear layer, which covers the cut dental surfaces, may break cohesively and fail

during polymerization shrinkage (Tao & Pashley, 1988). It was reported that shear bond strength to dentine could be improved by removing the smear layer (Powis, Folleras, Merson & Wilson, 1982). As a result pre-treating the cavity walls with acid solutions became a routine procedure before restoring with a GI or a RMGIC. Polyacrylic acid is an effective pre-treatment agent. The pre-treatment removes the smear layer and demineralizes the superficial dentine layer, allowing the HEMA in the RMGIC's to penetrate the exposed collagen network (Friedl, Powers & Hiller, 1995; Titley, Smith & Chernecky, 1996).

Adhesion

The improved adhesion to dentine compared to GIC's is probably due to the establishment of a hybrid zone and by molecular bond formation (Peutzfeldt, 1996). RMGIC's bond to tooth tissue by means of the carboxylic group of the polyalkenoate component (Rusz, Antonucci, Eichmiller & Anderson, 1992; Sidhu & Watson, 1995). Traditional GIC's as well as RMGI restoratives are water-based materials. They will therefore bond better to dentine surfaces that are not completely dehydrated as is the case with the new hydrophilic dentine bonding systems according to Kanca (1992b). According to Lin, McIntyre and Davidson (1992) light-activated glass ionomer penetrates through the smear layer into the dentinal tubules to a greater depth than chemical-cure glass ionomer. More mechanical interlocking with the substrate therefore takes place. Ion exchange occurs between glass ionomer and dentine at their interface.

Polymerization

Resin-modified glass ionomer cements harden by two reactions: (i) a slow acid-base reaction between the glass powder and organic acid, and (ii) an immediate photochemically induced polymerization of the resin component (Wilson, 1990). According to McLean, Nicholson and Wilson (1994) RMGIC's retain a significant acid-base reaction as part

of their overall curing process. In the absence of photoactivation, their properties depend on the strength of the glass ionomer setting reaction or chemically initiated free-radical methacrylate resin cure (Mitra, 1991). It has been reported that the acid-base reactions continue for weeks in the RMGIC's (Detrey, 1994). Curing takes place in 30 to 60 seconds by visible light exposure (Morabito & Defabianis 1997). Polymerization may be generated by either photoinitiators or by chemical initiators or by both. If chemical initiators are included, polymerization will begin on mixing (Hse *et al.*, 1999).

GIC's and RMGIC's must be mixed from a two-component system. The GIC and the self-cured resin elements must be kept separate to prevent reaction until it is needed (Berg, 1998).

Polymerization shrinkage

Due to polymerization shrinkage, higher bond strength is needed to prevent the material from pulling away from the margins and walls of the cavity during polymerization (Berg, 1998; Peutzfeldt, 1996). Light-cure glass ionomer liner-base formulations shrink 3,28 to 4,78% within 5 minutes after light exposure and the shrinkage continues for 12 hours. The shrinkage progresses with increased light activation time (Crim, 1993). Incremental placement techniques should therefore be used to limit polymerization shrinkage. According to Watson (1990), shrinkage can be reduced, by increasing the powder/liquid ratio.

Fluoride release

In resin modified ionomers, fluoride release is related to the type and amount of resin that is incorporated (Momoi & McCabe, 1993). Fluoride release levels off at between 30 and 90 days (Forsten, 1991). The levels of fluoride release of the RMGIC's are similar to that of the traditional GIC's (Peutzfeldt, 1996).

Advantages

RMGIC's provide better handling properties, reduced handling and setting time, more acceptable aesthetics and higher bond strength to dentine than conventional glass ionomers (Croll, 1992; Swift *et al.*, 1995). They have good sealing ability after thermocycling. Davidson and Abdalla (1994) reported that Fiji II LC RMGIC showed less microleakage than a conventional GIC even after load cycling. This agrees with the findings of Hallett and García-Godoy (1993) which indicates that cavities filled with glass ionomer/resin cement have significantly less leakage than similar cavities filled with glass ionomer cement. According to McCrary, Powers and Ladd (1994) RMGIC's possess higher mechanical properties than the conventional GIC's. Set RMGIC has improved tensile strength, compressive strength and elastic modulus when compared to conventional GIC's. The resin component makes it tougher and less brittle (Mathis & Ferracane, 1989; Uno, Finger & Fritz, 1996). The amount of resin in the material will therefore determine the physical and clinical behavior of the material (Berg, 1998). RMGIC'S exhibit a coefficient of thermal expansion similar to that of tooth structure. Fluoride ions are released from the hardened cement (Croll, 1998). RMGI's are less water-sensitive than conventional glass ionomers (Swift *et al.*, 1995) and light curing seams to solve the delayed setting reaction problem (Cho, Kopel & White, 1995). Curing can be done immediately if desired (Berg, 1998). RMGI's can bond directly to composite due to the production of a catalyst rich air-inhibited layer that can polymerize with a composite. They are also highly biocompatible with the pulp (Hse *et al.*, 1999).

Disadvantages

RMGI's are more difficult to use and less esthetic than resin composites, but better than GIC's. They must still be mixed and begin to set thereafter. Due to the resin content of the mixture, the potential of polymerization shrinkage begins with this material (Berg,

1998). RMGI cements do not have sufficient strengths to serve as complete crown restoration or in Class IV restorations (Croll, 1998).

1.2.4 POLYACID-MODIFIED RESIN COMPOSITES OR "COMPOMERS"

Introduction

In order to overcome the technique sensitivity and disadvantages of the RMGIC's, new materials containing acid-decomposable glass and acidic polymerizable monomers substituting the polyalkenoic acid polymer were developed. These materials became known as polyacid-modified resin composites or are generally referred to as "compomers" (El-Kalla & García-Godoy, 1998; García-Godoy & Hosoya, 1998; McLean *et al.*, 1994) and were introduced in the early 1990's (Andersson-Wenckert *et al.*, 1997).

McLean *et al.* (1994) offered a nomenclature categorization to distinguish RMGIC's from polyacid-modified resin composites. The name "Compomer" is a hybridization of **COM**posite and Glass Ion**OMER** (Berg, 1998). Although the term compomer has been criticized, it is widely used to describe water-free, single-component, light-cured composites consisting of an acid-soluble radiopaque fluoro-silicate glass in a matrix of acidic polymerizable monomers but in insufficient amounts to promote and acid-base reaction (Blunck, 1995; McLean *et al.*, 1994).

Dyract (De Trey Dentsply, Konstanz, Germany) and Compoglass (Ivoclar, Schaan, Liechtenstein) were the first two commercial compomers to appear on the market (Nicholson & Alsarheed, 1998). According to Croll (1998) other examples of compomers are Hytac (ESPE Dental Products, Loughborough, Great Britain), F2000 (3M Dental Products, St Paul USA), élan (Kerr GmbH, Karlsruhe, Germany).

Compomers combine the characteristics of both composites (resin component) and glass ionomers (polyalkenoic acid and glass filler component). The dental profession has rapidly accepted these new materials, largely due to their ease of use (Hse *et al.*, 1999), particularly in dentistry for children (Peters, Roeters & Frankenmolen, 1996). As a result of the rapid evolution in dental technology and the high turnover of new restorative materials, adhesive materials are today commercialized without sufficient proof of their clinical performance being provided (Maneenut & Tyas, 1995).

Components and constituents

All compomers have the following characteristics in common: They are single paste light-curing materials with fluoroaluminosilicate glass particles as fillers and at least two different resins for the matrix. The resins usually including a light-curable monomer like urethane dimethacrylate (UDMA) or Bis-GMA (Bisphenol-A-glycidyl-methacrylate) as well as a methacrylate resin with carboxyl groups (Hse *et al.*, 1999).

The acidic polymerizable monomers substitute the polyalkenoic acid polymers of the RMGIC's (Van Dijken, 1996). Due to the absence of water, the ion-leachable glass is partially silanized to ensure some bonding with the matrix (Ruse, 1999). The absence of water also prevents the material from setting in the container (Hse & Wei, 1997).

Pretreatment, adhesion and polymerization

Compomers were initially developed with the idea to build adhesiveness into the system at such a level that acid etching could be eliminated. However several compomers now do advise the use of acid etching for certain cavities. As in the case of composites, different concentrations of acids are used to etch enamel and remove the smear layer, creating irregularities in which resin can flow and,

after polymerization, result in mechanical interlocking (Gwinnett, 1976).

Different primer/adhesive systems have been developed for the different compomer restorative materials, by different product manufacturers. Syntac Single-component is used with Compoglass while F2000 primer/adhesive is used with F2000 (Hse *et al.*, 1999). Dyract AP makes use of Prime & Bond NT (Dyract AP Technical Manual, 1999). The composition of the different primer/adhesives makes them hydrophilic in nature and therefore more suitable for use on moist dentine surfaces. Once again mechanical interlocking is initiated by a water-tolerant primer permeating the spaces in the networked structure of the collagen, which was previously or simultaneously created by acid etching. In the fifth-generation bonding systems the primer and bonding agents are combined in the same bottle (Ferrari *et al.*, 1997a; Swift *et al.*, 1997). The compomer is initially bonded to the tooth structure by means of an intermediary bonding agent (Hse *et al.*, 1999).

The polymerizable methacrylate groups enable a free radical polymerization by light curing and an acid-base reaction if water is present. In the presence of water, the acid groups are expected to ionize and to interact with the basic glass components to produce a glass ionomer core analog with anticariogenic properties due to fluoride release (Eliades, Kakaboura & Palaghias, 1998). The acid-base reaction only takes place once the material is set and is in a water-containing environment (Abate, Polack & Macchi, 1997). Compomers do not have the ability to bond to hard tooth tissues directly (Martin, Paul, Luthy & Scharer, 1997).

Advantages

Compomers are esthetic, strong, are polymerized by light and release fluoride. They appear radiopaque on x-rays and are available in a

wide range of shades. Compomers are handled easily and require no mixing (Hse *et al.*, 1999).

Disadvantages

As is the case with composites, polymerization shrinkage still takes place. Due to lack in strength, compomers cannot be used in large stress bearing restorations and in general, are less resistant to wear and show more marginal discolouration than composites (Hse *et al.*, 1999).

Conclusion

Due to their chemical composition and setting reaction, compomers are linked more closely to composite resin materials than to glass ionomer cements (Marks, van Amerogen, Kreulen, Weerheijm & Martens, 1999). The resin component appears to be more responsible for the adhesion pattern of polyacid-modified composites attached with an acid etching technique than the glass ionomer component. Attin, Buchalla and Hellwig (1996) suggested that their adhesion is the result of micromechanical interlocking instead of a chemical bond to enamel.

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1.3 DYRACT, DYRACT AP, THEIR BONDING SYSTEMS, AND NRC

1.3.1. HISTORY OF DYRACT AND ITS BONDING SYSTEMS

Dyreact was introduced in 1993 as the first Compomer (Mass, Gordon & Fuks, 1999). It was developed with the objective to build adhesiveness into the system at a level sufficiently high to make acid etching unnecessary (Hse *et al.*, 1999).

Dyreact-PSA (Dentsply/De Trey, Konstanz Germany) Primer/Adhesive was introduced with Dyreact in 1993 (Dyreact Technical Manual, 1994). This system was later introduced in the United States as Prime & Bond (Dentsply/Caulk, Milford, DE). Dyreact-PSA was the first effective “one bottle bond.” This combined the separate primer and adhesive that were conventional at the time into one solution.

Continued research, development and improvements resulted in the introduction of Prime & Bond 2.0, Prime & Bond 2.1 and Prime & Bond NT. In 1995 Prime & Bond 2.0 (Dentsply/De Trey) was introduced in Europe and Asia (Hse *et al.*, 1999), while Prime & Bond 2.1 (Dentsply/Caulk & Dentsply/De Trey) was introduced in 1996 (Prime & Bond 2.1 Technical Manual, 1996).

Dyreact AP was introduced in 1997. At this stage it still made use of Prime & Bond 2.1 as bonding agent. Prime and Bond NT was introduced in 1998 as a self-priming bonding agent for amongst other things, composite restorations and Dyreact compomer materials (Dyreact AP Technical Manual, 1999; Prime & Bond NT Technical Manual 1998).

A conventional acid conditioning procedure was previously advised for occlusal stress-bearing class I, II and IV restorations. With the introduction of Prime and Bond NT, a second innovation, namely

NRC, was also introduced. This second innovation provides us with a new simplified technique of conditioning (NRC Technical Manual, 1998).

1.3.2. THE RESTORATIVE SYSTEMS

1.3.2.1. Dyract

Constituents: Dyract

- The **matrix** ($\pm 28\%$) of the final paste consists of two resins, namely 18% UDMA (urethanedimethacrylate) and 9% TCB (tetra-butane-carboxylic acid), (Eliades *et al.*, 1998; Friedl *et al.*, 1997; Gladys, Van Meerbeeck, Lambrechts & Vanherle, 1998; Hse *et al.*, 1999; Salama & El-Mallakh, 1997).

UDMA is highly biocompatible, while TCB is made up of a butane tetracarboxylic acid backbone with a polymerizable hydroxy-ethylmethacrylate (HEMA) side chain. The resultant new monomer contains two methacrylate groups as well as two carboxyl groups. The methacrylate groups can cross-link with other methacrylate terminated resins when initiated through radical polymerization while the carboxyl groups can undergo acid-base reactions to form a salt with metal ions and water (Hse *et al.*, 1999; Tyas, 1998).

- The **filler** (72%) is radiopaque SrAlFSiO_4 (Strontium fluoroaluminumsilicate glass) (Eliades *et al.*, 1998; Fritz, Finger & Uno, 1996; Salama & El-Mallakh, 1997; Tyas, 1998).

The finely milled reactive silicate glass filler (with a mean particle size of $2,5 \mu\text{m}$), accounts for 72 % (m/m) of the composition. It also contains 13 % (m/m) of fluoride, which is responsible for the fluoride release of Dyract (Hse *et al.*, 1999; Megid & Salama, 1997; Tyas, 1998).

- **Initiators, stabilizers** and **pigments** make up the remainder of the composition (Eliades *et al.*, 1998).

1.3.2.2 Dyract AP

The ingredients of Dyract AP are basically similar to the original Dyract. The organic matrix of the restorative has been modified by adding a small amount of a highly cross-linking monomer, that brings an enormous increase in hardness and strength of the matrix, almost equal to that of a hybrid resin composite, and considerably higher than the original Dyract. Dyract AP had its strontium-fluoro-silicate glass filler's particle size reduced to 0,8 μm and loading of about 73% by mass. The photoinitiator system was optimized in order to obtain further increases in strength. According to its manufacturer, Dyract AP is indicated for all cavity classes in anterior and posterior teeth, provided the width of class I and II cavities in posterior teeth are less than $\frac{2}{3}$ of the intercusp distance (Dyract AP Technical Manual, 1999).

Constituents: Dyract AP

In summary Dyract AP contains the following (Evancusky & Meiers, 2000; Gladys *et al.*, 1998; Salama & el Mallakh, 1997; Dyract AP Technical Manual, 1999):

- Polymerizable resins e.g PENTA (dipentaerythritol penta acrylate monophosphate), UDMA (urethane dimethacrylate), TEGDMA (triethylene glycol dimethacrylate)
- TCB resin (tetra-butane-carboxylic acid)
- Strontium-fluoro-silicate glass
- Strontium fluoride
- Photo initiators
- Stabilizers

Setting of Dyract AP

Dyract AP can only be hardened through photopolymerization. The setting reaction consists of two stages. The first stage is that of dominant free radical polymerization. Upon light-curing the polymerizable molecules of UDMA and the TCB resins are interconnected into a three dimensional network which is reinforced by means of the enclosed filler particles (Hse *et al.*, 1999; Megid & Salama, 1997). Dyract AP is an anhydrous formulation and therefore no ion exchange takes place at this stage. After the initial polymerization the Dyract AP begins to absorb water in the moist environment of the mouth. An ionic acid-base reaction as in glass ionomers can now take place. During the second stage of setting, the acidic conditions created by the carboxyl groups on the TCB molecules of Dyract AP, cause metal cations to be liberated from the reactive silicate glass which leads to the formation of hydrogels in the resin structure of the compomer (Hse *et al.*, 1999). The extent of this reaction is very limited due to the present rigidity of the material, but results in further cross-linkages of the matrix (McCabe, 1996). Water absorption can continue for months depending on the size of the restoration. The maximum level of water content is estimated at three per cent of water (m/m) at the most in Dyract (Dyract AP Technical Manual, 1999). The resulting volumetric change is considered to be low and insignificant (Attin *et al.*, 1995).

Adhesion of Dyract AP to the cavity wall

Theoretically Dyract AP therefore bonds to the cavity walls by means of two different mechanisms. One is the self-adhesive property of the restorative itself where the functional carboxyl (-COOH) groups of the TCB monomer can form ionic bonds with the calcium ions of the tooth surface. The second mechanism is adhesion to the tooth surface through the primer/adhesive system. Ionic bonds will form between the hydrophilic phosphate group of the PENTA resin and the calcium ions of the hydroxyapatite. The three methacrylate-based

resins in the adhesive will undergo free radical polymerization when light-cured (Hse *et al.*, 1999; Megid & Salama, 1997).

1.3.3. THE PRIMER/BONDING SYSTEMS

1.3.3.1. Dyract-PSA

As mentioned before, Dyract-PSA was introduced in 1993 (Barkmeier, Hammesfahr & Latta, 1999) as an adhesive consisting of a combination primer and bonding agent in one bottle. In its first version Dyract was marketed with Dyract-PSA to be directly applied on dentine and on enamel surface to increase adhesion. Dyract-PSA prime/adhesive liquid was developed with the stipulation that no etching of the tooth surface was to be necessary prior to application (Chersoni *et al.*, 1997).

Constituents: Dyract-PSA

In summary Dyract-PSA contains the following (Abate *et al.*, 1997a; Attin *et al.*, 1996; Gladys *et al.*, 1998; Perdigao, Lambrechts & Van Meerbeek *et al.*, Braem, Yildiz, Yucel & Vanherle, 1996; Swift *et al.*, 1997; Tulunoglu, Ayhan, Olmez & Bodur, 1998):

- PENTA (a hydrophilic monomer)
- TEGDMA (a hydrophobic monomer)
- Elastomeric urethane-modified Bis-GMA resin (a hydrophobic monomer)
- Fluoride
- Acetone (organic solvent)
- Photoinitiator (amine-campherquinone)
- Stabilizers

PENTA contains an acidic monomer made up of phosphoric acid with a polymerizable methacrylate group attached. It conditions the tooth structure and is responsible for the formation of ionic bonds to the inorganic part (calcium) of the tooth (Hse *et al.*, 1999). TEGDMA and

an elastomeric resin determine the level of cross-linking among the different monomers and the resilience (elasticity) of the cured PSA prime/adhesive. Elastomeric resins consist of rigid and elastomeric molecules that are highly cross-linked. This forms a network that can tolerate stress and in this way bonding and marginal seal is accomplished. The acetone acts as a solvent and carries the resins and helps to wet the enamel and penetrate the dentine surface (Abdalla & Davidson, 1998).

1.3.3.2. Prime & Bond 2.0

Prime & Bond 2.0 (Dentsply/De Trey) was introduced as a dentin-enamel bonding agent for composites (Hse *et al.*, 1999). For this material the total-etch technique was proposed.

Constituents: Prime & Bond 2.0

- PENTA
- Two additional unrevealed dimethacrylate resins
- Elastomeric urethane-modified Bis-GMA resin
- Acetone
- Photoinitiator
- Stabilizers

1.3.3.3. Prime & Bond 2.1

Prime & Bond 2.1 (Dentsply/Caulk & Dentsply/De Trey) was introduced worldwide as primer/bonding agent for composites and Dyract compomers. The application directions were changed with the introduction of Prime & Bond 2.1. The first application time was reduced from thirty to twenty seconds and the curing of the second application was eliminated. Prime & Bond 2.1 uses the elastomeric resin of Prime & Bond 2.0 plus the addition of cetylamine hydrofluoride, which provides release of fluoride ions. Once again the total-etch technique was proposed for stress-bearing class I, II and IV

restorations (Prime & Bond 2.1 Technical Manual, 1996; Barkmeier *et al.*, 1999).

Constituents: Prime & Bond 2.1

- PENTA
- Dimethacrylate resins
- Elastomeric urethane-modified Bis-GMA resin
- Acetone
- Photoinitiator
- Stabilizers
- Cetylamine hydrofluoride

(El-Kalla, 1999a; El-Kalla, 1999b; Evancusky & Meiers, 2000; Gordan, Boyer & Soderholm, 1998; Vargas *et al.*, 1997)

1.3.3.4. Prime & Bond NT

Prime & Bond NT is the successor of Prime & Bond 2.1. Prime & Bond NT nano-technology was introduced to adhesive dentistry in 1998 (Prime & Bond NT Technical Manual, 1998). The manufacturer has added the following (Perdigao, Baratieri & Lopes, 1999):

- nanofiller particles of average size of 0.007 μm
- a cross-linking agent
- a small resin molecule

The overall resin concentration of Prime & Bond NT has been raised in comparison to that of Prime & Bond 2.1. The higher resin concentration ensures saturation of the tooth surface, making the bonding agent less technique sensitive (Ferrari *et al.*, 1999).

A small resin (**D-resin**) has been added. The small resin molecule provides for better infiltration of the porous dentine structure, guaranteeing extremely high bond strengths (Perdigão *et al.*, 1999).

A crosslinking agent (**T-Resin**) leads to a denser network of the resin matrix, which could make the bonding agent tougher (Ferrari *et al.*, 1999).

The particle size of 0.007 μm of Prime & Bond NT **nanofiller** is $\pm 1/100^{\text{th}}$ of the size of the Dyract AP filler particle. The size of a conventional filler of Dyract AP is 1 μm (1 μm =1000 nm). Fillers are incorporated in composites or compomers to increase strength. Nanofillers allow an adhesive to be reinforced by tiny particles, while maintaining essential properties of high performance bonding such as low viscosity and good penetration properties. A nanofiller also increases the strength and toughness of a dental material. The nanofiller in Prime & Bond NT is nanoscale silica (Aerosil, Degussa Corp., Ridgefield, New Jersey) which has been functionalized by a special silanization process (Ferrari *et al.*, 1999; Perdigão *et al.*, 1999; Prime & Bond NT Technical Manual, 1998).

General characteristics

The manufacturer claims the following about Prime & Bond NT (Prime & Bond NT Technical Manual, 1998):

- it is a one bottle adhesive
- it is suitable for etched and unetched dentine and enamel
- long stability of bonds
- releases fluoride
- increased adhesive strength to both enamel and dentine
- increased marginal integrity
- sufficient film thickness for one-coat, one-cure technique
- it looks transparent seeing that the filler is too small to be visible

Composition of Prime & Bond NT: Components and functions

(Perdigão *et al.*, 1999; Prime & Bond NT Technical Manual, 1998)

- Di and Trimethacrylate resins:

UDMA resin - resin molecule of intermediate elasticity when cured

Resin R5-62-1 - elastomeric element in the cured network

T-Resin - small, crosslinking molecule

D-Resin - small, mobile resin molecule for good dentine infiltration

- **PENTA:** - adhesion promoter, wetting aid and crosslinker

- **Nanofiller:** - functionalised amorphous silica nanoscale filler for increased strength and crosslinking

- **Photoinitiators:** - initiate the light-curing reaction

- **Stabiliser:** - stabilizes material during storage

- **Cetylamine Hydrofluoride:** - fluoride source

- **Acetone:** - solvent and carrier for the resins; water displacer

Fluoride release

Prime & Bond 2.1 displays an initial burst of fluoride release, but it decreases in the first 10 weeks to below 0.1 microgram/cm². Prime & Bond NT has a more gradual fluoride release which decreases after 5 weeks to below 1 mg/cm², but at 22 weeks it is still well above 0.1 mg/cm² (Prime & Bond NT Technical Manual, 1998).

Shear bond strength

Perdigão *et al.* (1999) reported that the shear bond strength of Prime & Bond NT was not statistically different from that of Prime & Bond 2.1. However for each adhesive system individually, the shear bond

strengths to enamel was significantly higher than the shear bond strength to dentine.

Handling of Prime & Bond NT

According to its manufacturer (Prime & Bond NT Technical Manual, 1998), Prime & Bond NT can be used in three different ways:

- without conditioning the enamel and dentine
- in combination with phosphoric acid
- with a self-priming, non-rinse conditioner, namely NRC

The choice of procedure depends on the clinical conditions, e.g. the class of cavity (Ferrari *et al.*, 1999). As mentioned Prime & Bond NT contains a self-etching primer which is used as a coupling agent, and PENTA, an ester of phosphoric acid and adhesion promoting monomers in an acetone solvent. The primer conditions the tooth tissues and penetrates the enamel and dentine. The decalcified smear layer is not rinsed away, and a hybrid layer is formed, creating a micromechanical bond to tooth tissue. The manufacturer claims that adhesion is also obtained through ionic bonds between the hydrophilic phosphate groups of the PENTA and the calcium of the hydroxyapatite. Adhesion is also thought to be achieved through the hydrophilic TCB monomer (tetra-butane-carboxylic acid) present in the restorative (Andersson-Wenckert *et al.*, 1997).

1.3.3.5 Scanning electron microscope morphology

Prime & Bond NT

Ferrari *et al.*, (1999) reported the following scanning electron microscope morphology of the resin tags when Prime & Bond NT was applied in the three mentioned different ways:

- **Without conditioning** of the dental substrates:

The dentine samples did not clearly show hybrid layer formation. Resin tags were seldom observed, seeing that the primer/adhesive

material was only able to penetrate the smear layer in a few areas. The shape of the resin tags was narrow and their surfaces were smooth. Many areas did not show resin tag formation and the tubules were still plugged with smear plugs. The resin tags did not seal the tubular orifices completely. Adhesive lateral branches were not observed.

The enamel samples showed no resin tags and the characteristic etched pattern of enamel was not observed.

- In combination with **36% phosphoric acid**:

The dentine samples showed a hybrid layer between resin and dentine interfaces of 3-5 μm thickness. Resin tags of 10-30 μm length and adhesive lateral branches were also observed. The characteristic reverse cone-shape of resin tags was noted at higher magnification (x5000).

Characteristic uniform etch patterns were observed in the resin samples of the enamel group as well as resin tags penetrating the demineralized enamel surface.

- In combination **NRC** pretreatment:

The dentine samples presented a hybrid layer of between 1 and 2 μm thickness. Resin tags with adhesive lateral branches in some areas were also detected. The density of the resin tags was less evident than that observed in the phosphoric acid pretreated group. The resin tags were also shorter and narrower.

The enamel samples showed an etch pattern shallower and less uniform than that observed in the phosphoric acid pretreated group. Resin tag formation at the resin-enamel interface was observed.

Additional SEM reports for Prime & Bond NT as well as reports for Prime & Bond 2.1

Li, Tyas and Burrow (1999) reported a hybrid layer with a thickness of 1-2 μm , when Prime and Bond NT was applied to dentine after the application of NRC. Moodley *et al.* (2000) reported a hybrid layer of 2 μm and resin tags of 20 μm under the same conditions. Scanning electron microscopy reports by Perdigão *et al.* (1999), reported that phosphoric acid gel (Conditioner 36) demineralized intertubular dentine to a depth of 3.0 to 4.1 μm and that Prime & Bond NT as well as 2.1 gave hybrid layers 3.0 to 4.5 μm thick. Both adhesives resulted in a fully infiltrated hybrid layer. They also reported that the nanofiller of Prime & Bond NT penetrated the dentine tubules. The filler was also observed within the hybrid layer. Filled adhesives are designed to provide stress relief between the tooth and restorative material (Van Meerbeek, Willems and Celis *et al.*, 1993).

SEM reports for Prime & Bond 2.1 and Prime & Bond 2.0

Hybrid layer and resin tag length results obtained by Ferrari *et al.* (1997b) for Prime & Bond 2.1 were as follows:

In conditioned dentine a hybrid layer was formed between the resin and dentine interfaces. The thickness of the hybrid layer was 2-6 μm , and varied between samples and also at different sites of the same specimen. Resin tags of 10-30 μm length and adhesive lateral branches were observed.

In unconditioned dentine no hybrid layer was formed. A residual smear layer between the resin and dentine was observed. Resin tags were seldom observed.

El-Kalla (1999a) reported that Prime & Bond 2.1 showed heavy and deep infiltration of the dentine tubules, with much lateral branching

in etched cavities. In non-etched cavities there was no consistent hybrid layer and some dentine tubules exhibited shallow penetration, while others appeared empty. El-Kalla (1999b) also reported that the resin penetrated and formed resin tags to a mean depth of 62 μm , while the hybrid layer had a mean thickness of 5.4 μm .

Prati *et al.*, (1998) gave the following results for Prime & Bond 2.0 and Prime & Bond 2.1:

RIDL thickness:

P&B 2.0	deep dentine	3.0-6.5 μm
"	superficial dentine	2.0-4.0 μm

P&B 2.1	deep dentine	3.0-7.5 μm
"	superficial dentine	2.0-4.0 μm

Tag length:

P&B 2.0	deep dentine	20.0-120 μm
"	superficial dentine	4.0-5.0 μm

P&B 2.1	deep dentine	20.0-120 μm
"	superficial dentine	4.0-5.0 μm

Kälin, Paul, Schärer, Düggelin, Mathys and Guggenheim (1998) reported that when you apply two layers of Prime & Bond 2.1 after acid etching, the surface of the dentine appeared to be partially covered with liquid and showed "lake-like" areas of adhesive after the first application. Only superficial penetration into the collagen network appeared. Application of a second layer of Prime & Bond 2.1 resulted in a consolidation of the adhesive layer. The adhesive layer increased in thickness and was free of voids in many areas. However numerous internal voids were found in other areas.

SEM reports for self-priming adhesives and self-etching primer systems

Miyazaki *et al.* (1998) reported that the thickness of the resin-dentine interdiffusion zone was between 4-5 μm for self-priming adhesive systems, while a thin hybrid layer (1-1,25 μm) was observed for self-etching primer systems.

1.3.4. NON RINSE CONDITIONER (NRC)

As mentioned before, a conventional acid conditioning procedure was previously advised for occlusal stress-bearing class I, II and IV restorations. NRC was developed as a new simplified technique of conditioning. As the name of the product indicates, NRC (Non Rinse Conditioner) remains on the tooth surface after application and does not require rinsing. This simplifies the application and gives better control of the working field. NRC contains organic acids in an aqueous solution (Ferrari *et al.*, 1999; NRC Technical Manual, 1998).

Composition of Non Rinse Conditioner: Components and their functions:

Component	Functions
Itaconic acid	This organic acid primes the tooth surface. It copolymerizes with Prime & Bond NT. The carboxylic groups adhere to the calcium of the tooth surface.
Maleic acid	It conditions and cleans the tooth surface.
Water	Acts as a solvent

The maleic and itaconic acids contain polymerizable double bonds. The double bonds enable the NRC to copolymerize with the Prime & Bond NT matrix. As Prime & Bond NT has a low viscosity and is acetone based, it deeply penetrates the tooth surface and completely covers the NRC remnants as well as the debris (dissolved smear layer) on the tooth surface. The NRC remnants and "debris" gets

incorporated as filler particles. The functions of the NRC components indicate that it can be regarded as both conditioner and primer (Tanumiharja, Burrow & Tayas, 2000). It functions mainly as a conditioner to demineralize and expose collagen fibers on the dentine surface, but also contains priming components (Ferrari *et al.*, 1999; NRC Technical Manual, 1998).

NRC and removal of the smear layer

de Wet, van der Vyver, Eick and Dusevich (2000) evaluated the effect of different treatments on the smear layer using a field emission environmental scanning electron microscope. Phosphoric acid gel and liquid removed the smear layer. The gel demineralized the dentine for approximately 8 μm , while the liquid demineralized the dentine for approximately 6.5 μm . NRC penetrated the smear layer and modified it to a lesser degree. A demineralized zone of approximately 1.6 μm was formed. When the NRC treated surface was washed, it resulted in removal of part of the smear layer and opened up more dentinal tubules. The thickness of the demineralized zone was approximately 2 μm .



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1.4 MICROLEAKAGE

Introduction and definition

According to Kidd (1976), microleakage can be defined as the movement of bacteria, fluids, molecules or ions, or even air between the prepared cavity wall and the applied restorative.

Bergenholtz, Cox, Loesche and Syed (1982) as well as Brännström (1987) clearly documented the effects of bacterial leakage upon the dental pulp. Microleakage has a sensory component (Cox, 1992) as well as a pathological component (Bergenholtz *et al.*, 1982). The hydrodynamic fluid movement into patent dentinal tubules through the smear layer, results in hypersensitivity to thermal and osmotic stimuli and is referred to as the sensory component of microleakage (Cox, 1992; Pashely & Pashley, 1991c). Bacteria which have penetrated gaps along the axiopulpal floor as well as their metabolic products results in recurrent caries and pulpal pathoses and thus accounts for the pathologic component of microleakage (Bergenholtz *et al.*, 1982).

Besides pulpal irritation and recurrent caries, microleakage also results in tooth discolouration (Going, 1972). The ability of a material to prevent penetration can be evaluated by using a number of microleakage techniques reported in the literature (Yu, Davis, Joynt & Wiczkowski, 1992).

Different types of microleakage studies

Many different techniques have been used to demonstrate microleakage. Colour producing microorganisms (Seltzer, 1955); radioactive isotopes, such as Ca^{45} (Armstrong & Simon, 1951; Swartz & Phillips, 1961); fluorescent dyes, such as fluorescein (O'Brien, Espinoza, Leinfelder, Krainik & Krainik, 1971) and non-fluorescent dyes such as aniline blue (Hirsch & Weinreb, 1958), silver nitrate

(Eakle & Nakamoto, 1989) and basic fuchsin (Retief, O'Brien, Smith & Marchman, 1988) have all been used to demonstrate microleakage. Going (1972) reported that these microleakage studies are examples of qualitative microleakage studies.

Dye penetration studies are amongst the most popular techniques currently used to study *in vitro* microleakage (Yap, Ho & Wong, 1998). The silver-staining method has been used extensively in microleakage studies and is often referred to as the silver nitrate test (Wu, Cobb, Derman & Rupp, 1983). Microleakage studies normally quantify the movement of tracer molecules through gaps between the restorative material and the cavity wall (Sano *et al.*, 1995a).

Examples of quantitative microleakage studies includes the air pressure method, neutron activation analysis and a volumetric dye-recovery method (Kidd, 1976). Ferrari, Yamamoto, Vichi and Finger (1994) reported that *in vitro* microleakage tests performed offered results very similar to *in vivo* test results.

General facts about microleakage

Barnes, McDonald, Thompson, Blank and Shried (1994) found no statistically significant difference in microleakage between facial and lingual tooth enamel. Tay, Pang, Gwinnett and Wei (1995) confirmed that the extent of microleakage is not uniform along a restoration margin. Most microleakage studies do not take the density of the leakage into account, demonstrating only a two-dimensional picture of a three-dimensional leakage pattern (Mandras, Retief & Russell, 1993). Gale, Darvell and Cheung (1994) reported that microleakage is a three-dimensional phenomenon and different locations and angles of sectioning might result in completely different penetration scores.

Newer generation adhesives create a hybrid layer (resin-impregnated dentine) between resin and dentine with the use of a dentine/enamel

conditioner, a primer and a bonding resin (Inokoshi, Hosoda, Harnirattisai & Shimada, 1993). This results in an increase in the number of interfaces between the restoration and the tooth itself. Tay, Gwinnett, Pang and Wei (1995) hypothesized that systems using a total-etch technique to remove the smear layer completely and partially demineralize the underlying dentine, could have microleakage in any or a combination of at least eight regions within the dentine-restorative junction. Prati, Nucci, Davidson and Montanari (1990) reported that the time immediately after placement of a restoration is critical, since microleakage may occur during the first minute after placement. Cox (1994) emphasized the correlation between pulpal response and material biocompatibility in the absence of bacterial microleakage. Gwinnett, Tay, Pang and Wei (1995) reported that more extensive leakage occurred near the end surfaces of restorations and that a single longitudinal section through the center of a restoration appeared to underestimate the full extent of microleakage. Wieczkowski, Yu, Davis and Joynt (1992) reported that the ability of a restorative material/adhesive to seal the interface with tooth structure is the most significant factor in the prevention of the formation of future caries.

According to Owens, Halter and Brown (1998) as well as Fukushima, Nakamata, Okabe, Yamaga, Sunada and Iwaku (1991), microleakage occurs with all dental adhesives and restorative materials, even when using the new generation dentine adhesive systems with gingival margins on dentine/cementum. Retief DH (1994) reported that the ability to render a non-leaking composite restoration below the cemento-enamel junction still depended on the performance of the adhesive in resisting polymerization contraction and thermal, and occlusal loading stresses. Cardoso *et al.*, (1999) reported that a perfect seal still remains one of the most important and challenging features to consider when choosing an adhesive material.

Although new materials are often subjected to rigorous testing of bond strengths, no dental material is completely exempt from microleakage. However, the information is useful for comparative assessment of different materials (Morabito & Defabianis, 1997). Furthermore, microleakage can lead to staining, sensitivity or caries (Yap, Lim & Neo, 1995).

Microleakage and Prime & Bond NT

A few studies have been published dealing specifically with the microleakage associated with Prime & Bond NT.

The study of Cardoso *et al.* (1999) evaluated the microleakage of Prime & Bond NT without the influence of saliva contamination. The enamel margins were beveled and a cotton pellet was used to blot dry the cavity after etching. Prime & Bond NT was only able to eliminate microleakage at the enamel margins.

Moodley *et al.* (2000) compared the shear bond strength and microleakage of Dyract AP/NRC/Prime & Bond NT to Scotchbond Multi-Purpose Plus and F2000. Microleakage was evaluated with the use of a confocal scanning laser microscope. In the Prime & Bond NT specimens microleakage was found on the dentine side in 13 of the 15 specimens examined, while on the enamel side 2 of the 15 specimens showed microleakage.

A study performed by Tung, Estafan and Scherer (2000a) evaluated microleakage in which 36% phosphoric acid and Prime & Bond NT was used to prepare the cavities before Surefil resin composite was placed. Etchant was rinsed from the preparations with water after which the preparations were dried with an air syringe for 20 seconds. Of the 15 specimens, 3 of the specimens exhibited microleakage in enamel and 2 in dentine/cementum. Tung, Estafan and Scherer (2000b) investigated amongst others the microleakage of class V

lesions restored with a compomer material using Prime & Bond NT as the adhesive. The enamel cavosurface was beveled. A 20 second air-water spray was used to rinse off the etchant gel. One or 2 quick bursts of dry air was applied to obtain a moist dentine surface. No leakage was obtained on the enamel side. Leakage did occur on the dentine side, but the leakage on the enamel and dentine sides did not differ significantly from each other.

Factors that influence the tooth-restoration interface

The integrity of the tooth-restoration interface is dependent upon several factors. These factors include polymerization shrinkage on curing (Kaplan, Mincer, Harris & Cloyd, 1992); water sorption after curing; the difference between the linear coefficients of thermal expansion of the tooth and restorative material (Yap *et al.*, 1998); the adhesive bond strength (Davidson, de Gee & Feilzer, 1984); the cavity configuration, as well as thermal and masticatory stresses (Gwinnett *et al.*, 1995).

1.4.1. Polymerization shrinkage

Composite restorative resins undergo shrinkage during polymerization (Bowen, Rapson & Dickson, 1982). The difference in the coefficient of thermal expansion of tooth structure and restorative material, as well as polymerization shrinkage, produces stress within the adhesive layer (Asmussen, 1975; Hansen & Asmussen, 1985; Kemp-Scholte & Davidson; 1990a). This in turn introduces stress at the tooth/restorative interface (Bowen *et al.*, 1982; Davidson *et al.*, 1984b) and can lead to breakdown or gap formation in the adhesive bond between the restoration and the cavity walls. This could result in loss of marginal integrity if it is not resisted (Carvalho, Pereira, Yoshiyama & Pashley, 1996; Davidson, 1986; Feilzer, de Gee & Davidson, 1990a; Katoma & Winkler, 1994).

Wieczkowski, Joynt, Davis, Yu and Cleary (1992) found that polymerization shrinkage in hybrids is stronger than the chemical bond between glass ionomer cement and dentine, therefore opening a pathway for microleakage in glass-ionomer based resin restorations. Prati, Chersoni, Cretti and Mongiorgi (1997) reported that the shrinkage of resin composites and glass ionomer cements are sufficient to create open and permanent damage to margins which may lead to microleakage especially along dentine margins. They found in SEM studies that enamel margins were characterized by prism fractures around restorations. Dentine margins showed gaps along the dentine bonding agents and fractures in the bonding agent/composite interface. Several more authors also reported enamel microfractures, as a consequence of composite polymerization shrinkage (Staninec, Mochizuki, Tanizaki, Jukuda & Tsuchitani, 1986; Watson, Billington & Williams, 1991). Attin *et al.* (1995) reported that the resin content of compomers and light-cured glass ionomers, which varies among available products, produces polymerization shrinkage which is not evident in conventional glass ionomers. This could adversely affect marginal adaptation.

Feilzer, de Gee and Davidson (1990b) as well as Willems, Lambrechts, Braem and Vanherle (1993) reported that the hybrid layer is not able to sufficiently withstand the shrinkage stress of a high modulus composite e.g. Z100. Retief, Mandras and Russell (1994) suggested that shear bond strength of ± 21 MPa to dentine may reduce microleakage to near zero.

Two factors have a major influence on the results of comparative dye penetration studies. Firstly, the inorganic filler volume percentage which influences the polymerization shrinkage (Lai & Johnson, 1993) and secondly the elasticity which limits the residual shrinkage stresses (Feilzer, de Gee & Davidson, 1987).

1.4.2. Modulus of elasticity

Young's modulus or the modulus of elasticity, describes the rigidity or stiffness of a material (Phillips, 1991). According to Van Meerbeek, Willems and Celis *et al.* (1993) the resin-dentine interdiffusion zone or hybrid layer has a relatively low Young's modulus and may therefore also relieve polymerization contraction stresses.

The modulus of elasticity could influence the sealing ability of a material. If a material has a high modulus of elasticity, it is stiffer and has more internal stress. A low modulus of elasticity results in a greater elastic ability and more capacity to reduce contraction stress. The higher the degree of filler, the more stress is exerted on the bonding resin during polymerization. Resins with a higher filler content have a lower volumetric contraction, because of the higher filler content relative to the lower resin content, but they have a higher Young's modulus of elasticity. The higher stiffness of the high-modulus composites generally leads to higher shrinkage stresses during polymerization, which may tend to destroy the bond to the tooth structure, thereby increasing the chance of leakage. In turn, while a low-modulus material may shrink more than a high modulus material, its lower stiffness leads to a higher elasticity and thus a lower remaining contraction stress which can affect the resin-dentine interface (Schwartz, Summit & Robbins, 1996).

Kanca (1989) reported that microfills tend to exhibit less leakage than hybrids. Intermediate resins could be used to act as "elastic buffers" to relieve the polymerization contraction stresses within resin containing restorations (Swift, Triolo, Barkmeier, Bird & Bounds, 1996; Van Meerbeek *et al.*, 1993c). Dual-cure systems could relieve stress more effectively due to incorporation of air bubbles during mixing. Porosities have been shown to increase the elasticity of resin materials (Alster, Feilzer, de Gee, Mol & Davidson, 1992). Polymerization stress could also be reduced by a materials inherent

ability to undergo plastic deformation or flow, due to the ability of the molecules to slip into new positions and orientations, allowing the maintenance of the adhesive bond (Feilzer *et al.*, 1990b).

Tay *et al.* (1995a) reported that the presence of silver staining along the resin-hybrid layer interface suggests that contractional stresses during polymerization of the resin composite already created a separation of margins.

1.4.3. Light exposure and light intensity

Miyazaki, Fukuishi and Onose (1999b) reported that volumetric change of a compomer is amongst other things influenced by the duration of light exposure, light intensity and the environmental conditions to which the materials are exposed. Uno and Asmussen (1991) recommended using low light intensity, to allow the resulting reduced polymerization rate and the relaxation of the contraction stress, to get good marginal adaptation.

Miyazaki *et al.* (1999b) reported that the volumetric shrinkage for Dyract after 160 seconds was 2,7%. Increased light intensity as well as increased irradiation time resulted in increasing volumetric shrinkage. The polymerization shrinkage of compomers ranged from 2,1 to 2,7% (volumetrically). Davidson and de Gee (1984b) reported that polymerization shrinkage is greatest in the early stages of setting. Berg (1998) concluded that the shrinkage of composites ranged from 2 to 3,5%. Shrinkage of a composite, which is bonded circumferentially to the cavity walls, causes it to pull towards the centre of its mass (Berg, 1998). Versluis, Tantbirojn and Douglas (1998) concluded that composites do not shrink toward the light, but that the direction is predominantly determined by cavity shape and bond quality.

1.4.4. Hygroscopic expansion

The hygroscopic expansion of a restoration, when exposed to the oral fluids, partly counteracts the polymerization shrinkage (Fan, Edahl, Leung & Stanford, 1985), but does not necessarily compensate for it (Hansen & Asmussen, 1985). The water uptake and expansion will not re-establish the failed bonds that resulted from polymerization shrinkage (Rigsby, Retief, Russell & Denys, 1990).

Attin *et al.* (1995) reported that Fuji II LC glass ionomer cement expanded after curing and immersion in water, while Dyract (compomer) and Vitremer (glass ionomer) cement revealed a total volumetric loss. They concluded that water expansion is a factor that attributes to the reduction in microleakage.

Feilzer, Kakaboura, de Gee and Davidson (1995) reported that the better performance of marginal adaptation after twelve months compared to six months in compomers could be explained by water sorption and expansion of the material. Allen, Retief, Russell and Denys (1994) found that quantitative microleakage recorded after different storage periods was not significantly different. Although exposure to the oral environment resulted in a reduction or elimination of the dimensions of the marginal gaps caused by polymerization shrinkage at the tooth restoration interface, the adhesive bonds that were broken by polymerization shrinkage will not be re-established by hygroscopic expansion.

Toledano, Osorio, Osorio and Garcia-Godoy (1999) reported that resin modified glass ionomers show less or similar microleakage to Dyract. Curing contraction is often restricted by the cavity preparation resulting in polymerization contraction stress (Feilzer, de Gee & Davidson, 1988).

Perdigão *et al.* (1996) reported that one-bottle dentine bonding agents do not seem able to prevent gap formation (i.e. they cannot withstand shrinkage forces generated during the polymerization of resins).

Holderegger, Paul, Luthy and Scharer (1997) concluded that bond strength values superior to the forces generated during the polymerization of composite luting resins are only obtained after dual application of two or three-step dentine bonding agents.

Hansen (1986); Vargas and Swift (1994); Wieczkowski *et al.* (1992a) all suggested to apply thin incremental layers of composite into a cavity in order to minimize resin composite polymerization shrinkage.

1.4.5. Bond strength

Several factors influence the quality of the bond e.g. the thickness of the smear layer, the degree of etching, wetness and polymerization contraction (Pashley & Carvalho, 1997). Monteiro, Sigurjons, Swartz, Phillips and Rhodes (1986) reported that bond strength is more predictive of a material's retentive potential than its sealing ability. Kanca (1989) reported that there was no statistically significant correlation between bond strength and microleakage. Borem and Feigal (1994) reported that although it may seem logical for bond strength and microleakage to have an inverse relationship, leakage could take place around cluster areas or foci of adhesion.

According to Retief, Mandras and Russell (1994) bond strength of 20 to 24 MPa is thought necessary to resist three-dimensional stresses resulting from polymerization shrinkage of resin composite and prevent microleakage at the dentine-resin interface.

Titley, Chernecky, Rossouw and Kulkarni (1998) as well as Retief, Wendt, Bradley and Denys (1989) reported that storage of teeth in 0,1% thymol solution resulted in significantly lower shear bond strengths. However, Haller, Hofmann, Klaiber and Bloching (1993)

reported that there was not a significant difference in microleakage between freshly extracted teeth and teeth stored in thymol. Haller also reported that teeth stored in 1% chloramine gave results comparable to those of freshly extracted teeth.

1.4.6 The effect of acid etching

Conditioning of a cavity with acid increases the dentinal permeability. There is great individual variation in the permeability of teeth following acid conditioning of dentine (Youngson, Jones, Fox, Smith, Wood & Gale, 1999). Microleakage at the enamel margins of restorations etched with phosphoric acid has been eliminated (Fusayama & Kohno, 1989).

The ability of the dentine or cementum-material bond to resist microleakage is still controversial (Manhart, Chen, Mehl, Weber & Hickel, 2001). Sano, Yoshiyama and Ebisu *et al* (1995b) reported that bonding systems that etch deeper in dentine are more likely to show higher degrees of microleakage. Sano *et al.* (1995a) reported that strong adhesion heavily relies on the diffusion and curing of adhesive resins through demineralized dentine. They also reported that the adhesive resins could have difficulty diffusing through demineralized dentine as a result of the presence of water around collagen fibers, the depth of the demineralized zone and nonuniform distribution of monomers.

The presence of a layer of resin-infiltrated enamel is demonstrated by Tay *et al.* (1995a), this confirms the hybridization concept that was originally observed and described in enamel by Gwinnett and Matsui 1967. Perdigão *et al.* (1996) demonstrated that etched and bonded enamel produces a more consistent hermetic seal compared with dentine. Fusayama (1992) reported that the total-etching technique is more effective than etching of the occlusal enamel alone in reducing

microleakage along the cervical wall of Class V resin composite restorations.

1.4.7. Dry vs wet or moist bonding procedures

Tay *et al.* (1995a) demonstrated severe microleakage along the resin-dentine interface when water was not completely removed from the primer solvent. Resin globules formed along the surface of the hybrid layer instead of a continuous resin layer, preventing complete seal of the dentine tubules. Vargas and Swift (1994) found no differences in microleakage between the dry or moist bonding technique while Saunders and Saunders (1996) reported microleakage at both the enamel and dentine margins, but less microleakage at the gingival margin in the groups where acetone-based bonding agents and wet bonding were used.

1.4.8. Cavity configuration and bevelling

Dietrich *et al.* (2000a) found that beveled enamel margins showed significantly less enamel microcracks compared to unbeveled enamel margins regardless of the conditioner used. There were no significant differences in microleakage between butt-joint and beveled enamel margins of Dyract AP restorations. When enamel was conditioned with the non-rinse conditioner, Dyract AP total-bond restorations in beveled cavities showed significantly more perfect margins compared to butt-joint cavities. No differences between beveled and butt-joint cavity preparations with respect to perfect margins were observed when the enamel was conditioned with 36% phosphoric acid.

Hilton and Ferracane (1999) reported that vertical cavity walls without bevels exhibited pronounced leakage. Saunders and Muirhead (1992) reported that bevelling of enamel followed by etching virtually eliminates microleakage in enamel.

Dentine tubules run parallel to enamel rods, therefore beveling of dentine should expose opened tubules for efficient attachment of adhesive resins (Owens *et al.*, 1998). He also reported that beveling dentine (gingival margins) allows greater microleakage when current-generation adhesive systems are used.

Gwinnett *et al.* (1995) reported more severe microleakage along the end (embrasure) regions than along the center of the cervical margin when using Scotchbond 2 and P-50 as restorative material. Mixson, Eick, Chappell, Tira and Moore (1991), who made use of wedge-shaped cavity preparations, achieved similar results. Uno, Finger and Fritz (1997) concluded that cavity design had little influence on the microleakage of Dyract.

1.4.9. Co-efficient of thermal expansion

Phillips (1982) defined the linear coefficient of thermal expansion, as the change in length per unit length of the material when its temperature is raised or lowered by one degree. An ideal dental restorative material should have the same coefficient of thermal expansion as tooth structure seeing that it could facilitate microleakage (Sorensen, Dixit, White & Avera, 1991).

1.4.10. Masticatory stresses, thermocycling and dwell time

Yap (1998b) reported that thermocycling, load cycling and 3 months storage, with and without thermal load cycling, had no significant influence on microleakage at the enamel cement interface.

Microleakage at the dentine-cement interface was significantly greater for thermocycling, load cycling and long-term storage in artificial saliva. Doerr, Hilton and Hermes (1996) found that thermocycling had no effect on the microleakage of a conventional glass ionomer or that of resin modified glass ionomers.

Rossomando and Wendt Jr (1995) found that the need for thermocycling is dependent upon the extent a restorative is thermally conductive in relation to its mass. They further found that the dwell time should be clinically relevant, e.g. 10 seconds, and that it is only important if the restorative is thermally conductive. A wide range of dwell times have been proposed. Retief *et al.* (1988) reported an ideal dwell time of 15 seconds, while Wendt, McInnes and Dickenson (1992) justified an ideal dwell time in each bath of 15 seconds. Burger, Cooley and García-Godoy (1992) demonstrated that thermocycling time is not a significant variable.

1.4.11. *In vitro* vs *in vivo* microleakage studies

Microleakage studies have been carried out both *in vivo* and *in vitro*, but *in vitro* studies are more common (Taylor & Lynch, 1992). Several studies have concluded that higher values of microleakage occurred in *in vitro* restorations than in *in vivo* restorations (Barnes, Thompson, Blank & McDonald (1993).

Several studies (Jacobsen & Finger, 1993; Gwinnett & Kanka, 1992) have revealed similarity between *in vivo* and *in vitro* morphology concerning the relationship between resin and dentine. Ferrari *et al.* (1996a) evaluated the formation of the hybrid layer, resin tags and adhesive lateral branches *in vivo* and *in vitro*. All of the tested adhesive materials created a hybrid layer between conditioned dentine and the resin system, both under *in vivo* and *in vitro* conditions. They also found no essential differences in the morphology between the hybrid layers and the resin tag formation of the *in vitro* versus the *in vivo* samples, when the substrate conditions were kept wet. He concluded that *in vitro* tests could predict the results of *in vivo* clinical tests with respect to hybrid layer, resin tags and adhesive lateral branch formation.

1.4.12. Saliva Contamination

Until August 2001 no references could be found on the influence of saliva contamination on Prime & Bond NT specifically. However, several studies have been performed on the influence of saliva contamination on the different generations of Prime & Bond. Abdalla and Davidson (1998) as well as El Kalla and García-Godoy (1997) investigated the influence of saliva contamination on the shear bond strength of Prime & Bond 2.1 to enamel and dentine, if contamination took place just after etching and before adhesive application. Abdalla and Davidson (1998) found no significant influence. They gently air-dried the contaminated area before adhesive application. El Kalla and García-Godoy (1997) also found no significant influence whether the saliva was washed by water or just removed by blotting with cotton pellets.

El Kalla *et al.* (1999b) investigated the effect of saliva contamination on the resin tag formation of Prime & Bond 2.1. They found that hybrid layer formation and resin tag penetration was not influenced. The influence of saliva contamination on microleakage where Prime & Bond 2.1 had been used was included in a study by Evancusky and Meiers (2000). They found no significant effect on enamel, but found that microleakage was significantly increased on the dentine side of the restorations.

In a study performed by Fritz, Finger and Stean (1998), they investigated the effect of salivary contamination of enamel and dentine on the bonding efficacy of an experimental one-bottle resin adhesive. This experimental one-bottle resin adhesive (ARX) was a solution of UDMA, HEMA, and 4-META dissolved in acetone at equal weight parts that must be light-cured. The evaluation parameters were shear bond strength and marginal gap width. On the enamel side they found that ARX was relatively insensitive to salivary contamination, if the contamination occurred prior to light curing of

the adhesive and the contaminated area was carefully rinsed and blot dried. If the saliva was removed by an air blast the shear bond strength was significantly lower. On the dentine side, saliva-contaminated and air-dried dentine as well as the groups where the cured adhesive was contaminated with saliva, showed significantly lower mean bond strengths than the groups where the moist bonding technique was used and where saliva contamination was rinsed and blot dried.

Marginal gaps and dye penetration

Alterations of the materials due to all the factors mentioned could result in gaps at the tooth-restoration interface (Reeves, Fitchie, Hembree & Puchett, 1995). Microleakage occurs in these gaps between the restorative material and tooth structure and does not involve dentine permeability directly (Pashley & Carvalho, 1997).

Gwinnett and Kanka (1992) reported the absence of gap formation when a dentine adhesive system was used to create a hybrid layer between the resin and the dentine. The above mentioned adhesive systems made use of the total-etch technique (Bertolotti, 1990; Fusayama, 1992) together with the application of acetone-or alcohol-containing primer resins on a wet dentine substrate (Gwinnett, 1992; Gwinnett & Kanka, 1992). Several authors reported that a gap-free bond could be created with the use of an elastic intermediate layer of resin between the composite and adhesive resin. This may absorb the contraction stress of the composite during polymerization (Kemp-Scholte & Davidson, 1990b).

In 1995 Sano *et al.* (1995a) reported that penetration of silver nitrate along the dentinal wall was observed with or without gap formation in all specimens they tested. Sano *et al.* (1995b) reported similar results. Tay *et al.* (1995a) confirmed the earlier reports that gap-free restorations can be produced with the use of a total-etch technique

and acetone-containing dentine adhesives. They further reported that failure of the primer solvent to evaporate completely results in interfacial defects that are detectable under the SEM microscope.

Marginal gaps big enough to allow the leakage of bacteria would have to be in the region of 0.5-1.0 μm or larger (Dietrich *et al.*, 2000a).

Sano *et al.* (1995a) attributed the absence of gap formation in their '95 study in part to the elasticity of the adhesive layer, reduction of polymerization shrinkage of the composite, and the use of an intermediate low-viscosity resin. They further reported that the intermittent penetration of silver into the hybrid layer on cut dentine surfaces, indicated that the hybrid layer produced by the bonding systems used were not uniform. They presumed that regions of excellent adhesion between resin and collagen existed between areas where little bonding occurred. They therefore confirmed the presence of submicron spaces at the base of the hybrid layers that might allow water penetration.

Tay *et al.* (1995a) reported that the failure of the primer solvent to evaporate results in movement of fluid and bacteria through the interface into the tubules, and could later manifest as post-operative sensitivity and recurrent caries. Several authors reported that microleakage appeared to be initiated from the bonding resin-hybrid layer interface (Sano *et al.*, 1995a; Tay *et al.*, 1995a). Gwinnett *et al.*, (1995) reported that although leakage sites appear to be disconnected in individual sections, they are interconnected and originate from the cavosurface margins. Sano *et al.* (1995a) reported that leakage occurred within the acid-resistant hybrid layer when using All-Bond adhesive. Tay *et al.* (1995a) reported that under different handling conditions the site of microleakage initiation originated from the interface between the bonding resin and hybrid layer.

Pashley and Matthews (1993) reported that dentinal penetration depends on the concentration of the tracer and its diffusion coefficient, the thickness of the dentine and the surface area of the dentine available for diffusion. Christen and Mitchell (1966) showed that different concentrations of two dyes can vary between 5 minutes and 1 hour in penetration time. Fusayama and Terachima (1972) as well as Kidd, Joyston-Bechal, Smith, Allan, Howe and Smith (1989) reported that basic fuchsin solution, one of the most frequently used dyes in penetration studies, has a tendency to preferentially bind with carious dentine.

Tracers used to assess microleakage e.g. silver ions, are much smaller in magnitude. Dye penetration is therefore a severe test of microleakage (Douglas, 1989). Taylor and Lynch (1992) reported that marginal gaps allowing leakage of bacteria are expected to be in the region of 0.5-1.0 μm or larger. Microleakage studies making use of bacterial penetration therefore do not take gaps smaller than the above mentioned into account. Although bacterial penetration does not take place, these gaps allow toxins and other bacterial products, which could harm the tooth, to penetrate. They further reported that it is impractical to use dye particles with a diameter greater than that of the internal diameter of dentinal tubules (1-4 μm), seeing that it would not be able to penetrate.

Nanoleakage

Sano *et al.* (1994) reported that silver had penetrated into submicron-sized spaces beneath the bonded dentine surface. This penetration reveals the lack of a perfect seal, but it is not microleakage in the classical sense. To distinguish this form of leakage from typical microleakage, he suggested in 1995 that it should be referred to as nanoleakage (Sano *et al.*, 1995a; Sano *et al.*, 1995b).

If the etched dentine is dried too much, the collagen fibers may collapse, obstructing resin penetration and therefore hybrid layer formation. A space can therefore remain between undermineralized dentine and resin (Ferrari *et al.*, 1997b). The spaces that Sano *et al.* (1995a) referred to at the base of the hybrid layer are of the order of 20-100 nm in width. They therefore called the leakage that occurs within the hybrid layer, in the absence of gap formation, nanoleakage.

Sano *et al.* (1995b) demonstrated the nanoleakage of the hybrid layer using a silver nitrate staining technique and demonstrated silver accumulation within the hybrid layers. They speculated that leakage occurred in areas of demineralized dentine that was not completely filled by resin. This pathway may allow the penetration of bacterial products and oral fluids along the interface, with subsequent breakdown of the unprotected collagen (Li *et al.*, 1999).

It appears that bonding systems that etch deeper in dentine are more likely to show higher degrees of nanoleakage (Sano *et al.*, 1995a). Li *et al.* (1999) reported silver penetration throughout the entire thickness of the hybrid layer formed by the use of Prime and Bond NT after the application of NRC. Nanoleakage in the thermocycled species were essentially the same as in the non-thermocycled specimens. Nanoleakage identifies the exact location of the microleakage, e.g. within, above or below the hybrid layer; between the hybrid layer and the bonding agent; between the bonding agent and the resin composite.

CHAPTER 2

OBJECTIVES

The literature review emphasised the lack of studies specifically concentrating on the microleakage of compomers, as well as the effect of saliva contamination on the microleakage. Most studies on microleakage were done either *in vitro*, without saliva contamination or performed clinically with the use of rubber dam. The instructions for the use of Dyract AP and its bonding systems does not specifically advise the use of rubber dam.

The objective of this experiment was therefore, firstly, to determine and compare the qualitative microleakage of class V preparations restored with the above-mentioned compomer, bonded by the different systems, and secondly, to determine and compare the influence of saliva contamination on the microleakage when the different systems were used.

CHAPTER 3

MATERIALS AND METHODS

3.1 TEETH USED IN PROJECT.

One hundred and forty-four freshly extracted, non-carious, human premolars were used in this study. The teeth had all been extracted for orthodontic or periodontal reasons at the department of Maxillo-facial and Oral Surgery of the University of Stellenbosch. After extraction the teeth were immediately placed in physiological saline (distilled water + 9% NaCl) containing 1% thymol and stored at 4°C. The teeth were firstly scraped clean of gross debris with a no 15 blade^a, after which each tooth was polished as a whole with flour of pumice with a polishing wheel^b at low speed. After being rinsed under tap water the teeth were once again placed in physiological saline containing thymol and stored at 4°C. From the time of extraction to the finish of each batch of experiments no teeth were stored for a period longer than two months.

3.2. INITIAL TREATMENT OF THE TEETH.

All teeth were treated in the following manner:

- The teeth were randomly divided into eight groups of 18 teeth each.
- The apical 2 mm of each root was removed with a model trimmer^c using water as a coolant. A cavity was prepared at the cut root apices with an inverted cone carbide bur (ISO 006)^d with a slowspeed hand-piece^e. Each cavity was rinsed with water, dried with air and two layers of Polyvar^f cavity liner was applied. The cavities were then filled with Permite C^g amalgam; the amalgam was condensed with a small round

amalgam plugger and the restoration was finished with a ball burnisher.

- The teeth were then placed back in the physiological saline containing thymol and stored at 4°C.

3.3 PREPARATION AND RESTORATION OF THE CAVITIES AS DETERMINED BY EACH EXPERIMENTAL GROUP.

3.3.1 Preparation of the cavities

Cavities were prepared in the following manner in all eight groups of teeth just before the restorations were placed:

A class V cylindrical cavity was prepared on the buccal side of each tooth on the cemento-enamel junction. Each cavity was 1,5 mm deep and 3 mm (Figure 1a) in diameter and was prepared partly in enamel and partly in dentine. A cylindrical round diamond bur (ISO 012)^d was used in a highspeed Kavo hand-piece^h with water cooling. A new bur was used for each group of experiments. After preparation the cavities were washed with water and dried with a single blast of filtered air from a triple syringe held 2 cm away from the cavity. Oil free compressed air was obtained by filtering the air through a Wilkerson filter attached to a laboratory air lineⁱ.

3.3.2 Saliva collection

Fresh saliva samples were collected prior to every group of experiments. Saliva was obtained before breakfast from 4 donors and mixed to minimize individual characteristics. The saliva was kept at room temperature in a sterile container and used in the experimental groups within 4 hours of collection.

3.3.3 The experimental groups.

In order to compare the different bonding systems and determine the influence of saliva contamination on microleakage at different application stages, the eight experimental groups were as follows:

Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT ^j + LC + Dyract AP ^j + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner ^j (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

3.3.4 Restoration of the cavities

Restoration of the cavities is summarized in Table 1. The application techniques of the materials and saliva contamination were as follows:

Application of De Trey Conditioner 36^j gel (36% phosphoric acid)

After each cavity was dried in groups 2, 5 and 6, De Trey Conditioner 36 gel (Lot no. 9607146, Expiry date 2001-07) was applied starting at the enamel margins after which it was spread to the rest of the cavity. The enamel was conditioned for 15 seconds while the dentine was

conditioned for 10 seconds. The gel was removed by rinsing the cavity with water for 15 seconds. The cavity was once again dried with a single burst of air from a triple syringe held 2 cm away from the cavity.

Application of Non-Rinse Conditioner

NRC (Figure 2a), (Lot no. 9806000156, Expiry date 2001-04) was dispensed into a dappen dish and applied with an applicator tip^h in sufficient amounts to the enamel and dentine of groups 3, 7 and 8. It was left undisturbed for 20 seconds after which the excess NRC was blown away with a single burst of air from a triple syringe held 2 cm away from the cavity.

Application of Prime & Bond NT and curing

One layer of Prime & Bond NT (Figure 2b), (Lot no. 9806000602, Expiry date 2000-05) was applied in the following manner. The Prime & Bond NT was dispensed into a dappen dish. The dappen dish was kept covered to prevent evaporation of the acetone and a fresh drop was dispensed for every third cavity. An applicator tip was used to flood the entire cavity with Prime & Bond NT. It was left undisturbed in the cavity for 30 seconds after which the excess was blown away with air from a triple syringe for 5 seconds. The Prime & Bond NT was then light-cured for 20 seconds. The curing-light was held 5 mm away from the cavity. A new bulb was placed in the curing-light at the start of each group of experiments. The output of the curing-light was also measured before each group of experiments with a Cure Rite Visible Curing Light Meter^k to ensure exposure of 350 ± 50 mW/cm². An Optilux 150 curing-light was used for all light-curing required^l.

Application of Dyract AP

Dyract AP (Figure 3), (Lot no. 9806001203, Expiry date 2000-05) was placed immediately after the application and curing of the Prime & Bond

NT. The Dyract AP was dispensed directly into the prepared cavity. All cavities were filled in three increments. The first two increments were placed obliquely against the occlusal and gingival walls of the cavity and the third increment was used to slightly overfill the cavity. Each increment was condensed in the cavity with a teflon flat plastic instrument. Each increment was cured individually. The first two increments were light-cured for 40 seconds each while the third increment was cured for 50 seconds. The curing-light was held 5 mm away from the filling. The entire cavity was exposed to the light.

Saliva contamination

The saliva was dispensed into the cavity with an applicator tip. The cavity was flooded with saliva.

In groups 4, 6 and 8 the cavities were contaminated immediately after the Prime & Bond NT had been light cured. A single burst of air from a triple syringe was used to remove the excess saliva from the cavities.

In group 5 each cavity was contaminated with saliva after the acid etch had been washed out and the cavity had been dried. In group 7 the contamination was done after the excess NRC had been blown away. The excess saliva was blown away in both groups 5 and 7 with a single burst of air.

3.3.5 Finishing of restorations

The teeth were immersed in distilled water after each restoration was placed. When all 18 cavities in each group had been restored, the final finishing of the restorations (Figure 1b) was done with a series of Sof-Lex discs^m, to ensure that all overhangs had been removed. On completion, the teeth were once again stored in distilled water at room temperature for 24 hours.



Figure 1.
(a) Tooth with prepared cavity and (b) tooth with filling

Figure 2.
A bottle of (a) NRC and (b) Prime & Bond NT

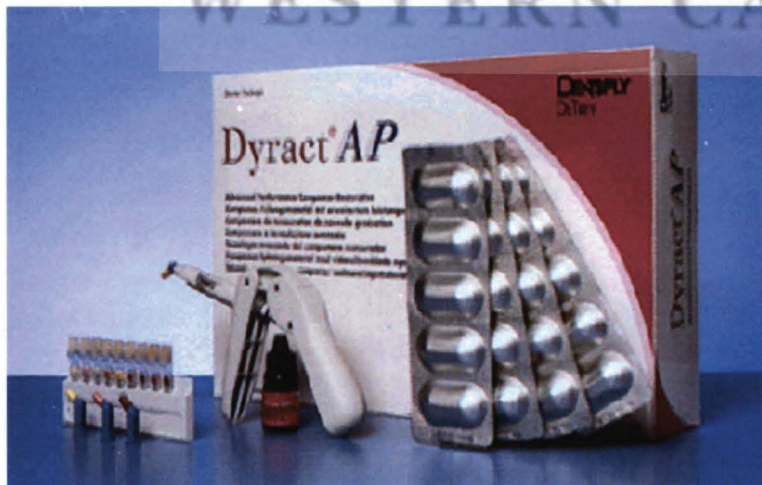


Figure 3.
Dyract AP composites, applicator gun and shade guide

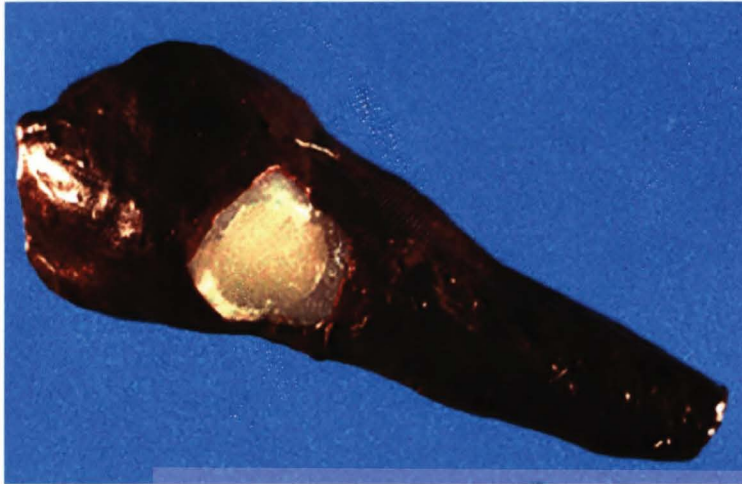
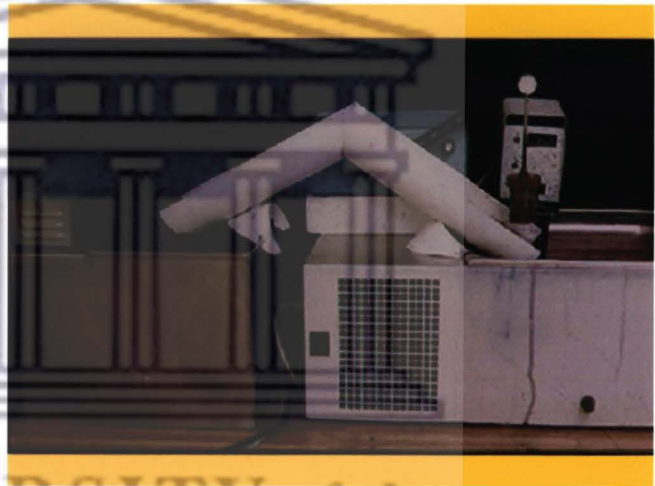


Figure 4.
Filled tooth covered in nail varnish except for filling and 1mm surrounding filling.

Figure 5.
Cycling machine used to thermocycle filled teeth in basic fuchsin solution.



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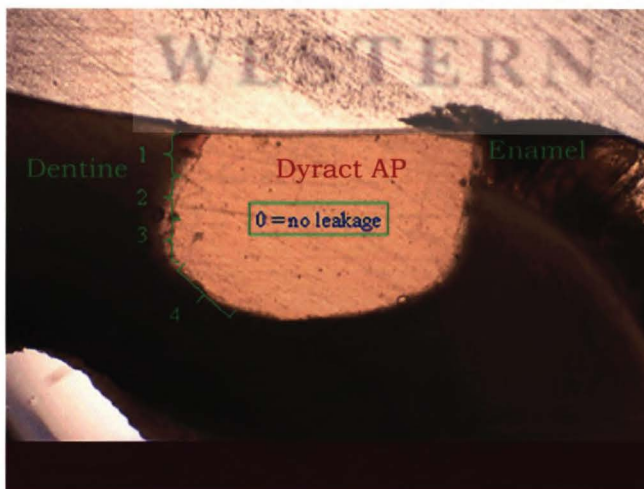


Figure 6.
Criteria used for microleakage evaluation (micro-leakage = 1 in example)

3.4 MICROLEAKAGE TESTS

After 24 hours the restored teeth were removed from storage and coated with two layers of nail varnishⁿ. The tooth surfaces were all covered except for 1mm around the restorations (Figure 4), which were left free of varnish. The groups of teeth were then thermocycled (Figure 5), in a 0.5% basic fuchsin^o (Lot 7350420P; Expiry date 2003-04) solution for 500 complete cycles, in a temperature cycling machine between 8°C and 50°C and with a dwell time of 15 seconds.

The teeth were removed from the thermocycling machine; the gross nail varnish was scraped off with a no 15 blade and the teeth were polished with flour of pumice and rinsed with tap water to remove all the remaining nail polish and pumice. The teeth were embedded in casting resin^p and allowed to cure overnight. Vertical sections were cut in a bucco-lingual plane through the restorations using a water-cooled Minitom circular saw^q. On average four sections of 600 µm thickness were cut of each restoration. The maximum amount of sections was cut of each restoration. The cut sections were gently rubbed on grit P600^r waterproof sandpaper to smooth them.

The extent of microleakage was scored separately on both the enamel and dentine/cementum margins on both sides of each section, according to the criteria listed below (Figure 6). Scoring was done immediately after sectioning. The sections were placed on a glass slide and examined under a Wild light microscope^s at X60 magnification to evaluate microleakage (Figure 7).

Criteria:

- 0 = no leakage
- 1 = dye penetration up to one-third cavity depth
- 2 = dye penetration to two-thirds cavity depth (Figure 8)
- 3 = dye penetration to full depth of cavity
- 4 = dye penetration extending along the floor of the cavity (Figure 9)

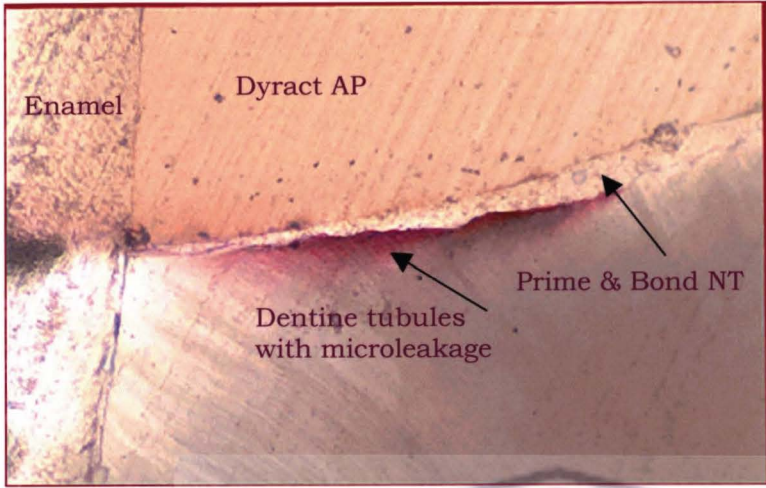


Figure 7.
X60
Enlargement of
micro-leakage
in dentine
tubules.

Figure 8.
Microleakage value
of 2 extending more
than one third of
the cavity depth.

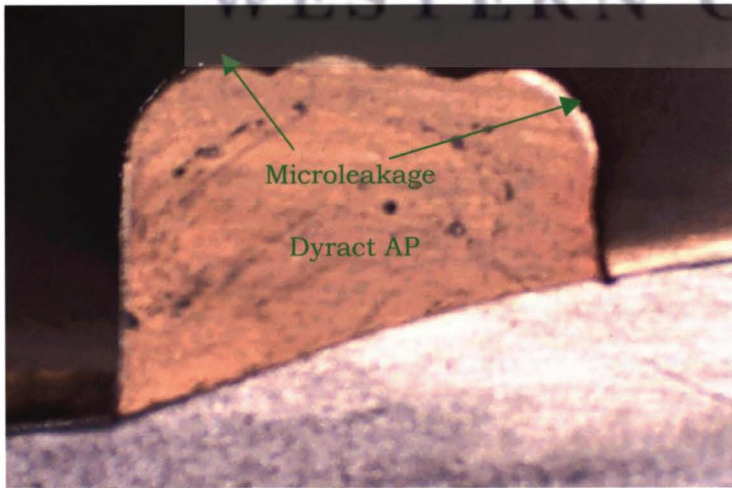


Figure 9.
Microleakage value of 4.

For the sake of consistency, scoring procedures were immediately performed on all sections cut, by two independent investigators who previously had synchronized their technique. The same two investigators were used for all eight groups of experiments. The percentage of each score in each group was calculated by the investigators.

The original scores for each section of both investigators were supplied to the statistician. The data was then statistically analyzed using Kruskal-Wallis one way ANOVA on ranks (significance at $p < 0.05$).



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3.5 LIST OF MANUFACTURERS

- a. Swann Morton LTD. Sheffield, England
- b. Denta Olymp. Herm. Metzдорff, Dortmund, Germany-Allemagne
- c. C.H. Wilhelm Wassermann, Feinwerk, Hamburg, Germany
- d. Komet, GEBR. BRASSELER GmbH & Co. KG, Lemgo, Germany
- e. Microo-Mega, Besancon, France
- f. Lorvic Corporation, St. Louis, USA Southern Dental Industries, Australia
- g. Oral-B Laboratories, Sandton, South Africa
- h. Kavo, Kaltenbrecht & Voigt, Germany
- i. Johnson Controls, Inc., Milwaukee, USA
- j. Dentsply DeTrey, Konstanz, Germany
- k. Dentsply, Caulk, Milford, USA
- l. Demetron/Kerr, Danbury, USA
- m. 3M Dental Products, St. Paul, France
- n. Xpress Finish, Maybelline, New York, USA
- o. BDH Laboratory Supplies, Poole, England
- p. Fobrogas, Foukes Bros. (PTY) LTD, Cape Town, South Africa
- q. Minitom, Struers, Copenhagen, Denmark
- r. 3M, Johannesburg, South Africa
- s. WILD HEERBRUGG, Switzerland

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CHAPTER 4

RESULTS

DATA

The percentage of each score in each group was calculated and is represented in Table 2 for enamel and in Table 3 for dentine.

Table 2. Percentages of microleakage for every group: EMAMEL

Criteria Depth of dye penetra- tion in cavity 0=none 1=one- third 2=two- thirds 3=full depth 4=along floor	Group 1 Prime & Bond NT + Dyract AP	Group 2 Phosp horic acid + Prime & Bond NT + Dyract AP	Group 3 Non Rinse Condi- oner (NRC) + Prime & Bond NT + Dyract AP	Group 4 Prime & Bond NT + Saliva + Dyract AP	Group 5 Phosp horic acid + Saliva + Prime & Bond NT + Dyract AP	Group 6 Phosp horic acid + Prime & Bond NT + Saliva + Dyract AP	Group 7 Non Rinse Condi- oner (NRC) + Saliva + Prime & Bond NT + Dyract AP	Group 8 Non Rinse Condi- oner (NRC) + Prime & Bond NT + Saliva + Dyract AP
	%	%	%	%	%	%	%	%
0	6	16	39	0	14	28	2	20
1	48	68	42	28	50	48	21	39
2	34	16	3	33	25	8	43	18
3	3	0	8	33	11	8	29	18
4	9	0	8	6	0	8	5	5

Table 3. Percentages of microleakage for every group: DENTINE

Criteria Depth of dye penetration in cavity 0=none 1=one-third 2=two-thirds 3=full depth 4=along floor	Group 1 Prime & Bond NT + Dyract AP	Group 2 Phosphoric acid + Prime & Bond NT + Dyract AP	Group 3 Non Rinse Conditioner (NRC) + Prime & Bond NT + Dyract AP	Group 4 Prime & Bond NT + Saliva + Dyract AP	Group 5 Phosphoric acid + Saliva + Prime & Bond NT + Dyract AP	Group 6 Phosphoric acid + Prime & Bond NT + Saliva + Dyract AP	Group 7 Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + Dyract AP	Group 8 Non Rinse Conditioner (NRC) + Prime & Bond NT + Saliva + Dyract AP
	%	%	%	%	%	%	%	%
0	37	24	29	27	22	0	15	5
1	23	26	3	28	28	3	37	0
2	10	3	11	17	11	11	12	5
3	10	8	8	0	3	3	12	5
4	20	39	49	28	36	83	24	85

The percentages were also graphically illustrated with pie charts (Figures 10 to 25).

Percentages of microleakage for group 1:

Figure 10: Enamel

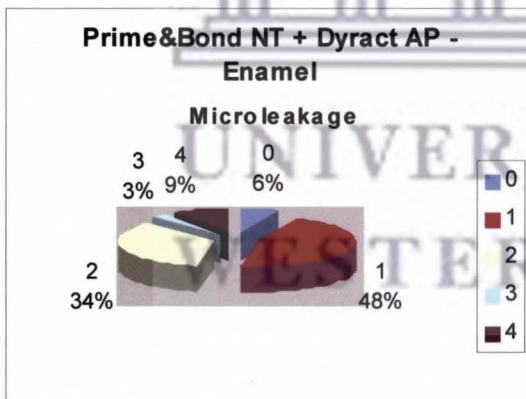
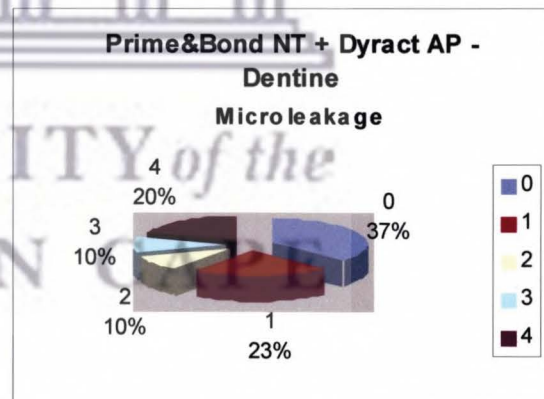


Figure 11: Dentine



Percentages of microleakage for group 2:

Figure 12: Enamel

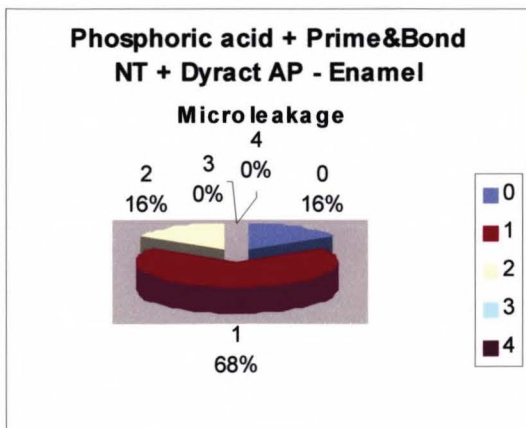
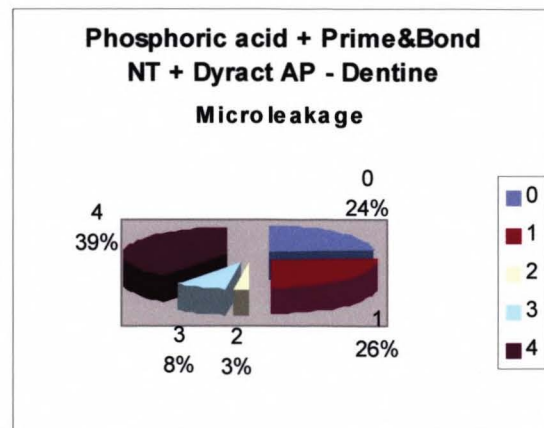


Figure 13: Dentine



Percentages of microleakage for group 3:

Figure 14: Enamel

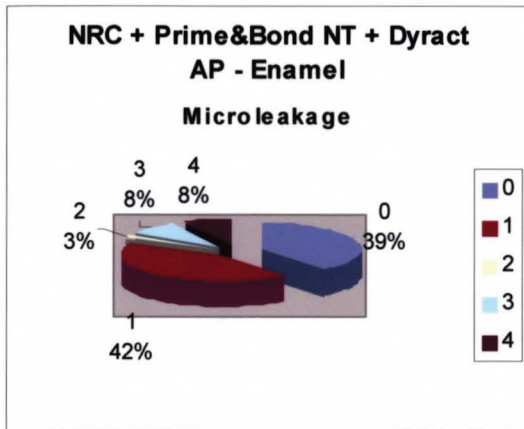
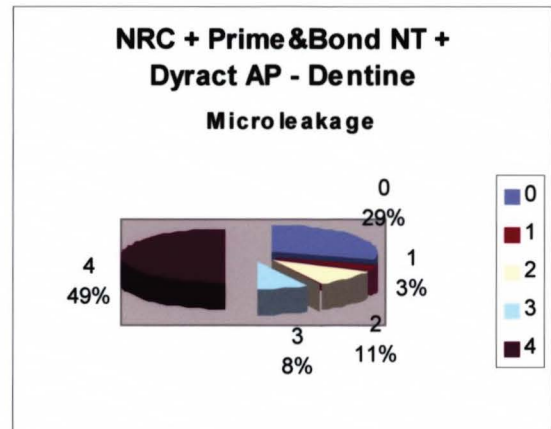


Figure 15: Dentine



Percentages of microleakage for group 4:

Figure 16: Enamel

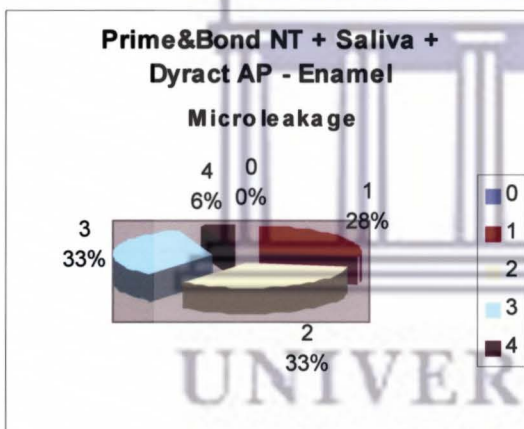
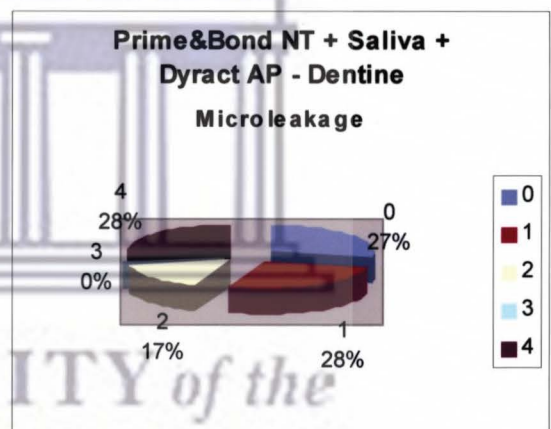


Figure 17: Dentine



Percentages of microleakage for group 5:

Figure 18: Enamel

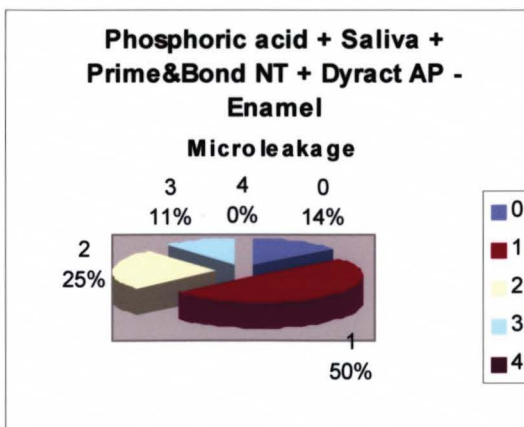
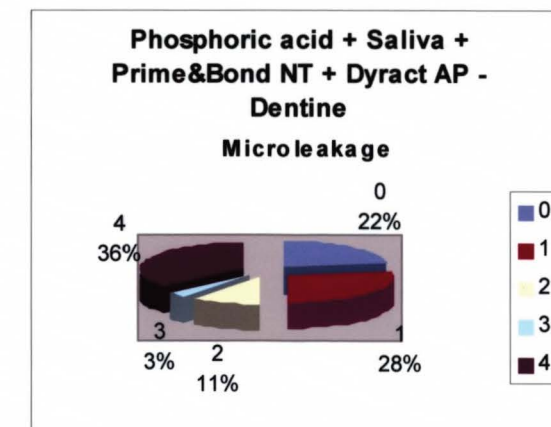


Figure 19: Dentine



Percentages of microleakage for group 6:

Figure 20: Enamel

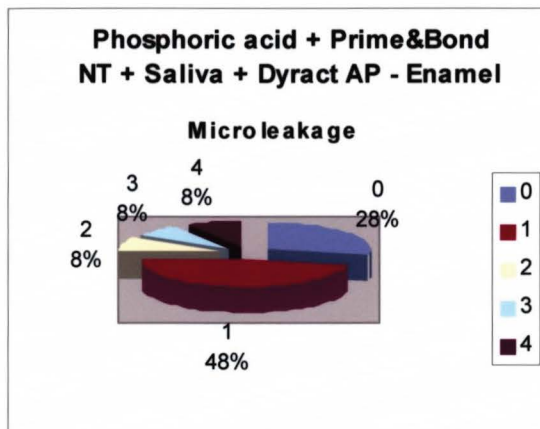
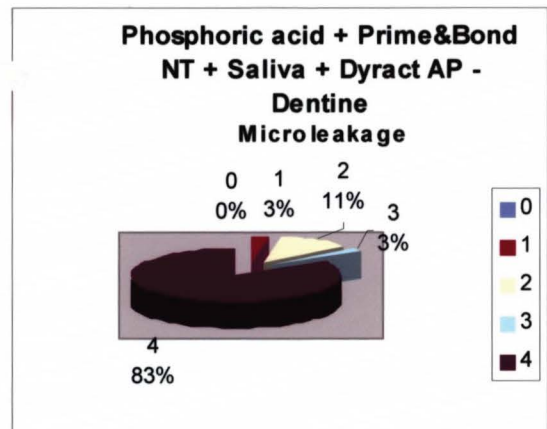


Figure 21: Dentine



Percentages of microleakage for group 7:

Figure 22: Enamel

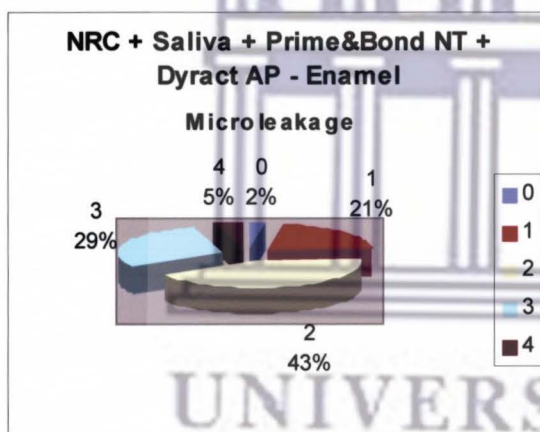
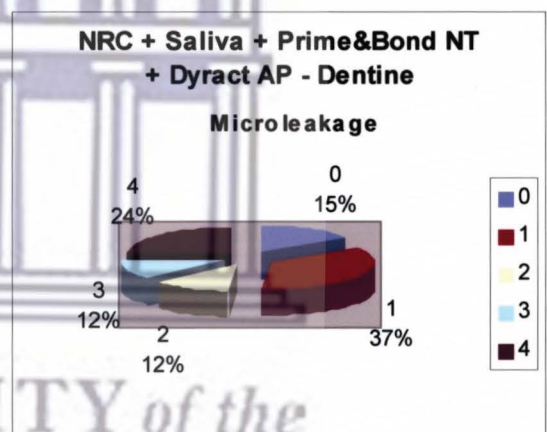


Figure 23: Dentine



Percentages of microleakage for group 8:

Figure 24: Enamel

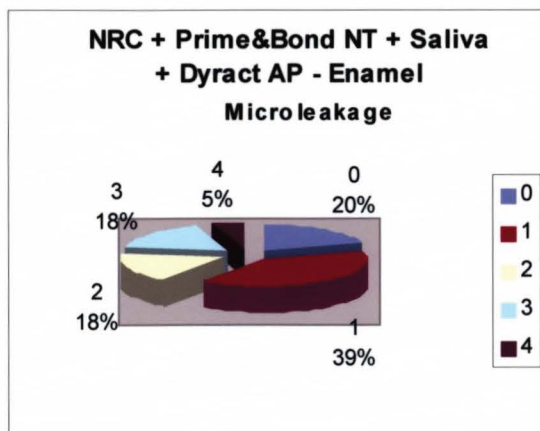
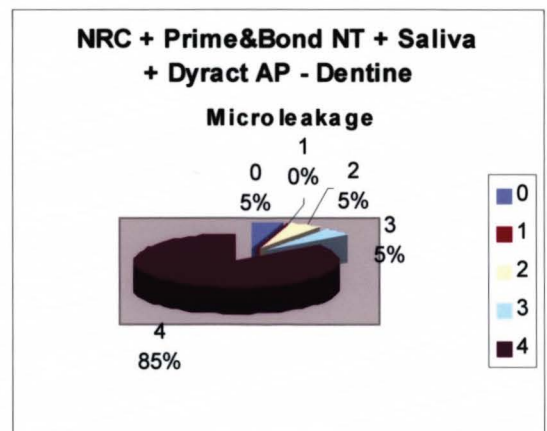


Figure 25: Dentine



STATISTICS

The data was statistically analyzed using Kruskal-Wallis one way ANOVA on ranks (significance at $p < 0.05$). A summary of the means, number of sections scored, the standard deviation, the median as well as the values at the 25% and 75% levels for enamel microleakage is represented in Table 4.

Table 4. Summary of means, number of sections scored, standard deviation, median, 25% and 75% levels for microleakage on the enamel side.

ENAMEL						
Smallest N for any variable: 505						
	ENS	ENS	ENS	ENS	ENS	ENS
	Means	N	Std.Dev.	Q25	Median	Q75
G_1:1	1.276786	56	0.738054	1	1	1.5
G_2:2	0.737705	61	0.616486	0	1	1
G_3:3	0.766129	62	1.144186	0	0	1
G_4:4	1.672414	58	0.769779	1	1.5	2
G_5:5	0.921875	64	0.751817	0	1	1.5
G_6:6	0.753623	69	0.905957	0	0.5	1
G_7:7	1.383333	60	1.018335	1	1	2
G_8:8	0.896104	77	1.02706	0	1	1
All Grps	1.033531	507	0.944016	0	1	1.5

Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT ^j + LC + Dyract AP ^j + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner ^j (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

Table 5 presents the statistically significant differences at a 95% confidence level for microleakage on the enamel side. It was found that Groups 1 and 4 differed significantly from all the groups except group 7. They also differed significantly from each other. Group 7 differed significantly from all the groups except groups 1 and 4. Groups 2,3,5,6 and 8 only differed significantly from groups 1,4 and 7.

Table 5. Statistically significant differences ($p < 0.05$) - Enamel side. Shaded areas indicate areas of significance.

LSD Test; Variable: ENS								
	{1}	{2}	{3}	{4}	{5}	{6}	{7}	{8}
	M=1.2768	M=.73770	M=.76613	M=1.6724	M=.92188	M=.75362	M=1.3833	M=.89610
G_1:1 {1}		0.001203	0.002065	0.018605	0.030583	0.001223	0.521724	0.015726
G_2:2 {2}			0.860189	2.07E-08	0.250377	0.919376	8.24E-05	0.302
G_3:3 {3}				4.72E-08	0.328938	0.936346	0.000156	0.394814
G_4:4 {4}					4.7E-06	1.42E-08	0.079831	8.26E-07
G_5:5 {5}						0.278899	0.004263	0.864808
G_6:6 {6}							7.64E-05	0.337021
G_7:7 {7}								0.001654
G_8:8 {8}								

Table 6 presents the means, number of sections scored, the standard deviation, the median as well as the values at the 25% and 75% levels for microleakage on the dentine/cementum side.

It was found that Groups 6 and 8 differed significantly from groups 1,2,3,4,5 and 7. Group 1 differed significantly from groups 2,3,5,6,7,8; Group 2 from 1,4,6,8; group 3 from 1,4,6,7,8; group 4 from 2,3,6,8; group 5 from 1,6,8; group 7 from 1,3,6 and 8.

Table 6. Summary of means, number of sections scored, standard deviation, median, 25% and 75% levels for microleakage on the dentine/cementum side

DENTINE/CEMENTUM						
Smallest N for any variable: 505						
	DES	DES	DES	DES	DES	DES
	Means	N	Std.Dev.	Q25	Median	Q75
G_1:1	1.018868	53	1.414085	0	0	2
G_2:2	1.862903	62	1.68204	0	1	4
G_3:3	2.104839	62	1.746512	0	2.5	4
G_4:4	1.192982	57	1.469191	0	1	2
G_5:5	1.6875	64	1.684429	0	1	4
G_6:6	3.376812	69	1.21995	4	4	4
G_7:7	1.575	60	1.564503	0	1	3.25
G_8:8	3.596154	78	1.050843	4	4	4
All Grps	2.146535	505	1.735859	0	2.5	4

Table 7 again presents the statistically significant differences between the groups ($p < 0.05$).

Table 7. Statistically significant differences ($p < 0.05$) - Dentine/Cementum side. Shaded areas indicate areas of significance.

LSD Test; Variable: DES								
	{1}	{2}	{3}	{4}	{5}	{6}	{7}	{8}
	M=1.0189	M=1.8629	M=2.1048	M=1.1930	M=1.6875	M=3.3768	M=1.5750	M=3.5962
G_1:1 {1}		0.00247	0.000103	0.538625	0.015546	4.68E-17	0.047196	1.02E-20
G_2:2 {2}			0.364118	0.014159	0.507138	9.76E-09	0.284207	1.93E-11
G_3:3 {3}				0.000867	0.114894	1.29E-06	0.049052	6.34E-09
G_4:4 {4}					0.06769	1.68E-15	0.164302	4.45E-19
G_5:5 {5}						1.32E-10	0.673079	1.19E-13
G_6:6 {6}							1.77E-11	0.371221
G_7:7 {7}								1.38E-14
G_8:8 {8}								

A summary of the means of both enamel and dentine/cementum microleakage is graphically represented in Figure 26. The summary of the experimental groups is given in table 1.

Figure 26. Summary of the means of both enamel and dentine/cementum microleakage.

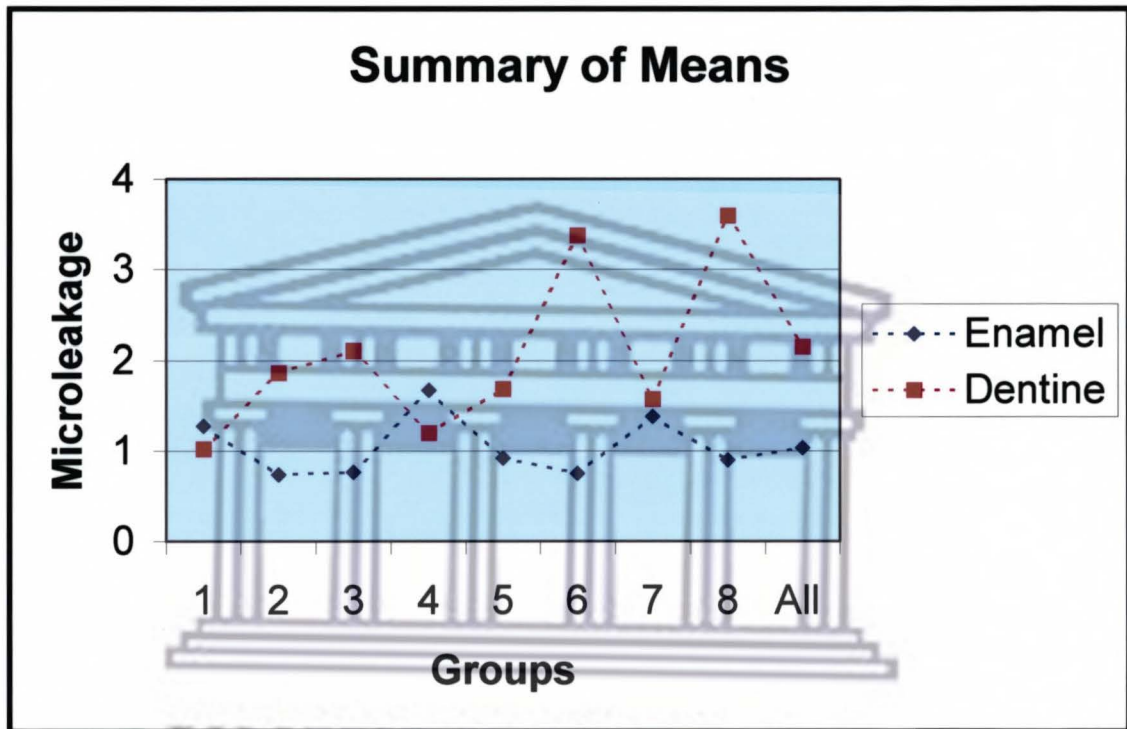


Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT ⁱ + LC + Dyract AP ⁱ + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner ^j (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

BOX PLOT SECTION

Box plot diagrams were also compiled which graphically show the average microleakage of the different experimental groups in enamel as well as in dentine/cementum. The line dividing each rectangle indicates the median for each experimental group, while the top and the bottom of each rectangle indicates the top 75% and bottom 25% of the values respectively while the blue lines indicate the 10% and 90% values respectively. Abnormally high or low readings are indicated by single blue dots.

Figure 27. Box Plot diagram showing the average microleakage of Groups 1 to 3 in Enamel

Kruskal-Wallis One-Way Anova on Ranks

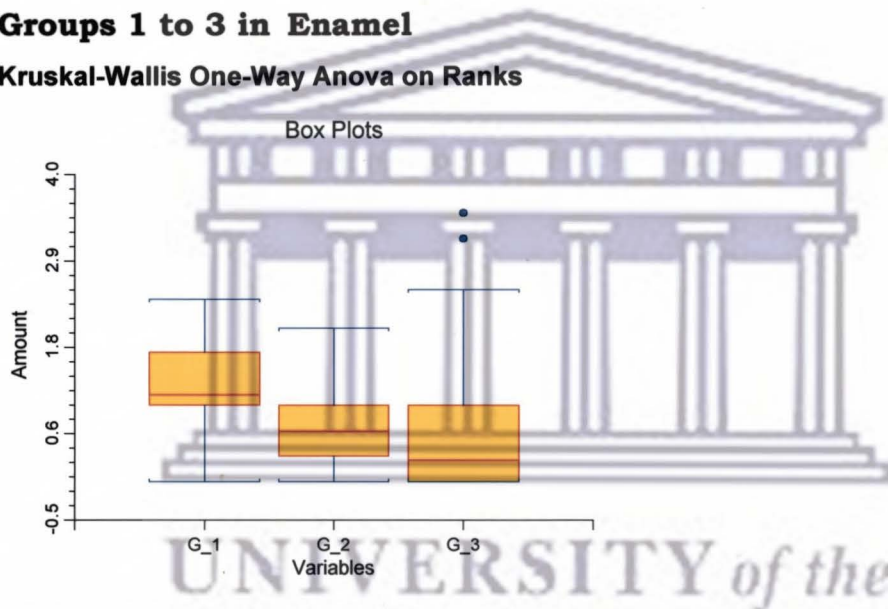


Figure 28. Box Plot diagram showing the average microleakage of Groups 4 to 8 in Enamel

Kruskal-Wallis One-Way Anova on Ranks

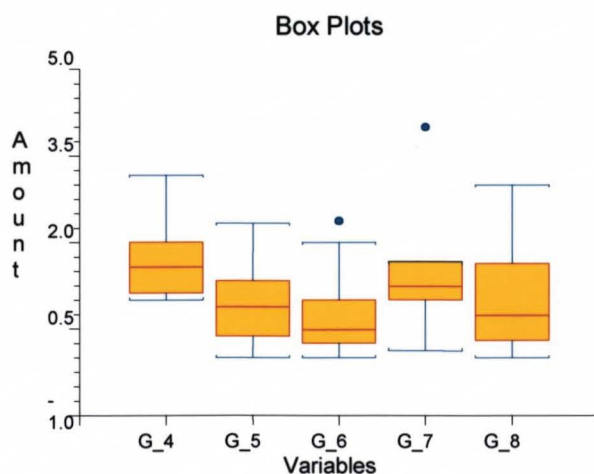


Figure 29. Box Plot diagram showing the average microleakage of Groups 1 to 3 in Dentine/Cementum
Kruskal-Wallis One-Way Anova on Ranks

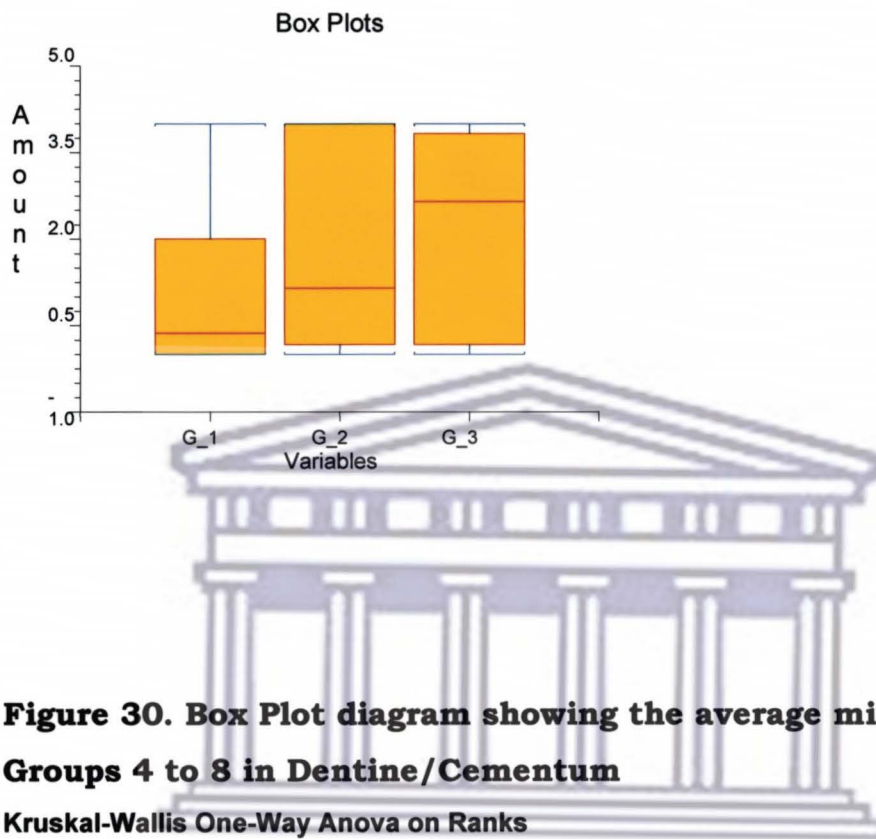
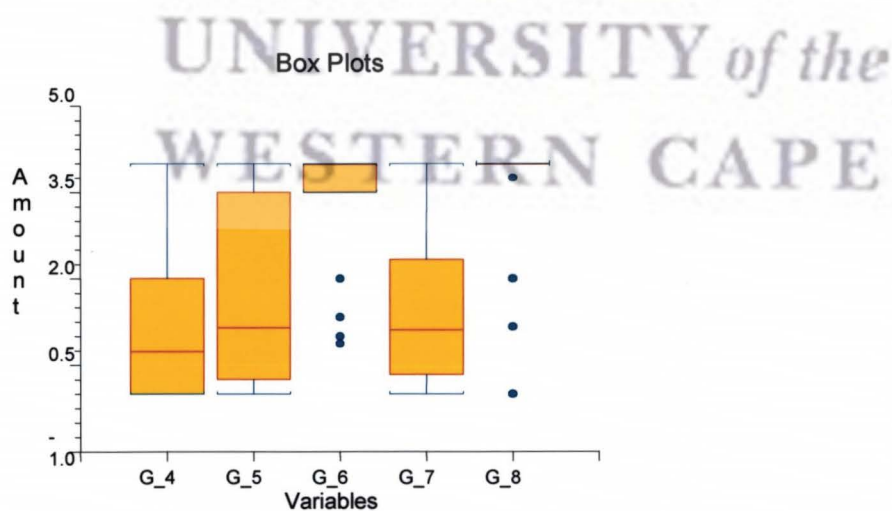


Figure 30. Box Plot diagram showing the average microleakage of Groups 4 to 8 in Dentine/Cementum
Kruskal-Wallis One-Way Anova on Ranks



CHAPTER 5

DISCUSSION

Contamination by blood and/or saliva is a major clinical problem during restorative dental treatment. Restorative materials must possess sufficient marginal sealing ability to avoid microleakage. The only way to ensure that saliva contamination does not take place is by using a rubber dam. However, virtually none of the manufacturers of compomers advocate the use of a rubber dam in their instructions. Marshall and Page (1990) found that very few private practitioners make use of a rubber dam at all. This study was stimulated by the idea of what effect contamination could have on the sealing ability of compomer restorations and therefore on the microleakage and longevity of them.

In order to simulate the clinical situation as close as possible, the study was designed in a specific manner. To ensure the worst case scenario, it was decided upon an *in vitro* study, due to the tendency of *in vitro* studies to result in higher microleakage values (Barnes *et al.*, 1993) than *in vivo* studies. There were no differences found in the morphology of the hybrid layers and resin tag formation of *in vitro* versus *in vivo* samples in a study by Ferrari *et al.* (1996a). Dye penetration studies such as those with basic fuchsin are amongst the most popular techniques used to study *in vitro* microleakage (Yap *et al.*, 1998a).

A cylindrical diamond bur was used to prepare the cavities seeing that diamond burs are mainly used for cavity preparation in modern day dental surgeries. Uno *et al.* (1997) concluded that cavity design

had little influence on the microleakage of Dyract. Dietrich *et al.*, (2000a) found no differences between beveled and butt-joint cavity preparations with respect to perfect margins when enamel was conditioned with 36% phosphoric acid. In this experiment the cavity margins were therefore not beveled.

Several authors (Kanca, 1992a; Kanca, 1992b; Gwinnett, 1992) all demonstrated that bonding to moist dentine delivered higher bond strengths than bonding to dry dentine. Tay *et al.* (1996a) reported that large amounts of water on the dentine surface could result in poor bonding. A critical amount of water is therefore necessary on the dentine surface to prevent water from evaporating and the resultant collapse of the collagen fibrils. At the time that these experiments were performed, the Dentsply Company did advocate the moist bonding technique for Dyract AP. However, it was not clearly indicated how to practically achieve the instruction of “gently air dry”. Cavities were therefore dried with a single blast of air instead of the traditional 15 second drying time.

The fillings in this experiment were placed in three increments and each was individually light-cured to minimize polymerization shrinkage (Linden & Swift, 1994). The reason being that amongst other things, light intensity and the duration of light exposure influences volumetric change (Miyazaki *et al.*, 1999b). Uno & Asmussen (1991) recommended using low light intensity to allow a reduced polymerization rate and relaxation of the contraction stress. Attin *et al.* (1995) reported that the resin content in compomers, which varies among available products, contributes to polymerization shrinkage, and this could adversely affect marginal adaptation. To keep microleakage to a minimum an ideal dental restorative material should have the same co-efficient of thermal expansion as tooth structure. The difference in the co-efficient of thermal expansion of tooth structure and restorative material, as well as polymerization

shrinkage, produces stress within the adhesive layer (Hansen & Asmussen, 1985; Kemp-Scholte *et al.* 1990a). This introduces stress at the tooth/restorative interface that could result in loss of marginal integrity (Carvalho *et al.*, 1996b; Katoma *et al.*, 1994).

Expansion of a restoration due to water absorption partly counteracts the polymerization shrinkage (Fan *et al.*, 1985). However, it does not necessarily compensate for it (Hansen & Asmussen, 1985) seeing that the water uptake and expansion will not re-establish the bonds which failed due to polymerization shrinkage (Rigsby *et al.*, 1990). Teeth were therefore only stored for 24 hours before thermocycling.

Rossomando and Wendt (1995) reported that thermocycling is dependent upon the extent a restorative is thermally conductive in relation to its mass, while a dwell time of 15 seconds was justified by Retief *et al.* (1988). Yap (1998b) reported a significant difference in microleakage on the dentine-cementum interface after thermocycling. Therefore, the filled teeth were thermocycled for 500 complete cycles with a dwell time of 15 seconds.

In this experiment it was hypothesised that results on the enamel side, from the worst microleakage to the least microleakage could be in the following sequence: Group: 4; 1; 7; 5; 8; 3; 6; 2 (detail of groups in table 1) based on the reasons discussed following Table 1.

Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT [†] + LC + Dyract AP [‡] + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner [†] (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

Ferrari *et al.* (1999) demonstrated with scanning electron microscope reports that when enamel was etched with 36% phosphoric acid, a deeper etch pattern was formed than when etching was performed with NRC. Resin tags were produced in both cases with the application of Prime & Bond NT. When no form of etching was applied to enamel, no resin tags were formed with the application of Prime & Bond NT and the characteristic etched pattern of enamel was not observed. It would be expected that a deeper etch pattern on the enamel side would result in better resin penetration and in turn in less microleakage. Saliva proteins could clog an already etched enamel surface and decrease adhesive penetration and in turn result in more microleakage.

However the analysis of the present study indicated microleakage from the worst leakage to the least leakage on the enamel side in the following sequence:

Group: 4; 7; 1; 5; 8; 3; 6; 2 (detail in table 1)

Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT + LC + Dyract AP + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

The only unexpected result was therefore that group 7 resulted in more microleakage than group 1. However, statistically their results did not differ significantly.

Other studies involving microleakage and Prime & Bond NT have shown that it is possible to eliminate microleakage on the enamel side (Tung *et al.*, 2000a; Tung *et al.*, 2000b; Moodley *et al.*, 2000; Cardoso *et al.*, 1999) if some form of etching had taken place during

cavity preparation. None of the studies were able to totally eliminate microleakage on the dentine side even if the cavity had been blot dried with a cotton pellet or extremely gently air-dried to ensure that the dentine still remains moist. Currently no studies are available on the influence of saliva contamination of Prime & Bond NT, but Evancusky and Meiers (2000) found that saliva contamination had no significant effect on the microleakage on the enamel side of Prime & Bond 2.1.

In the present study, etching followed by curing of the bonding agent and in turn followed by saliva contamination (groups 6 and 8), resulted in statistically significantly less microleakage on the enamel side than when no form of etching was performed and no saliva contamination had taken (group 1). When saliva contamination took place immediately after etching with 36% phosphoric acid and before Prime & Bond NT was applied (group 5) this also produced statistically significantly less microleakage than when no form of etching was applied and no saliva contamination had taken place as in group 1. Microleakage was therefore only significantly influenced if no form of etching was applied or if a week form of etching was applied and saliva contamination took place before curing of the bonding agent. These results support the fact that with no form of etching no characteristic etch pattern of enamel and no resin tags are produced. On the enamel side the results correspond to those achieved by Evancusky & Meiers (2000). Etching therefore seems to be crucial in order to eliminate microleakage at the enamel margin, even in the absence of saliva contamination. Manufactures however do not routinely advise etching and often only advocate it in large cavities or stress bearing areas (Dyract AP Technical Manual, 1999).

In this experiment it was also expected that microleakage at the dentine margin would be ranked from worst microleakage to least microleakage as follows: Group: 4; 8; 6; 1; 7; 5; 3; 2 (details of groups in table 1).

Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT [†] + LC + Dyract AP [†] + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

The reasons for this are probably that mechanical preparation results in the surfaces being covered by a film of denatured protein and ground hydroxyapatite, which is referred to as the smear layer. The smear layer extends into the dentine tubules to form plugs (Pashley, 1984) and could be incorporated into the hybrid layer or modified or removed by acid etching (Pashley *et al.*, 1997). Removal of the smear layer by acid etching causes an increase in surface permeability (Bowen *et al.*, 1984). Stronger etchants remove the smear layer more thoroughly and therefore unplug the dentinal tubules more effectively. Ferrari *et al.* (1999) reported that etching with 36% phosphoric acid followed by Prime & Bond NT application, produced a hybrid layer and dense resin tags of 10-30 μm length. NRC etching produced a thinner hybrid layer and less dense resin tags while no etching did not result in clear hybrid layer formation and the resin tags did not completely seal the tubular orifices.

Water fills the spaces around the collagen fibrils and the resins must diffuse through this water to form a hybrid layer (Nakabayashi *et al.*, 1982). Mechanical interlocking is initiated by a water tolerant primer permeating the spaces in the networked structure of the collagen, created by acid etching. As a result of a decrease in surface tension,

air-drying results in the water that was supporting the collagen network to evaporate and this in turn also results in shrinkage or collapse of the collagen fibrils (Pashley & Carvalho, 1997). A region of unprotected collagen fibers may exist if the resin does not completely infiltrate and surround the decalcified collagen fibers (Miyazaki *et al.*, 1998). Acetone solvents act as "water chasers" (Abdalla & Davidson, 1998) and remove the water molecules by dissolving it. The primer monomers are dissolved in acetone and diffuse into the spaces previously occupied by water. (Pashley & Carvalho, 1997). Wet bonding therefore prevents collagen fibers from collapsing (Kanca, 1992a; Saunders & Saunders, 1996). If dentine is dried too much the resulting collapse of the collagen fibers around the tubules before primer application could cause a decrease in resin penetration.

The analysis of the present study indicated microleakage from the worst microleakage to the least microleakage on the dentine/cementum side in the following sequence:

Group: 8; 6; 3; 2; 5; 7; 4; 1 (details of groups in table 1).

Table 1. Experimental groups (LC = Light-cured)

Group 1	Prime & Bond NT + LC + Dyract AP + LC
Group 2	Phosphoric acid + Prime & Bond NT + LC + Dyract AP + LC
Group 3	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC Dyract AP + LC
Group 4	Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 5	Phosphoric acid + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 6	Phosphoric acid + Prime & Bond NT + LC + Saliva + Dyract AP + LC
Group 7	Non Rinse Conditioner (NRC) + Saliva + Prime & Bond NT + LC + Dyract AP + LC
Group 8	Non Rinse Conditioner (NRC) + Prime & Bond NT + LC + Saliva + Dyract AP + LC

Unexpected results were therefore found in groups 1, 2, 3 and 4 on the dentine side. This could possibly be explained as follows:

In the present experiment cavities were dried after rinsing or saliva contamination with a single gust of air. In the literature review we can clearly see the importance that the dentine must be kept moist (Kanca, 1992a; Kanca, 1992b) in order to prevent the collagen fibers

from collapsing and in turn to ensure that the hydrophilic monomers penetrate deep into the demineralized collagen network. Due to the deeper demineralization of the collagen network produced with stronger etchants it is more prone to collapsing if too much water is removed. The utmost importance of understanding what the manufacturers imply with "moist dentine", and how to achieve it, becomes clear seeing that 3 seconds of air drying resulted in significantly lower bond strengths than blotting dry a cavity (De Goes ne, *et al.*, 1997).

The fact that etching causes deeper demineralization and that the collagen network is more susceptible to collapse in these cases, if the dentine is not dried in the correct manner, could explain the performance of groups 1 and 4. It is speculated that saliva contamination immediately after etching could "overwet" the demineralised area and therefore compensate for water loss if "overdrying" had taken place in combination with deeper demineralization. This could also explain why groups 5 and 7 gave statistically better results than groups 2 and 3. Although group 7 performed better than group 5 there was no statistical difference between their results.

Groups 6 and 8 were etched, and the Prime & Bond NT cured before saliva contamination. Seeing that the resin was already cured, the "wetting" action of the saliva was probably not able to compensate for the "overdrying" of the dentine. Further, the saliva proteins could have clogged the remaining demineralized tubules and formed a film between the Prime & Bond NT and the Dyract AP, thus allowing microleakage. The above mentioned facts combined with deep demineralization possibly resulted in the significantly more microleakage in groups 6 and 8 than in all the other groups.

The fact that group 4 performed statistically better than groups 6 and 8 indicates that the etching of groups 6 and 8 played a bigger part in the microleakage. Not only was the demineralization of the collagen network and its possible collapse due to “overdrying” much worse in groups 6 and 8, but one layer of Prime & Bond NT is possibly not enough to infiltrate all the demineralized dentinal tubules. Platt, Almeida, Gonzalez-Cabezas, Rhodes and Moore (2001) reported that a double layer of Prime & Bond NT gave significantly better results.

No studies are currently available on the influence of saliva contamination of Prime & Bond NT. Abdalla and Davidson (1998) and El Kalla and García-Godoy (1997) investigated the influence of saliva contamination on the shear bond strength of Prime & Bond 2.1 if contamination took place just after etching and before adhesive application. No significant influence was found provided that the saliva was washed by water and extremely gently dried or just removed by blotting with cotton pellets. Saliva contamination did not influence hybrid layer formation and resin tag penetration of Prime & Bond 2.1 (El-Kalla *et al.*, 1999b). It was found to have no significant effect on the microleakage on the enamel side, but significantly increased microleakage on the dentine side of the restorations (Evancusky & Meiers., 2000).

There are also other factors that could contribute to microleakage on the dentine side. If the Prime & Bond NT is dispensed long before use or if the bottle of Prime & Bond NT is not properly closed, the acetone could evaporate, resulting in the concentration of acetone decreasing, which in turn could limit the ability of the adhesive to penetrate the collagen network. Saliva proteins could also clog up the dentinal tubules therefore preventing penetration. The deeper the demineralization, the more saliva proteins could adhere to the dentinal tubules.

Prime & Bond NT contains nanofillers which are absent in Prime & Bond 2.1 (Ferrari *et al.*, 1999). Although Prime & Bond NT is a filled adhesive, it has a low viscosity because of its nanofiller composition (Perdigão *et al.*, 1999). Tay, Moulding and Pashley (1999) found that nanofillers from Prime & Bond NT were congested around patent tubular orifices but were not found within the interfibrillar spaces of the hybrid layer. They concluded that aggregation of the nanofillers within the adhesive resulted in filler clusters that are too large to infiltrate the interfibrillar spaces of the hybrid layer. They also concluded that retention of ground substance within the demineralized intertubular collagen matrix may also have prevented the infiltration of the nanofillers. Although Li, Burrow and Tyas (2000) found that the nanofillers did penetrate the hybrid layer, they also found silver nitrate infiltration and therefore microleakage throughout the hybrid layer.

Although we would expect Prime & Bond NT to penetrate deeper into more demineralized dentine, form longer resin tags as well as a thicker hybrid layer and have less microleakage, the contrasting results achieved in this study corresponds to results achieved by Sano *et al.* (1995b). They reported a higher degree of microleakage when deeper demineralization of dentine had been produced. They referred to the leakage that occurs within the hybrid layer as nanoleakage.

Hashimoto, Ohno, Endo, Kaga, Sano and Oguchi (2000a) reported that after prolonged acid conditioning, there was an increase in the demineralized dentine zone without resin impregnation, within the hybrid layer. This resulted in lower bond strengths. The increase in the demineralized dentine zone without resin impregnation, can be the reason for the increased microleakage in etched dentine achieved in this study.

Research by Hashimoto, Ohno, Kaga, Endo, Sano and Oguchi (2000b) on the degradation of the resin-dentine bond over a period of 3 years in the oral cavity, revealed degradation of the hybrid layer over the 3 years. This degradation could contribute to nanoleakage and microleakage along the dentine margins. However, the above mentioned did not have an influence on the present study due to the short storage period of the filled teeth.

The results of the experiment shows that the dentine could have been dried too much, and therefore differs from results obtained by Evancusky and Meiers (2000) on the dentine margin. However, it underlines the absolute technique sensitivity of these materials. This once again highlights the fact that enamel and dentine/cementum are two completely different entities that should be treated in two completely different ways. By trying to treat them simultaneously by the total etch technique and by applying one-bottle adhesives to them, we are most probably increasing the risk of technique sensitivity and decreasing the durability of our restorations.

Although no studies currently exist which corresponds 100% to the present study, microleakage studies on Prime & Bond NT (Cardoso *et al.*, 1999; Moodley *et al.*, 2000) and studies on the influence of saliva contamination of Prime & Bond 2.1 (Evancusky & Meiers, 2000) partly support the results.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

- ◆ The current experiments with Dyract AP and Prime & Bond NT could not eliminate microleakage at either the enamel or the dentine/cementum margins of the restorations.
- ◆ Microleakage was statistically significantly reduced on the enamel side by acid etching with NRC or with 36% phosphoric acid.
- ◆ Saliva contamination did not significantly increase microleakage on the enamel side when 36% phosphoric acid was used to etch the enamel.
- ◆ The absence of etching resulted in the least microleakage on the dentine side.
- ◆ If saliva contamination took place after etching and prior to Prime & Bond NT application, it resulted in less microleakage on the dentine side than when the contamination took place after curing of the Prime & Bond NT.

RECOMMENDATIONS

- ◆ Enamel should always be etched in order to eliminate microleakage.
- ◆ Manufacturers should be emphasizing the “wet bonding” technique more and clearly indicate in their instructions how this can be achieved, preferably by dabbing the cavity with a moist cotton pellet.
- ◆ More time and money should be spent on the improvement of materials that do not emphasise the difference between enamel and

dentine and the effects of the same pre-treatment on them so severely. RMGI's could be such an example.

- ◆ The influence of the nanofillers in Prime & Bond NT and the ability of Prime & Bond NT to seal dentinal tubules against microleakage must be more thoroughly researched.



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